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X-Ray Fluorescence (XRF) Analysis in Masonry Heritage Building Conservation in Malaysia

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ABSTRACT

There are many historical masonry heritage buildings in Malaysia which built during the British colonial period. Most of the masonry heritage buildings were dilapidated due to high dampness, salt contamination and other factors. The damaged original lime plaster or mortar require replacement to safeguard the masonry heritage building. According to the Guidelines on Heritage Building Conservation Malaysia (2017), the authenticity of the heritage building including the building materials should be conserved (p.70). Masonry is referred to the art and building of structures built from individual units such as brick, stone, concrete block, glass block and other unit masonry (Masonry, n.d.). However, the quality and durability of the masonry structure is depending on the materials used, the composition and quality of the mortar, the workmanship, and the construction or assembly pattern of the masonry structure. Hence, it is crucial to analyse the composition of the materials used in masonry heritage building conservation. This paper discusses the literature reviews and research on x-ray fluorescence (XRF) analysis conducted in masonry heritage building conservation projects in Malaysia. The data was collected from several literature reviews and documentation such as final conservation report and scientific test report of x-ray fluorescence (XRF) analysis conducted in masonry heritage building conservation projects in Malaysia. The data was compiled and analysed to reveal the process of conducting x-ray fluorescence (XRF) analysis and interpret the analysis result for practical use in masonry heritage building conservation in Malaysia. This research findings outline the basic procedure in interpreting the result of x-ray fluorescence (XRF) analysis for heritage conservation project. Furthermore, the basic procedure shall act as a reference and technical guidance for future heritage building conservation projects.

Keywords: X-ray fluorescence (XRF), conservation, masonry heritage building

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INTRODUCTION

Application of appropriate diagnostic tests and conservation methods or techniques are important to ensure a successful masonry heritage building conservation project. In heritage building conservation, the decision-making tool should be evidence-based practice as the risk normally involves an element of uncertainty (Douglas & Ransom, 2013). In the diagnostics stage of a masonry heritage building conservation project, the evidence and cause factors of building defects can be observed or revealed by application of scientific tests such as x-ray fluorescence (XRF) analysis. X-ray fluorescence (XRF) analysis is commonly used to analyse major elements such as silicon dioxide (SiO_2), titanium dioxide (TiO_2), aluminium oxide (Al_2O_3), ferric oxide (Fe_2O_3), manganese (II) oxide (MnO), magnesium oxide (MgO), calcium oxide (CaO), sodium oxide (Na_2O), potassium oxide (K_2O) and phosphorus pentoxide (P_2O_5).

LITERATURE REVIEW

Heritage is defined "as something of significance or value that is inherited from one generation to another" (Guidelines on Heritage Building Conservation Malaysia, 2017, p.68). According to National Heritage Act 2005 (Act 645) section 2, building means "a building or groups of separate or connected buildings which, because of their architecture, their homogeneity or their place in the landscape, are of outstanding universal value from the point of view of history, art or science" (p.95). Conservation is a process of protecting, safeguarding and maintaining a tangible heritage from any damage due to natural factors and human factors such as improper restoration methods and vandalism. It includes preservation, restoration, reconstruction, rehabilitation and adaptation or any combination of it. The main objective of heritage conservation is to protect and safeguard the heritage building with proper conservation approaches and techniques. Authenticity is the key aspect in heritage conservation especially for the heritage buildings which are listed as Heritage Site and National Heritage Site in Malaysia under National Heritage Act 2005 (Act 645). The original building materials are the important value of a heritage building as they are historical evidence from the past. The original building materials contain the information of the building technology and knowledge from the past. Hence, the original building materials must be conserved with scientific methods to safeguard the authenticity of a heritage building. Scientific method is defined as "A method of procedure that has characterized natural science since the 17^{th} century, consisting in systematic observation, measurement, and experiment, and the formulation, testing, and modification of hypotheses." (Scientific Method, n.d.). Scientific tests on the existing building materials such as x-ray fluorescence (XRF) analysis must be conducted before the restoration works to provide sufficient information in decision making stage. Scientific test is a significant tool to diagnosis heritage building defect and building material performance. All interventions in the built environment incur risks (Pereira Roders & Douglas, 2008). In heritage building conservation, application of appropriate or proven conservation techniques is the key to ensure a good conservation project. Thus, the decision-making tool in heritage building conservation shall be evidence-based practice. Evidence is defined as something that is visible, measurable and recordable (Douglas & Ransom, 2013). Several scientific expert systems can be used to study the chemical, physical and properties of mortar, brick and masonry and building defects diagnosis (Lourenco, van Hees, Fernandes, & Lubelli, 2014). An accurate building diagnosis is critically important where there is no substitute for a thorough understanding of a building's behavior and its response to changes over time. This only can be done by conducting scientific tests such as x-ray fluorescence (XRF) (David, 2008). Besides that, many masonry heritage buildings in Malaysia were built with lime. According to the lime cycle, lime exists in three forms which are calcium carbonate ($CaCO_3$), calcium oxide (CaO) and calcium hydroxide [Ca $(OH)_2$]. Lime exists in the form of calcium carbonate ($CaCO_3$) in the nature which can be found as limestone, shells, chalk and coral rocks (Stafford & Michael, 2003). Calcium carbonate ($CaCO_3$) will release carbon dioxide (CO_2) when it is heated in a kiln at temperature around 900°C and forming quicklime, calcium oxide (CaO) (Stafford & Michael, 2003). Quicklime does not exist in natural environment. Once the quicklime, calcium oxide (CaO) is reacted with water molecules (H_2O), it will be converted into calcium hydroxide [Ca $(OH)_2$] which known as slaked lime or hydrated lime. Slaked lime will be kept in lime pit for at least one month or more longer time to settle down and form lime putty. Carbon dioxide (CO_2) from the atmosphere will be slowly absorbed by slaked lime, calcium hydroxide [Ca $(OH)_2$] to form calcium carbonate ($CaCO_3$) and this process is known as carbonation (Stafford & Michael, 2003). In masonry heritage building conservation in Malaysia, lime putty or calcium hydroxide [Ca $(OH)_2$] is used as building material for wall plaster, mortar, lime wash, lime concrete and others. In general, the ratio of putty lime to aggregate (clean sand) for lime plaster (base or first layer) is 1: 3 by volume (Tan, 2015). As calcium oxide (CaO) is one of the major elements which can be detected by x-ray fluorescence (XRF) analysis, the existence and percentage of calcium oxide (CaO) in building material such as plaster and mortar can be detected and identified by conducting x-ray fluorescence (XRF) analysis.

