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Durability of autoclaved construction materials of sewage sludge-cement-fly ash-furnace slag



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HIGHLIGHTS

• New construction materials are made from dewatered sewage sludge with autoclave process.

• Autoclaved specimens exhibit good long-term performance.

• The gel-like and honeycomb-like hydrated products of autoclaved samples are katoite and C-S-H phases.

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ABSTRACT

In the present work, we demonstrate an alternative for the final disposal of sewage sludge by using it as an additive in a mixture with cement, fly ash and furnace slag, which can potentially be used to develop newly promising construction materials by autoclave curing. The dewatered sewage sludge is obtained with fly ash and lime. These physical conditioners contribute to both dewatering process and solidifying/stabilizing of sludge.

Various mechanical properties such as flexural strength, compressive strength and the toxicity characteristic leaching procedure (TCLP) were evaluated. To evaluate long-term performance, different types of accelerated attacks, i.e. freezing-thawing cycles, accelerated carbonation, wet-dry cycles, and heat-cool cycles were also determined. The obtained test results were indicated that the autoclaved samples exhibit good long-term performance after evaluations of different durability tests. XRD patterns show that the hydration products of autoclaved samples are katoite and C-S-H phases, which mainly contribute to strength of autoclaved products. Morphologies of autoclaved samples also demonstrate the existence of the gel-like and honeycomb-like hydrated products. The results show that this new construction material could be applied as many construction and building materials, i.e. landfill liners and building blocks. © 2013 Elsevier Ltd. All rights reserved.

1. Introduction

The disposal of sewage sludge is becoming an increasing concern in many urban municipalities. Some traditional methods for disposal of sludge have been developed, such as applications of landfill, incineration, and utilization in agriculture [1]. Since sewage sludge contains heavy metals, organic matters, and high content of water, these traditional disposal methods could face long-term risk of environmental pollution or problems of uneconomical energy-consuming drying process. Therefore, many researchers have been seeking alternatives for the final disposal of sewage sludge by using it as a component in construction and building materials. Tay et al. carried out the research in the area of sludge reutilization as building bricks, lightweight aggregates, and cementitious materials [2]. Valls et al. demonstrated the application of dried sewage sludge or wetsludge as an additive in mortar or concrete [3–5]. Katsioti et al. investigated properties of stabilized/solidified admixture of cement–bentonite or cement–jarosite/alunite and sewage sludge as new construction materials [6–8].

Although many researchers have shown potential applications of sewage sludge utilized as building and construction materials, further investigations should be carried out. Firstly, long-term performance of non-conventional construction materials with the addition of sewage sludge should be paid more attentions [5,9]. Secondly, some construction or cement materials with the addition of sewage sludge have been prepared by traditional sintering process at higher temperature [2,10,11]; some composite binders with the addition of sewage sludge have been prepared and cured at room temperature [3–8]. Autoclave process could improve longterm performance of construction and building materials compared with curing condition at room temperature [12]. However, autoclave process for construction materials containing sewage

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sludge was seldom investigated in the previous literatures. Thirdly, the addition of wet sewage sludge causes lumps in mixtures because of its high water content and high organic compounds content [13]. Such inhomogeneous lump is potential the Achilles' heel when specimens are subjected to environmental erosion.

Dewatered sewage sludge conditioning with organic polymer i.e. PAM was commonly used in previous researches. However, fly ash and lime, inorganic materials, were proved as effective physical conditioners since fly ash and lime could act as skeleton builders to form a more porous and incompressible cake structure [14,15]. Conditioning with skeleton builders will produce cakes of high solids content that can be more easily disposed of. At the same time, fly ash and lime can be used in solidification/stabilization of sewage sludge and preparation of construction materials. In the present work, sewage sludge was conditioned with fly ash and lime in dewatering process, and then dewatered sludge was used in the following preparation of construction materials. This novel route combined the sludge dewatering with the following reutilization of dewatered sludge, which made the mixture homogenised easily and lumps diminished effectively because fly ash and lime could remain uniform rigid lattice structure in dewatered sludge cake with higher solids content and lower organic compounds content. The flow-sheet of the whole route combined the sludge dewatering with the following reutilization of dewatered sludge as construction materials is presented in Fig. 1. Dewatered sludge cake with addition of cement, fly ash and furnace slag were uniformly blended in a mechanical mixer. The homogenous mixture was pressed into shape with a hydraulic machine at a pressure of 20 MPa. Then shaped brick specimens were cured by autoclave process.

