

Unbaked materials from mixtures of waste sludge of a water purification plant, fly ash, and water glass

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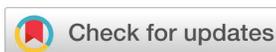
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ABSTRACT

In water purification plants, a large area of urban land is using to store waste sludge (WS). The waste sludge from water filtration plants is aluminosilicate, which can form a geopolymer. However, the waste sludge has low alkaline activity, so it must be used in combination with fly ash (FA) to create geopolymer products. Fly ash is a solid waste containing amorphous silica and it has high alkaline activity, so that it is suitable for treatment by the geopolymer method. The geopolymerization of waste sludge from water purification plants is a relatively new method. The geopolymer is a binder formed by the chemical reaction between aluminosilicate materials and alkaline activated solutions. The alkaline activated solution used in these experiments was water glass (WG). The water glass is the solution of sodium silicate ($\text{Na}_2\text{O}\cdot n\text{SiO}_2$) dissolved in water. The research results of geopolymer materials from the mixture of fly ash, the waste sludge of Thu Duc water purification plant in Ho Chi Minh City (Vietnam), and water glass (WG) were introduced in this study. The activated Al_2O_3 and SiO_2 oxides in the fly ash and the waste sludge can be dissolved in the water glass and polymerized into a geopolymer material. The test samples had pressed at a high pressure of 225 MPa to form cylindrical ones weighing approximately 3 grams, height about 18 mm, and 10 mm in diameter. These samples were then cured at 110 °C for 24 hours and at room temperature (30 ± 5 °C). The methods of Fourier infrared spectroscopy (FTIR) and scanning electron microscope (SEM) had used to detect the microscopic structure and geopolymer bond formation of the samples. The compressive strength of the tested samples at 28 days old was higher than 3.5 MPa, the pH was less than 12.5, meeting the Vietnamese National Standards for unbaked materials (TCVN 6477:2016) and National Technical Regulation on environmental impact (QCVN 50:2013 / BTNMT), respectively. The results show a new approach of solidifying the waste sludge for further applications such as the manufacture of geopolymer concretes or landfill materials.

Key words: unbaked material, fly ash (FA), waste sludge (WS), water glass (WG), alkaline activated solution, geopolymer

INTRODUCTION

Waste sludge (WS) from water purification plants is composed mainly of aluminosilicate. The WS is usually treated by methods as landfills, making ceramic bricks¹⁻³ ... However, due to the areas of landfill sites, WS management is a growing global problem². It is necessary to study the treatment of WS by new methods. In recent years, the treatment of the WS by polymerization is also being interested in many studies⁴⁻⁶.

Geopolymer is a binder formed by the chemical reaction between aluminosilicate materials and alkaline activated solutions. The alkaline activated solutions are composed of strongly alkaline solutions such as sodium hydroxide (NaOH), potassium hydroxide (KOH), soda ash (Na_2CO_3), calcium hydroxide ($\text{Ca}(\text{OH})_2$), and water glass (WG: $\text{Na}_2\text{O}\cdot n\text{SiO}_2\cdot m\text{H}_2\text{O}$)^{2,3}. Fly ash (FA), silica fume, kiln slag, and metakaolin are the industrial wastes

used as raw materials to produce the geopolymers⁴⁻⁷. Unbaked materials made from WS of the Thu Duc water purification plant, Vinh Tan fly ash, and the WG are introduced in this research.

Sodium silicate is a generic name for chemical compounds with the formula $\text{Na}_2\text{O}\cdot n\text{SiO}_2$ (molar ratio $n \sim 1 \div 3.75$ named module of sodium silicate) and soluble in water with various amounts. The solution of the sodium silicate in water is called water glass or liquid glass. Activated Al_2O_3 and SiO_2 oxides in aluminosilicate can be dissolved in an alkaline solution to form a similar bonding circuit of a WG. The bonding circuit is $\text{M}\{-(\text{SiO}_2)_z - \text{AlO}_2\}_n \cdot w\text{H}_2\text{O}$, wherein M is a cation such as K^+ , Na^+ , Ca^{2+} , and “n” is a degree of polycondensation, z is 1,2,3 [8]. When dehydrated, the WG condenses to form a gel-like polymer circuit with the features of poly (sialate) $\text{M}_n - (-\text{Si} - \text{O} - \text{Al} - \text{O} -)_n$ and poly (sialate - siloxo) $\text{M}_n - (-\text{Si} - \text{O} - \text{Al} - \text{O} - \text{Si} - \text{O} -)_n$ ⁷⁻⁹. For increasing the rate of

