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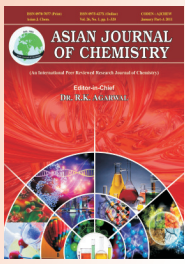

Preparation and Properties of Sludge and Coal Gangue Composite Polymer†

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In view of a larger number of wastewater treatment sludge and coal-product of waste rock disposal problems of environmental resource, studied the scientific preparation of the both things into new gelling geological polymer. Let 40 % sludge (< 5 μm) after roasted at 900 ºC for 45 min displace gangue to make the geopolymer has good compressive strength. XRD and SEM analyze the preparation of the novel geopolymer mechanism. The result shows that the effective composition such as CaO, MgO and SiO_2 of thermally activated sludge and the alkaline substances K_2O and Na_2O provided the alkaline environment to the polymerization reaction, Conducive the depolymerization of gangue aluminosilicate phase and the dissolution and diffusion of the depolymerized aluminosilicate complexes, accelerated the polymerization reaction. The results of this study enriched the selection of raw materials of geopolymers, helped the resource utilization of industrial wastes such as the sludge and waste rock containing the aluminosilicate phase.

Keywords: Thermally activated sludge, Coal gangue, Geopolymer, Performance analysis.

INTRODUCTION

Geopolymer is silicon-aluminium inorganic materials generated through mineral condensation of a tetrahedral aluminum silicon-cell of amorphous inorganic polymer three-dimension network. The excellent performance characterization of geological polymer such as high strength, high toughness, corrosion resistance, fire resistance, heavy metals stabilization and solid preparation make it award-winning attention[1].

For nearly 30 years, research on geopolymer has already developed from metakaolin geopolymer that consume kaolin resources to the stage of development using industry waste geological polymer.

Sludge is subsidiary product that produced in the production process in municipal sewage treatment plant, it contains large amounts of organic matter and rich in nitrogen, phosphorus, potassium and other nutrients, while containing heavy metals, bacteria and pathogens and so on. Emissions without any treatment will not only cause serious pollution to the environment, but also a serious waste of resources[2,3]. Gangue is the process of coal mining and washing, emissions-10 % to coal production, National "Twelve-Five" put forward the plan by 2015, coal gangue comprehensive utilization rate of industrial solid waste reaches 75 %. Energy-saving emission reduction target[4], this research has practical significance of recycling, environmental protection, in line with social reality of the need for efficient use and recycling of resources, expand common disposal of a variety of industrial solid waste in cities and towns, particularly developing a silicon-containing aluminum and calcium-containing industrial waste utilization of resources.

EXPERIMENTAL

Determination of optimum roasting temperature of sludge: The sludge used in the test is from Huainan city pioneered the first sewage treatment plant. Main chemical composition see Table-1. The main composition of sludge is similar to clay, apart from some orgainic matter. And it contains a certain amount of heavy metals, viruses, harmful substances such as pathogens and parasite eggs. Improper disposal of sludge will lead to be serious secondary pollution to the environment.Thermal activation process not only removes the free water, bound water and organic matter of dried sludge, can also make the phase change of inorganic mineral composition in sewage sludge occur which results in better activity and makes it easier to mix evenly with other raw material. The experiment for thermal-activated sludge by TG-DTA and XRD analysis is mapped as shown in Figs. 1 and 2. Fig. 1 shows that at 51.6 ºC appear an obviously endothermic peak appear

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TABLE-1
CHEMICAL COMPOSITION OF SLUDGE (wt. %)

Chemical composition	SiO_2	$A1_2O_3$	Fe_2O_3	MgO	CaO	SO_3	K_2O	Na_2O	LOI	Total
Percentage (%)	13.2	2.21	0.90	5.63	40.7	-	0.33	0.27	36.5	99.7

