

Investigation of Red mud, Rice Husk and Anthill based Geopolymer composite for Engineering applications

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Abstract

Quite a lot of geological surveys concede that Guinea is home to 33% of the world's known bauxite reserves. The industrial scale conversion of raw materials such as bauxite into products or intermediates releases huge quantities of waste posing environmental concerns. Red mud RM is a solid waste resulting from the digestion of bauxite ores with caustic soda for alumina production, treating and disposal of RM has become a concern. Rice is the second largest produced cereal in the world, it is composed by approximately 20% of rice husk RH, which contains fibrous materials and silica. Due to its high percentage in the grain composition, the RH is considered a by-product that creates disposal and pollution concern. Rice husk ash RHA is an industrial waste produced by burning rice husk primarily for the generation of electricity. Because of its high silicon content, RH has become a source for preparation of several engineering materials. The alkali activation converts industrial waste into valuable engineering materials called geopolymers GP. In comparison to ordinary portland cement OPC, GP are produced with significant reduction of energy consumption and CO₂ emissions. They represent alternative binding materials with huge potential for civil engineering applications or for waste management [1]. In this chapter RM, RH and anthill AH soil are used to prepare a ternary blended material for building purposes in laboratory conditions. The effect of blending ratio on the compressive strength were investigated. Raw materials and the final products are characterized by several methods: XRD, SEM.

Keywords: Bauxite, red mud, rice husk ; anthill, geopolymer; compressive strength

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I. INTRODUCTION

Due to the growth of population and the needs for infrastructure, it is obvious that the demand for space, natural resources, and energy will grow. The increase of industrial activity gives rise to an increase of quantity of waste that can have a negative impact on the environment and human life. Among the solid wastes we can cite red mud, fly ash, ground granulated blast furnace slag, rice husk ash.

In recent years, there is a growing demand of development of new low-cost technologies that use the industrial wastes causing damages to health and environment. Inorganic polymers or geopolymers have been recently gaining attention as an alternative binder for concrete because of its potential to lower the environmental impact of construction, to utilize waste as raw materials of aluminosilicates, and to enhance the material performance.

The involvement of OPC production to greenhouse gas production in the world is estimated to be approximately 1,35 billion tons per year or about 7% of the total greenhouse gas emissions into environment [2].

Investigations suggested that the production of a tonne of OPC releases as much as an equivalent tonne of CO₂. In this way it is the most energy-intensive material produced after steel and aluminium [3]. Because of the heavy and growing demand for concrete, OPC producing industries still do not anticipate in the foreseeable future any major changes to reduce CO₂ emissions.

Fig.1 [5] illustrates the quantities of CO₂ emitted by material binders depending on their OPC content. It shows that in the earlier period, binders were made exclusively of OPC with about 1000.

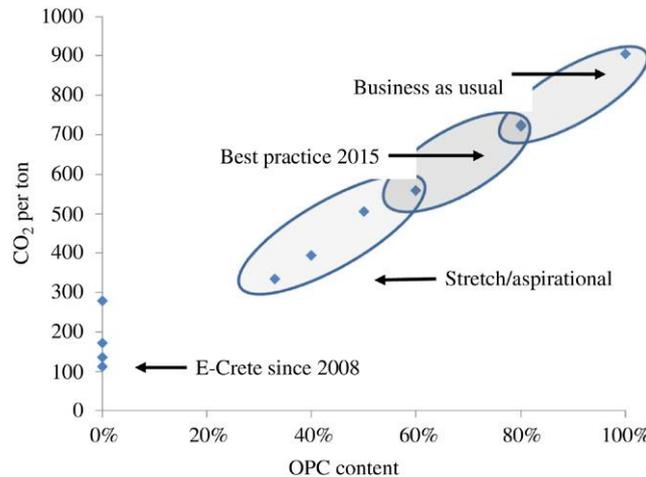


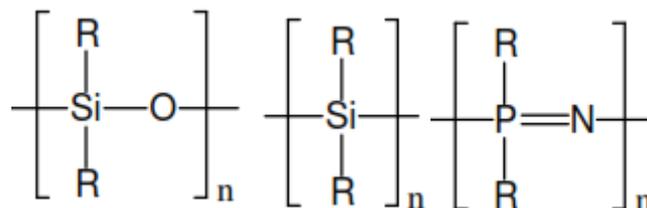
Fig.1 [5]:CO₂ emissions of variouscement binders as a function of OPC content.

