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Use of Waste Glass Cullet and Nepheline Syenite in The Development of Wall Linings for Petrochemical Reactors: Suitability and Comparative Studies

EJIKEME. M. E.[1], ABUH, MARK. A.[2], ONOH, MAXWELL. I.[3], AYABIE. BONIFACE. U.[4], USHIE FRANCIS A[5]

[1, 3]Dept. of Chemical Engineering, Enugu state University of Science and Technology, ESUT, Agbani, Enugu State Nigeria.

[2]Dept. of Materials and Energy Technology, MET, Projects Development Institute (PRODA), Federal Ministry of Science, Technology and Innovation (FMST&I), Enugu, Nigeria

[4]Standards Organization of Nigeria-National metrology institute, Enugu

[5]Henley & Ascot Ltd, 14/16 olokonla road, Lekki-Ekpe expressway, Lagos state

***Abstract:** The research focus on the use of glass cullet (GW) and nepheline syenite (NS) in the development of linings for petrochemical reactors walls. The ash used was rice husk ash (RHA) obtained from rice husk after burning at 700^0C. The Chemical profile of GW and NS were evaluated using Atomic Absorption Spectroscopy (AAS) and result obtained showed lower percentage of SiO_2 (56.23%) in NS than in GW (71.13%). The physical tests showed higher flexural strength (FS) of ≥35mpa for GW linings (RHAGW) which was within the ISO 13006 B1a standard at 1100^0C. Thermal conductivity values were higher for NS linings (RHANS) (0.32 – 1.98w/mK) than RHAGW (0.22 – 1.91) which is probably due to more closure of pores, higher content of Fe_2O_3 and conductive metallic impurities making NS very unsuitable for linings production. Thermal gravimetric analysis (TGA) showed higher weight loss with RHANS (26%) than with RHAGW (17%) making only RHAGW suitable for production of linings. The X-ray diffractogram (XRD) shows the presence of quartz and mullite while RHANS showed the presence of microcline, albite hermatite, andalusite, orthoclase and nepheline. Results obtained showed that GW formulations gave better porcelain linings suitable for corrosion resistant reactors*

***Keywords:** RHANS, Kaolin/Quartz/Ash, TGA, RHAGW and XRD.*

I. INTRODUCTION

Reactor wall linings are usually of high alumina, Magnesia-chromite, Magnesite, Magnesia-Chrome, and Alumino-zirconia base. The performance of these wall linings is reduced if it is operated outside the temperature it was designed to withstand. Understanding the mechanical and chemical composition of materials used for the design of these linings help in their careful selection as additives to modify the property during production (Potgieter 2004). Petrochemical industries use linings of various basic refractories and porcelain for walls and pressure vessel on its hot face sides (Chidambaram, 2018). The need to improve on wall linings presently in use arises due to cost and operation life. Present walls are either too expensive to acquire or easily fail leading to accumulation, absorption and adsorption.

Waste glass is associated with serious disposal issues as it is not biodegradable, constitutes nuisance and very difficult to dispose as it can remain active wherever found or thrown even for thousands of years. Nepheline syenite ($K_2O.Na_2O.2Al_2O_3. 4SiO_2$) sometimes mistaken for granite due to similar physical appearance on the other hand, is a light-colored, silica-deficient, and feldspathic rock. It contains high ratio of alkaline (Na_2O+K_2O)/acid (SiO_2) and Alkaline (Na_2O+K_2O) / amphoteric (Al_2O_3). Nepheline syenite is naturally occurring with very high deposits in Nigeria and commonly used in place of granite as reinforcement for concrete. After utilization, remnants of nepheline syenite (NS) remain littered in the dumpsites constituting also nuisance which needs proper reuse or cleanup. Emphasis on use of waste glass as additive for concrete and other advance materials applications have been reported in literature (Hooi and Min, 2017; Singh and Singh, 2016; Subba et al, 2023; Shayan and Xu, 2006; Alhaji etal, 2019; Ogundairo et al, 2019) but no use as additive for reactor walls formulated with a blend of Kaolin (50%) Quartz and ashes from organic wastes has been documented nor did the comparison with nepheline syenite reported anywhere. Finding alternative applications for these