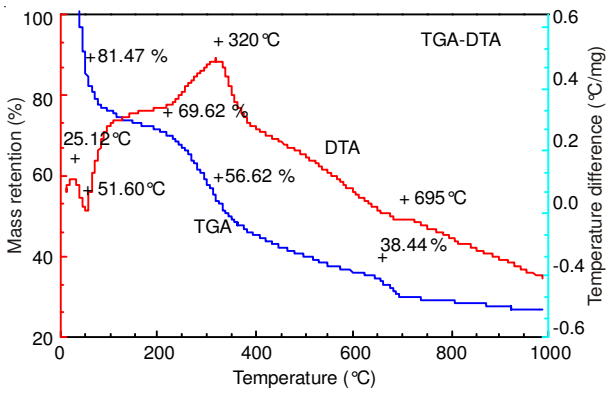


Fig. 1. TG/DTA thermal analysis of sludge

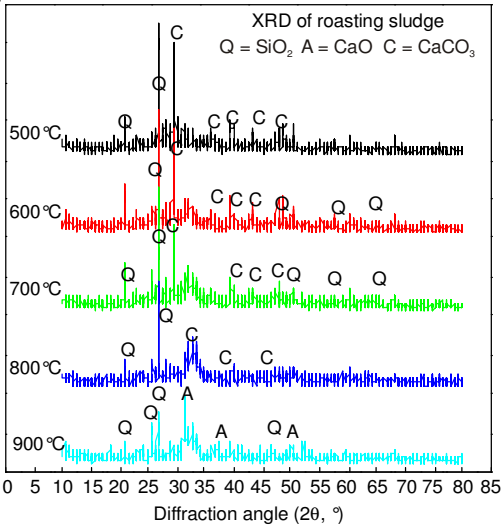


Fig. 2. XRD content phase analysis of thermally-activated sludge

at 51.6 ℃. This stage is mainly caused by free water evaporation endothermic. As the temperature rises at 320 ℃ a highlighted thermal spike appears due to combustion of organic matter volatile. When the temperature continued to rise beyond 695 ℃, DTA curve absorbed heat. This stage is mainly caused by inorganic mineral phase composition, morphologic changes and decomposition of carbonate crystals induced. At 800-900 ℃, the change of weight loss rate is very small, showing that sludge components are basically stable. According to Fig. 2, phase components of sludge calcined to maintain stability, so we can determine that 900 ℃ the optimum thermal activated temperature for sludge.

Coal gangue and its main components formation: Coal gangue in different regions have a certain different composition for different formation conditions. The coal gangue used procured from Huainan Mining Company, Xinzhuang zi coal preparation plant. Chemical composition is shown in Table-2.

Due to the high contents of Si, Al or something other element and the exist of volcanic activity and chemical activity[5] in coal gangue, it can be used for the preparation of geopolymer, this is one of the main ways of dispose coal gangue. Before coal gangue used in preparation of geopolymer, it should be crushed and through 60 mesh sieve.

Alkaline activators-sodium silicate: The water glass was used as the sodium silicate to stimulate the activity of sewage sludge and coal and to provide the alkalinity needed in the preparation of geopolymer[6], improve the properties of geopolymer by forming Al-O/Si-O, Si-O-Si/Si-O-Al keys. Solid content of water glass is 41.17 %, containing Na_2O 20.25 %, SiO_2 20.92 %, water 58.83 %, module M = n(SiO_2)/n(Na_2O) = 1. The dosage (Na_2O content introduced) in experiment is 10 %.

Sample preparation: Sludge mixed to coal gangue, the dosage range from 10 to 5 %, the detail shown in Table-3.

TABLE-3
TEST RATIO (WT %)

Number	1	2	3	4	4	6
Sludge (%)	0	10	20	30	40	50
Coal gangue (%)	100	90	80	70	60	50

Replace coal gangue with 10-50 % thermally activated sludge and alkali-activate by adjusted water glass. High quality of water-cement ratio is 0.25, water include those from water glass and those we added. Molding in 40 mm × 40 mm × 40 mm mould, then the sample conserved for 3d, 28d at room temperature 23 ℃, test the compressive strength of sample conserved with setted age. Finally confirm the best dosage of sludge according to mechanical properties. Preparing samples with 40 mm × 40 mm × 40 mm steel mold testing, demold after 24 h and continues to conserve to 3 days and 28 days, then examine the compressive strength.

RESULTS AND DISCUSSION

Test and analysis of compressive strength of sample prepared: Characterizing the activity of gelled material by analyzing compressive strength of cementing materials is a intuitive and effective evaluation methodologies. The main key indicators of this experiment mainly is compressive strength of geopolymer key indicators, pay close attention to ratio of raw materials when compressive strength is higher and analyze from micro-structure of sample. Test the compressive strength using universal testing machines after 3 days and 28 days conservation. Compressive strength test results shown in Fig. 3.

Analysis shows that the compressive strength of geopolymer compound prepared by sludge and coal are higher than the pure gangue in preparation of geopolymer. Strength

TABLE-2
CHEMICAL COMPOSITION OF GANGUE (wt. %)

Chemical composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	SO_3	K_2O	Ignition loss
Percentage (%)	20.61	25.33	4.39	10.39	0.08	2.91	3.28	0.9	35.69

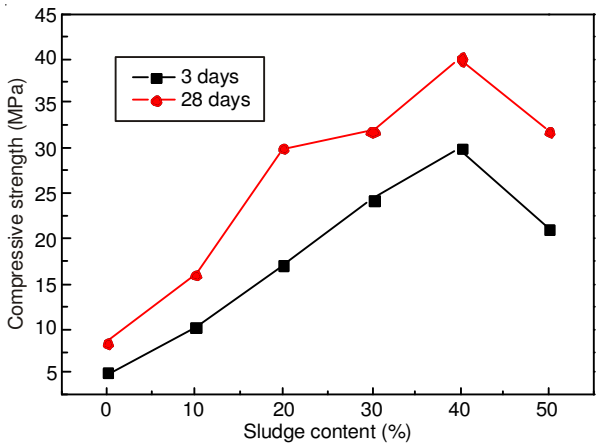


Fig. 3. Compressive strength of geopolymer prepared of sludge and coal gangue

of geopolymer of 28 days are higher than that of 3 days under 23 ºC conservation. The strength of geopolymer gradually increases when the ratio of sludge range from 10 to 40 %, but the strength has a decline trend when the ratio of sludge range from 40 to 50 %; compressive strength reached the highest when sludge mixed volume for 40 % in the preparation, obviously this ratio is a better proportion.