One of the reasons for developingresearch on geopolymers are partlyenvironmental in order to reduce CO₂ emissions and footprint relative to OPC, and partlyeconomical in particularwhenindustrialbyproductssuch as RK, RHA can beused as aluminosilicate source or puzzolanas.Inthisstudy, engineering properties of a GP materialproducedfrom the ternaryblend of RM, RHA, AHsoilandmixed in an alcali-solution are investigated.

I. GEOPOLYMERS

Generally apolymeris a molecule, consisting of the repeatedsequence of the same pattern, the monomer (from Greek monos: one or only one, and meros: part), connected to eachother by covalent bonds. In the molecule A-A-A-A-A-A-A = [-A-]. The constituent unit isA;itisformed of a group of atomsthatrepeatsitself, At the molecularlevel, mostpolymers are in the form of "long and flexible thread. The chemicalreactionsallowing to passfrom a monomer A to the macromolecule [-A-] are calledpolymerization.

In addition to polymersthatcontainonlycarbon or other 'organic' atoms in thechain, polymerscontaining 'inorganic' atoms can alsobeformed,These compounds are of interestbecausetheyoffer the opportunity to developnew technological applications due to theirreactivity, structure, and physicalproperties, The mostwidelydevelopedcontainsilicon as polysiloxanes and polysilanes.Anothercommon class are the polyphosphazenes,



Polysiloxanes Polysilanes Polyphosphazenes

Polysiloxanesrepresent the most important inorganicpolymerswith regard to commercial applications : medical:prosthetics, catheters, contact lenses, drugdelivery capsules; non medical: e.g,elastomers, adhesives, lubricants, water repellents, molds,cosmetics.Polysilanes have a polymer backbone containingonlysiliconatoms. A siliconchainallowselectrondelocalisation of sigma electrons. Many uses of polysilanesderivefrom the electronmobility. Most uses are in the electronicsindustry as photoresists and precursors to siliconcontainingmaterials [6].

A novel type of binding material with different labels, but generally known as “geopolymers”, appears like an option to reduce greenhouse gas emissions from the cementous material production [7]since it has a very small greenhouse footprint when compared to traditional concretes [8]. The technique still utilizes industrial wastes or by-products in their formulation and it can offer a possibility of a solution to the immobilization of toxic and radioactive wastes[9]. This explains the growing interest in academic and commercial areas recently, where the potential for CO₂ emission reductions can be appointed as the key factor [10].

I.1. Nomenclature and definition

The term geopolymer was first used by J. Davidovits in the 1970's for alkali-activated alumina-silicate binders [11]. After Davidovits, the "geo-" part refers to the Greek *Gaia* which means earth, as geopolymers mainly consist of aluminium and silicon oxide which are the most common materials in the earth's crust. Other authors attribute the prefix "geo" to the constitutive relationship of the binder to geological materials such as natural stone and/or minerals [12]. The "polymer" part refers to the composition of the geopolymers which is a three-dimensional network of monomers consisting of aluminate and silicate tetrahedra.

The term *Geopolymer* covers a class of aluminosilicate materials with potential application in a number of engineering areas, basically as a substitute for OPC and for innovative high-tech composites. Advanced composite materials are products for space, aerospace, defense, marine, petrochemicals and effluent treatment etc. The geopolymerization process involves a chemical reaction between red mud and alkali metal silicate solution under highly alkaline conditions. The product of this reaction is an amorphous to semi-crystalline polymeric structure, which binds the individual particles of red mud transforming the initial granular material to a compact and strong one.

GP can be prepared from a range of aluminate and silicate materials such as metakaolin, flyash, blast furnace slag, rice husk ash and other *puzzolana*. Pozzolanas are defined as siliceous or siliceous and aluminous materials which in themselves possess little or no cementing property, but will in a finely dispersed form in the presence of water chemically react with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties [13].