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materials after serving their intended function is important since they now constitute wastes and nuisance to environment. Understanding why feldspar is utilized in porcelain formulation helps in selection of alternative materials. Feldspar (orthoclase) commonly utilized introduces K_2O, Al_2O_3 and SiO_2 with the K_2O acting as flux. Kaolin is readily available and easily sourced as with silica from obtained from milled river sand but feldspathic rocks are not easily accessible. In the absence of feldspar, ashes rich in fluxing oxides are used alongside materials with high silica (glass former) content. This explains the consideration of wastes glass and nepheline syenite which are rich in silica as alternative materials to feldspar and hence the reason for this research. . If successfully applied, they can discourage the frequent excavation for orthoclase ($K_2O.Al_2O_3.6SiO_2$) which causes negative environmental impact when exploited. To this end therefore, this research aims to evaluate the suitability and comparative uses of the glass waste (GW) and nepheline syenite (NS) in the development of chemically inert, impervious wall linings for corrosive chemical reactor.

II. MATERIALS AND METHOD

Equipment used in this research is: Meter balance (Metler Toledo with sensitivity of 0.001), drying oven (DHG-9101-1SA, 75L, Gallenkomp, England), Hood type electric furnace (Cone art kiln inc, model BX 119D, Canada), Pan mill (Quanzhou Xinhaineng machinery co ltd, model PSJ-SS5040 China), rotary ball mill (William Boulton Ltd, model F.S 8470, England), 300µm sieve(BS), digital display double-Arm tensile testing machine (LT-10kn, model no; 202200534, accuracy level 1, Luotai precision instruments, Dongguan co.ltd, china), Pycnometer (Pyrex), scanning electron microscopy, Atomic Absorption Spectroscopy (AAS, Buck Scientific model 210), X-ray diffractometer, XRD, Thermal Gravimetric analyzer/Differential Thermal analyzer (TGA/DTA), and thermal conductivity tester (Thermtest TLS – 100)

Specimen Preparation: Assorted glass wastes were obtained, washed and dried to remove oily, organic and inorganic contaminants including nylons and paper labels. Samples was then then crushed with the pan mill before pulverized with the rotary ball mill to particle size ≤200µmBS. Same procedure was applied in processing the nepheline syenite and were separately stored in air dried containers for the study.

Plate 1 Samples of glass wastes used for this study. (A) Wastes alcoholic beverage bottles. (B) Wastes laboratory glass wares. (C) Wastes glass after pan milling. (D) Powdered glass wastes (GW) after ball milling

Plate 2 Samples of nepheline syenite (NS) used for this study. (A) Hip of NS supplied. (B) Crushhed NS from panmill. (C) Dewatered milled NS from ball nill using POP mould. (D) Powdered NS obtained

kaolin: quarts: ash ratio of 50:20:30 was used as the sample and GW and NS were each introduced using a line mix of 100% sample, 95/5% (representing 95% sample and 5% ashes) , up to 70/30 %. Every formulation was tested at Temperature, T (^{0}C) of 900 – 1200. Each batch of specimen was manually mixed to homogeneity as described by Pena et al, (2016). Calculated amounts of water was then added and mixed vigorously followed by wedging to achieve moldable plasticity. The moldable mix was dry-pressed into rectangular, cylindrical and spherical metallic molds to obtain test specimens for the study as reported (Abuh et al, 2014).

