TREATING WASTE SLUDGE FROM WATER-PURIFICATION PLANTS WITH THE GEOPOLYMERIZATION METHOD

OBDELAVA ODPADNEGA BLATA IZ ČISTILNIH NAPRAV Z METODO GEOPOLIMERIZACIJE

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Prejem rokopisa - received: 2020-06-05; sprejem za objavo - accepted for publication: 2021-07-19

doi:10.17222/mit.2020.070

Treatment of the sludge from water-purification plants is becoming more and more urgent due to the inability to increase its storage area. To avoid CO_2 emissions, the use of non-Portland cement binders is recommended. The application of geopolymerization of waste sludge (WS) from water-purification plants is a novel solution. Curing conditions including high temperature, pressure or microwaves enhance the formation of geopolymer bonds. This paper presents the results of a research on the treatment of the WS of the Thu Duc water-purification plant (Vietnam) with the geopolymerization method. Solid phases were prepared by mixing the WS and fly ash (FA). The FA proportions of the solid phases were (10, 40, 70) w/%. The alkali-activated solution (AAS) was a mixture of a 40 w/% NaOH 6M solution and 60 w/% water glass (WG: Na₂O.nSiO₂ with n = 1.75 and volumetric density ρ = 1.40 kg/L). The geopolymer materials were mixtures containing an 80 w/% solid phase and a 20 w/% liquid phase of the AAS. Geopolymer samples were formed in a cylindrical steel mold with a diameter of 10 mm at a high pressure. The samples were cured in a 112 W microwave oven for 30 s or in a dryer at 110 °C for 24 h. The compressive strength and volumetric density of both sample groups were determined and compared to each other. The formation of geopolymer bonds was investigated using XRD, FTIR and SEM.

Keywords: waste sludge, water, purification plants, fly ash, water glass, alkali-activated solution (AAS), geopolymer, microwave oven, dryer, curing

Obdelava odpadnega blata iz vodnih čistilnih naprav postaja vedno bolj pomembna in nujna zaradi pomanjkanja ustreznih prostorov za skladiščenje ali nezmožnosti povečanja skladiščnih prostorov. Da bi se izognili emisiji CO_2 , avtorji predlagajo uporabo veziv cementov, ki ne temeljijo na Portland cementnih vezivih. Najnovejša rešitev je geopolimerizacija odpadnega blata (WS; angl.: waste sludge) iz čistilnih naprav. Pogoji toplotne obdelave pri visoki temperaturi in tlaku v mikrovalovni peči pospešijo tvorbo geopolimernih vezi. V članku so predstavljeni rezultati raziskave obdelave odpadnega blata iz vietnamske čistilne naprave »Duc water purification plant« s pomočjo geopolimerizacijske metode. Trdne faze so bile pripravljene z mešanjem odpadnega blata in dimnega pepela v različnih razmerjih (10, 40, 70) mas. %. Aktivirana alkalna raztopina je bila mešanica 40 mas. % NaOH 6M raztopine in 60 mas. % vodnega stekla (Na₂O.nSiO₂ z n = 1,75 in volumsko gostoto ρ = 1,40 kg/L). Geopolimerni material je predstavljal mešanico 80 mas. % trdne faze in 20 mas. % tekoče faze oz. aktivirane alkalne raztopine. Vzorci geopolimerne mešanice so bili oblikovani pod visokim tlakom v cilindričnih modelih premera 10 mm. Vzorci so bili nato toplotno obdelani v 112 W mikrovalovni pečici za 30 sekund, ali pa so jih v sušilniku sušili 24 ur pri 110 °C. Sledila je določitev tlačne trdnosti in volumske gostote vzorcev ter medsebojna primerjava lastnosti vzorcev obdelanih na oba načina. Tvorba geopolimernih vezi je bila analizirana z metodami rentgenske difrakcije (XRD), Fourierjeve transformacijske infrardeče spektroskopije (FTIR) in vrstične elektronske mikroslopije (SEM).

Ključne besede: odpadno blato, voda, vodne čistilne naprave, dimni pepel, vodno steklo, aktivirane alkalne raztopine (AAS), geopolimer, mikrovalovna pečica, sušilnik, toplotna obdelava

1 INTRODUCTION

Waste sludge (WS) from a water-purification plant is not hazardous but it occupies a large area of urban land, so the management of WS from water-treatment plants is becoming a global problem.¹⁻⁴ Conventional solutions for WS treatment are giant landfill or storage tanks. Using large areas of urban land is a big problem because it is very expensive. Moreover, WS still releases unwanted substances into the surrounding environment. Utilization of WS as a raw material for baked or unburnt

bricks is considered an effective solution in terms of environmental treatment.^{5,6} However, in practice, the quality of these bricks is not high so it is difficult to use them.