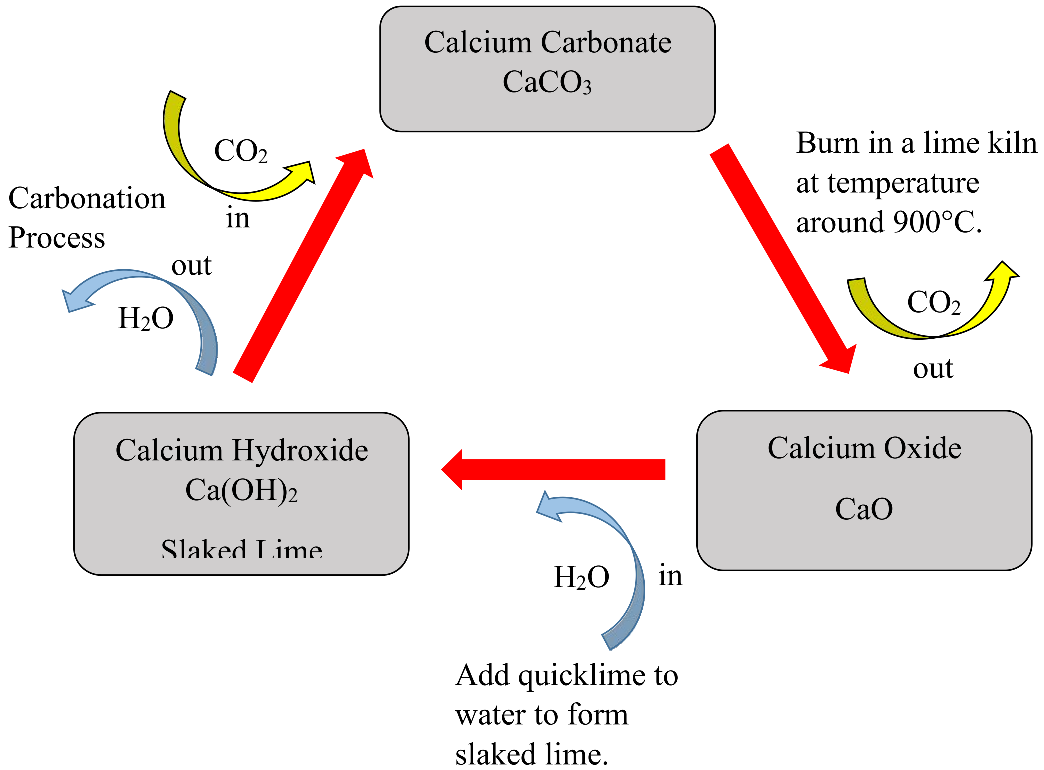

Figure 1: The life cycle.

