

SYNTHESIS OF CALCIUM SILICATE HYDRATE (CSH) FROM VIETNAM RICE HUSH

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ABSTRACT

Vietnam is an agricultural country, which produces lot of rice and its by product is rice husk ash (RHA). The RHA is considered as waste in agriculture, and treated by burn in the open air. This process causes air pollution, thus attracted Vietnamese researchers to find alternative method to reduce the impact of rice husk ash on environment. The research group in Department of Ceramic Materials aims to reuse rice husk ash as source of silica (SiO₂). It needs to emphasize that the main content of rice husk ash is silica. This research reports new technology to mixing RHA and CaO with the Ca/Si molar ratio of 1.0, in order to synthesize calcium silicate hydrate (CSH) such as tobermorite (C₅S₆H₅) and xonotlite (C₆S₆H) as environmental materials. The advantage of our study is to utilize the Vietnam RHA and to reduce the environmental impacts by using hydrothermal treatment technique.

Key words: Calcium silicate hydrate, rice hush ash, hydrothermal treatment, ceramic.

1. INTRODUCTION

Vietnam is an agricultural country, which produces lot of rice and its by product is rice husk ash (RHA). The RHA is considered as waste in agriculture, and treated by burn in the open air. This process causes air pollution, thus attracted researchers to find alternative method to reduce the impact of rice husk ash on environment [1-3]. The research group in Department of Ceramic Materials aims to reuse rice husk ash as source of Silica (SiO₂) [4-8]. The obtained silica can be used as stating materials to reaction with calcium source to form calcium silicate hydrate (CSH). By this chemical reaction, we can utilize the Vietnam rice hush for sustainable development.

2. METHODOLOGY

Preparation of Vietnam Rice Husk Ash (VRH): Rice husk is burned at 600 °C with the heating rate of 10 °C/min (Naberthem 1400, Nabertherm, Germany), then soaking for 4 hours to complete burning. The phase composition of obtained VRHA is characterized using X-ray Diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). The chemical composition of VRHA is analyzed using X-Ray Fluorescent (XRF) method.

Preparation of CaO: CaO is used from the commercial without purified (Xilong

Chemical, China). The phase composition of commercialized CaO using XRD.

Hydrothermal treatment the mixture of VRHA and CaO: The mixture of VRHA and CaO is mixed with the Ca/P molar ratio of 1.0 with the moisture of 10% (weight percent) then pressing at 30 MPa to form the compact disk with diameter of 9 mm. The compacted cylinder is hydrothermally treated at different temperature levels for 24 hours to obtain calcium silicate hydrate such as tobermorite and xonotlite.

Phase analysis: The powder X-ray Diffraction (XRD) patterns of disk samples were recorded with a vertically mounted diffractometer system (Bruker-AXS: D8 ADVANCE, Germany) using Ni filtered CuK α generated at 40 K ν , 20 mA.

The chemical compositions of sample were characterized by XRF (ZSX, Rigaku, Japan) operated at 40 kV and 40 mA.

The bonding chemical of sample was characterized by FTIR analysis: the sample were ground into fine powder, mixed with KBr powder at the ratio 1:200. Infrared spectra were measured at a resolution of 2 cm⁻¹ using a Fourier transform infrared (FTIR) spectrometer (PerkinElmer 2000, USA).

3. RESULTS AND DISCUSSIONS

The chemical composition of RHA is shown in Table 1:

Table 1. The chemical composition of RHA (weight percent)

SiO ₂	K ₂ O LOI	CaO Total	P ₂ O ₅	MgO	Al ₂ O ₃	MnO	Fe ₂ O ₃	SO ₃	other
92.7	3.16 0.63	1.33 100	0.596	0.466	0.306	0.291	0.242	0.126	0.153

The phase analysis of VRHA is given in Figure 1, indicating that VRHA is composed of crystalalite, which shows the peak at 22° (PDF# 01-082-0512).