This work studied long-term performance of new construction material specimens made from dewatered sludge with conditioners of fly ash and lime as skeleton builders. Mineral phases and microstructure characteristics were investigated on specimens after being subjected to different types of accelerated attacks.



Fig. 1. The schematic of this study.

2. Experimental

2.1. Raw materials

2.1.1. Characterization of raw sewage sludge

Raw sludge (RS) used was the mixture of primary and secondary sludge that came from Longwangzui Wastewater Treatment Plant in Wuhan City, where municipal wastewater of 150,000 m³/d can be treated with an anaerobic–anoxic–oxic process. The main characteristics of RS are shown in Table 1. The X-ray diffraction was used to identify crystalline minerals in dried sludge sample. Significant amount of quartz was detected, shown in Fig. 2. The concentrations of heavy metals in the digestion solution of dry sludge were measured by standard method of USEPA 3050, as shown in Table 2.

2.1.2. Dewatered sewage sludge

Dewatered sewage sludge was prepared by filter press with the inorganic conditioner of fly ash and lime. Both dosages of fly ash and lime were 50 g/L (volume of RS with water content of 98.5%). Thus the mass ratio of dry sludge:fly ash:lime was 1.5:5:5 in dewatered sludge cake. Dewatered sludge cake with the content of water of about 45% was dried for several days in the ambient air. Then it was used as a component of construction material specimens when the water content of dewatered sludge was less than 30%.

2.1.3. Lime

Quick lime was used as a skeleton builder in sludge dewatering. Lime also played an important role in pozzolanic reaction between reactive SiO_2/Al_2O_3 in fly ash and lime under humidity conditions. The content of the free-CaO was 60 wt% in used lime. The chemical compositions of lime are shown in Table 3.

2.1.4. Fly ash

Fly ash was collected from electrostatic precipitator in a local coal-combustion power plant. Fly ash was used as another skeleton builder in sludge dewatering. At the same time, fly ash was used as a component of the mixture of autoclaved construction materials. Fly ash could provide reactive SiO_2 and Al_2O_3 that could take place pozzolanic reaction with lime and water. The chemical compositions of fly ash are shown in Table 3.

Table 1

Characteristics of raw sludge.

pН	Water content (%)	COD (mg/L)	TSS (g/L)	VSS (g/L)	VSS/TSS (%)
7.2	98.5	13090.6	13.0	7.5	57.7



Fig. 2. XRD patterns of dry raw sludge.

Table 2

Concentrations of heavy metals in dry raw sludge (mg/kg dry sludge).

Cr	Cu	Pb	Zn	Cd
7.2	176.5	153.3	265.7	76.5

Table 3

Chemical compositions of fly ash and furnace slag (wt%).

Type of raw material	SiO ₂	CaO	Al_2O_3	MgO	Fe ₂ O ₃	TiO ₂	P_2O_5	SO ₃	LOI ^a
Fly ash	42.39	2.99	30.04	-	7.72	1.13	0.26	1.37	4.15
OPC	20.60	58.80	5.40	3.30	2.90	-	-	2.40	4.00
Lime	6.70	60.10	-	1.50	-	-	-	-	22.60
Furnace slag	49.45	7.34	29.84	-	3.16	0.98	0.08	0.46	5.12

^a LOI = loss of ignition.