With geopolymerization techniques, it is possible to use large amounts of hazardous and non-hazardous wastes to fabricate new products as well as minimizing the environmental impacts of solid waste.^{7–13} Indeed, the technology can transform industrial solid waste containing silica or aluminosilicates into useful products thanks to the flexibility and capability to immobilize and stabilize the wastes inside a geopolymer network.^{7,8,10} In principle, any waste material containing a proper amount of

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silica and alumina can serve as an activator for the geopolymerization technique.^{13–15} Nowadays, the geopolymerization technique can be considered a new trend in the wastewater treatment.^{3,7,11}

To produce geopolymer concrete, the raw materials must be dissolved into an alkaline solution and then condensed to form geopolymer bonds. As the alkaline activity of WS is low, it is not used as an independent material for geopolymerization. It is necessary to add to WS an alkali activator such as fly ash (FA),12,13 metakaolin14 or rice-husk ash15 and then heat WS to 900 °C to increase its alkaline activity.16 The curing process for geopolymer specimens is usually carried out under normal atmospheric conditions or in a dryer at 65-115 °C.11-15 Microwave power has also been found to shorten the curing time of geopolymer concrete. Microwave energy provides a direct delivery of energy to microwave-absorbing materials, allowing volumetric heating of the samples.^{17,18} Thus, the rate of strength development of geopolymer concrete can also improve.15

In the mentioned studies, the method of forming samples by mainly casting WS into steel molds, curing it under different conditions and determining mechanical characteristics, rarely provides the formation of geopolymer bonds.

The research results for the samples from alkali-activated WS + FA mixtures with a NaOH6M + WG solution, formed with a semi-dry pressing method at a relatively high pressure (225 MPa) are presented in this paper. The samples were cured under different conditions, treated in a microwave oven for 30 s or dried in a drying oven at 110 °C for 24 h. The volumetric density and compressive strength of the samples were compared to show the properties of each curing method. The microstructure of the samples cured in the microwave oven was detected with modern methods such as XRD, SEM, FTIR.

2 EXPERIMENTAL PART

Preparation of the AAS: The chemicals used were caustic soda containing 99 w/% NaOH, commercial WG with a volumetric density of 1.40 kg/L and silicon module n = 1.75. The AAS was prepared by mixing the chemicals in the ratio of 40 w/% of the NaOH 6M solution and 60 w/% of WG. After being mixed, the AAS must be cured at room conditions for 24 h before use.

Preparation of alkali- activated samples: The raw materials were WS (Thu Duc District, Vietnam) and FA (Vinh Tan thermal power plant, Vietnam). The alkali-activated samples were prepared by mixing 80~w% of solid phases (WS and FA) with 20~w% of the AAS (the liquid phase). The contents of FA in the alkali-activated samples FA/(solid + AAS) were (10, 40, 70)~w%.

The alkali-activated samples were pressed at 225 MPa in a cylindrical steel mold to form a green body with a diameter of 10 mm, height of 18 mm and weight of about 3 g. Then the specimens were cured in two different conditions. The first sample was cured in the microwave oven (SANYO EM-S2182W with a power of 112 W and frequency of 2.45 MHz) for 30 s. The second one was cured in the dryer at 110 °C for 24 h.

Compressive-strength and volumetric-density measurement: The volumetric density of both sample groups was determined after (1, 3, 7, 10, 14) d. The volumetric density is defined as the ratio of the mass to the total volume (including the interparticulate void volume). The sample volume is equal to the bottom area times the height. Based on the data of volumetric density, other characteristics of the samples could be determined after a curing time of 7 d. It was the time when the volumetric density of the samples was almost unchanged. The compressive strength of both sample groups was determined with the DTU-900 MNH machine with a loading speed of 3 kN/min.

