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Investigation of Concrete Strength Made with Nanoparticles and Metakaolin in Acid and Sulphate Solution

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Research Article

Abstract

The investigation of concrete strength with nanoparticles and metakaolin in acid and sulphate solutions was the aim of this study. The nanoparticles were obtained from rice husk through sol-gel method and beneficiated metakaolin from kaolin to replace cement in concrete. The compressive strength results at 56 days were computed for various mixes. From the results, mix number 5 (1%NS 20%MK), 10(2%NS 15%MK) and 15(3%NS 10%MK) had the highest compressive strength of 39.36N/mm^2, 40.20N/mm^2 and 41.78N/mm^2 showed the most strength development at 28days respectively. This is 10.7%, 9.8% and 14.8% strength increment over the control sample. The water absorption test of the concrete samples revealed that mix number 10 had the least water absorption at 69% less than the control sample. After 28days of 5% sulphuric acid and 5% magnesium sulphate immersion, mix number 10 also showed a compressive strength reduction by 9.7% and 7.2% more than the compressive strength before immersion. The sample with no additive was found to have 30.8% and 23.8% compressive strength reduction more than the compressive strength before immersion. The scanning electron microscopy (SEM) of the samples showed that, mix 10 had a more compact, homogenous and dense structure after 28days of immersion which explains the high strength. Energy Dispersion X-ray spectroscopy (EDS) also showed that the Ca/Si of mix 10 which was 0.83 and 0.65 after 28days of immersion in acidic and sulphate solutions respectively was within the range of optimal performance of concrete. X-ray diffraction analysis showed the development of ettringite, gypsum and silica gel as a result of the disintegration and reaction of the chemicals with hardened material.

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Keywords
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1. Introduction

The ability to resist weakening and disintegration over time under the influence of disintegrating agents has to be considered during concrete production. Concrete exposed to an aggressive environment is a major concern as regards to the durability of the composite material. Acid rain, polluted water, sewage water, etc are environmental phenomena that significantly affect the durability of the concrete structure. (Ejeh, 2017) stated that the durability of concrete will give the necessary information about the quality of the concrete as it is the resistance to weakening and disintegration over some time. Concrete structures, when exposed to the aggressive or harsh environment results in the deterioration due to acid, chloride and sulphate attacks which eventually leads to corrosion failure (Jayaranjini and Vidivelli, 2016). The strength of concrete alone is not enough for the durability of the material, the degree of resistance to the harshness of the environment in which the concrete structure is exposed over its service life is extremely important. The deterioration rate is a factor of the chemical attack and depends on the type and nature of the chemicals and the permeability nature of the concrete material. These factors significantly affect the reaction products due to the acid, salts, sulphates, and alkali chemical processes. The phenomena which occur at the nano and micro scale affect the durability properties and the over the behaviour of the structural element (Tawfik, *et al* 2019). The structure and bulk properties of the composite material can be improved using nanotechnology, which will result in the improvement of stability, durability, and sustainability of the concrete composite. Cost-effectiveness, long-lasting, and high-performance concrete which can produce a material with unprecedented use. The nano-mechanical reinforcement given to cement by supplementary cementitious materials (SCM) like metakaolin, silica fume, fly ash, nanoparticles, etc is the most desirable properties which civil engineering are taking advantage of. Other advantages include the reduction in the quantity of cement needed to obtain similar or better strength of concrete but reducing the cost and the environmental impact and also the reduction in the time needed for construction to be completed as nanoparticles are capable of producing high-strength concrete with less curing time due to their pozzolanic reactions and filler effect with cement Tawfik *et al.*, (2019).

In civil engineering projects, concrete is extensively used construction material and it is also very important across the globe. The use of this material comes with some serious concerns about its durability in some environments, especially in an aggressive environment. Environmental conditions like acid rain, seawater, polluted water, and salts sulphates which causes deterioration to concrete that are permeable. Nabil (2006) stated that a well-compacted concrete that is less porous with fewer voids is more durable. Concrete with the ingress of moisture, acid, and sulphates will seriously diminish the strength and durability of it. This is a result of voids within the concrete composite that serves as pathways for the ingress leading to deterioration due to the formation of ettringites and corrosion of reinforcements. Thus, this paper aims to study the performance of concrete with nanoparticles from agricultural waste (Rice Husk) and metakaolin in aggressive environments with respect to strength deterioration and ettringite formation. This will be achieved by obtaining nanosilica from rice husk and used in concrete production to study the effect of the production nanosilica on the durability properties of concrete.

2. Literature Review

Concrete mechanical behaviour depends on micro and nanoscale phenomena that occur within the structural element. The molecular structure of concrete can be modified at nano-level using nanotechnology which leads to the material's bulk properties improvement. The volume stability, durability, mechanical and concrete's sustainability can also be improved using nanotechnology (Tawfik et al, 2019). The excellent benefits accompanying the use of the nanotechnology allows for the production of cost-effective composite material, long-lasting products of cement with high-performance ability. The mechanical reinforcement provided by the nanomaterials is the most desired property for cement-based structural materials. Nanomaterials offer some advantages to concrete production and they include; high-strength concrete production, reduction in the amount of cement used for concrete production (cost and environmental impact reduction) and less curing time need for concrete produced with nanomaterials (Morsy *et al.*, 2011).