2.1.5. Furnace slag

Furnace slag is the bottom slag provided from a local coal-combustion power plant. Furnace slag was used as the aggregate with the size of <3 mm in the mixture of autoclaved construction materials, and furnace slag could form a permeable structure that could remain porous under high pressure. The chemical compositions of furnace slag are also shown in Table 3.

2.1.6. Portland cement

Ordinary Portland cement (OPC) 32.5 was used as a component in the mixture of autoclaved construction materials. OPC could improve the initial strength of specimens. The chemical compositions of OPC are also shown in Table 3.

2.2. Preparation of construction material specimens

The mixing proportions of specimens are listed in Table 4. The additions of dewatered sludge were designed as 40%, 50%, and 60%, respectively. The calculated proportions of specimens could be deduced and summarized in Table 5 since the mass ratio of dry sludge:fly ash:lime was 1.5:5:5 in dewatered sludge cake.

The mixtures were uniformly blended in a mechanical mixer. Suitable amount of water was added in the blending procedure. Steel mould of internal dimension $80 \text{ mm} \times 38 \text{ mm} \times 18 \text{ mm}$ was used to keep the size of brick specimens as one third of the size of standard brick in China. Brick specimens were pressed into shapes with a hydraulic machine at a pressure of 20 MPa.

2.3. Autoclave treatment

The following curing process was carried out with a steam at the temperature of 180 °C and at the pressure of 0.80 MPa for 4 h in a local autoclaved fly ash brick plant. Some organic matters in raw sewage sludge could volatilize in autoclave process, so the gases released from the closed container in autoclave process should be treated. On the other hand, steam disinfection and sterilization is an effective method of eliminating or killing all forms of pathogens in autoclave process, although raw sewage sludge contains pathogens and becomes infection source. Autoclaved specimens were tested for compressive strength, flexural strength, and durability after staying in the air for over 24 h. All of test procedures followed Chinese test methods for wall bricks (GB/T 2542-2003) [16].

2.4. Freezing-thawing cycles

Durability of autoclaved specimens was investigated for 15 freezing and thawing cycles [12,17]. One cycle comprised of freezing the specimens at -20 °C for 5 h and then of thawing in water of 20 °C for 3 h. After 15 cycles, the compressive strength and weight loss of dry specimens were measured.

Table 4

The designed proportions of autoclaved specimens (wt%).

Mix designation	Dewatered sludge	Fly ash	Furnace slag	Cement
S1 S2 S3	40.0 50.0 60.0	11.0 4.6	43.0 39.4 34.0	6.0 6.0

Table 5

The calculated proportions of specimens according to the compositions of dewatered sludge cake (wt%).

Mix designation	Dry raw sludge	Fly ash	Quick lime	Furnace slag	Cement
S1	5.0	28.6	17.4	43.0	6.0
S2	6.5	26.4	21.7	39.4	6.0
S3	7.8	26.1	26.1	34.0	6.0

2.5. Accelerated carbonation

Carbonation is a normal process in construction materials reacting with atmospheric carbon dioxide. Since normal carbonation process was slow, a mixture of gas with more than 60% (v/v) carbon dioxide was used to accelerate the reactions. The photo of equipment for accelerating carbonation is shown in Fig. 3. The Phenolphthalein indicator was used to determine the depth of carbonation. If the phenolphthalein is colorless in the core of broken specimens, it can be indicated that carbonation is completed. The coefficient of carbonation (K_c) could be expressed in the following equation [16]:

$$K_c = \frac{R_c}{R_0} \tag{1}$$

where R_c is the average compressive strength of 10 completely carbonated specimens, and R_0 is the average compressive strength of 10 comparative uncarbonated specimens.

2.6. Wet-dry cycles

Durability of autoclaved specimens was investigated in 60 wetting and drying cycles. One cycle comprised of heating the specimens at a temperature of 27 °C, 40 °C, or 60 °C respectively for 16 h, of cooling for one hour and then of immersing in water of 20 °C for 7 h. After every 10 cycles, the compressive strength and weight loss of dry specimens were measured [18].