Test method. The oxide analyses of samples evaluated using AAS. The XRD (Dandona Ndona, model AL – 2700A, serial no 2322) pattern was displayed in the computer interface attached to the equipment as peaks were automatically identified by the instrument software. The TGA analysis was carried out using Shimadzu model TA-60WS. At the specified testing, 20 - 25 mg of sample was measured into platinum pan and heated in nitrogen atmosphere at a temperature range between 20^0C to 1200^0C with controlled heating rate $10^0C/min$. Thermtest's portable measurement system, the TLS-100 was used for thermal conductivity test. Measurements are performed in accordance with ASTM Standard D5334

III. RESULTS AND DISCUSSIONS

3.1 Chemical Composition of Samples.

Table1 Chemical profile of nepheline syenite used (AAS)

	Composition (%)	Spread1	Amount	Ratio	Spread 2	Ratio 2
SiO_2	56.12	Acid	56.23	30.4	Acid	3
TiO_2	0.11					
Al_2O_3	23.14	Amphoteric	26.4	14.3	Amphoteric	1
Fe_2O_3	3.26					
K_2O	6.56	Alkaline	16.43	9	Basic	1
Na_2O	9.87					
CaO	1.61	Alkaline Earth	1.85	1		
MgO	0.24					
LOI	0.86					

Table 1 shows the oxide profile of the Nepheline syenite used in this study. The oxides distribution shows that basic oxides were very high (20.54%) with the alkaline oxides (K_2O and Na_2O) constituting about 80% of basic oxides composition. the composition is obtained from the AAS reading and so expressed. The spread1 represents the aggregation of the oxides into alkaline (oxides of group 1 elements), alkaline earth (oxides of group 2 elements), amphoteric (group 13) that share the boundary between metals and non-metals (Al_2O_3 and Fe_2O_3) and acid oxides (SiO_2 and TiO_2). Both alkaline and alkaline earth are added to form the basic oxides as shown in spread 2. The amounts and the ratios are computed from their corresponding composition (%) as presented in tables 1 – 3. The Basic: Acid oxide ratio was 1:3. The closer the ratio is, the easier the melting of glass formers to form glass-like coating and mullitization (Gaultieri 2011, Hongxu et al, 2023)

Table 2 chemical profile of cullet (Glass Wastes) used

Name	Composition (%)	Spread1	Amount	Ratio	Spread2	Ratio2
SiO_2	71.13	Acid Oxides	71.21	46	Acid	46
TiO_2	0.08					
Al_2O_3	1.39	Amphoteric	1.55	1	Amphoteric	1
Fe_2O_3	0.16					
Na_2O	14.18	Alkaline	14.21	9	Basic	17
K_2O	0.03					
CaO	10.62	Alkaline Earth	11.45	7		
MgO	0.83					

The basic: acid oxide ratio was 17: 46 which translates to 1:3 as shown in table 2. This means that for every 1 mole of basic oxide (fluxes) available, 3 moles of acid oxide (glass formers) are present to be melted. Similar trend is observed in table 1. Difference is in the distribution of the alkaline and alkaline earth oxides. The alkaline oxides are the low temperature fluxes ensuring that vitrification is obtained at lower temperatures. The alkaline earth metals are mid-temperature fluxes which require higher temperature to vitrify the composite. The ratios of these oxides to acid oxides determines how easily the composite can be melted to obtain glass-like porcelain texture. Alkaline: acid oxide for GW is 14:71 which is 1: 5 as presented in table 2 while same ratio was 1/3 for NS as shown in Table1. This explains why the NS is likely to melt at lower temperature than GW. A composite would have a lower melting point if the alkaline oxides are higher than the alkaline earth oxides in the basic oxides distribution (Dasilva et al, 2012; Eke, 2010).