Conventional concrete is most of the produced concrete. This convention concrete is not very good indicators for concrete with good corrosion resistance to aggressive environment. The collapse of the concrete matrix (composite material) which occurs as a result of the influence of chemical and physical processes if the environment is Concrete corrosion (Setina et al, 2013). Due to the interaction of concrete with external influences, the physical and mechanical properties of concrete can be lost as chemical attacks may deteriorate concrete over time. An unfriendly environment where concrete is used as a construction material is a concern. When a durable and good concrete is exposed to an aggressive environment, it will retain its quality serviceability and original form without any sign of failure which may come from different sources such as acid rain, contaminated water (oil spillage, offloading and loading of crude oil), soil salt (sulphates), etc.

Researchers and engineers have anticipated the developments in advancing concrete technology using nanoscience for the next generation which is focused on sustainability objectives and desired functionality (Shafiq et al 2019). The use of SCMs and nanotechnology in concrete, will not only improve the strength of concrete but also decrease the deterioration of the material as a result of ettringite formation as acid and sulphate salts react with CSH which leads to expansion and cracks in concrete, thereby giving a pathway for external chemical to corrode internal reinforcement leading to structural failure and reduction in the durability of the composite material. The mechanical and durability properties of concrete with modified metakaolin (MK) and nanosilica (NS) was studied by Shafiq et al (2019). They carried out two phases of trials. In the first phase, they prepared concrete mixes to achieve the desired value of the 28 days compressive strength. They used the experimental results to perform statistical analysis using the response surface method (RSM) in the second phase. They concluded that 10% MK + 1% NS replacement of cement showed superior concrete with mechanical and durability. Tawfik et al (2019) experimented on concrete and cement mortar with MK and NS and came to a conclusion that, when cement is replaced by MK at 20% in the mortar, the strength is increased by 19% and the flexural strength by 16% over the control mix.

Chaudhary and Sinha (2018) investigated the effect of nano-silica addition on the variation of compressive strength and weight loss of concrete cubes subjected to an aggressive environment in terms of different chemical solutions of sulphuric acid, sodium sulphate, and sodium chloride. In their study, they prepared 6 different mixes of concrete of M35 grade by incorporating 0%, 0.5%, 1.0%, 1.5%, 2.0% and 2.5% nano-silica by weight of cement. After 60 days submerged in 5% sulphuric acid solution (H_2SO_4), sodium sulphate solution (Na_2SO_4) and sodium chloride solution (NaCl) came to a conclusion that concrete with 1.5% NS addition by weight of cement, shows a better result for acid, alkali and chloride ion resistance of concrete. Ashok et al (2017) used two nanomaterials in mortar to study the mechanical and durability properties by varying the replacement from 0.5 – 2.0% with 0.4 as w/c ratio. They drew a conclusion that cement mortar with nanomaterials resulted in higher strength both compressive and tensile as compared to plain mortar. Shah and Jamani (2017) conducted research on concrete properties with supplementary cementitious materials metakaolin and nanosilica and concluded that concrete with 2%NS and 10MK improved the mechanical properties as well as resistance to chemical attacks.

3. Materials and Methods

3.1 Materials

For this research, the materials used include the following: cement, metakaolin, nanosilica, fine aggregate, coarse aggregate, 5% sulphuric solution and 5% magnesium sulphate solution.

3.2 Experimental Methods

Cement: the specific gravity, and fineness test was done according to BS EN 196-1 (2011). The oxide composition analysis was done using XRF.

Metakaolin: XRF analysis was performed to determine the oxide compostion.

Coarse aggregate: the ACV, AIV and specific gravity test were done according to BS EN 1097-2 (2010)

Fine aggregate: the specific gravity and silt content test were done according to BS EN 1097-2(2020). The particle size distribution was done according to BS EN 933-1 (2012).

Nanoparticles preparation from rice husk:

To obtain nanosilica, the following steps are followed: Converting Rice Husk to Rice Husk Ash (RHA) and Nanosilica from Rice Husk Ash (RHA).

3.2.1 Converting Rice Husk to Rice Husk Ash (RHA)

Rice Husk was obtained from Gboko Rice Mill, Gboko Local Government Area of Benue State in Nigeria. The Rice Husk was thoroughly washed with water to remove stones and other solid debris. Rice husk was air-dried at room temperature for a week and then burnt to collect the RHA. The rice husk was burnt at 750^{o}C. The resulting Rice Husk Ash (RHA) is allowed to cool down and sieved through BS sieve size 75μm and its colour was grey. X-ray flourence (XRF) analysis was performed to obtain the chemical and oxide composition of the Rice Husk Ash (RHA).