METHODOLOGY

From year 2009 until Jan 2019, there are seventy-one (71) buildings or monuments in Malaysia have been listed under National Heritage Act, section 67 as National Heritage Site. Majority of these heritage buildings are masonry structural buildings. Hence, the building type selected for this study is masonry heritage building which is the major typology of heritage building in Malaysia. The data was collected from several literature reviews and documentation such as final conservation report and scientific test report of x-ray fluorescence (XRF) analysis conducted in masonry heritage building conservation projects in Malaysia. The data was compiled and analysed to reveal the process of conducting x-ray fluorescence (XRF) analysis and interpret the analysis result for practical use in masonry heritage building conservation in Malaysia.

DISCUSSION AND FINDINGS

X-ray Fluorescence (XRF)

X-ray fluorescence (XRF) analysis is conducted by the "emission of characteristic "secondary" (or fluorescent) X-rays from a material that has been excited by bombarding with high-energy X-rays or gamma rays" (Amptek, n.d.). X-ray fluorescence (XRF) analysis is a quantitative and qualitative analytical tool used for elemental analysis of building materials such as plaster and mortar in masonry heritage building conservation in Malaysia (Tan & A Ghafar, 2017). Besides, it is commonly used to analyse major elements such as silicon dioxide (SiO_2), titanium dioxide (TiO_2), aluminium oxide (Al_2O_3), ferric oxide (Fe_2O_3), manganese(II) oxide (MnO), magnesium oxide (MgO), calcium oxide (CaO), sodium oxide (Na_2O), potassium oxide (K_2O) and phosphorus pentoxide (P_2O_5) (Tan & A Ghafar, 2018).

Table 1: Ten major elements which can be identified by XRF analysis.

No.	Major Elements
1	Silicon dioxide (SiO_2)
2	Titanium dioxide (TiO_2)
3	Aluminium oxide (Al_2O_3)
4	Ferric oxide (Fe_2O_3)
5	Manganese (II) oxide (MnO)
6	Magnesium oxide (MgO)
7	Calcium oxide (CaO)
8	Sodium oxide (Na_2O)
9	Potassium oxide (K_2O)
10	Phosphorus pentoxide (P_2O_5)

Figure 2: X-ray fluorescence spectrometer.

Sample Preparation

To ensure the accuracy of the building material analysis, prior to x-ray fluorescence (XRF) analysis, sample shall be prepared according to related procedures. There are several sample preparation methods for x-ray fluorescence (XRF) analysis, such as loose powder, pressed pellets, fused bead and others. In masonry heritage building conservation in Malaysia, pressed pellet and fused bead methods are the common sample preparation methods in laboratory.

Pressed Pellet

The representative portion of each sample will be pulverised into approximately 20 µm grain size using motorised grinding machine. The sample and binder (Boreox or Boric acid) will be weighed and mixed well. Ratio of sample to boric acid is 1:5. The boric acid will be served as backing material. The mixture will be poured into die. Press the mixture to required force using high pressure presser. After that, the specimen will be analysed for ten (10) major elements and other minor elements by using x-ray fluorescence (XRF) spectrometer. Furthermore, the loss of ignition value (L.O.I.) will be derived by igniting 1g of the sample at 1050 ºC for an hour. The reduction in weight of the ignited sample compared to initial weight will be analysed as L.O.I. value.

Fused Bead

The representative portion of each sample will be pulverised into approximately 20 µm grain size using motorised grinding machine. Then, the specimen will be produced by igniting 0.5g of the sample and 5.0g of the spectruflux (Di-Lithium Tetraborate) at 1050 ºC for 20 minutes before casted into a glass disc. After that, the specimen will be analysed for ten major elements by using x-ray fluorescence (XRF) spectrometer. Furthermore, the loss of ignition value (L.O.I.) will be derived by igniting 1g of the sample at 1050 ºC for an hour. The reduction in weight of the ignited sample compared to initial weight will be analysed as L.O.I. value (Tan & A Ghafar, 2017).