2.7. Heat-cool cycles

Durability of autoclaved specimens was investigated in 60 heating and cooling cycles. One cycle consisted of heating the specimens at a temperature of $27 \,^{\circ}$ C, $40 \,^{\circ}$ C, or $60 \,^{\circ}$ C respectively for 6 h, and of cooling the samples at room temperature of $20 \,^{\circ}$ C for 18 h. After every 10 cycles, the compressive strength and weight loss of dry specimens were measured [18].

2.8. Microstructure characterization of construction material specimens

Broken brick specimens after strength tests were immersed and saturated in ethanol for 24 h in order to end hydration reaction, and then were dried at 40 °C for 48 h. A small section of dried sample was prepared for morphology analysis after coating with gold using SEM (JSM-5610LV and Sirion200); and the crystalline phase was investigated using powder XRD technique (D/Max-3B).



Fig. 3. The equipment of accelerated carbonation.

 Table 6

 Comparison of mechanical strengths before and after freezing-thawing cycles.

Mix designation	Dry raw sludge	Lime content (%)	Strength before freezing-thawing cycles		Results after freezing- thawing cycles	
			Flexural strength (MPa)	Compressive strength (MPa)	Compressive strength (MPa)	Weight loss (%)
S1 S2 S3	5.0 6.5 7.8	17.4 21.7 26.1	6.57 8.88 3.79	23.70 36.92 14.80	22.67 36.20 -	0.74 0.67 17.50

Table 7

Results of accelerated carbonation experiments.

Mix	Compressive strength	Compressive strength	K _c
designation	without carbonation (MPa)	after carbonation (MPa)	
S1	18.75	17.32	0.92
S2	32.12	27.41	0.85
S3	16.83	17.39	1.03

3. Results and discussion

3.1. Mechanical strength compared with results after freezingthawing cycles

The mechanical strength of autoclaved specimens is shown in Table 6. Results of freezing-thawing experiments are also comparatively shown in Table 6. From Table 6, the mechanical strengths of both S1 and S2 are much higher than that of S3. Firstly, since the amount of sludge in S3 is higher than the amount of sludge, more organic matters contain in S3, which could be contributed to the decrease of the strength of S3. Secondly, the content of lime in the S3 exceeds the necessary amount for hydration reaction. Therefore, both flexural and compressive strengths and the ability of resistance to freezing-thawing cycles decrease significantly. As a result, weight loss is as large as 17.5% for S3 specimen after 15 freezing-thawing cycles, and their compressive strengths measurementcould not be carried out because of destroying the shape of specimens with higher weight loss.

3.2. Accelerated carbonation

Results of accelerated carbonation tests are shown in Table 7. From Table 7, the coefficient of carbonation (K_c) of S1 and S2 is less than 1, which demonstrates that carbonation reaction could decrease the mechanical strength of autoclaved samples of S1 and S2. However, K_c of S3 is more than 1, indicating that carbonation process is positive for S3 because the carbonation process can reduce the proportion of free excessive Ca(OH)₂, which is somewhat soluble, and is transferred into calcium carbonate which fills the pores and is relatively insoluble [5].

3.3. Wet-dry cycles

The effect of wetting and drying cycles on the compressive strength and flexural strength of autoclaved samples kept at different temperatures is plotted in Fig. 4. In Fig. 4a and b, both compressive strength and flexural strength of S1 and S2 reduce after 10 cycles. However, both compressive strength and flexural strength do not reduce significantly from 10 cycles to 60 cycles. Most of compressive strengths of S1 and S2 keep strength of over 10.00 MPa after cycling; and most of flexural strengths of S1 and S2 retain strength of over 3.00 MPa after cycling, which indicates



Fig. 4. Effect of wetting and drying cycles on strength of autoclaved samples of (a) S1, (b) S2, and (c) S3 at different temperatures.

that autoclaved samples of S1 and S2 have a good resistance to the attack of wet-dry cycles.

