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Synthesis and thermal behavior of geopolymer-type material from waste ceramic

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HIGHLIGHTS

• Waste ceramic was collected, grinded and screened to synthesize geopolymer.

• The synthesized geopolymer showed a maximum 28 d compressive strength of 71.1 MPa.

• The geopolymer showed a higher compressive strength after heat treatment of 1000 °C.

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ABSTRACT

Waste ceramic was activated by alkali hydroxides and/or sodium/potassium silicate solutions to synthesize geopolymer-type material in this study. The synthesized geopolymer pastes were characterized by mechanical test, TG-DSC, SEM, XRD, as well as FT-IR analyses. And the thermal behavior of synthesized geopolymer was determined in terms of compressive strength evolution by exposure to 100, 200, 400, 600, 800, and 1000 °C. The synthesized geopolymer pastes exhibited a maximum 28-day compressive strength of 71.1 MPa and favorable anti-thermal properties by showing a higher compressive strength of 75.6 MPa after heat treatment of 1000 °C. The results indicate that waste ceramic could serve as a satisfying source material for thermostable geopolymer.

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1. Introduction

As the world's largest ceramics producer and consumer, China produces over one million tons of all kinds of waste ceramics (tiles, pan forms, blocks, and so on) which are just land filled or stacked every year. With increasingly stricter restrictions on landfills in China, ways for comprehensive utilization of waste ceramics have to be explored. Reutilization of waste ceramic as part of the feedstock for the production of ceramics has been practiced, but the amount of wastes reused in that way is still negligible [1]. Efforts have also been made in the cement industry to use recycled ceramic and calcined clays as alternative cementitious materials [2–5]. However, according to Sanchez De Rojas [6] and Goncalves et al. [4], the increase of the cement replacement by calcined clay materials would result in the increase in total porosity and the reduction in strength. Nowadays, there are several papers [6–10] that study the possibility of using ceramic wastes in substitution of natural aggregate (sand or gravel). Though a good workability can be obtained using waste ceramic as aggregates, the studies in this field showed that some problems arise. Senthamarai and Manoharan [11] studied the properties of concrete with electrical ceramic waste aggregate where he found that the compressive, splitting tensile and flexural strengths of ceramic waste coarse aggregate concrete were lower than conventional concrete. Results observed by Cachim [12] indicated that brick residuals could be used as partial replacement of natural aggregates without reduction of properties for 15% replacement and with reductions up to 20% for 30% replacement. Binici [13] reported a decrease in both abrasion and chloride resistances when using crushed ceramic as fine aggregate substitution. Hence ways to compressively reutilize waste ceramic should be expanded.

Geopolymers are a class of inorganic polymers synthesized by polycondensation of [SiO₄] and [AlO₄] tetrahedral in alkali activated aqueous solutions [14,15], which was firstly introduced by Joseph Davidovits in the late 1970s [16]. According to Davidovits [16,17], geopolymers are polymeric silicon–oxygen–aluminum three-dimensional materials containing a variety of amorphous to semi-crystalline phases. Based on such a unique structure, geopolymers exhibit higher mechanical strength, much more excellent chemical, fire and heat resistances, lower thermal conductivity and shrinkage than ordinary Portland cement (OPC) [18]. Moreover, as







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Komnitsas and Zaharaki [19] studied, geopolymers can generally deliver a great reduction in CO₂ emission and require less energy compared with OPC. Thus geopolymers can be regarded as a 'green concrete' [19,20]. Due to the sound properties as well as significant environmental benefits of geopolymer products, geopolymer technology has been drawing great interest [21–23].

On one hand, large number of scholars have developed a variety of raw materials such as metakaolin [24,25], fly ash [18,26–28], and slag [29,30] to synthesize geopolymer matrixes of excellent performances. All these materials could supply the polymerization reaction with sufficient silica and aluminum [20,31]. On the other hand, waste ceramic is a kind of typical silicon rich material, therefore using waste ceramic as source material to manufacture geopolymer should be an efficient and environmental friendly integrated utilization of it. However, no dedicated study has been reported about systematic characterization of the synthesis and high-temperature properties of geopolymer pastes using waste ceramic as source material.