I.1. Sustainability of GP development

The primary function of a geopolymer is to act as a cementitious binder and replace OPC in civil engineering manufacture or provide complementary products. Some of the advantages that geopolymers have over OPC is their high compressive and flexural strength, their very high temperature resistance, a high resistance to acid, and the ability to utilize multiple waste or byproduct streams [14].

Geopolymer binders have the potential to incorporate cations, anions, and organic species within their three-dimensional structure [15]. OPC can also trap impurities, though the acid resistance of geopolymer binders allows for a much wider range of safe receiving environments. A significant ecological and marketing benefit reported for geopolymer binder over OPC is the reduction in CO₂ emissions. One tonne of OPC releases 0,55 t of CO₂ from the calcination of limestone and the combustion of carbon-based fuel for heat and power generation produces an average additional 0,40 t of CO₂. Comparatively, geopolymer production creates only between 0,2 and 0,5 t of CO₂ per tonne of product, depending on inclusion of lifecycle and transport factors [16].

An industrial sustainable approach, utilizing such residues more effectively, would maximize the community benefit from the consumption of limited resources and reduce the requirement for production of natural, virgin resources, with its associated environmental impacts, [17].

The use of multiple and varied feedstock in GP synthesis has led to a focus on understanding the chemical basis of the amorphous reactive components. This in turn allows GP products to be formulated with predictable performance properties, without the requirement for trial and error on the basis of new or variable feedstock flows [18].

II. FEEDSTOCK FOR GP PRODUCTION

The main parent materials for the large-scale industrial production of GP include: concentrated caustic solution with dissolved silica or alumina ;



Figure 2 : a)RM

b) AH

a source of caustic soluble alumina. These materials must be available in sizeable quantities and at appropriate cost for commercial application. Basing on their origin, caustic soluble alumina or caustic soluble silica are natural (clay minerals) or industrial (mainly by-products).

In Guinea a substantial quantity of RM as caustic soluble alumina results from the production of alumina at the ACG plant; and the conversion of the huge bauxite reserves into alumina will produce an equivalent quantity of RM. Moreover the increase of rice production and its treatment will deliver RH containing enormous reserve of caustic soluble silica. In this way suitable conditions and framework for a sustainable GP industry are set.

II.1. Red mud

RM is characterized by strong basicity even with a high water content, because of the presence of huge amount of sodium hydroxide used to extract silicates and alumina. The amount of bauxite required to produce one ton of alumina and, thus the resulting amount of RM varies considerably depending on each deposit or district as shows Fig.3 [19].

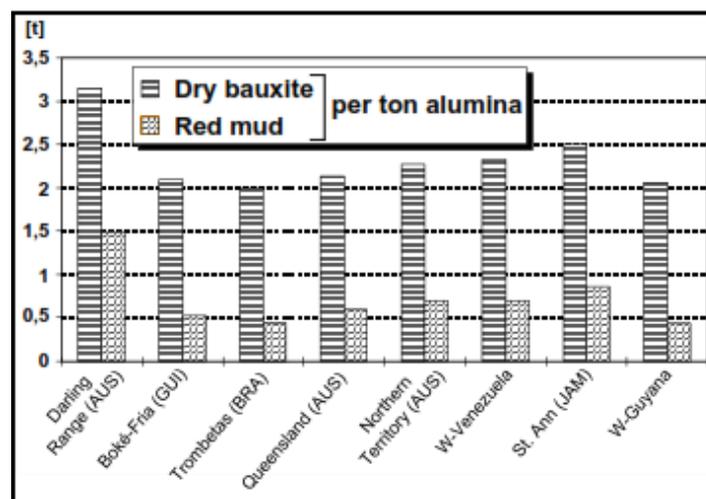


Fig.3 [19] : Bauxite required and red mud produced per ton of alumina for eight selected major bauxite districts.

The amount of the RM generated, per ton of the alumina processed, varies greatly with the type of the bauxite ore used from 0,3 ton to 2,5 tons for high and very low grade bauxites respectively. The storage of large quantities of RM is expensive (between 1 and 2% of the price of alumina), requiring large disposal areas (about 1 km² for five years of production for a factory producing 1 million tons of alumina per year) and causing serious environmental problems [20].