In Fig. 4c, both compressive strength and flexural strength of autoclaved samples of S3 do not show tendency of reduction after cycling. Moreover, the increase of strength is significant after wet–dry cycles at 60 °C. It suggests that hydration reaction could continue to generate in wet–dry cycles, which is demonstrated in following investigations of the microstructure.

3.4. Heat-cool cycles

The effect of heating and cooling cycles on the compressive strength and flexural strength of autoclaved samples at different temperatures is plotted in Fig. 5. In Fig. 5a and b, both compressive strength and flexural strength of S1 and S2 reduce after 10 cycles. However, most of compressive strength and flexural strength of S1 keep strength of over 10.00 MPa and 3.00 MPa, respectively after cycling, and most of compressive strength and flexural strength of S2 keep strength of over 20.00 MPa and 5.00 MPa, respectively after cycling, which manifests excellent resistance to the attack of heat–cool cycles. In Fig. 5c, both compressive strength and



Fig. 5. Effect of heating and cooling cycles on strength of autoclaved samples of (a) S1, (b) S2, and (c) S3 at different temperatures.

flexural strength of autoclaved samples of S3 do not showtendency of reduction after cycling. Moreover, the increase of strength is significant after heat-cool cycles at 60 °C. Generally, results of heatcool cycles are consistent with those of wet-dry cycles in Fig. 4.

In current studies, different types of accelerated attacks, i.e. freezing-thawing cycles, accelerated carbonation, wet-dry cycles, and heat-cool cycles were investigated separately. The interactions of the several factors will be considered in further study, e.g. under simultaneous wet-dry cycle and heat-cool cycle.

3.5. Hydration products and morphologies

XRD patterns of autoclaved samples of S1, S2, and S3 are shown in Fig. 6. In general, mineral phases of three autoclaved samples are the same, comprising calcite, quartz, and hydration products i.e. katoite and C–S–H phases, in which hydration products mainly contribute to the strength of autoclaved products. However, calcite phase is the strongest major phase in S3 instead of quartz in S1 and S2. It indicates that more excessive calcium hydroxide transformed into a large amount of calcite in autoclaving curing for S3. Excessive calcite phases, not entering into hydration reaction, have a



Fig. 6. XRD patterns of autoclaved samples of S1, S2, and S3.

significantly negative impact on the strength of autoclaved samples, which is consistent with strength results in Table 6.

SEM images of autoclaved samples of S1, S2, and S3 are shown in Fig. 7. As shown by arrow-A, spherical fly ash particles are covered with a continuous "gel-like" coating, which is the typical morphology of hydration products. It is suggested that this coating is holding fly ash and other agglomerated particles together. As shown by arrow-B in Fig. 7a and b, the honeycomb-like hydrated products, known to be distinct C–S–H compounds, which are also identified in XRD pattern in Fig. 6, are found to be widespread on surface of solidified sludge mixed with agglomerates. However, in Fig. 7c, much less gel-like hydrated products surround the agglomerates for S3 compared with S1 and S2 (in Fig. 7b and c). More pores are significantly observed around particles for S3 compared with S1 and S2. It is consistent with the lower strength of S3 compared with S1 and S2 in Table 6.

3.6. Comparison of microstructure characterization after heat-cool cycles

Strength results between dry-wet cycles and heat-cool cycles are generally consistent with those presented in Sections 3.3 and 3.4. Therefore, microstructure characterization was selectively investigated after heat-cool cycles in this section. The XRD patterns of original S2 and S2 attacked after 60 heat-cool cycles at 60 °C are comparatively shown in Fig. 8. Mineral phases of both original S2 and S2 after cycles are the same, i.e. calcite, quartz, katoite, and C-S-H. However, relative intensity of different mineral phases changes significantly between original samples and samples attacked in 60 heat-cool cycles. Firstly, the intensity ratio of strongest peak of calcite to that of quartz dramatically decreases from original S2-S2 after 60 cycles. Secondly, intensities of corresponding peaks of typicalhydration products of katoite and C-S-H in S2 after 60 cycles are apparently stronger than those of original S2, which is positive for durability of solidified sludge samples. It is suggested that strengths of autoclaved samples could not decrease significantly after attacks of 60 heat-cool cycles, which is consistent with strength results of durability in Fig. 5.