In this study, waste ceramics were creatively introduced to discuss its probability for geopolymer synthesis. Focus would be on the synthesizing process concerning selection of best activating solution. An optimal geopolymer product will be selected for further high temperature tests in terms of compressive strength gains or losses after exposure to high temperatures (100, 200, 400, 600, 800, and 1000 °C). The geopolymer products were characterized by mechanical testing, Thermogravimetric Analysis-Differential Scanning Calorimetry (TG-DSC), Scanning electron microscopy (SEM), X-ray diffraction (XRD), as well as Fourier Transform Infrared Spectroscopy (FT-IR) analyses. Results obtained in this study will enrich the studies of geopolymer and provide a promising alternative way to reutilize waste ceramic economically and environmentally.

2. Experimental procedure

2.1. Materials

The ceramic used in this study was derived from municipal waste collection to enhance the social value of this research, it's a mixture of titles, pan forms, blocks and so on. The ceramics were first ultrasonic washed to remove contaminants such as paper scraps, metal, plastic, or organic matters. And the dried ceramics were crushed, pulverized in a ball mill for 45 min and then screened and measured on a Mastersizer 2000 laser analyzer (Malvern, UK). Fig. 1 shows the particle size distribution of ceramic powder, the average particle size (d50) of milled ceramic is $30.17 \ \mu m$. Fig. 2 shows the SEM image of the grounded ceramic powder, which consists exclusively of irregular, coarse and angular particles. 9800XP⁺ X-ray fluorescence spectrometer (XRF) (ARL, Switzerland) and ARL XTRA X-ray diffractometer (Thermo, Switzerland) were employed for analyzing the chemical compositions and crystal phases of waste ceramic powder, and the results are listed in Table 1



Fig. 1. Particle size distribution of ground waste ceramic.



Fig. 2. Scanning electron microscope image of waste ceramic powder.

and Fig. 3, respectively. The main chemical compositions of waste ceramic are SiO_2 and Al_2O_3 , with the major crystalline phases of being quartz (SiO_2) and albite (NaAlSi₃O₈).

An industrial grade sodium silicate solution (SiO₂ = 26.5%, Na₂O = 8.5%, and H_2O = 64.8; molar ratio of SiO₂/Na₂O, 3.2), sodium and potassium hydroxides (analytical grade), as well as deionized water, were employed in this investigation to prepare activating solutions. The activating solutions can be divided into three groups, i.e. groups A, B, and C. Each group consists of three solutions, making a total of nine activators. Activators A1–A3 are mixtures of sodium silicate and sodium hydroxide; B1–B3 are blendings of sodium silicate and potassium hydroxide solutions; and C1–C3 are sodium and/or potassium hydroxide solutions. Compositions of all the activating solutions are detailed in Table 2. All alkaline solutions were stored for at least 24 h before use.

2.2. Geopolymer synthesis and analysis

Geopolymers were synthesized by mixing ground waste ceramic with each activating solution with a liquid/solid ratio of 0.4, respectively, as summarized in Table 3. The 5 min mixing process was followed by casting the slurries in triplet moulds of 20 mm cubes and another 5 min vibration to remove entrained air bubbles. The moulds were then sealed with polyethylene film and set in a standard curing box at 60 °C with 100% humidity under ambient pressure. After 24 h of setting, the samples were demoulded and subjected to curing at 60 °C standard curing box in sealed polypropylene boxes for further 27 d.

The compressive strength values of synthesized geopolymers were measured using a NYL-300 compressive strength testing apparatus (Wuxi Jianyi, China), with the force applied at a rate of 1.0 kN/s. Geopolymer of the highest compressive strength was selected and subjected to high-temperature performance test. The selected specimens were calcined in a muffle furnace at 100, 200, 400, 600, 800, and 1000 °C for 2 h, respectively. Compressive strength gains or losses of the calcined specimens were then measured. The results reported were the average of three replicates.

Simultaneous TG-DSC was carried out on a STA 449C (Netzsch, Germany) thermal analyzer to determine the mass loss history of selected geopolymer at elevated temperatures. The sample was heated from 40 to 1100 °C in an inert nitrogen environment with the heating and nitrogen purging rates kept constant at 10 °C and 25 ml min⁻¹, respectively. The ground waste ceramic as well as selected geopolymers before and after thermal exposures was characterized via SEM, XRD, and

Table	1
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Chemical composition of waste ceramic.

Chemical composition	Content (wt%)
SiO ₂	65.52
Al ₂ O ₃	21.00
CaO	6.00
K ₂ O	3.31
MgO	1.95
Fe ₂ O ₃	1.11
Na ₂ O	0.36
TiO ₂	0.20
SO ₃	0.17
BaO	0.15
LOI ^a	0.14

^a LOI, loss on ignition at 960 °C.

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