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condensation, the unbaked materials can be applied to the treatments in the atmosphere by drying at 65 - 110 °C^{8,10} or using microwave energy¹¹.

When using WS to fabricate unbaked materials by the geopolymer method, due to the weak alkaline activity of the WS, the substances with better alkaline activity such as FA, silica fume often need to be mixed¹. The use of strongly alkaline substances influences the surroundings, first of all, is pH. Using WG binders can reduce the impact of environmental pH less than using NaOH, KOH. Furthermore, WS is relatively inexpensive, easy to use, and non-toxic.

This research presented some results on the physico-mechanical properties and environmental pH effects of unbaked materials made from WS, FA, and WG binder. The bonding and microstructure of the materials had to be studied by Fourier infrared spectroscopy (FTIR) and scanning electron microscope (SEM).

EXPERIMENTAL METHODS

Raw materials

The raw materials are WS from Thu Duc water purification plant, FA from Vinh Tan thermal power plant, alkaline activator solution is water glass WG. The chemical composition of the raw materials was determined using the X-ray Fluorescence method.

The alkaline activity of WS depends on the solubility of the oxides in alkaline solution with different concentrations. The amount of activated SiO₂, Al₂O₃, and Fe₂O₃ oxides of the WS dissolved in NaOH solution was determined in referring to Vietnamese National Standards (TCVN 7572-19: 2006)⁹.

The mixing ratio of FA/(FA+WS) changed from 10-70 (% by weight). To achieve the compressive strength of at least 3.5 MPa (refer to Vietnamese National Standards TCVN 6477:2016¹²) and to reduce the amount of alkali discharged into the environment (National Technical Regulation QCVN 50:2013/BT-NMT¹³), the chosen alkaline activated solution was WG. The mixing ratios of WG and the solid (FA + WS) mixture were 6, 8, 10, 12, and 14 (% mass fraction). The (FA + WS) mixture and WG must be thoroughly mixed before being formed by pressing.

Forming

The weight of each test sample was 3 g and pressed in a steel mold to form a cylindrical specimen with height $h = (18 \pm 1)$ mm, diameter $d = (10 \pm 0.2)$ mm (Figure 1). The samples had pressed at 225 MPa pressure, and the moisture content of the mixture was about 7-8%. This pressure value was the parameter

for the cohesion and high density of shaped samples. As the pressure increases further, the sample density hardly increases anymore¹¹. After forming, the samples had cured under two different conditions: 1. At room temperature (average about (30 ± 5) °C) for 24 hours, and 2. In a Venticell laboratory dryer (MMM Medcenter Einrichtungen GmbH) at 110 °C for 24 hours. Then, both sample groups were cured at room temperature (30 ± 5) °C at the same time until the determining properties. The properties identified were volumetric density and compressive strength.

Determination of the properties

The physico-mechanical properties of the specimens were determined after 28 days of curing. This is the time that the specimen weight has been not changed, the properties were considered stable¹¹. Volumetric density was determined by equation $d = m/V$ (therein m is the weight of the sample and V is its volume, calculated by $V = \pi d^2 h / 4$). Compressive strength was determined by the DTU-900 MNH equipment (loading speed 3 kN / min). Chemical composition was analyzed using the X-ray Fluorescence method by the XRF- Thermo ARL ADVANT'X spectrometer. The pH was determined daily during curing time of 28 days according to ASTM D3987-12 (2020)¹⁴ with the Hanna Instruments HI221 pH meter.