3.2.1 Nanosilica from RHA

There are three (3) major steps involve in the preparation of nanosilica from Rice Husk Ash (RHA). The steps are: In the first step, metal oxide impurities are removed from RHA by treatment with 1M hydrochloric acid and heat treatment, the second step involves filtering and oven drying the treated RHA and the final step, the acid treated RHA was calcined at 750^{o}C to obtain nanosilica.

Step 1: Removal of Metal Oxide Impurities from RHA

For metal oxides impurities to be removed from RHA, sieved RHA is added of 1M HCl. The resulted mixture of RHA and diluted HCl is then stirred and heated, continuously stirred for 2h at 80^{o}C using a programmeable burner. All glass ware were used in order not to introduce new oxide impurities to the mixture and to ensure metal oxide impurities are evolved as gases at elevated temperature during the process. The equation of reaction is given below

$$RHA + HCl \xrightarrow[heat\ \&\ stirred\ continously]{80^{0}C\ (2\ h),soaked\ overnight} SiO_2\ (Ash) + MCl + H_2O$$

Where M = Na; K; Ca; Fe; Al, Mg,…

The resulting mixture is let standing overnight for 24hours to cool and further remove any other oxide impurites in RHA that was not removed during the heating. The acid treated RHA is the filtered.

Step 2: Washing, Filtration and Oven drying Treated RHA

The residue from the filtration in step 1 is then washed with distilled water and dried at 110°C in an electric oven. The aim of washing with distilled water is to neutralize the effect of the acid (HCl). The XRF analysis was performed on the treated RHA to obtain the oxide and chemical composition.

Step 3: Ball milling and calcination of the Oven Dried Treaded RHA

The oven dried sample was ball milled to break the lump within the residue and then it is calcined at 750^{o}C for 3hours. The colour of the material changes from gray to white and the particle size if further broken down to the desire size.

3.3 Concrete Mix design

The mix design used for the productin of nanosilica-metakaolin conctete (NM-Concrete) was 1:2:3 for grade 30 concrete. The materials were measured by weight using the weighing machine and concrete samples were made with varying amount of nanosilica and metakaolin from 1 - 3% and 5 - 25% respectively by weight of cement and labelled.

3.3.1 Compressive strength of NM-concrete cubes

The concrete cubes will be used to test for the compressive strength after 56days of curing. This control specimen will be made of only cement without metakaolin and nanosilica. The concrete samples were cured in water for 28days, after which the samples were airdried to achieved constant weight and then immersed in 5% sulphuric acid solution and another set in 5% magnesium sulphate solution for 28 days to determine the resistance to chemical attack of the concrete samples with metakaolin and nanosilica. The percentage deterioration in strength was calculated using equation 1 after 28days of immersion

$$\text{Loss in compressive strength, } f_{lc} = \frac{f_{cb}-f_{ca}}{f_{cb}}\ x\ \frac{100}{1} \qquad (1)$$

Where f_{lc} = Loss in compressive strength, f_{cb} = compressive strength before immersion, f_{ca} = compressive strength after immersion

3.3.2 Water Absorption of NM-concrete

The water absorption ability was determined after oven drying the samples for 72hours and immersing in water for 48hours to determine permeability of the samples which is a function of the voids present in the concrete sample. The water absorption was calculated using equation 2

$$\text{Water Absorption} = \frac{M_S-M_O}{M_O}\ x\ \frac{100}{1} \qquad (2)$$

Where M_s is the weight of saturated sample, and M_o is the weight of dry sample (after oven drying for 24hrs)

3.4 Scanning Electron Microscopy/Energy Dispersion X-ray Spectroscopy (SEM) and X-Ray Diffraction analysis

SEM/EDS and XRD analysis were carried out after 28days of immersion in acidic and sulphate solutions to study the performance at micro-level and mineralogical characteristics of the concrete samples and also to determine the residual composition of the samples after 28days of immersion in acidic and sulphate solution.

4. Results and Discussions

4.1 XRF analysis

Table 4.1 shows the XRF oxide compostion of cement, metakaolin and nanosilica.

Table 4.1: Oxide composition

Oxide Composition	Cement	Metakaolin	Nanosilica
CaO	64.45	0.66	0.278
SiO_2	21.55	55.00	94.259
Al_2O_3	5.28	35.20	1.068
Fe_2O_3	3.95	0.82	0.759
MgO	1.85	0.21	0.593
SO_3	1.50	-	0.278
Alkalis	0.65	-	-
Loss of ignition	1.44	6.40	-

The specific gravity and fineness of cement is shown in table 4.2

Table 4.2: Specific gravity and fineness of OPC

Cement	Value	BS EN 196-2 (2016)
Specific gravity	3.19	3.15
Fineness test (%)	6	⩽10%

4.2 Aggregate properties

Table 4.3 shows the aggregate properties of fine and coarse aggregate which shows all were within the code specification.

Table 4.3: Test values for fine and coarse aggregate

	Silt content (%)	Specific gravity	Impact value (%)	Crushing Value (%)
Fine aggregate	3.98	2.63	-	-
Coarse aggregate	-	2.78	17.2	18.06
Code specfication	8	2.4-3.0	<30	<30

The particle size distribution graph is shown in Figure 1. It was observed from the graph that the fine aggregate were of zone 2.