Geopolymer XRD analysis: 28d XRD pattern of the geopolymer produced from alkali-activated-sludge and coal gangue is shown in Fig. 4.

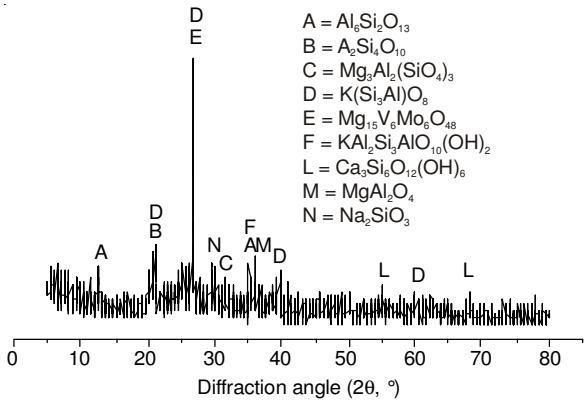
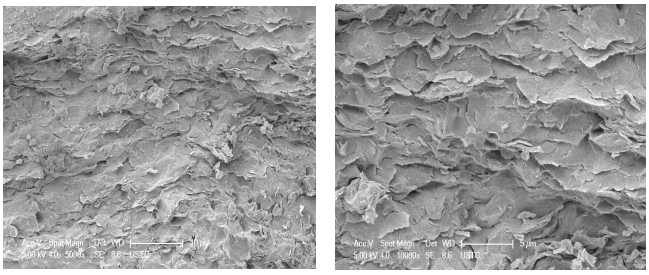


Fig. 4. 28d XRD pattern of the geopolymer prepared of sludge and coal gangue

XRD shows that the geopolymer aggregate product contains a lot of silica and aluminum oxide and higher concentrations of alkali metal polymers,even though its compositions are very complex; alkalinity is an important factor affecting the properties of geopolymer. The appearance of more alkali metal polymers comes mainly from the coal except for alkaline activators provided by alkali metals. The adequate alkalinity conditions accelerate the form rate of geological polymer, make geological polymer reunion more closely around the gangue particles. Due to the existence of chemical bond such as Al-O, Si-O, Si-O-Si, Si-O-Al, *etc.* silicon oxygen tetrahedron and aluminum oxygen tetrahedron structure of geological polymer has formed and then developed three dimensional mesh structure. It makes geological polymer cross linked closely, thus the polymer has better compressive strength and other performances.

Geological polymer by scanning electron microscopy (SEM) analysis: 28d SEM of sludge and coal gangue composite polymer is showed in Fig. 5(a-b).



(a) (b)

Fig. 5. 28d SEM map of the thermal activated sludge-coal gangue compound geological polymer

The analysis shows that the microscopic structure of coal is graininess through inorganic non-metallic materials microstructure. Volcanic activity of gangue comes from SiO_2 and Al_2O_3 decomposed from clay minerals. After thermal activation process, dried sludge are mainly including CaO, MgO and SiO_2 phase. And calcium components and alkaline material K_2O and Na_2O can be used as the alkaline activator of polymerization reaction provided more better alkaline environment,which increase the alkalinity of reaction system, conducive to the depolymerization of silicon aluminum phase of gangue and the dissolution and proliferation of silicon-aluminum complexes, accelerate polymerization reaction. Therefore, after joining the thermally activated sludge, geopolymer gelation is tightly wrapped around coal gangue particles.

Conclusion

XRD, DTA-TG analysis at different temperatures of thermally activated sludge shows that the best roast temperature of sludge is 900º. According to the experiment scheme which optimum water cement ratio is 0.25, sludge volume is 40 %, the polymer blocks with the dimension 40 mm × 40 mm × 40 mm are produced and the compressive strength reaches 39.8 MPa after the compressive strength of test specimens through maintenance in 23 ºC for 28-day. Alkalinity is an important factor affecting the formation and properties of geopolymer through XRD analysis. And geopolymer gelation is tightly wrapped around coal gangue particles through SEM analysis which increas the compressive strength of geopolymer. Study results open up new areas of industrial solid waste for better utilization of sludge and waste rock containing silicon aluminum.

REFERENCES

1. E. Wang, W. Ni and H. Sun, *Mult. Util. Miner. Res.*, **2**, 30 (2005).
2. Z.-F. Zhang, G.-H. Wang, B. Yu and H.-L. Zhang, *Min. Metall.*, **16**, 69 (2007).
3. L. Yang, S.-Q. Luo and Y.-M. Zhang, *Chin. J. Environ. Eng.*, **4**, 1177 (2010).
4. B.-W. Gu and P.-M. Wang, *J. Build. Mater.*, **1**, 6 (2009).
5. X.-H. Wang, W. Ni and Y. Huang, *Mult. Util. Miner. Res.*, **2**, 30 (2011).
6. A.-G. Wang, D.-S. Sun and P.-H. Hu, *J. Hefei Uni. Technol.*, **31**, 617 (2008).