The sample group with the best mechanical strength (10% WG) was selected for FTIR analysis (Bruker Tensor 27 spectrometer) and the microstructure was analyzed by scanning electron microscopy (Hitachi S-4800 FE-SEM) for determination of bonds in the sample.

THE RESULTS AND DISCUSSIONS

Chemical composition of raw materials, solubility of oxides in alkaline solution and SEM of WS, FA.

WS was taken from the Thu Duc water purification plant (Ho Chi Minh City, Vietnam), FA was from Vinh Tan thermal power plant (Binh Thuan province, Vietnam), and WG was a commercial product. Chemical composition of raw materials is shown in Table 1. The amount of oxides dissolved in NaOH solution with different concentrations is shown in Table 2.

From the data in Table 1, the module of the WG (Na₂O:nSiO₂, n: molar ratio) was calculated: $n = 1.74$.

The results show that the dominant dissolved component was Al₂O₃.

Scanning electron microscope (SEM) images of WS and FA are shown in Figure 2.



Figure 1: Image of the samples

Table 1: Chemical composition of raw materials (% wt.)

Oxides	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	K ₂ O	CaO	Na ₂ O	Others	LOI*
FA	48.93	11.22	26.19	1.38	6.94	1.52	-	2.27	1.55
WS	28.14	27.81	20.65	2.64	1.30	1.55	-	1.30	16.54
WG	52.67	0.04	1.59	-	-	-	31.24	0.46	14.00

(*) Loss on ignition at 1000 °C.

Table 2: SiO₂, Al₂O₃, and Fe₂O₃ oxides (% wt.) of WS dissolved in NaOH solution

Amount of dissolved oxides (% wt.)	Concentration of NaOH solution (M)									
	1	2	3	4	5	6	7	8	9	
SiO ₂	0.46	0.71	0.90	1.10	1.35	1.67	1.89	2.06	2.24	
Al ₂ O ₃	1.56	3.08	3.21	4.47	5.42	6.51	7.59	8.90	10.12	
Fe ₂ O ₃	0.03	0.03	0.05	0.06	0.09	0.13	0.17	0.19	0.20	

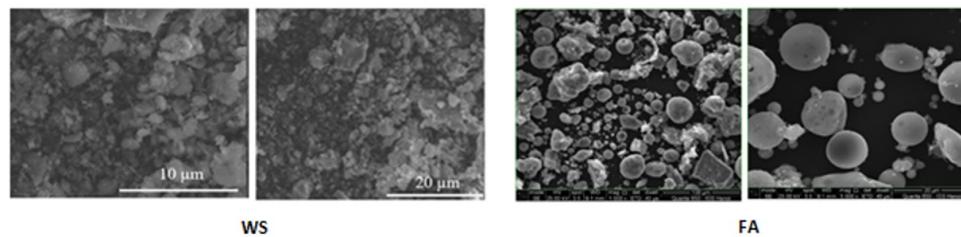


Figure 2: SEM images of WS and FA

Physicomechanical properties of unbaked materials

Figure 3 showed the compressive strength of the samples under different curing conditions after 28 days of age.

The compressive strength of the test samples (Figure 3) under both curing conditions showed that the compressive strength of the 10% FA and 40% FA samples were higher than 3.5 MPa. The compressive strength

of the 40% FA sample was higher than the 10% FA sample when the WG content was 6-8%. However, with the 10-14 % WGs, the compressive strength of the 40% FA sample was lower than that of the 10% FA sample. The compressive strength of the 10% FA and 10% WG samples was the highest. Therefore, the compressive strength, in this case, depends on the ability to form polymer bonding circuits in the tested samples.