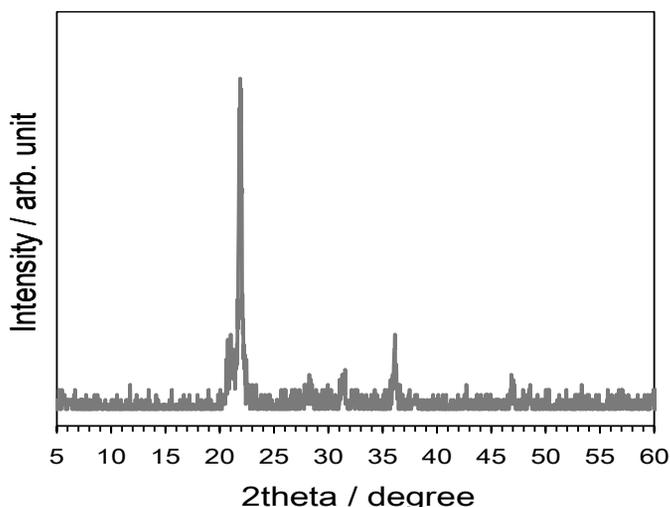


Figure 1. XRD pattern of VRHA

The FTIR of VRHA is given in Figure 2, indicating that the main chemical bonding of VRHA is O-Si-O, go well with XRD data given in Figure 1.

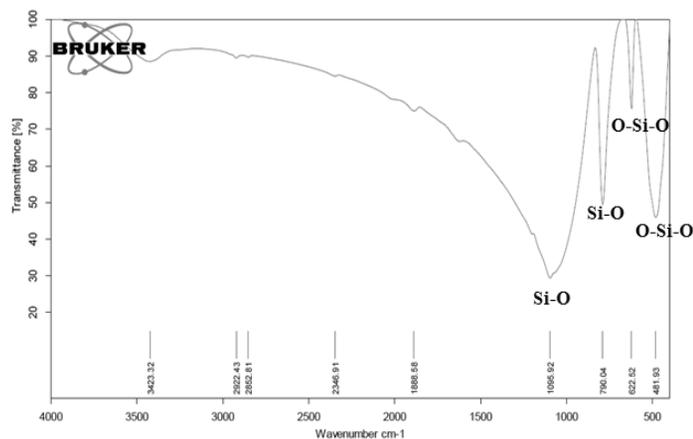


Figure 2. FTIR spectrum of VRHA

The phase analysis of CaO is given in Figure 3, indicating that commercialized CaO is purified (which the peak of CaO at 32°, 37° and 54° corresponding to PDF# 01-077-2376) and can be used for further reaction.

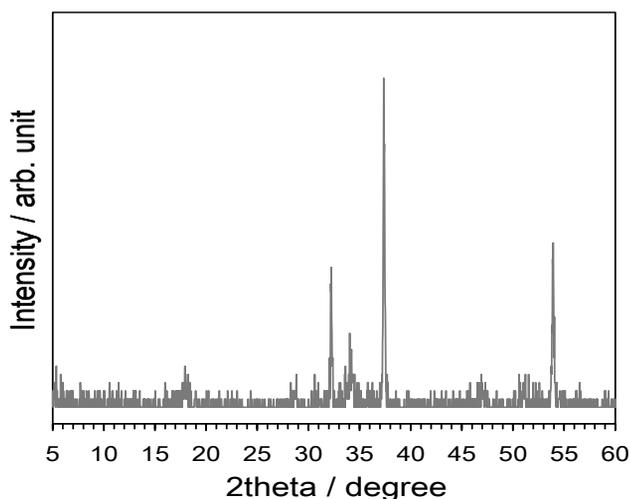


Figure 3. XRD pattern of CaO

The phase analysis of mixture of VRHA/CaO before and after hydrothermal treatment at 110 °C, and 200 °C for 24 hours.

Before hydrothermal treatment, the phase composition of sample is cristobalite and Ca(OH)₂. The present of Ca(OH)₂ is given by hydration of CaO and water during the mixing process. After hydrothermal treatment, we can observe the new phase of Calcium Silicate Hydrate (CSH), Tobermorite (at 110 °C for 24 hours) and Xonotlite (at 200 °C for 24 hours). The morphology of sample before and after hydrothermal treatment at 110 °C and 200 °C for 24 hours also is given at Figure 5.

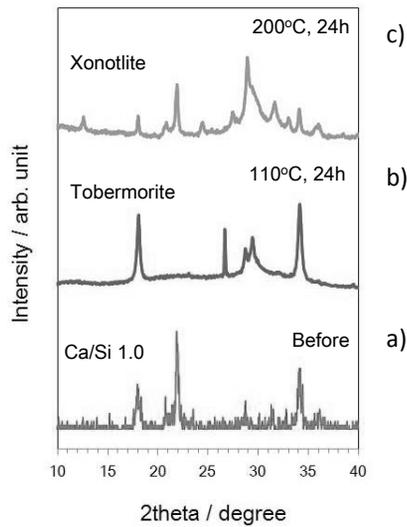


Figure 4. XRD pattern of mixture of VRHA/CaO before and after hydrothermal treatment at different temperature levels: (a) before; (b) 110 °C to obtain Tobermorite; and (c) 200 °C to obtain Xonotlite.