Interpretation of X-ray Fluorescence (XRF) Analysis Result for Practical Use in Heritage Building Conservation

In masonry heritage building conservation projects in Malaysia, especially the masonry heritage buildings with dilapidated wall plaster and mortar due to high dampness, salt contamination, cracks and other factors where re-pointing and re-plastering works are required. X-ray fluorescence (XRF) analysis was conducted to identify the ratio of silicon dioxide, calcium oxide and other elements in existing wall plaster and mortar of masonry heritage building. Silicon dioxide (SiO_2) is referred as aggregate such as sand, while calcium oxide (CaO) is lime exists in the plaster or mortar. Loss of ignition (L.O.I) are the elements lost during the burning process which can be organic element, moisture or other elements. It is referred as bonding agents. The result of the x-ray fluorescence (XRF) analysis was served as reference for the preparation of new plaster and mortar. X-ray fluorescence (XRF) analysis also reveals the condition of the existing plaster and mortar for the heritage building. In general, the ratio of putty lime to aggregate (clean sand) for lime plaster (base or first layer) is 1: 3 by volume (Tan, 2015). However, the original composition of plaster and mortar for heritage buildings may be different due to workmanship or building technology of different period in the past.

Table 2: XRF analysis result of plaster sample (first layer) KM-1 to KM-3 for Fort Margherita Sarawak (2013).

Element	KM- 1 (Wt %)	KM- 2 (Wt %)	KM- 3 (Wt %)
Na_2O	0.1182	0.2043	0.3098
MgO	0.5180	0.4351	0.4636
Al_2O_3	2.5555	2.4439	2.2666
SiO_2	30.0301	38.3911	31.9780
P_2O_5	0.0658	0.0632	0.0606
K_2O	0.1962	0.2338	0.3044
CaO	58.5550	49.7501	56.7733
TiO_2	0.4687	0.4253	0.4517
MnO	0.0325	0.0268	0.0148
Fe_2O_3	1.9018	1.6121	0.9002
L.O.I.	3.7671	4.6887	4.1551

Source: XRF analysis result prepared by School of Materials & Minerals Resources Engineering, Universiti Sains Malaysia compiled in the Final Report for Restoration and Upgrading Works of Fort Margherita, Petrajaya, Kuching Sarawak (Tan, 2014).

Table 2 above shows the ratio of calcium oxide to silicon dioxide for sample KM-1, KM-2 and KM-3 are 1.95 : 1, 1.3 : 1, 1.78 : 1. The x-ray fluorescence (XRF) analysis result shows the existing wall plaster has higher percentage of calcium oxide which is lime. It is different with the referencing composition of lime plaster (base or first layer) which is 1 : 3. Hence, it can be assumed that the existing plaster may not in good condition or it was changed in the past. Further study of Fort Margherita's background shows the building was restored in the past. The composition of plaster will affect the structural integrity of the masonry heritage building as the wall plaster is important protection layer for the old bricks in the masonry walls. The ratio of aggregate (silicon dioxide), calcium oxide and other elements in the new plaster and mortar is important to safeguard its authenticity and ensure the conservation work quality. Hence, the new plaster mock-ups are required to be produced on site by referring to the x-ray fluorescence (XRF) analysis result of the existing plaster and the referencing composition of lime plaster. The new plaster mock-ups must be exposed to sunlight and rainwater for at least one month before carrying out the Schmidt hammer rebound test to identify its compressive strength.

Basic Procedure on Interpretation of X-ray Fluorescence (XRF) Analysis Result for Masonry Heritage Building Conservation

A basic procedure on interpreting the result of x-ray fluorescence (XRF) analysis was derived from the research findings and summarised as below:

I. Identify the two major elements (silicon dioxide and calcium oxide) in the result of x-ray fluorescence (XRF) analysis.
II. Calculate the ratio of silicon dioxide to calcium oxide by referring to the result.
III. Use the referencing ratio of silicon dioxide to calcium oxide as 3 : 1. If the ratio of silicon dioxide to calcium oxide is vary or much different to 3 : 1, it can be predicted that the existing plaster may not in good condition and further inspection is required on site.

Note: there must be sufficient quantity of samples (at least three) taken from different areas of a wall to avoid bias result. The information such as the sample is taken from which layer (base, middle or finishing) should be recorded as well.

CONCLUSION

In conclusion, masonry heritage buildings in Malaysia, especially the National Heritage, have unique cultural and historical significance to the particular community and country. Thus, the masonry heritage buildings should be conserved according to the scientific methods. The decision-making tool shall be evidence-based practice to safeguard its authenticity. The scientific tests such as x-ray fluorescence (XRF) analysis shall be conducted in all masonry heritage building conservation projects to ensure the quality of the conservation works, especially for the conservation of masonry heritage buildings with dilapidated or damaged wall plaster and mortar due to high dampness, salt contamination, cracks and other factors where re-pointing and re-plastering works are required. This research outlined the basic procedure in interpreting the result of x-ray fluorescence (XRF) analysis for heritage conservation project. Furthermore, the basic procedure shall act as a reference and technical guidance for future heritage building conservation projects.

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