II.2. Rice husk and Rice husk Ash

Rice is the second largest produced cereal in the world. Its production is geographically concentrated in Asia with more than 90 percent of world output. Analysis of the Guinea's food balance sheet shows rice as the major food produced in the country, accounting for 61% to 69% of available cereals.

Rice is composed by approximately 20% of RH, which contains a fibrous material and silica. It is reported that approximately 0,23 tons of RH is formed from every ton of rice produced [21]. Therefore, due to its high percentage in the grain composition, the husk is considered as a byproduct in the mills and creates disposal and pollution problems. It is one of the most intractable agricultural wastes known to man, because it's tough, woody and abrasive nature along with high silica content makes its proper disposal very difficult. RHA is an industrial waste produced by burning rice husk primarily for the generation of electricity, a kind of sustainable biomass energy. The main component of the ash is silica (90–95 wt. %), existing predominantly in amorphous and partly in crystalline phases. Due to its high silicon content, RHA has become a source for preparation of elementary silicon and a number of silicon compounds [22], especially silica, silicon carbide [9], Silicon and silicon compounds are utilized in semiconductors, advanced ceramics, optical fiber,

II.3. Anthill

Anthills or termite mounds are natural clay soils that are very cohesive in nature, and therefore must have a high plasticity index [23]. They are made up of soil grains that are coated with sticky rapidly hardening secretions from the recta and mouths of ants

IV. MATERIAL AND METHODS

In this study, RM, RHA and AH constitute the materials for the preparation of the geopolymer. Red mud was used as the primary source of reactive alumina, it was provided by the Alumina plant ACG/Fria in Guinea. The raw RH was brought from a local rice mill in Sinko; Prefecture of Beyla; Guinea. It was burned at 600°C for 1 hour, grinded for 30 minutes and then processed into RHA in the Laboratory for Material Research; Polytechnic Institute, University of Conakry. Anthills were collected in savanna districts of Dinguiraye and Siguirii in Guinea. Sodium hydroxide (purity quotient: 99%, Merck KGaA, Germany), and deionized water were purchased at the Pharmacie Centrale de Guinée in Conakry; Guinea. RM, RHA and AH processing involves drying, crushing, grinding and determination of the chemical composition. The raw materials were initially oven dried to constant weight at 50°C during 72h to ensure that all the moisture had been removed. They were then crushed and grinded to pass a 0,30 mm mesh sieve using a pestle and mortar until a suitable ratio for coarse particles to fine particles was achieved. RM, RHA and AH as fine particles were characterized by X-ray diffraction (Shimadzu diffractometer, model XD-7A, with radiation Cu-K α) and by scanning electron micrograph (SEM; Model 2000FX, JEOL Co). The mineral composition was determined by atomic absorption spectrometry (Analytik JENA, Vario 6, Germany). Chemical elemental analyses were performed by an energy-dispersive X-ray spectroscopy (EDXS). The mix-ratios were adapted from [28] where the specimen TGPx stands for ternary geopolymer and x for the mix ratio as shows table 1.

Table 1: raw materials entering in the specimens amount (w %)

| Specimen | Proportion (%) | | |
|----------|----------------|------|------|
| | RM | RHA | AH |
| TGPx | | | |
| TGP1 | 100 | 0 | 0 |
| TGP2 | 0 | 100 | 0 |
| TGP3 | 0 | 0 | 100 |
| TGP4 | 50 | 50 | 0 |
| TGP5 | 50 | 0 | 50 |
| TGP6 | 0 | 50 | 50 |
| TGP7 | 66 | 17 | 17 |
| TGP8 | 17 | 66 | 17 |
| TGP9 | 17 | 17 | 66 |
| TGP10 | 33,3 | 33,3 | 33,3 |

For the mixture design, 3 replicates of each specimen were prepared.

The 10 molar NaOH solution as alkali activator was added at 20% by weight of dry material to the powder mixture. The mixing of the blends was carried out by Heidolph ST-1 Laboratory stirrer at two different speeds; 100 rpm for 2 min and 200 rpm for 15 min to insure their homogeneity and avoid bubbles and agglomeration into the sample and to make certain the reaction between the powder and NaOH solution, resulting in the formation of a product having a paste consistency.

The resulting products were immediately poured into cubic molds 50x50x50 mm and left at room temperature. Curing was carried out by keeping the geopolymer specimens during 28 days in a laboratory ambient; the specimens were then demolded.