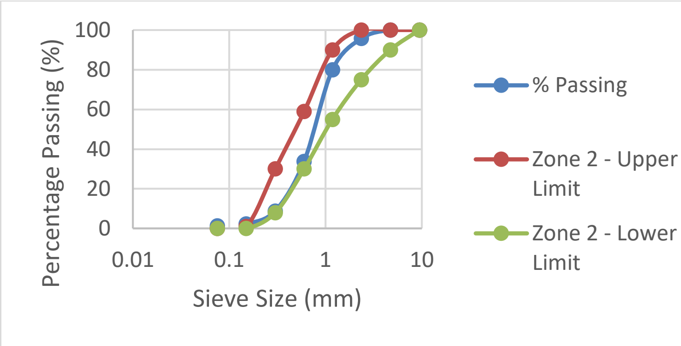

Figure 1: Particle Size Analysis for fine aggregate.

4.3 Compressive strength result

When 10%, 15% and 20% MK are added to 1% NS, at 56days, there is a significant increase in strength development at 9.2%, 9.7% and 10.7% respectively over the control. Also the addition of 10%, 15% and 20% MK to 2% NS yielded a compressive strength of 38.55N/mm^2, 40.20N/mm^2 and 39.37N/mm^2 respectively as shown in Figure 2.

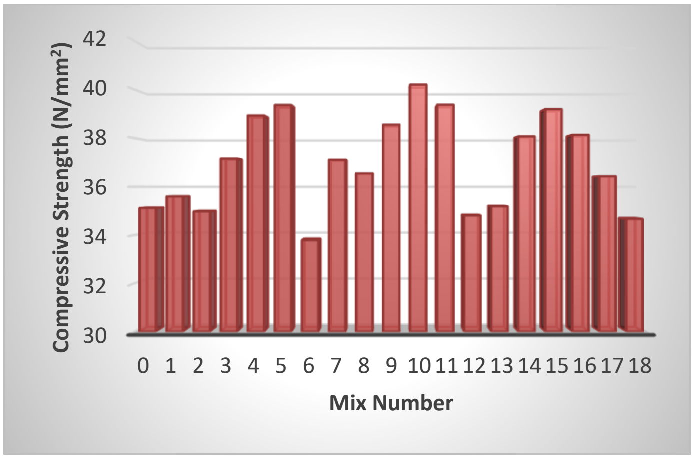

Figure 2: Compressive Strength of concrete with nanosilica and metakaolin

Similarly, at 56days, the increment of strength against for control at 7.7%, 10.4% and 7.9% for 5%, 10% and 15% MK addition to 3% NS. This is as a result of the increase in pozzolanic activity with the addition of MK to the concrete mix and the production of tricalcium silicates (C_3S) and dicalcium silicates (C_2S). This pozzolanic activity increase is as voids which are filled up and the nucleating effect of the silicon oxide (at nanolevel) present which reacts with the excess CaOH produce by the OPC to yield more C-S-H which is responsible for the strength of concrete, especially C_2S. From the results, the optimum mix was mix number 10 (2%NS 15%MK).

4.4 Water absorption

The test result shown in Figure 3 shows that the inclusion of nanosilica (NS) and metakaolin (MK) can improve considerably in the resistance of water penetration of the mortar mix. A significant reduction in water absorption by marginal replacement of cement was observed. At 28 days curing water absorption values decreased considerably with an increase in NS content up to 1%.

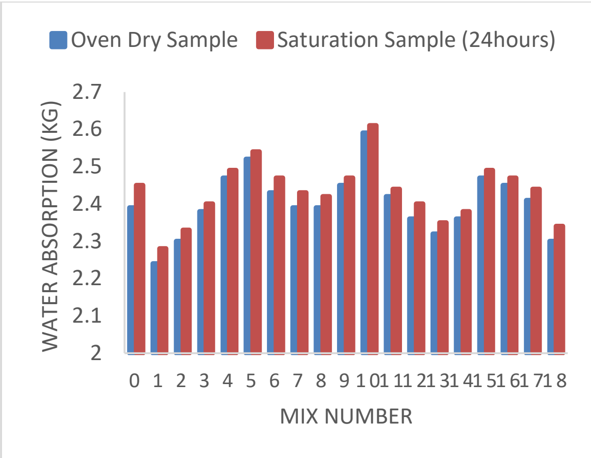

Figure 3: Water Absorption Test Results

At 2% NS, the value was lower compared to that of control. Similarly, when nano-silica and metakaolin used as a replacement for cement the water absorption in the mixes was decreased by more than two times compared to the control mix. Mix number 10 lead to substantial improvement in water absorption by about 69.6% decrease when compared with the control mix (mix number 0). The increase in impenetrability can be accomplished by two phenomena. Nanosilica particles generate a large number of nucleation sites for hydration products and induce a homogeneous distribution of C-S-H gel and hence lesser pore structure. Nanoparticles act as a retarder or blockage of passages connecting capillary pores and water channels in cement mortar.