The XRD patterns of original S3 and S3 attacked after 60 heatcool cycles at 60 °C are comparatively shown in Fig. 9. In general, trend of XRD patterns of S3 keeps consistent with above analysis on XRD patterns of S2 in Fig. 8.

SEM images of S2 and S3 after attacks of 60 heat-cool cycles are shown in Fig. 10. From morphologies in Fig. 10, more gel-like hydration products cover and hold the sludge and fly ash agglomerates, and more well-grown crystals fill open pores after heat-cool cycles. Therefore, SEM results also demonstrate a good



Fig. 7. SEM images of autoclaved samples of (a) S1, (b) S2, and (c) S3.



Fig. 8. Comparison of XRD patterns between the original S2 and S2 after 60 heat-cool cycles at 60 $^{\circ}\text{C}.$



Fig. 9. Comparison of XRD patterns between the original S3 and S3 after 60 heat-cool cycles at 60 $^{\circ}\text{C}.$



Fig. 10. SEM images of autoclaved samples of (a) S2 and (b) S3 after 60 heat-cool cycles at 60 °C.

Table 8

Results of the leaching tests of autoclaved samples (mg/L).

Samples	Cu	Zn	Pb	Ni	Cd	Cr
S1 S2 S3 Chinese regulatory standard [19]	0.2717 0.1672 0.3224 100	0.0527 0.0381 0.0701 100	0.1227 0.1182 0.1046 5	0.0501 0.0320 0.0803 5	0.0266 0.0261 0.0250 1	0.2990 0.2784 0.2577 15

durability of autoclaved samples mixed with dewatered sewage sludge after attacks of heat-cool cycles.

3.7. Leaching tests results

TCLP results on three autoclaved samples are summarized in Table 8. The concentrations of heavy metals leached from autoclaved samples are much lower than the regulatory limits accepted in China [19], which is consistent with the leaching results of heavy metals in construction materials or cement materials made from sewage sludge [10,11]. The lower leachability characteristics of the autoclaved samples could be attributed to the heavy metal ions solidified/stabilized in hydration products. The long-term environmental risk assessment of the autoclaved brick will be investigated in the further study, such as column leaching test.

5. Conclusions

Autoclaved samples were prepared from dewatered sewage sludge mixed with fly ash, cement, and furnace slag. Some conclusions can be made:

- (1) In general, the autoclaved samples exhibit good long-term performance after evaluation of different durability tests, i.e. freezing-thawing cycles, accelerated carbonation, wetdry cycles, and heat-cool cycles if suitable content of lime is controlled. Then this new construction material could be applied as many construction and building materials, i.e. landfill liners and building blocks.
- (2) XRD patterns show that mineral phases of autoclaved samples are calcite, quartz, and hydration products of katoite and C–S–H phases, in which hydration products mainly contribute to strength of autoclaved products. Morphologies of autoclaved samples also demonstrate the existence of the gel-like and honeycomb-like hydrated products, which cover and hold sludge agglomerates.
- (3) Microstructure characteristics are comparatively investigated between autoclaved samples and samples after attacks of heat-cool cycles. More hydrated products of katoite and C-S-H phases are identified in samples after attacks of heat-cool cycles, and more gel-like hydration products and more well-grown crystals are also observed in samples after attacks of heat-cool cycles. It is suggested that autoclaved samples do not decrease strength significantly after heat-cool cycles and show good resistance to the attack of heat-cool cycles.
- (4) Sludge was solidified/stabilized by a large quantity of hydration products, so heavy metal ions were solidified by hydration products, and the concentrations of heavy metals leached from autoclaved samples are much lower than the regulatory limits accepted in China.

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