The compressive strength of the synthesized RM-RHA-AH presumed geopolymer materials were tested on a testing machine. The compressive strength was measured on a Shimadzu apparatus (Model: AG-X/R Refresh). The water absorption test was also performed after the ASTM C140.

V. RESULTS AND DISCUSSION

Chemical composition of RM, RH, RHA and AH

The grinded and sieved RM, RHA and AH subjected to characterization before mixing, are presented in Fig.4a ; b and c relatively. The mechanical processing of the material into finest particles did not affect their color.



Fig.4 : a) GrindedRMb) RHAc) AH

The analysis shows several minerals in parent materials such as Al₂O₃, SiO₂, Fe₂O₃, Na₂O, K₂O, CaO, TiO₂ and others (table 2). Substantial quantities of Al₂O₃ and SiO₂ as monomers required for the building of GP are present.

Table 2 : Chemical composition of RM, RH, RHA and AH

| Raw Material | Al ₂ O ₃ | SiO ₂ | Fe ₂ O ₃ | Na ₂ O | K ₂ O | CaO | TiO ₂ | Others | LOI |
|--------------|--------------------------------|------------------|--------------------------------|-------------------|------------------|------|------------------|--------|-------|
| RM | 18,98 | 4,52 | 49,90 | 2,60 | 0,05 | 0,87 | 5,62 | 0,94 | 16,52 |
| RH | 0,06 | 23,77 | 0,13 | - | 1,08 | 0,25 | - | 0,81 | 78 |
| RHA | 1,12 | 90,90 | 0,54 | - | 4,66 | 1,41 | - | 0,60 | 0,77 |
| AH | 11,75 | 65,14 | 5,67 | 0,31 | 2,15 | 1,1 | 1,51 | 2,73 | 9,64 |

As main constituent of RHA and AH, SiO₂ contributes much to the formation of the skeletal structure of GP. The figure 5 below shows its predominance in RH and AH ; 90% and 65,14% respectively. While the Fe₂O₃ is relatively high in RM and AH. The high LOI of RM and AH due to the burning of organic compound, may contribute to the formation of pores in the presumed GP specimens.

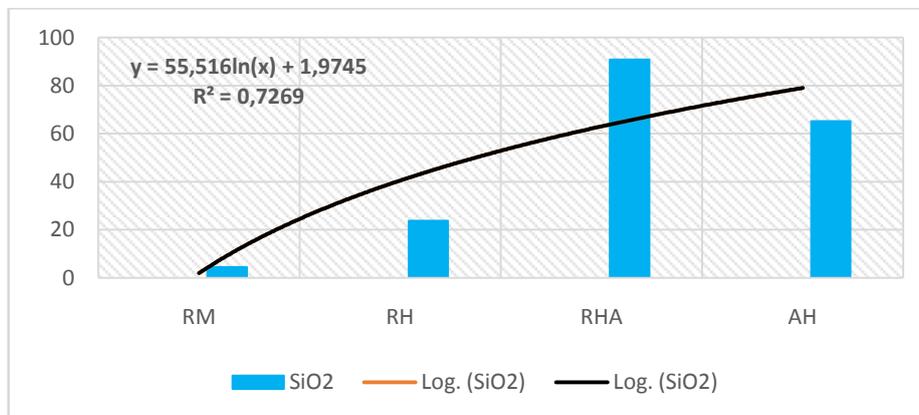


Fig.5: The silica content in parent materials

V.1. Microstructure of parent materials

RM

The XRD diagram (Fig.6) below shows some unidentified peaks and a few sharp peaks that are mainly from hematite and calcite, but no observable broad humps, suggesting that the amorphous phases are not present at large quantity.