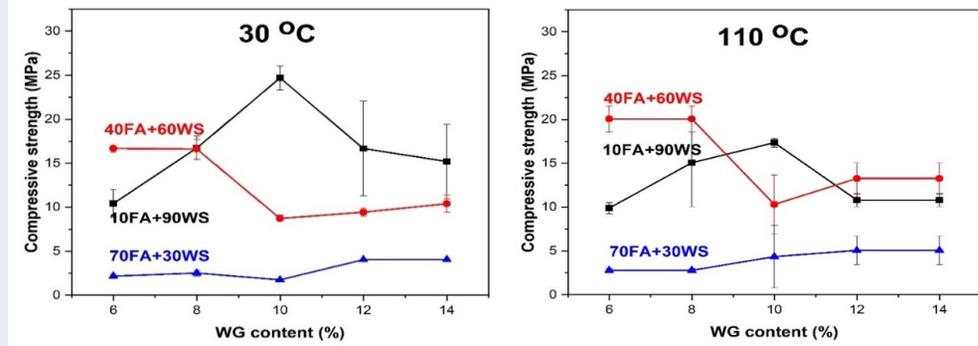


Figure 3: Compressive strength of the samples cured at 30°C (left) and at 110°C (right) after 28 days of age.

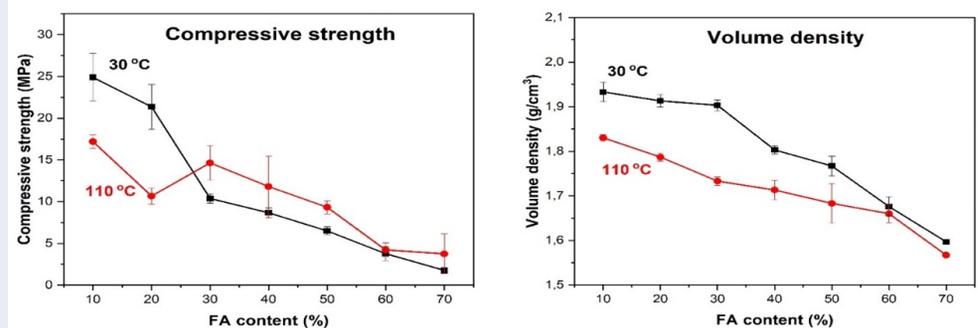


Figure 4: Influence of FA content to volumetric compressive strength (left) and volume density (right) of the 10% WG-containing samples

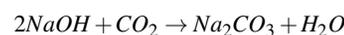
The volumetric density of the samples cured at room temperature was higher than that dried at 110 °C. The compressive strength of the samples cured at room temperature with a 10 – 20% FA content was also greater than that of the samples cured at 110 °C (Figure 4). This result pointed out that the rate of water evaporation too fast did not increase the compressive strength.

The change of pH according to curing time

The pH changes of the samples according to curing time are plotted in Figure 5.

The plots of the pH change over curing time in Figure 5 indicated that the pH increased with an increase in the FA content. That can be explained by the higher pH of FA (11.5) than that of WS (7.5). Besides, the results also showed that the pH of the samples decreased over the curing time. The 10% FA sample at 28 days of age had the lowest pH, such as the pH value of the (10FA+90WS)+10WG samples treated 110 °C only is 9.1 (Figure 5). The pH value decreased over curing time because of the formation of a product layer on

the surface, which prevents diffusion of Na⁺ ions and the reaction of alkaline NaOH solution with CO₂ in the air^{15,16}:



Analysis of bond formation by FTIR and microstructure by SEM

Figure 6 illustrate the FTIR spectra of raw material and 10% WG sample cured at 110 °C for 28 days. Several characteristic bonds are indicated on these FTIR spectra.

On the FTIR spectrum of FA, the band in the 1033-538 cm⁻¹ wavenumber corresponds to the oscillating region of the T-O-T bond (T is the Si and Al tetrahedra) of the aluminosilicate minerals^{15,17,18}. The band of 3441-3696 cm⁻¹ corresponds to the oscillation of the H - O - H and -OH bond¹⁸. The band in the 469.2 cm⁻¹ is characteristic of quartz^{12,19}, and the band in the 2360 cm⁻¹ corresponds to the oscillation of CO₂¹⁵. The existence of CO₂ groups on FTIR spectra is evidence of the possibility of reaction alkaline