Chemical composition determined with the X-ray fluorescence method (XRF): A sample was pressed in a 5-cm mold and exposed to an X-ray source at an acceler-

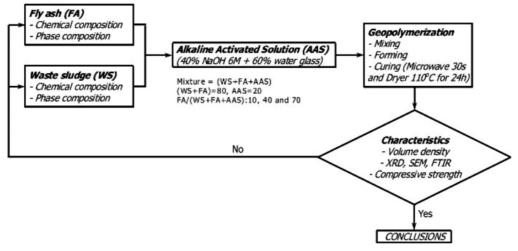


Figure 1: Schema of the experimental procedure

Table 1: Chemical compositions of WS, FA and WG (w/%)

Oxides	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	K ₂ O	CaO	Na ₂ O	Other	LOI*
WS	31.18	30.81	22.88	2.91	1.44	1.72	_	1.30	7.84
FA	48.93	11.22	26.19	1.38	6.94	1.52	_	2.27	1.55
WG	52.67	0.04	1.59	_	_	_	31.24	0.46	14.00

(*) LOI - Lost on Ignition up to 1100 °C for 1 h

ating voltage of 40 kV to obtain its chemical composition using ARL ADVANT'X Thermo.

Phase analysis carried out with X-ray diffraction (XRD): A sample was ground into powder, then pressed in a sample holder and exposed to X-ray irradiation (Bruker D8 Advanced) with 2θ varying from 5° to 70° at 40 kV and 40 mA.

Morphology analysis carried out with scanning electron microscopy (SEM): A sample was put on a carbon tape and coated with Pt. The microstructure images were investigated with SEM using a FESEM S4800 Hitachi machine.

Chemical bonding analysis carried out with Fourier-transform infrared spectroscopy (FTIR): A sample was mixed with KBr powder with a weight mixing ratio of 1:200 and then pressed to into a transparent pallet. The geopolymer bonds in the sample were determined with FTIR using a Bruker Tensor 27, with a wavenumber scan of 500–4000 cm⁻¹.

The experimental procedure is summarized in Figure 1.

3 RESULTS AND DISCUSSION

3.1 Chemical compositions of raw materials

Table 1 shows the chemical compositions of WS, FA and WG. The WS and FA raw materials contain high aluminosilicate resources including 31.18-48.93~w/% SiO₂ and 22.28-26.19~w/% Al₂O₃. It is noted that both materials also have a high iron content (Fe₂O₃), 30.81~w/% in the WS and 11.22~w/% in the FA.

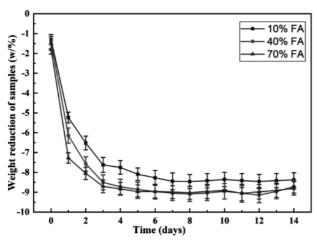


Figure 2: Graph of weight reduction (w/%) of the samples over curing time (d)

3.2 Engineering and microstructural properties of geopolymer

3.2.1 Engineering characteristics

Experimental results of weight reduction (w/%) of AAS over curing time (days – d) are shown in **Figure 2**. The influence of the FA content on the volumetric density (g/cm³) and compressive strength (MPa) of the alkali-activated samples were studied and evaluated in **Figures 3** and **4**.

The weight reduction (w/%) of the FA (10, 40, 70) (w/%) samples depends on the curing time (d). These samples were cured in the microwave oven for 30 s and then left in the air. On the experimental curves in **Figure 2**, the weight reduction of the samples is unchanged after more than 4 d in the atmosphere condition. It is based on these results; the characteristics were tested only on alkali-activated samples after 7 d to ensure their stability.

Figure 3 shows a graph of the relationship between the volumetric density (g/cm^3) and the content of FA (w/%), while **Figure 4** shows a graph of the compressive strength (MPa) and the content of FA (w/%). In these experiments, amounts of (10, 20, 30, 40, 50, 60, 70) w/% of FA were added to determine the volumetric density and compressive strength. There were two sample groups in these experiments. The first group was cured in the microwave oven for 30 s and then left in the air. The second one was cured in the dryer at 110 °C for 24 h and then left in the air.

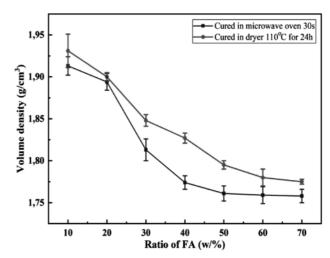


Figure 3: Graph of the volumetric density (g/cm^3) depending on the FA content (w/%) and curing conditions

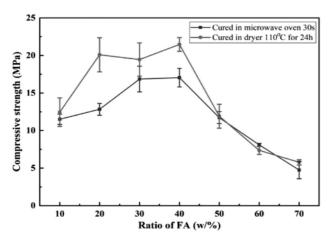


Figure 4: Compressive strength depending on the FA content (w/%) and curing conditions

In **Figure 3**, when the FA content is increased, the volumetric density of the samples is decreased. In **Figure 4**, the sample with 40 *w*/% FA has the highest compressive strength.