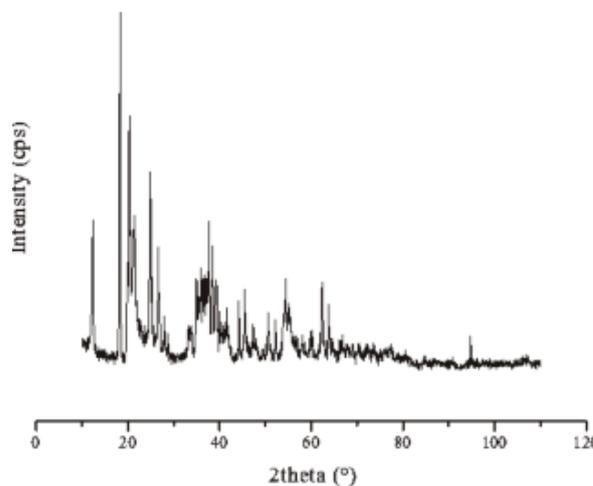


Fig.6. XRD diffractogram of raw RM

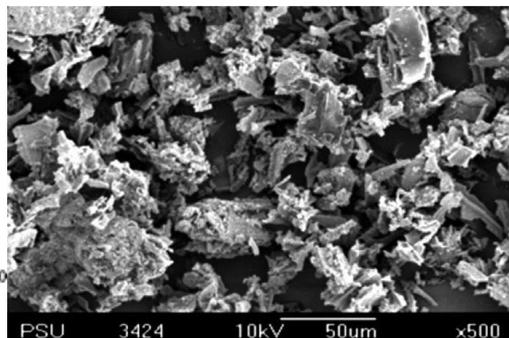


Fig.7. SEM micrograph of raw RM

RM contains mainly hematite (Fe_2O_3), diaspor ($\text{AlO}(\text{OH})$), gibbsite ($\text{Al}(\text{OH})_3$), calcite (CaCO_3), quartz (SiO_2), anatase (TiO_2), and goethite (FeOOH). By comparison with its chemical composition (table 1), alumina mainly presents as amorphous phases. Thus RM provides mainly Al (in the form of amorphous Al_2O_3 or dissolved NaAlO_2) and NaOH but little Si to geopolymerization [29].

The SEM micrographs of raw red mud (Fig.7) have a rough surface, different shapes, and different particle sizes. It has a relatively porous microstructure with the presence of dispersed particles. The micrographs show that the particles of the raw RM were roughly crystallized and the hematite was detected in the form of aggregates.

RHA

The RHA X-ray diagram (Fig.8) indicates that it is formed by silica in the crystalline form, resulting from the predominant presence of cristobalite ($2\theta = 21,9$) [30]. Both forms: crystalline and amorphous silica are governed by the burning temperature. RHA shows 2 huge and broad humps at $5-15$ and $15-30^\circ 2\theta$, respectively, indicating the presence of amorphous phases. A few sharp peaks also indicate the presence of crystalline phases such as quartz and carbon. By comparison with its chemical composition (table 2), silica in RHA is present as both amorphous and crystalline (quartz) phases. Furthermore, the overwhelming majority of silica is in amorphous form, which agrees with the previous studies.

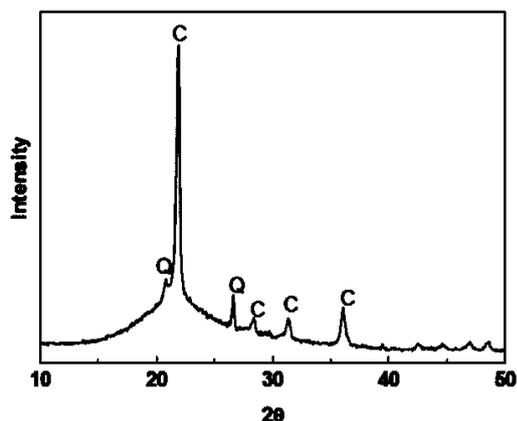


Fig.8. XRD diffractogram of RHA

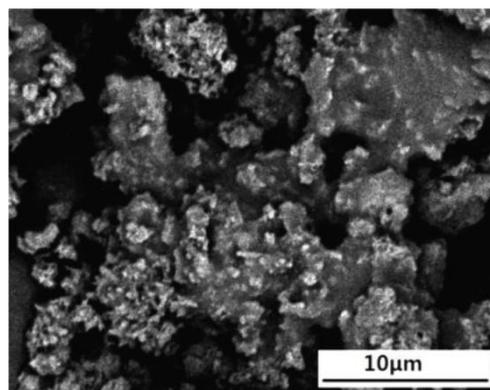


Fig.9. SEM micrograph of RHA

The micrograph of RHA (Fig.9.) shows a porous and multifaceted particle shape and size. The foremost constituents of rice husk comprise hydrated silica, cellulose and hemicellulose component totaling a 55–60% and lignin component of approximately 22%. The porous and honeycomb morphology seen can be credited to the burning out of the organic component in the rice husk during combustion. The hydrated silica afterward polymerizes to form a skeletal silica.