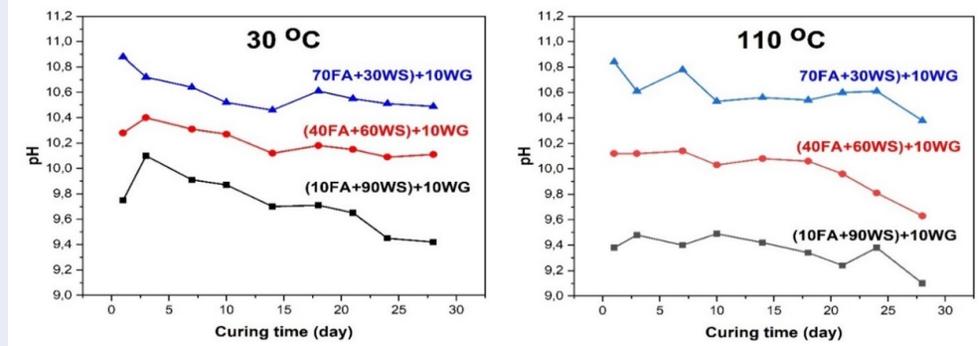


Figure 5: The pH changes according to curing time at 30°C (left) and at 110°C (right)

NaOH solution and CO₂ in the air.

On the spectrum of WG, the band in the 3440-2700 cm⁻¹ wavenumber region characterizes the O-H oscillation of free water. On the FTIR spectra of the alkaline-activated samples, this band has narrowed. It is indicated that the dehydration was occurred in the geopolymer bond-forming process. The band in the 1569 cm⁻¹ characterizes the OH⁻ group in structure, and 1080 cm⁻¹ characterizes the groups - Si - O - Si - with bridging oxygens of the geopolymer materials^{16,19}.

Comparing the characteristic bands on the FTIR spectra of WS and 10% WG activated samples cured in a dryer at 110 °C indicated that they are almost identical. That means the weak activity of WS in the geopolymerization reaction. In other words, the WS acts as a filler. Meanwhile, the characteristic peaks on the FTIR spectra of the WG and FA changed very clearly: the range from 3440 to 2700 cm⁻¹ on the WG spectrum no longer appears on the spectrum of all 10% activated samples. That are (10FA + 90WS) + 10WG, (40FA + 60WS) + 10WG, and (70FA + 30WS) + 10WG. Thus, the WG has lost water, condensed, and cured to form bonds in the material²⁰. The characteristic peaks of the Si - O - Si bond at 1080 cm⁻¹ on the WG spectrum shift to 950 - 980 cm⁻¹ in the FTIR spectrum of 10% WG activated samples.

The microstructure of the samples investigated using the SEM. Figure 7 shows SEM images of WG-activated samples and WS-free samples under different curing conditions.

Comparing the SEM images of the WG-activated and WG-free samples in Figure 7 exhibits the round-shaped FA particles in the WG-activated sample completely deformed, almost participated in the geopolymerization reaction. The non-sphere fuzzy regions

show the gel structure of the geopolymers that consisted of a WG-activated sample.

CONCLUSIONS

Unbaked materials can be fabricated from a mixture (40% FA + 60% WS) and 10% WG by a relatively high forming pressure of 225 MPa. This material has the compressive strength R_n following the standard of unbaked material (R_n > 3.5 MPa, according to Vietnamese Standards TCVN 6477: 2016). The pH of this material decreases with the time of storage and meets the National Technical Regulation QCVN 50: 2013 / BTNMT¹³ on environmental impact. The geopolymer bonds in the material are formed mainly by WG condensation. The results suggest a new method of solidifying WS as aggregate for further applications such as fabrication of geopolymer concrete or fill material.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this article.

AUTHORS' CONTRIBUTION

The contribution of each author in this article follows the State Council for Professorship.

