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Synthesis of sodium waterglass from white rice husk ash as an activator to produce metakaolin-based geopolymer cements

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ABSTRACT

Rice husk from source in Cameroon was leached in HCl (5 M) to remove the most of metallic ingredients and then calcined at 600 °C in order to produce white rice husk ash. This white ash were applied for producing sodium waterglass with different molar ratios SiO_2/Na_2O (0.31; 0.47; 0.62; 0.78; 0.93; 1.09 and 1.25) and then used to synthesize metakaolin-based geopolymer cements. The obtained white rice husk ash shows the loss of crystalline mineral and reveals high amorphous silica with quartz as impurity. Geopolymers GPi (i varying from 1 to 7) were obtained using different synthesis sodium waterglass (NWG) with a mass ratio NWG/MK=0.87. It could be observed that the 28 days compressive strength (4/5/7/9/32/34/36 MPa) increase with increasing the molar ratios SiO_2/Na_2O defined in this work in the course GP1/GP2/GP3/GP4/GP5/GP6/GP7. The micrographs show the formation of more geopolymer gels when the molar ratios SiO_2/Na_2O in alkaline activators are between 0.93 and 1.25. Sodium waterglass from white rice husk ash proved to be an effective alkaline activator in geopolymers preparation. It can be concluded that it is possible to replace quartz sand and sodium carbonate which is responsible to greenhouse gas emitted during the production of commercial sodium silicate solution by using rice husk as silica sources.
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1. Introduction

Geopolymers recently emerged as a new class of inorganic aluminosilicate polymeric materials. The similar materials were synthesized for the first time in 1940 by Purdon [1] and again in the late 1950s by Glukhovsky [2]. The term geopolymer was introduced by Davidovits [3] in the early 1970s to denote their inorganic polymers and structural similarity to organic polymers and is commonly used nowadays [3,4]. Geopolymers are a new class of inorganic polymers that have been considered as good candidate materials for many applications including fire resistant and refractory panels, adhesives and coatings, waste encapsulation material, etc. These inorganic polymers are processed by polycondensation of aluminum and silicon monomeric or oligomeric species in metal alkali-activated solutions [3]. The geopolymer precursors can be obtained from different aluminosilicate sources such as clays, metakaolin, volcanic scoria, etc. Commercial sodium silicate solutions which are added to sodium hydroxide solution and then mixed with the aluminosilicate source to activate the dissolution and polymerization process are very expensive compared to sodium hydroxide pellets.

Rice is one of the major food crops in the world. Its production generates a great amount of waste in the world namely rice husk. Cameroon is among of countries which produce rice in Africa. Rice is grown mainly in two agro-ecological zones, the Western Highlands (North-West and West Regions) and the Northern Region (North and Far-North Regions), but it's also found on smaller areas in the Centre, South-East and East Regions. The major rice cultivation projects in the country are in Maroua and Kousseri in the Far North Region and Ndop in the North-West Region [5]. The production of rice in the different Region of Cameroon generates a high amount of rice husk. Presently, having no commercial value in itself, rice husk usually ends up burned in open spaces, thus causing environmental pollution and disposal problems. In order to conserve energy and resources, efforts have been made to burn the husk under controlled conditions and to use the resultant material as silica sources to produce sodium waterglass which is

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very important during the synthesis of geopolymers.

The manufacture of commercial sodium silicate solution is energy intensive because the syntheses involve silica sand and sodium carbonate treated at approximately 1400 °C [6]. For the synthesis of sodium carbonate, energy is expended via the Solvay manufacturing process i.e. ammonia reacts with CO_2 (derived from calcined limestone) which is then introduced to brine which reacts to create sodium carbonate. In the synthesis of the sodium silicate solution, sand and soda ash (sodium carbonate) are mix and melt, a process that requires significant energy due to high temperature and pressure needed. The total CO_2 emission during the synthesis of sodium silicate estimated at 1.514 kg CO_2 emitted per Kg of sodium silicate [7]. This process is considered very expensive due to the energy consumption with the fuel burning to reach high temperature. Besides producing considerable air pollution by emissions such as carbon dioxide, dust, nitrogen and sulphur oxides [8]. Previous researchers [9–11] used waste glass to synthesis sodium waterglass. But during their process, they used sodium carbonate to enhance the dissolution of silica while the synthesis of sodium carbonate required other chemical product such as brine, ammonia and calcined limestone. The calcination of limestone emitted CO_2 at the atmosphere. The synthesis of sodium waterglass from pure silica obtained by only rice husk ash with sodium hydroxide pellet and distilled water as starting materials and then using its to produce geopolymer cements could be great interested.