Table 3: Oxides distribution in RHA

Oxide	RHA	Spread1	Amount	Ratio1	Spread2	Ratio 2
SiO_2	82.29	Acid Oxide	82.29	29.4	Acid Oxide	21
TiO_2	0					
Al_2O_3	2.82	Amphoteric	3.97	1.4	Amphoteric	1
Fe_2O_3	1.15					
CaO	1.73	alkaline earth	4.05	1.4	Basic	2
MgO	2.32					
K_2O	2.38	Alkaline	2.8	1		
Na_2O	0.42					

Table 3 also shows the chemical profile of the used RHA and the oxide spread. Clearly the basic: acid oxide ratio is 1:12. This acid oxide from silica is amorphous and atoms are arranged in randomly in a continuous disorderly network. This makes for easier vitrification at elevated temperatures of 1000^{0}C and above. However, the ratio is not high enough when compared with the distribution in GW and NS. The introduction of RHA in the kaolin/quartz mix normally is to reduce shrinkage and thermal shock by creating pores as the volatile elements are thermally degraded at extreme temperatures (1000^0 – 1200^{0}C).

3.2 Flexural strength: Flexural strength observed for GW reinforced materials was higher than with NS especially at 1100^{0}C and 10% addition (38.570mpa) which is higher than ISO 13006B1a standard of ≥35mpa recommended for porcelain (Dondi et al, 1995). The NS sample achieved weaker strength of 34.271mpa under same conditions. Which is clearly lower than recommended standard. This implies that the GW can be safely utilized as alternative for development of wall linings

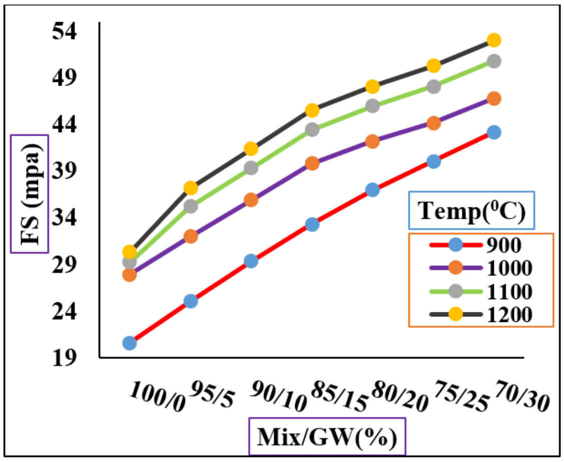

Figure 1: response of flexural strength of RHA and TA base mix to cullet (GW) addition

The strength of the GW samples increased with increase in GW addition and increase in temperature as shown in figure 1. Fewer fluxing oxides such as the alkaline and alkaline earth oxides were present in GW and their interaction in breaking the strong Si – O, and Al – O bond would be minimal as their little presence would rather aid the mullitization and vitrification of the crystalline and amorphous silica (Zhou et al, 2013).

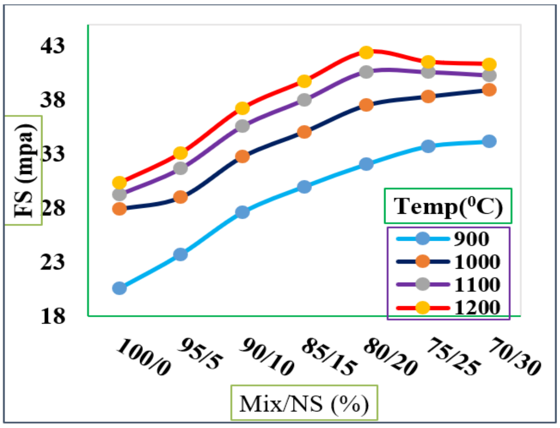

Figure 2: response of flexural strength of composite to NS addition

Figure 2 shows a drop in the FS of NS samples at above 20% addition which suggest use beyond 20% should be discouraged especially at higher temperatures of 1100^0 – 1200^0C. At lower temperatures of 900 – 10000C, increment beyond 20% did not reduce the FS. This observed trend at higher temperature is possibly due to cleavage of the strong Si – O , Al – O, and Si – Al bonds influence by more fluxing oxides from the NS and subsequent reformation of weak Si – K, Si – Fe, Si – Na, Al – Fe and other bonds which compromises the strength of the composite (Dondi et al, 1995). The basic oxides are fluxes and the more they are in a batch recipe, the more the ease of mullitization and vitrification (Dimitrov and Komatsu, 2005; Zhou et al, 2013)

3.3 Cold crushing strength (CCS): CCS was higher for: the GW than the samples with NS at temperatures above 1100^0C. Common trend observed was that as the GW/NS addition increased, and as temperature also increased, CCS for GW increased while NS decreased.