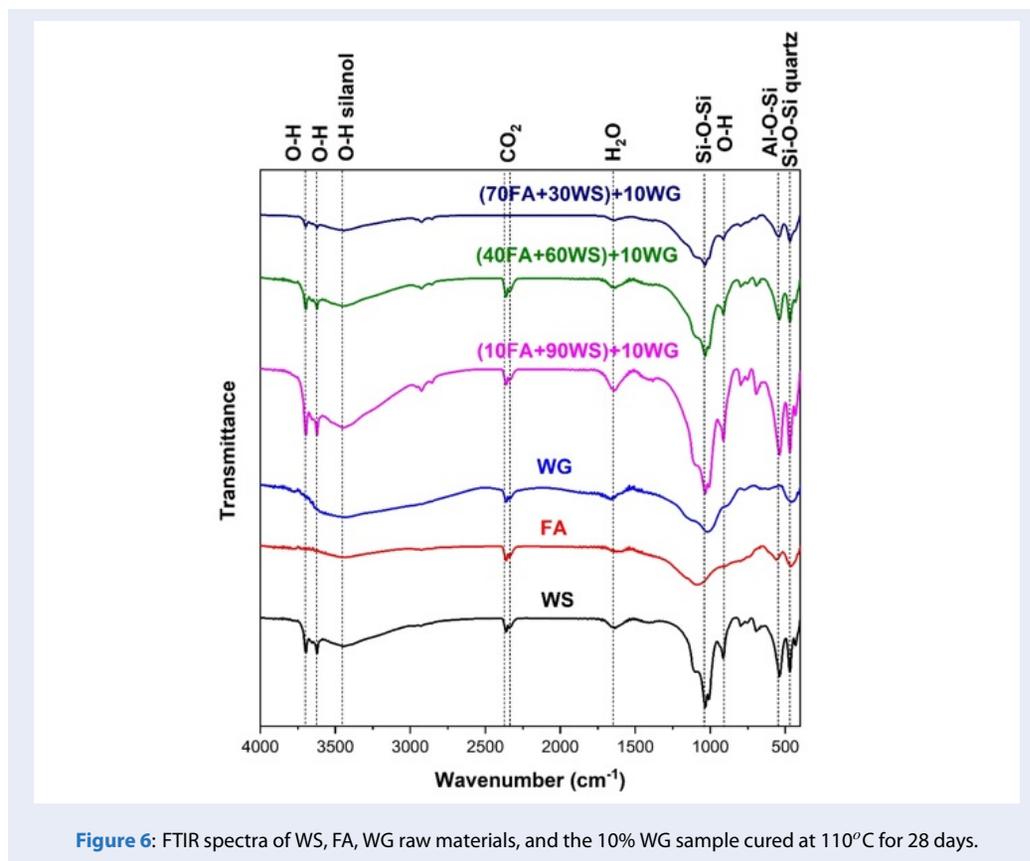


Figure 6: FTIR spectra of WS, FA, WG raw materials, and the 10% WG sample cured at 110°C for 28 days.

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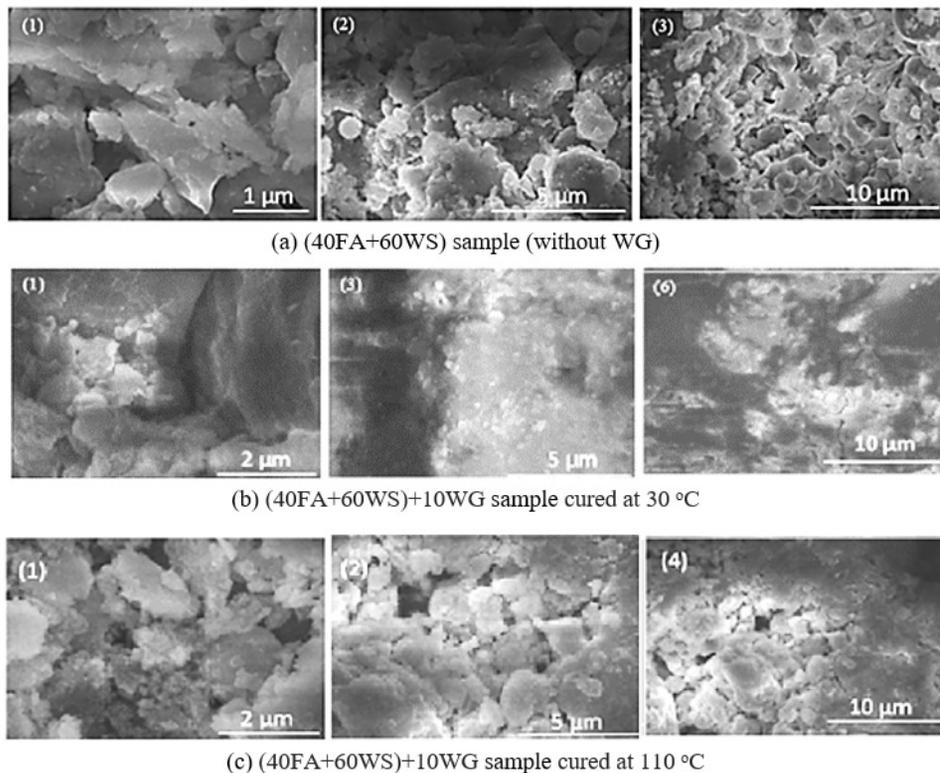