The purpose of this work was to investigate the possibility to synthesize sodium waterglass from white rice husk ash and used its as an activator to produce metakaolin-based geopolymer cements. The process used in this work consumes low energy compared to current melting method used to produce commercial sodium waterglass. This process may decrease significantly the amount of greenhouse gas emission due to the manufacture of commercial sodium waterglass using Na_2CO_3 and sand (SiO_2). The starting materials were characterized by their chemical and mineralogical compositions and infrared spectroscopy. The effects of the molar ratios SiO_2/Na_2O used for the synthesis of sodium waterglass on the formation of metakaolin-based geopolymers have been studied using infrared spectroscopy, TG analysis, X-ray diffractometry (XRD), environmental scanning electron microscope (ESEM) coupled with energy dispersive X-ray spectroscopy (EDX) and compressive strength.

2. Materials and experimental procedures

2.1. Materials

Kaolin used in this work was extracted from Mayouom in the West Region in Cameroon and rice husk was collected from a local agro-industry from Ndop, Department of Ngoketundjia, Region of North-West in Cameroun. Kaolin was crushed in a ball mill with a porcelain jar and spheres of high alumina as grinding medium and then passing through a 90 mesh sieve. The powder of this kaolin was calcined at 700 °C for 4 h at a heating rate of 1 °C/min in order to get a highly reactive metakaolin (MK). This temperature was chosen according to the findings of Elimbi et al. [12]. NaOH pellet is the commercial product by Merck (KGaA, 64271 Darmstadt, Germany, with 99% purity) was used as sodium source. Analytical grade HCl solution (Merck and Co.) was also used as the acid treatment. The kaolin was previously studied by Djangang et al. [13]. They reported that this kaolin consists of approximately 82.3% of kaolinite, 8.0% of illite, 4.0% of anatase, 0.9% of goethite and 2.9% of quartz. Rice husk ash was previously studied by Tchakouté et al. [14] and the chemical composition of rice husk ash and pure rice husk ash are shown in Table 1.

Table 1
Chemical composition of K, RHA and PRHA in mass percent.

Oxide	K [13]	RHA [14]	PRHA [14]
SiO_2	44.58	83.05	93.49
Al_2O_3	35.80	1.82	0.96
Fe_2O_3	0.79	0.58	0.28
K_2O	0.94	5.65	0.96
TiO_2	3.96	0.10	0.05
MgO	/	3.59	0.75
Na_2O	/	0.13	0.11
CaO	/	0.69	0.56
SO_3	/	0.34	0.09
P_2O_5	0.37	3.81	0.68
ZnO	/	0.03	0.01
MnO	/	0.15	0.03
Rb_2O	/	0.03	0.004
SrO	/	0.003	/
ZrO_2	/	0.005	0.003
LOI	13.28	/	/

LOI: Loss on ignition at 1000 °C.

2.2. Experimental procedures

2.2.1. Purification of rice husk ash

In order to remove the metallic ingredients and produce completely pure silica, rice husk was leached in HCl (5 M) for 24 h. After leaching, the ash was thoroughly washed with water until neutral pH and then dried in air. The dried rice husk was burned at 600 °C for 2 h at a heating rate of 5 °C/min to obtain pure white rice husk ash (PRHA).

2.2.2. Synthesis of sodium waterglass

Seven sodium waterglass were prepared by adding each different mass of the powder of pure rice husk ash to sodium hydroxide pellets with different molar ratios of SiO_2/Na_2O are 0.31; 0.47; 0.62; 0.78; 0.93; 1.09 and 1.25. The assembly was mixed with a 200 mL of distilled water for 2 h at 80 °C using a magnetic stirrer to enhance the dissolution of silica. The obtained waterglass were stored at ambient temperature for at least 1 d before being used to allow a full silica dissolution and equilibration [15]. The sodium waterglass (NWG) obtained from the aforementioned molar ratios are labeled NWG1, NWG2, NWG3, NWG4, NWG5, NWG6 and NWG7 respectively (shown in Table 2).

2.2.3. Synthesis of geopolymer cements

Geopolymer cements were prepared by adding each NWG gradually to metakaolin in a mortar and mixed for 5 min, obtaining series of geopolymer cements GP1, GP2, GP3, GP4, GP5, GP6 and GP7 which correspond to the geopolymers obtained from NWG1, NWG2, NWG3, NWG4, NWG5, NWG6 and NWG7 respectively. The NWG/MK mass ratio was kept constant at 0.87 obtaining a suitable workability. The fresh geopolymer paste samples were rapidly molded into cylindrical PE-containers with a diameter of 30 mm and height of 30 mm [16]. During hardening of the geopolymer paste samples, the samples were covered with a thin film of polyethylene for 24 h at the ambient atmosphere

Table 2
M ratios SiO_2/Na_2O content in the synthesis sodium waterglass from rice husk ash and compressive strength of geopolymer cements and standard deviation.

	NWG1	NWG2	NWG3	NWG4	NWG5	NWG6	NWG7
SiO_2/Na_2O	0.31	0.47	0.62	0.78	0.93	1.09	1.25
Compressive strength (MPa)	3.58	5.13	6.50	9.28	31.74	33.74	36.29
Standard deviation (MPa)	0.13	0.12	0.20	0.08	0.63	0.34	0.46

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