



The effect of hydrothermal treatment time on the synthesized of Xonotlite using glass cullet (GC) and Calcium hydroxide

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ABSTRACT

In this study, the effect of hydrothermal temperature of glass cullet (GC) and $\text{Ca}(\text{OH})_2$ was study. The batch-mixed sample with the Ca/Si 1.0 were hydrothermal reacted at different temperature such as 100, 150 and 180°C for 48 hour to find the suitable temperature to form the xonotlite. The data indicate that at 180°C for 48 hour, the peak of xonotlite at 2theta of 30 degree is highest, thus suggest for further study on the effect of hydrothermal reaction time to form the xonotlite.

Introduction

Up to date, the recycling of wasted glass cullet in Vietnam is the severe problem. Yearly, Vietnam produce 450 melting ton of glass container at OI-BJC glass company located at My Xuan Industria zone, also 500 melting ton of sheet glass at Viglacera glass company at Binh Duong province, and also 900 melting ton of sheet glass at Chu Lai sheet glass company. Briefly around 20% of the glass production recycles of glass cullet, leading to melting of at least 1550°C, thus consume the fuel energy and release the CO_2 emission to the atmosphere. In order to reduce the CO_2 footage during the recycling of wasted cullet, the research group at Faculty of Materials Technology, Ho Chi Minh City University of Technology (HCMUT) has proposed the new idea to use wasted glass as raw materials to supplied Calcium and Silica simulanesouly to fabrication the building materials by using

hydrothermal treatment techniques [1-3]. However, these techniques require the high pressure during the shaping process, thus prevent the application of wasted glass cullet. In order to convert the silica content to Xonotlite [$\text{X}, \text{Ca}_6(\text{Si}_6\text{O}_{17})(\text{OH})_2$], our research group propose to use wasted cullet and Calcium hydroxide as raw materials with the Ca/Si molar ratio of 1.0, follow by casting method (with no pressure) and indirect hydrothermal treatment at 180°C for 48 hour. The Xonotlite mineral is classified as Calcium Silicate Hydrate mineral materials (CSH) [4-9] owing the environmental friendly property since it can be used as heavy metal adsorbent materials as well as thermal insulated materials.

Experimental

Materials: The wasted glass cullet (WGC) was supplied by Viglace glass company. The WGC was grounded by

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fast-grinding machine with the power of 3kW (3A model, Tuan Tu Agriculture manufacturer, Vietnam), then passed through the 0.45-mm sieve to collect the fined glass cullet (GC). The $\text{Ca}(\text{OH})_2$ was supplied by Xilong Chemical (China) without purification.

Synthesize Xonotlite by hydrothermal reaction: the GC and $\text{Ca}(\text{OH})_2$ were mixed with Ca/Si molar ratio of 1.0 to form the 6-mm diameter disk, follow by hydrothermal reaction at different hydrothermal reaction temperature such as 100, 150 and 180°C for 48 hours in the present of distilled water to study the effect of hydrothermal reaction time to the formation of Xonotlite mineral materials. During the hydrothermal reaction, the 6-mm disk were contacted only with the steam water to prevent the washout phenomena as show in Figure 1.

Indirect contact

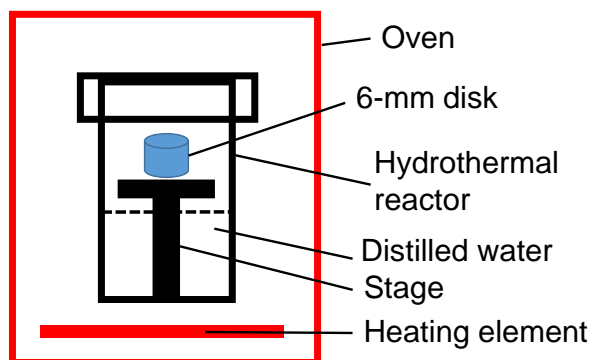


Figure 1: The schematic layout of hydrothermal reactor used. The 6-mm disk were contacted only with the steam water to prevent the washout phenomena. The hydrothermal set was placed inside the oven to supply the heat

The research flow chart to synthesized the xonotlite mineral using GC and $\text{Ca}(\text{OH})_2$ was shown on Figure 2. In this study, the effect of hydrothermal reaction temperature on the formation of xonotlite materials was focused.