Figure 7: SEM images of the (40FA+60WS) sample without WG and (40FA+60WS)+10WG sample cured at 30°C and at 110°C

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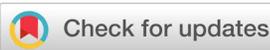
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TÓM TẮT

Trong các nhà máy lọc nước, một phần lớn đất đô thị đang được sử dụng để chứa bùn thải (WS). Bùn thải từ các nhà máy lọc nước là alumino silicate, có thể tạo thành geopolymer. Tuy nhiên, bùn có hoạt tính kiềm thấp nên phải sử dụng kết hợp với tro bay (FA) để tạo sản phẩm geopolymer. Tro bay là chất thải rắn có chứa SiO₂ vô định hình nên có hoạt tính kiềm cao, thích hợp để xử lý bằng phương pháp geopolymer. Quá trình tạo geopolymer hóa bùn thải từ các nhà máy lọc nước là một phương pháp tương đối mới. Geopolymer là chất kết dính được hình thành do phản ứng hóa học giữa vật liệu alumino silicate và dung dịch kích hoạt kiềm. Dung dịch kích hoạt kiềm được sử dụng trong các thí nghiệm này là thủy tinh nước (WG). Thủy tinh nước là dung dịch của natri silicate (Na₂O.nSiO₂) hòa tan trong nước. Kết quả nghiên cứu vật liệu geopolymer từ hỗn hợp tro bay, bùn thải của nhà máy lọc nước Thủ Đức, Thành phố Hồ Chí Minh (Việt Nam) và thủy tinh nước (WG) được giới thiệu trong nghiên cứu này. Các oxit Al₂O₃ và SiO₂ hoạt tính trong tro bay và bùn thải có thể hòa tan trong thủy tinh nước và polyme hóa thành vật liệu geopolymer. Các mẫu thử nghiệm đã được ép ở áp suất cao 225 MPa tạo thành những mẫu hình trụ nặng khoảng 3 gam, chiều cao khoảng 18 mm và đường kính 10 mm. Các mẫu này được bảo dưỡng ở 110 °C trong 24 giờ và sau đó ở nhiệt độ phòng (30 ± 5 °C). Các phương pháp quang phổ hồng ngoại Fourier (FTIR) và kính hiển vi điện tử quét (SEM) đã được sử dụng để phát hiện cấu trúc vi mô và sự hình thành liên kết geopolymer của mẫu. Cường độ nén của các mẫu thử nghiệm 28 ngày tuổi cao hơn 3,5 MPa, pH nhỏ hơn 12,5, đáp ứng Quy chuẩn quốc gia Việt Nam về vật liệu không nung (TCVN 6477: 2016) và Quy chuẩn kỹ thuật quốc gia về tác động môi trường (QCVN 50: 2013) / BTNMT, tương ứng. Kết quả cho thấy một cách tiếp cận mới trong việc hóa rắn bùn thải cho các ứng dụng khác như sản xuất bê tông geopolymer hoặc vật liệu san lấp.

Từ khoá: vật liệu không nung, tro bay (FA), bùn thải (WS), thủy tinh nước (WG), dung dịch hoạt tính kiềm, geopolymer

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