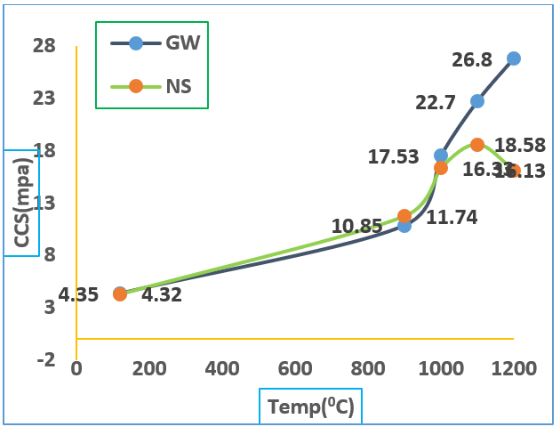

Figure 3 Response of cold crushing strength (CCS) of composite to GW and NS addition

This decrease in CCS with NS addition than with GW at same ratios and temperature is possibly due to development of pin-holes and degradation of the Si – O bond and Si – Al bond followed by rearrangement and formation of weaker bonds such as Si – K, Si – Fe, AL – Ca and other bonds. These pin-holes creates stress points in the composite which increases the tendency to crack and crush under applied pressure as shown in figure 3 above. At 1000^0C, the fatigue resistance of the NS samples greatly reduced and at 1200^0C when pinholes were very visible and the hardness was lost unlike with GW which showed higher fatigue resistance with increase in temperature as presented in figure 3.

Plate 3 face valuation of nepheline syenite samples at different ratios for NS showing pinholes.

Plate 4 Comparative view of 30 % and 20% glass waste reinforced wall linings.

The high amount of basic oxides in the NS completely forms a viscous liquid due to crystallisation of primary mullite from pseudo-amorphous products with a consequent segregation of a silica deficient amorphous phase and subsequent formation of an alkaline melt (Gaultieri et al, 2011) originating from the melting of the oxides forms weakened products upon cooling which develops network of cracks and large pinholes. These tiny network of cracks and large pin-holes greatly impacts the FS weakening the product

3.4 Thermal conductivity. This measures how readily a material conducts heat. Wall linings for reactors are expected to have low heat conduction to be able to retain energy input within the reactor for effective process and production. Therefore the lower the heat conduction, the better the wall lining. Values obtained for GW composites were higher than for NS and were in the range of 0.24 – 0.84 at 1100^{0}C which were within the standard values of 0.01 to 1.1 K (W/m.k) for good porcelain and refractory fireclay bricks (Gilmore, 2013; Cengel and Ghajar, 2012; Cengel and Boles, 2015).

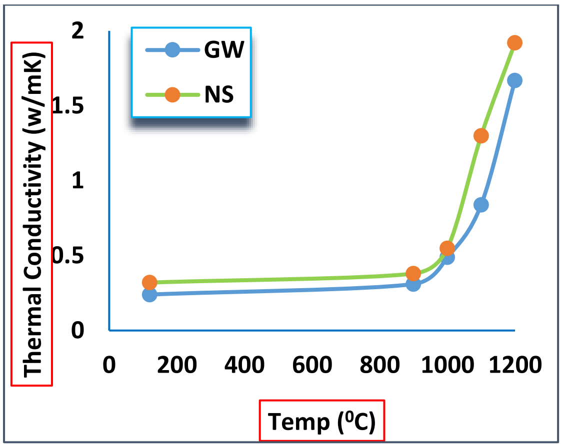

Figure 4 Response of thermal conductivity of samples to GW and NS addition

Figure 4 shows similar trend for the GW and NS composite irrespective of temperature. The higher The GW samples could withstand more number of cycles without cracking than the NS samples. All the NS samples appeared to have brownish colouration with different shades depending on the quantity of NS added. Higher temperature favours the closure of pores within the material and this reduces thermal insulation. These closure of pores compel the surface to conduct heat as shown in figure 4.