AH

Fig.10 represents the XRD diagram of an anthill soil. The qualitative analysis of the AH as depicted by the XRD pattern shows sharp high peaks for SiO_2 , Al_2O_3 , Fe_3O_4 and sharp but shorter peaks for K_2O , MgO , MnO , Na_2O and CaO . The peaks indicate that the samples contain mainly silica and aluminosilicate phases. Some other oxides were also present but in very negligible proportions.

The SEM image of the AH is presented in Fig.10. The micrograph revealed that the particles that made up the material agglomerated together on its rough surface.

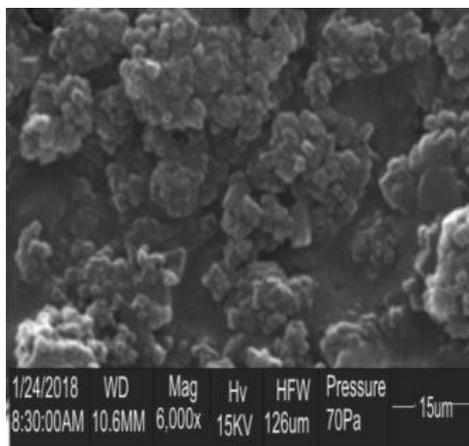


Fig.10. SEM micrograph of AH

V.2. Properties of the GP specimens

There are actually four parent materials involved in the synthesis of RM-RHA-AH based geopolymers: RM, RHA, AH and sodium hydroxide solution. As shown in XRD and SEM characterisation, only amorphous phases in raw materials participate in the geopolymerization reaction. Among the three parent materials, RM provides Al_2O_3 , NaOH and NaAlO_2 ; RHA provides amorphous SiO_2 ; AH provides SiO_2 and Al_2O_3 ; while the sodium hydroxide solution provides NaOH .

To facilitate the interpretation of the results, it should be noted that TGP1, TGP2 and TGP3 are respectively parent materials RM, RHA and AH not mixed with others. The cubic GP specimens after 28 days curing at ambient laboratory temperature are present in Fig.11 below. The predominant color is that from RM.



Fig.11 : GP specimens after 28 days curing

Figure 12 shows the SEM micrograph of 33,33-33-33,33 RM-RHA-AH geopolymer. The SEM analysis illustrates the finest contours and surfaces indicative of the extent of dissolution and polycondensation that occurred during geopolymerization for this mixture. The micrograph revealed a lesser number of unreacted particles and the matrices appeared dense. These samples were prepared with relatively high NaOH molarity. This is in line with the earlier report which indicated that the use of solution with high molarity could accelerate the geopolymerization process [31].

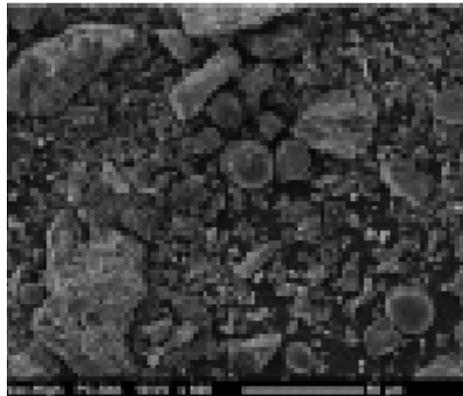


Fig.12. SEM micrographRM-RHA-AH

The experimental test done one the presumed GP specimens are presentedon fig.13 and fig.14