4.4 Acid Resistance Test

After 28days of immersion in 5% H_2SO_4 solution as shown in Figure 4, it can be seen that from the results that, the samples with the highest compressive strength were mix number 4, mix number 5, mix number 10, mix number 11 and mix number 15 at 33.33N/mm^2, 34.56N/mm^2, 36.30N/mm^2, 33.47N/mm^2 and 33.99N/mm^2 respectively. This represented a 14.4%%, 12.2%, 9.7%, 15%, and 13.3% reduction in compressive strength. With respect to the control, there was a significant strength reduction from 35.12N/mm^2 to 24.32N/mm^2 representing 30.8% strength reduction. The reaction between H_2SO_4 and cement constituents of concrete results in the conversion of $Ca(OH)_2$ and C-S-H to calcium sulphate (gypsum) as shown in equation 3.

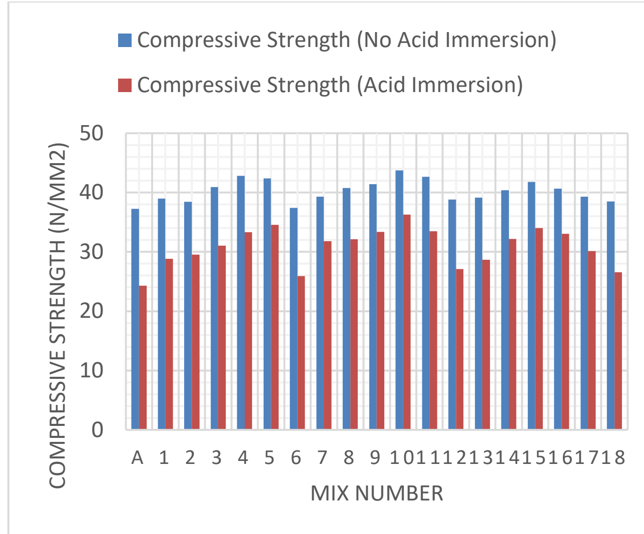

Figure 4: Acid Resistance of concrete samples at various replacements.

The formation of this calcium sulphate results in the softening of the concrete. This calcium sulphate (gypsum) further reacts with calcium aluminate to form calcium sulfoaluminate (ettringite) as shown in equation 4. These ettringite further reacts with the acid to for silica gel. This gel can be easily disintegrated. The XRD mineralogy analysis confirmed the high presence gypsum, ettringite, portlandite (Ca(OH)2) and tobermorite (C-S-H) as shown in Figure 5 and 6.

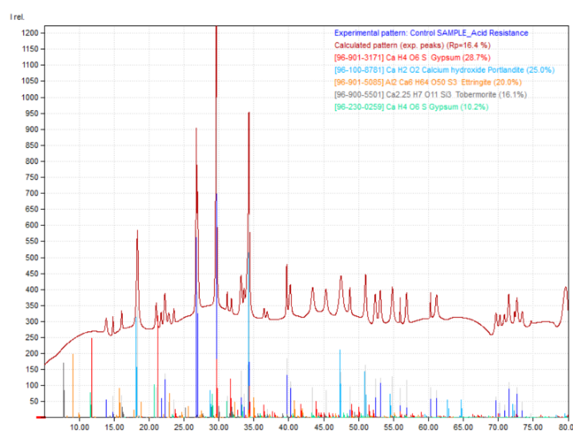

Figure 5: XRD Analysis of Control Sample After 28days of Acid Immersion

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4.2H_2O \quad (3)$$

Acid *Gypsum (Calcium Sulphate)*

$$3CaO.2SiO_2.3H_2O + H_2SO_4 \rightarrow CaSO_4.2H_2O + Si(OH)_4 \quad (4)$$

C-S-H *Acid* *Gypsum (Calcium Sulphate)* *Silica gel*

$$3CaSO_4 + 3CaO.Al_2O_3.6H_2O + 25H_2O \rightarrow 3CaO.Al_2O_3.3CaSO_4 + 31H_2O \quad (5)$$

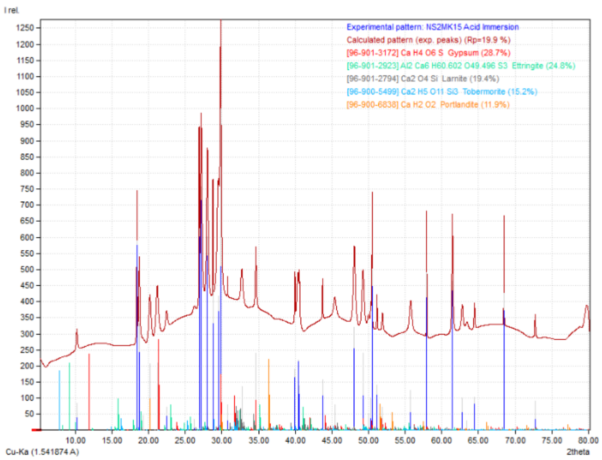

Figure 6: XRD Analysis of mix10 after 28days of Acid Immersion

But when nanosilica and metakaolin are added to concrete, there is a reduction in the amount of $Ca(OH)_2$ present in the hardened concrete, which leads to the significant reduction the reaction between the sulphuric acid and $Ca(OH)_2$. Thereby reducing the formation of ettringite and silica gel. This can be seen as the sample containing nanosilica and metakaolin showed less reduction in strength as compared to the control sample. Mix number 10 showed the most resistance to acid attack with a 9.7% loss in strength as compared to the control sample with 30.8% loss in strength.