Materials characterizations method:

The phase analysis using powder-type XRD: the raw materials such as GC and $\text{Ca}(\text{OH})_2$ sample, the mixture before and after hydrothermal treatment was ground and put in XRD machine (Bruker D8 Advance, Germany) with the 2theta scanning from 5 to 60 degree, operation at 40kV and 40mA.

The chemical composition analysis using Xray Fluorescing (XRF): the sample was characterized by Xray at 40kV (ARL Advant, Thermo Scientific).

The morphology analysis using SEM: the sample was scattered on Carbon tape stick to Copper substrate, and carried the SEM analysis (Hitachi S-4800) at 10kV.

The chemical bonding of samples were analysis using FTIR: the sample(s) were mixed with KBr with the ratio of 1:200, press to make the transparent pellet, and carried out under infrared ray (Thermo Nicolet iS50, ATR)

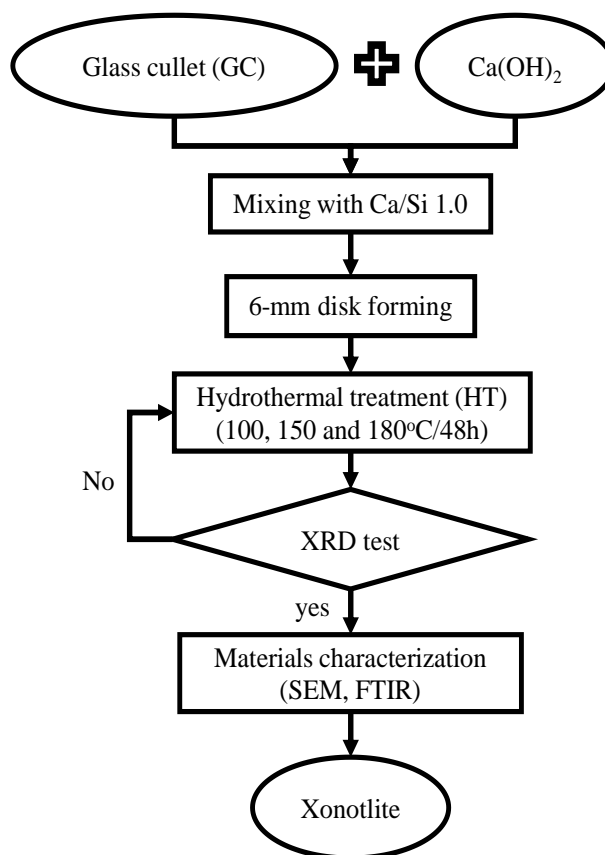


Figure 2: The research flow chart to synthesized the xonotlite mineral using GC and $\text{Ca}(\text{OH})_2$ using hydrothermal reaction. In this study, the effect of hydrothermal reaction temperature on the formation of xonotlite materials was focused

Results and discussion

Raw materials study: The XRD diffraction of glass cullet (GC) was shown at Figure 3. In general, the GC shown the low crystallinity of quartz (PDF#46-1045) with is the main composition is SiO_2 . It need to emphasized that SiO_2 is also the main component of glass culler. In addition the XRD pattern of $\text{Ca}(\text{OH})_2$ also shown on Figure 4, with the high crystallinity of portlandite (PDF#44-1481) at 2theta of 18 and 32 degree. These XRD indicated that the main component of GC and $\text{Ca}(\text{OH})_2$ were Calcium and Silica containing, with further analysed by XRF analysis.

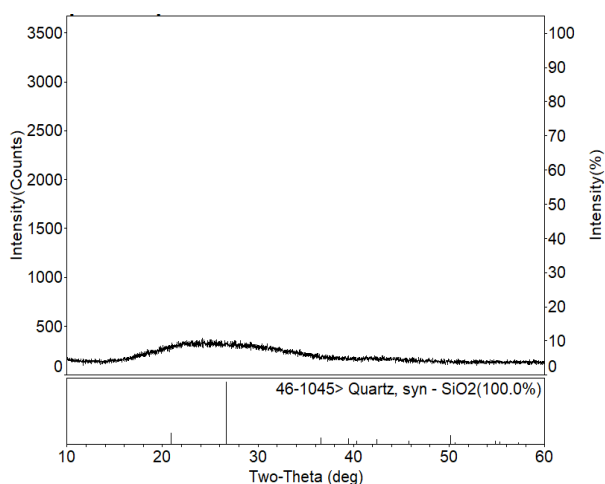


Figure 3: The XRD pattern of GC shown the low crystallinity of quartz (PDF#46-1045)