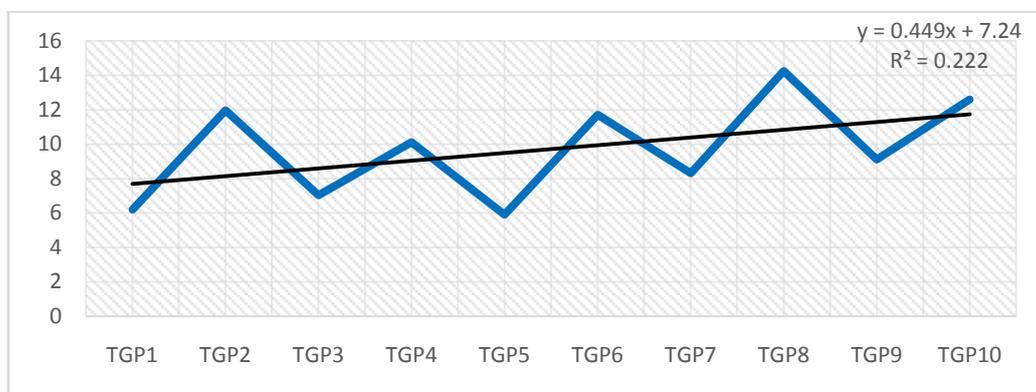


Fig.13: Compressive strength (MPa; 28 days)

The 28-day compressive strength of the specimens range from 5,89 to 14,25 MPa, Specimens TGP8 and TGP10 corresponding to RM-RHA-AH mix ratios of 17-66-17 and 33,3-33,3-33,3 respectively were above 11,7 MPa, i.e. the ASTM C55 prescribed strength standards for the concrete brick. Among the 10 (ten) specimens, 4 are conform to the above standards. They are characterized by a high content of SiO₂ in 2 parent materials RHA and AH. It may be thought that the presence of SiO₂ is the predominant factor which contributes to the resistance of the GP to compression.

With regard to water absorption, the TGP8 specimen has the lowest value (165 kg/m³) whereas TGP9 has the highest value (387 kg/m³). However, the water absorption values of the geopolymer were enough lower than 288 kg/m³ which is the prescribed limit according to ASTM C55 requirements for lightweight concrete brick material. TGP2=RHA.

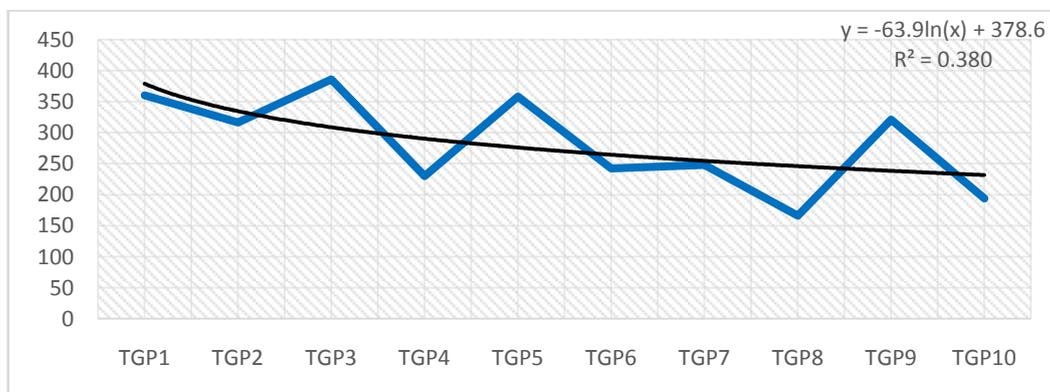


Fig. 14: Water absorption (kg/m³)

VI. CONCLUSION

The central purpose of this study is to point out that an industrial waste can be turned to raw material or valuable intermediate. The main idea was to mitigate the environmental concern raised by dumping of the plentiful industrial wastes as land fillers. Thus, the management of the RM resulting from the conversion of the enormous bauxite reserves may become a sustainable industrial opportunity. In this study, with the additional aim of including all three parent materials, a ternary blend of 0-50-50; 17-66-17 and 33,33-33,33-33,33 respectively by mass ratio of RM-RHA-AH was developed that was able to meet the requirements of the target application. These GP materials showed a water absorption lower than 288 kg/m^3 the prescribed limit according to ASTM C55. In addition, the incineration of RH to produce energy and the use of the resulting RHA product as cementitious material, contribute to recycling according to the zero waste principle. The high content of SiO_2 and in AH could draw attention to this natural resource for its sustainable valorization.

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