Also, strength reduction due to acid attack is as a result of penetration of the acid through the concrete pores to destroy the cement gel binder leading to the formation soft and soluble gypsum (calcium sulphate hydrate) that reacts with C_3S to form ettringite. Expansion of concrete specimen is as a result of the formation of secondary ettringite. The migration of these sulphate ions through diffusion which is slow a process. The pozzolanic reaction of nanosilica and metakaolin, leads to a dense, compact and better resistance of penetration and diffusion of the sulphate ions through concrete.

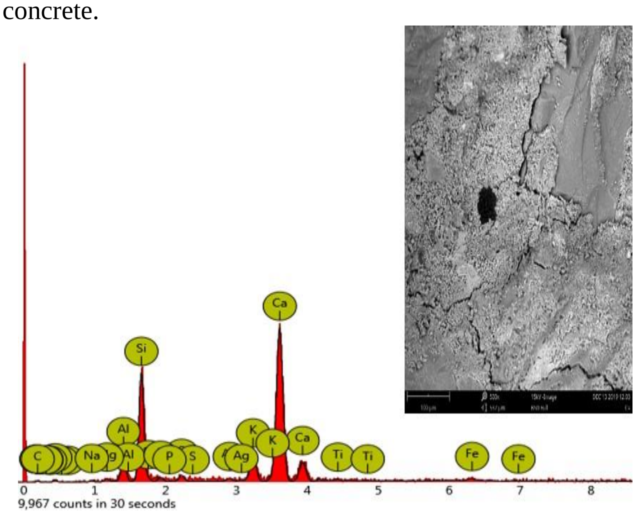

Figure 7: SEM/EDS of control Sample

The SEM/EDS of the control sample (Mix No 0) and optimum mix (Mix No 10) are shown in Figure 7 and Figure 8 respectively. According to Kunther *et al* (2017), for the concrete to perform optimally, the Ca/Si should be within 0.55 – 1.8. The EDS analysis shows that the residual Ca/Si of the control sample was 2.23 which fell outside the required range of 0.55 – 1.8 showing the presence of high calcium components like ettringite and gypsum while that of the optimum mix was 0.83 which fell within the allowable range of 0.55 – 1.8, which explains while the optimum replacement had higher strength after immersion in acidic medium. Similarly, the SEM analysis showed a scally and broken microstructure which could be associated with the debonding of materials while the optimum mix showed a dense state which can be associated with a compact and dense material related to strength.

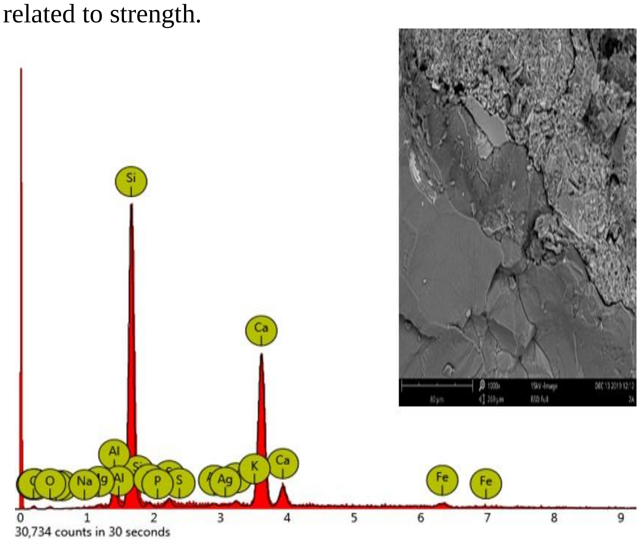

Figure 8: SEM/EDS of Optimum Mix

4.4 Sulphate Resistance Analysis

From the results as shown in figure 4.9, it was noticed that there was a considerable drop in the strength of concrete when immersed in sulphate solution for the control specimen from $35.12N/mm^2$ to $28.37N/mm^2$ representing a 23.8% drop in strength. The optimum replacement of (mix number 10) shown a considerable resistance of the sulphate attack as the strength reduction was from $40.2N/mm^2$ to $37.51N/mm^2$ representing 7.2% strength reduction. The sulphate (Mg_2SO_4) attack on concrete is severe as there is a double component attack on $Ca(OH)_2$ and C-S-H. Other replacements also showed considerable resistance to sulphate attack.