The volumetric density and compressive strength of the sample group cured in the microwave oven are lower than those of the samples cured in the dryer. This can be explained with a faster evaporation rate in the microwave oven, due to which not many geopolymer bonds were formed in the samples. The compressive strength of samples reached the maximum when the FA content was optimal. In this case, the optimal content was 40 *wl%* FA.

3.2.2 FTIR, XRD, SEM analyses

Figure 5 shows the FTIR spectra of fresh WG, FA, WS and alkali-activated samples with FA contents of

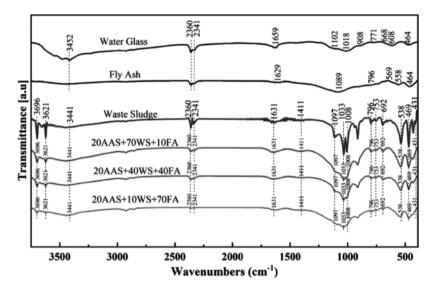


Figure 5: FTIR spectra of WG, FA, WS and the alkali-activated samples with FA contents of (10, 40, 70) w/% after curing in the microwave oven for 30 s

Table 2: Comparison of FTIR spectra of WG, FA, WS and AAS

Wave number (cm ⁻¹)				Bonds	V:1	Dof
WG	FA	WS	AAS	Bollus	Vibration	Ref.
3696	_	3696	3696	-OH groups on the surface of the octahedral [AlO ₆] ⁹	Stretching	23
_	_	3653	_			
_	_	3621	3621	-OH groups on the surface of the octahedral [AlO ₆] ⁹	Stretching	20-23
3452	3451	3441	3441	Н-О-Н	Stretching	20,21
2361	2361	2360	2360	Н-О-Н		20,21,28,29
2341	2341	2341	2341	H-O-H (vibrations of hydrogen bonds between Si(OH) ₄		20,21
201629	1629	1631	1631	Н-О-Н	Stretching	20,21
1411	_	1411	1411	O-C-O (calcite)		27,28
1102	1089	1097	1097	Si-O-Si quartz	stretching	22
1018	_	1033	1033	Si-O-Al (of clay minerals)	stretching	11,13
_	_	1008	1008	Si-O-Al (of clay minerals)	stretching	11,13
908	_	913	913	Si-O-Al	stretching	13,21–23
_	797	796	796	Si-O-Si (of quartz)	bending	8,15,19
771	_	753	753	Si-O-Si in [SiO ₄] ⁴⁻	stretching	21
668	_	692,	692	Si-O		24–26
_	558	538	538	Al-O-Si	bending	23
464	464	469	469	O-Si-O	bending	19,21,24
i0-	_	431	431	Si-O-Al		14,15

(10, 40, 70) w/% after curing in the microwave for 30 s. Comparing these FTIR spectra (**Table 2**), we find some changes that show the formation of geopolymer materials.

The characteristic bands of SiO_2 in the FA at wavenumbers of (1089, 797, 464) cm⁻¹ were shifted to (1097, 796, 469) cm⁻¹ for the alkali-activated samples. The wavenumbers correspond to the network of WG (group vibrations Si–O–Si) where the main bands at (1102, 1018, 908) cm⁻¹ were shifted to (1097, 1008, 1033) cm⁻¹ or overlapped by the FTIR spectra of WS. There is a small shift from 771 cm⁻¹ in the band of the FTIR spectrum of WG to 796 cm⁻¹ in the band of the FTIR spectrum of the hardened sample. This is related to an increase in asymmetric vibrations of isolated $[SiO_4]^{4-}$ -tetrahedra.

The FTIR spectrum of WS is expanded compared to the FTIR spectra of the alkali-activated samples. The expansion of these bands indicates the solubility of the aluminosilicate phase in the raw material and the formation of a new geopolymer-specific amorphous gel. FTIR spectra are most typical in polymer systems, appearing in a wavenumber range of 990–1090 cm⁻¹ showing the asymmetrical stretching of T-O-Si (T is Si or Al). The formation of this new amorphous phase is also most clearly observed on the FTIR spectrum of the alkali-activated samples with 40 w/% of the FA content.