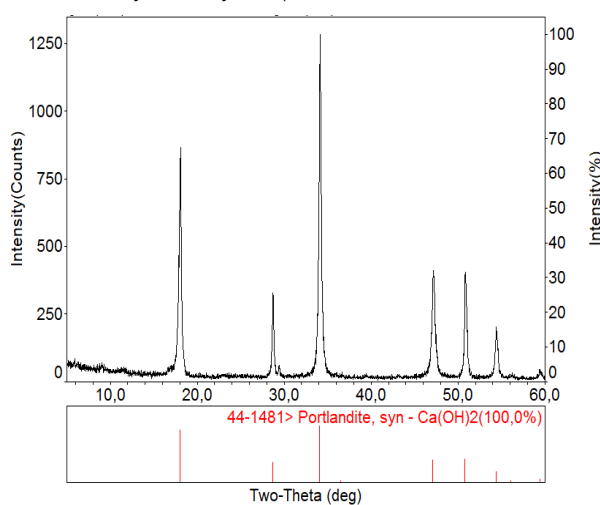


Figure 4: The XRD pattern of Ca(OH)₂ shown the main peak of Portlandite (PDF#44-1481)

The chemical composition of GC and Ca(OH)₂ were given in table 1 by XRF analysis. Based on these result, our research rroup can batch-mixed the GC and Ca(OH)₂ with the Ca/Si molar ratio of 1.0

Table 1: Chemical analysis of the GC and Ca(OH)₂

Oxide (wt.%)	SiO ₂	Na ₂ O	CaO	¹ LOI	Other	Total
GC	69.50	14.94	12.74	0	2.82	100
Ca(OH) ₂	-	-	75.6	24.4	-	100

¹LOI: loss on ignition

Study the sample before and after hydrothermal treatment: Figure 5 shown the effect of hydrothermal reaction temperature such as 100, 150 and 180°C for 48 hour on the synthesized of xonotlite by using XRD analysis. The peak of Xonotlite (PDF#23-0125) at 30 degree increase upon increase the hydrothermal reaction temperature , indicating that 180°C/48h is the good hydrothermal treatment condition to form xonotlite

Figure 5: The XRD pattern of the 6-mm disk sample before and after hydrothermal treatment (HT) at 100, 150 and 180°C for 48 hours (Figure 5c, 5d, 5e and 5f). In addition Figure 5a and b show XRD patterns of raw materials. The left XRD patterns shown the 2theta degree vary from 10 to 60, while the right XRD pattern zoom the 2theta degree vary from 25 to 35. Code: C-Ca(OH)₂ (PDF#44-1481); X-Xonotlite (PDF# 23-0125)