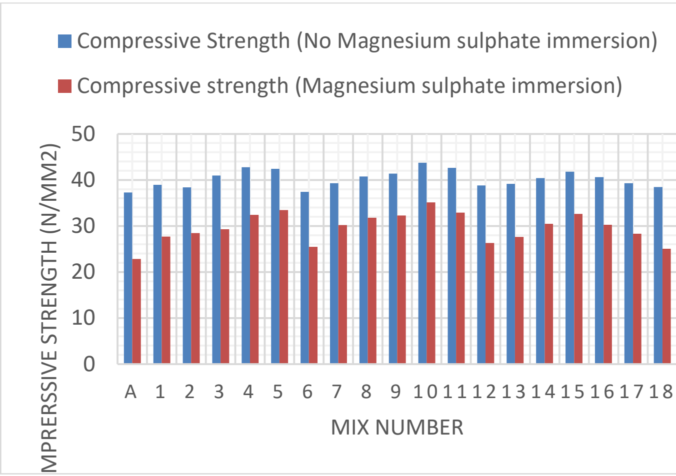

Figure 9: NM-concrete immersed in magnesium sulphate

With the aid of X-ray diffraction, the mineralogical phases of the samples after 28days of immersion in 5% MgSO4 solution and it's shown in Figure 10 and 11 for the control sample and optimum sample (optimum strength). From the XRD mineralogical phases, it was noticed that the predominant in the samples were Gypsum, Ettringite, Brucite, Portlandite (Calcium hydroxide) and C-S-H. Portlandite ($Ca(OH)_2$) had a considerable reduction in the quantity as shown in XRD as compared the XRD analysis at 28days. This is due to the reaction of $MgSO_4$ with $Ca(OH)_2$ to form gypsum (calcium sulphate) and brucite (Mg(OH)2)as shown in equation 6

$$\mathbf{Ca(OH)_2 + MgSO_4 + 2H_2O \rightarrow \underset{Gypsum}{CaSO_4.2H_2O} + \underset{Brucite}{Mg(OH)_2}}$$

Magnessium sulphate also reacts with C-S-H to form additional gypsum and brucite as shown in the equation 7

$$3CaO.2SiO_2.3H_2O + 3MgSO_4 + 6H_2O \rightarrow 3(CaSO_4.2H_2O) + 3Mg(OH)_2 + 2SiO_2$$

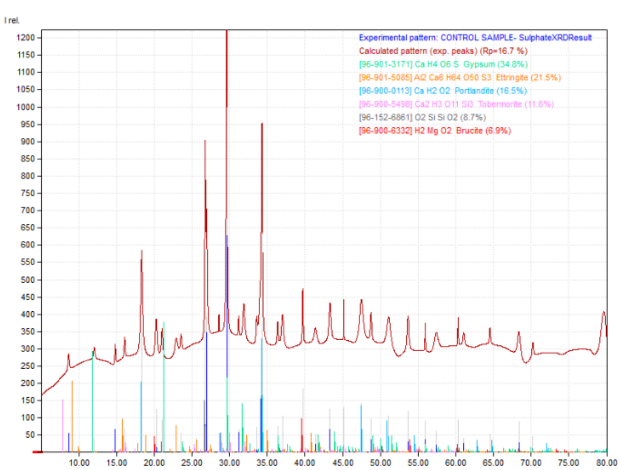

Figure 10: XRD Analysis of Control Sample after 28days Immersion in 5% $MgSO_4$

From the XRD results of the control sample and the sample with the optimum strength (mix number 10), the content of gypsum (calcium sulphate) is 34.8% and 25.0% respectively. This is as a result of the reaction with $Ca(OH)_2$ and can be seen in the reduction in the quantity of $Ca(OH)_2$ from the XRD analysis from 24.3% (before immersion) to 16.5% (after immersion) in the control sample representing a 47.3% reduction. The presence of brucite ($MgSO_4$) and silica gel (SiO_2) is responsible for the strength reduction are the silica gel (SiO_2) is a result of the breaking of bonds between the C-S-H molecules and the substitution of Ca^{2+} molecules with Mg^{3+} molecules in the C-S-H configuration. Mg^{3+} is a stronger reducing agent than Ca^{2+} in the electrochemical series, which is why it displaces Ca^{2+} in the reaction. The presence of ettringite is, as a result, the reaction between gypsum and tricalcium aluminate (C_3A) in the concrete as shown in the equation below. Ettringite is responsible for the concrete expansion and subsequent cracks in concrete.