On all FTIR spectra, there is a band at the position of 1432 cm⁻¹ corresponding to the asymmetric stretching vibration of O–C–O bonds.²⁸ This band indicated the reaction of carbon dioxide (from the air) with WG:

$$2Na_2O.nSiO_2.mH_2O + CO_2 \rightarrow$$

$$Na_2CO_3 + Na_2O.nSiO_2(m-1)H_2O + H_2O \qquad (1)$$

Depending on the curing condition, the water in the structure of polymer molecules $nSiO_2(m-1)H_2O$ continues to decrease and geopolymer bonds are formed.

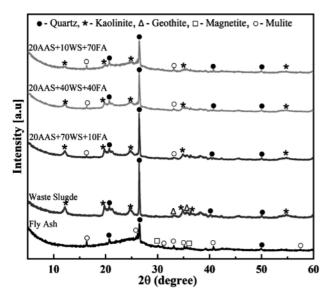


Figure 6: XRD spectra of the AAS with (10, 40 or 70) w/% FA

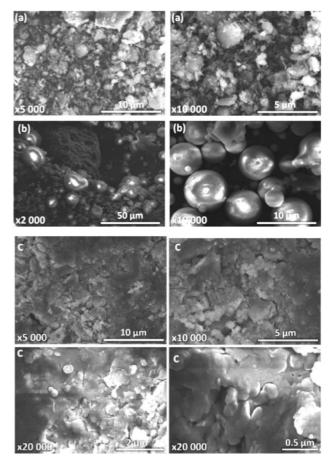


Figure 7: SEM images: a) WS, b) FA, c) microwave-cured geopolymer sample with 40 w/% FA

The XRD spectra of the AAS samples cured in the microwave oven for 30 s are shown in **Figure 6**. Comparing the changes between the XRD spectra (**Figure 6**), it is clear that reactions of the AAS with aluminosilicate solid mixtures occurred. The characteristic peaks for the mullite crystals (symbol M on the XRD spectra in **Figure 6**) show a decreased intensity with the FA content of 10–70 *w*/%. In the XRD pattern of the sample with 10 *w*/% FA, these peaks are almost invisible. On the other hand, broad bands became sharp, indicating that amorphous phases were dissolved.

On the XRD spectra of alkali-activated samples, the intensity of characteristic peaks of kaolinite and quartz minerals is much weaker than on the XRD spectra of the raw materials (WS and FA). This proves that the raw materials reacted with the AAS. If only compared with the angle 2θ of 26.3° , 30 the trace of analcime crystals is unknown because it was overlapped by the kaolinite peaks.

Figure 7 shows the microstructure of the 40 w/% FA alkali-activated sample. This sample was selected for the SEM analysis because of the highest mechanical strength, exhibiting the ability to create good polymerization reactions. The spherical FA particles were dissolved and they disappeared; there are only insoluble solid WS particles distributed in the amorphous matrix

that acted as the binder. Hence, the spherical FA particles reacted with the alkaline activators to form an alumino-silicate amorphous matrix. The mechanical strength of these materials is closely related to the strength of the chemical bonds formed when in contact with the inorganic solid/amorphous geopolymer matrix.

5 CONCLUSIONS

The compressive strength of the alkali-activated samples depends on the volumetric density, the FA content and the curing time. The initial compressive strength of the samples cured in the microwave oven for 30 s or in the dryer for 24 h was similar (13 MPa and 15 MPa, respectively). This shows that the microwave treatment saves time in the early stages. Microwave-cured samples had a high initial compressive strength but later it was lower than that of the samples cured in the dryer. For a long time, the difference in the strength was negligible. The evaporation rate is the cause of this phenomenon.

In both curing conditions, the maximum compressive strength was obtained when the FA content amounted to 40 w/%. It can be considered as the best proportion in the geopolymerization reaction between the AAS and FA. With the concentration of 40 w/% FA, the compressive strength of the sample reaching 15 MPa (in the microwave) or 20 MPa (in the dryer at 110 °C for 24 h) was relatively high compared to the other, untreated, materials.

The geopolymer bonds of the alkali-activated sample were shown by very small displacements on the FTIR or XRD spectra. In addition, the high compressive strength and SEM images with the amorphous matter also provide evidence of a geopolymer formation. Due to its weak alkaline activity, WS acts only as a filler in geopolymer concrete.

Acknowledgment

We acknowledge the provision of time and facilities by the Ho Chi Minh City University of Technology (HCMUT), VNU-HCM, for this study.

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