Plate 5 Comparative view of NS and GW samples at.20% addition

The brownish colour observed in NS base samples as shown in plate 3 and 5 is due to presence of elevated amounts of iron (III) oxide (Fe_2O_3) When values of Fe_2O_3 >2 %, sample products will change colour depending on the percentage involved (Singer and Singer, 1993; Gupta, 2008).

3.5 TGA of products: The TGA line shows a gradual downward movement corresponding to decomposition of volatile organics at moisture content loss as seen in the curve which corresponds to 105.4^{0}C.for RHANS and RHAGW samples respectively shown in figure 5 – 6. This decomposition corresponds to 3% loss in mass.

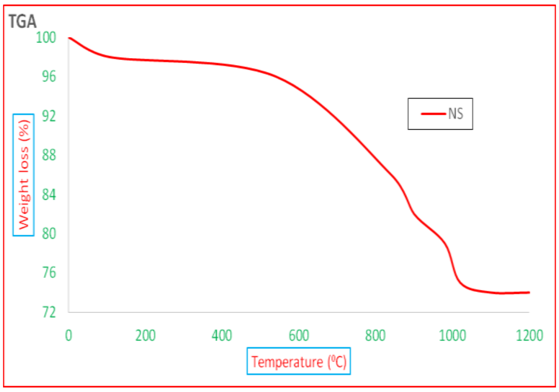

Figure 5 Response of NS composite containing GW (RHANS) to TGA treatment

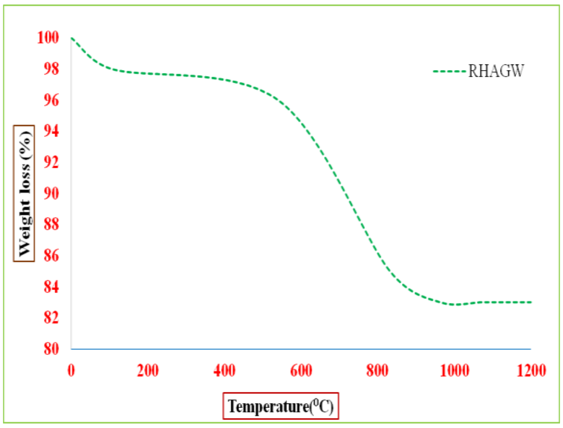

Figure 6 Comparative Response of RHAGW

Another peak is observed at 540^{0}C which corresponds to allotropic transformation of α-quartz to β (Nandi et al. 2015) represents a 6% loss for RHAGW and due to carbonisation and decarbonation of organic impurities (Maeda et al, 2001; Ramadhansyah, 2012).

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Again, this loss at 540^{0}C represents dehydroxylation or transformation of kaolinite to metakaolinite ($Al_2O_3.2SiO_2$) as shown below

$$\underset{\text{Kaolinite}}{Al_2O_3.2SiO_2.2H_2O} \rightarrow \underset{\text{Metakaolinite}}{Al_2O_3.2SiO_2} + 2H_2O\uparrow$$

The loss of weight at 800^{0}C for RHANS and 830^{0}C for RHAGW is due to de-hydroxylation of kaolin and interaction of unstable inorganic components of the formulation. The weight loss at this point was about 18%.for RHANS and 13% for RHAGW. Two exothermic peaks were observed at 1020^{0}C and 1110^{0}C for RHANS and one peak at 1070^{0}C for RHAGW. The peak at 1020^{0}C corresponds to slight weight loss which is probably due to higher interaction of unstable alkaline oxides to form mullite and amorphous quarts or spinel and represents about 25% loss for RHANS. The peak at 1070^{0}C and 1110^{0}C is due to nucleus formation of mullite. Total mass loss is about 26% for RHANS and which is significantly higher than 17% for RHAGW.