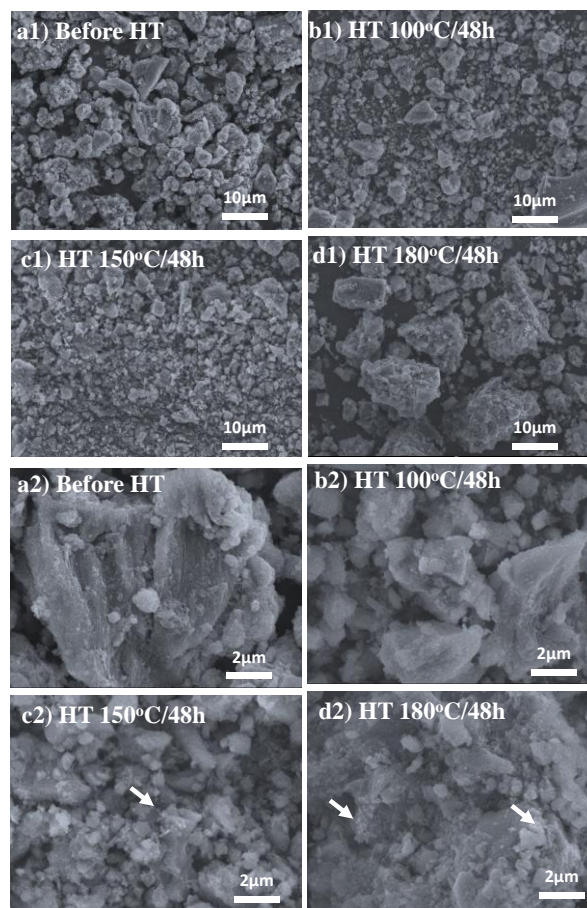


Figure 6: The SEM image of sample before and after hydrothermal treatment at the magnification of 2.000X (a1, b1, c1 and d1) and magnification of 10.000X (a2, b2, c2 and d2). The hydrothermal condition were shown at before hydrothermal treatment (HT) as Fig. 6a1, 6a2; at 100°C for 48h as Fig. 6b1, 6b2; at 150°C for 48h as Figure 6c1, 6c2 and at 180°C for 48h as Fig. 6d1, 6d2

Figure 6 shown SEM image of 6mm-disk sample before and after hydrothermal treatment at different temperature reaction condition. Basically, before hydrothermal reaction (Fig. 6a1 and 6a2), the sample had smooth surface, however after hydrothermal reaction temperature for 100, 150 and 180°C for 48h, we can observed the precipitated of xonotlite on the surface of sample, indicating the formation of xonotlite (Fig. 6d1 and 6d2) as shown in the white arrow. Based on the XRD data in Fig. 5, we can conclude that the main phase of $\text{Ca}(\text{OH})_2$ still remain, and the new phase of xonotlite can be observed at 2theta of 30 degree.

Figure 7 shown the FTIR spectra of sample before and after hydrothermal treatment at 100, 150 and 180°C for 48 hours. The peak of OH could be observed at 3600cm^{-1} , while the peak of xonotlite could be observed at 1000cm^{-1} at Fig. 7b, 7c and 7d. Basically there is no peak different between the reaction temperature of 150°C and 180°C except the clear peak at 700cm^{-1} thus suggest the 180°C is the optimum temperature for forming xonotlite.

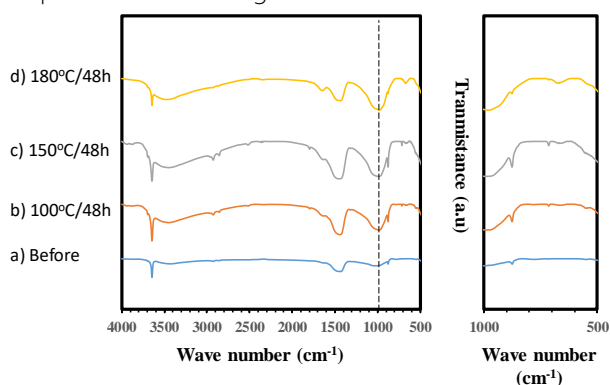


Figure 7: The FTIR spectra of the 6-mm disk sample before and after hydrothermal treatment (HT) at 100, 150 and 180°C for 48h

This research needs to put into the context of experiment conditions. In hydrothermal technique, it is important to identified the hydrothermal reaction such as hydrothermal temperature as well as hydrothermal duration time. In this research, we focus on study the effect of hydrothermal reaction such as 100, 150 and 180°C for 48h on the formation of xonotlite. The finding from XRD, SEM and FTIR data shown that the 180°C for 48h is the optimum condition to form xonotlite. Thus, the next research on study the effect of hydrothermal duration time at 180°C will be carried out. The use of synthesized xonotlite from fined flint glass cullet may offer several benefit for heavy metal removal or thermal insulator in heat industry. The silica, xonotlite can be conjugated with various function groups for specific target detection such as heavy

metal ions (Pb^{2+} , Cu^{2+} , Cd^{2+} , Cr^{3+} , Cr^{6+} etc.) for environmental application. The surface area of synthesized xonotlite can be increase by adding the pore agent during the hydrothermal reaction, thus suggest that can be used in biomaterials as well [10].

Conclusion

In this work, we use fined flint glass cullet ar precursor to supplu both Ca and Si. The GC was batch-mixed with $\text{Ca}(\text{OH})_2$ at Ca/Si 1.0 and hydrothermal reacted at different temperature of 100, 150 and 180°C for 48 hours. The finding data indicating that the optimum temperature for hydrothermal reaction is 180°C for 48 hours, and suggest my research group to further study on the effect of hydrothermal duraiton time.

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