We can observe the morphological change of sample before and after hydrothermal treatment with the increase of hydrothermal treatment temperature. At 110 °C and 200 °C, we can observe the new pore, while the morphology transitionally changes from polyonal-like shape to needle-like shape and these needle-like shape crystals are interlocked together (Figure 5c). The size of new needle-like shape also increased with the increase of hydrothermal treatment temperature (Figure 5b, 5c).

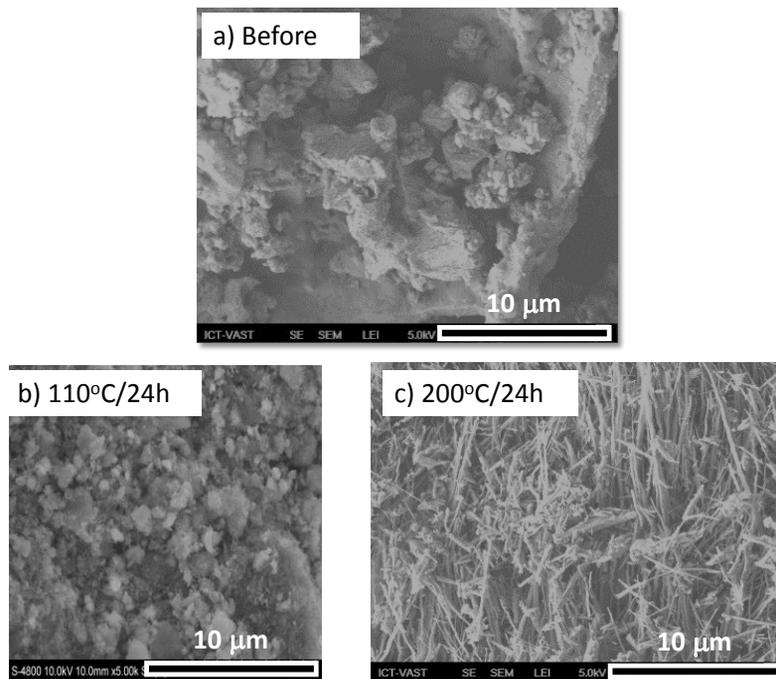


Figure 5. SEM images of sample before and after hydrothermal treatment at different temperature levels: (a) before; (b) 110 °C; and (e) 200 °C.

4. CONCLUSIONS

By using hydrothermal treatment of the compaction of VRA and CaO with the molar ratio of Ca/Si 1.0, we can synthesize Tobermorite ($C_5S_6H_5$) at 110 °C and Xonotlite (C_6S_6H) at 200 °C. Both Tobermorite and Xonotlite are calcium silicate hydrate, and can be used as environmental materials. Thus, this research can contribute to the sustainability of Vietnam rice husk industry.

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TÓM TẮT

TỔNG HỢP KHOÁNG CALCIUM SILICATE HYDRATE (CSH) TỪ NGUỒN NGUYÊN LIỆU TRÁU

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Việt Nam là nước nông nghiệp, sản xuất nhiều gạo và sản phẩm phụ là trấu. Trấu được xem là chất thải nông nghiệp và được đốt bỏ. Quá trình này gây ô nhiễm môi trường, thu hút nhiều nghiên cứu để tận dụng trấu. Nhóm nghiên cứu ở bộ môn Ceramic tận dụng trấu như nguồn cung cấp silica (SiO₂). Cần nhấn mạnh rằng thành phần chính của trấu là silica. Nghiên cứu này công bố kỹ thuật mới phối trộn tro và CaO với tỷ lệ mol Ca/Si 1.0 để tổng hợp khoáng xonotlite như vật liệu môi trường. Ưu điểm của nghiên cứu này là tận dụng nguồn trấu Việt Nam và giảm tác hại lên môi trường bằng phản ứng thủy nhiệt.

Từ khóa: Calcium silicate, trấu, thủy nhiệt, ceramic.