3.6 XRD of products

RHANS: The peaks at 26^0 is topped by a broad peak between 43^0 and 58^0 and another peak at 51^0, indicating that the silica phase is partly crystalline and partly amorphous. These sharp peaks are indicative of crystalline silica while the broad peak is attributed to amorphous silica (David et al, 2022; Dogruyol, 2024).

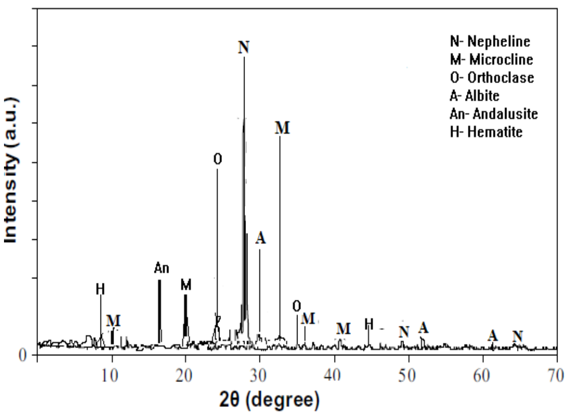

Figure 7: XRD of RHANS

The mechanism of forming mullite phase is explained by inter-diffusion of aluminum and silicon ionic species under high calcination temperature in the alumina–silica contact zone. (Hussein, 2016). The spectrum for RHAGW clearly shows that quartz is the main component of the final product with mullite being significant but with traces of plagioclase as shown in figure 9.

3.7.2 XRD of RHAGW

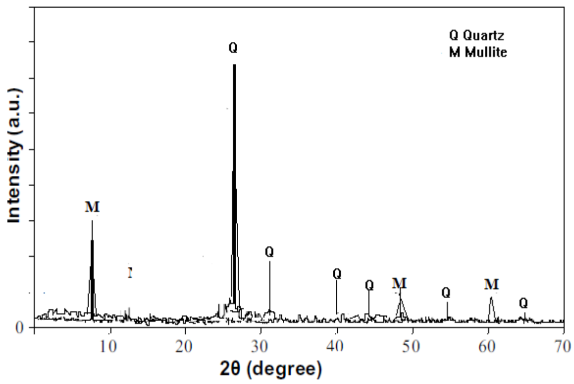

Figure 8 XRD of RHAGW

The spectra show two main components being quartz and mullite. The quart is the principal component of the wall lining produced at 1200^{0}C, and this result also corroborates the EDX measurement of higher percentage of quarts in the product. The presence of mullite coating explains the glassy lustre of the final product.

IV. CONCLUSION

It can be deduced from this study that;

1. Only glass waste are suitable for use in the development of wall linings for petrochemical reactors. This is reflected in the higher flexural strength, cold crushing strength, lower thermal conductivity, and wider alkaline – acid oxide ratio.
2. The incorporation of glass wastes into the composite is limited to 30% at temperatures less than 1200^{0}C to avoid deformation.
3. If Nepheline syenite must be used, maximum composition is 30% at temperatures ≤ 1100^{0}C and the high content of Fe_3O_2 is enough deterrent.
4. The thermal gravimetric analysis results shows higher degradation of NS reinforced linings (RHANS) (26%) against GW linings (RHAGW0 which had weigh loss of 17%
5. The XRD profile shows that GW reinforced linings comprised of mullite and quartz while NS linings (RHANS) contained nepheline, microcline, albite, andalusite, orthoclase and hematite which further explains the unsuitability of NS for linings production due to high mineral contents.

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