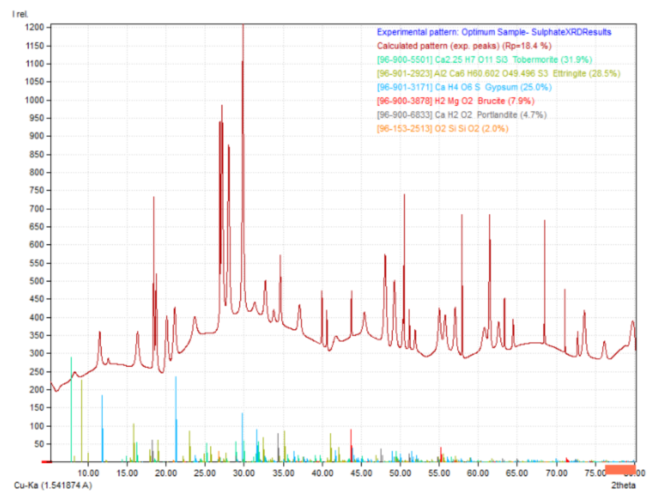

Figure 11: XRD Analysis of Mix 10 after 28days of immersion in 5% $MgSO_4$

SEM/EDS of the concrete samples after 28days of immersion in sulphate solution is shown in Figure 12 and Figure 13 for the control and optimum sample respectively. According to Kunther *et al* (2017), for the concret to perform optimally, the Ca/Si should be within 0.55 – 1.8. The EDS analysis showed that the residual Ca/Si for the control sample is 2.25 which is out the allowable range for the Ca/Si ratio for concrete. This high calcium content can be attributed to the formation of calcium elements like ettringite and gypsum. The optimum mix had a Ca/Si ratio 0.65 which is within the allowable range of 0.5 - 1.8 which can be the reason for the high residual strength after sulphate immersion.

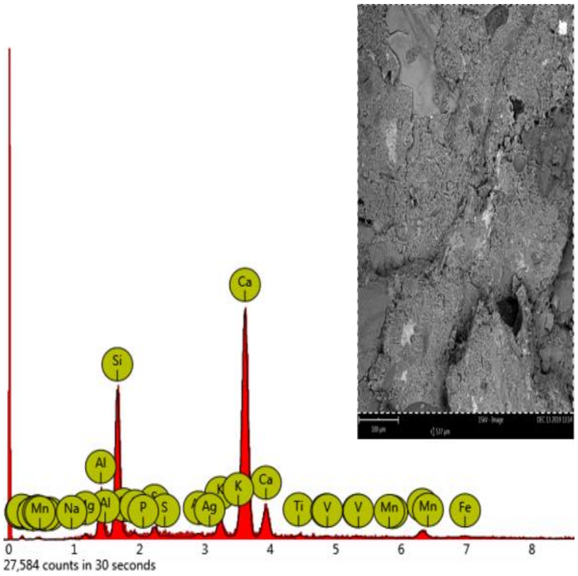

Figure 4.12: SEM/EDS of control sample

Similarly, the SEM analysis showed high formation of needle-like features which can be attributed to the ettringite that causes swelling, spalling and cracks in concrete. The optimum sample also showed the need-like formations but also showed a densed and compact structure. This could be the reason for high strength after immersion in sulphate solution.

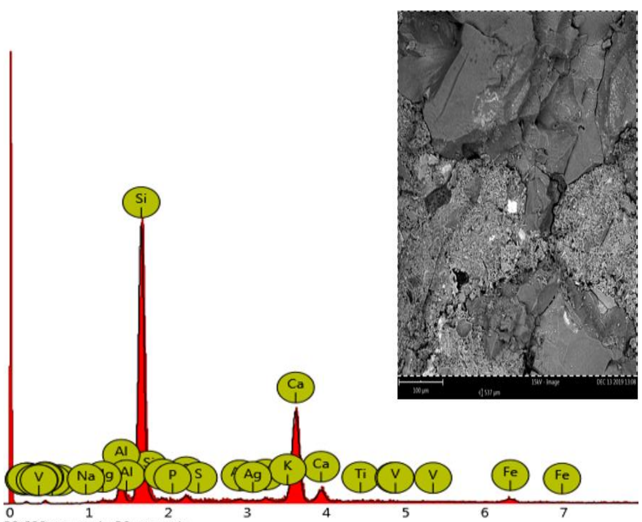

Figure 4.13: SEM/EDS of optimum sample

5. Conclusion

i. The nanosilica was obtained from rice husk was found to be 94% pure.

ii. Water absorption of the concrete samples was calculated after 28days of immersion in water and the sample with the least water absorption capability was mix number 10 at 0.77%.

iii. Acid resistance test was carried out on the concrete samples and after 28days of immersion in 5% H_2SO_4 solution, it was observed that the concrete sample, mix number 10 had the highest residual strength at 36.30N/mm^2 which is 33% more than the residual strength of the control sample at 24.31N/mm^2.

iv. Sulphate resistance test was also carried out on the concrete samples after 28days of immersion in 5% $MgSO_4$. It was observed that the concrete sample, mix number 10 had the highest residual strength at 35.10N/mm^2 which is 35% more than the residual strength of the control sample after 28days of immersion.

v. The XRD analysis was also carried out to determine the mineralogy of the concrete acid resistance after 28days immersion in 5% H_2SO_4 solution and sulphate resistance after 28days of immersion in 5% $MgSO_4$ solution. After 28days of immersion in 5% H_2SO_4, the mineralogy result showed the high presence of Gypsum and Ettringite which are responsible for concrete deterioration, especially swelling, crack development and spalling. Also after 28days of immersion in 5% $MgSO_4$ solution, the mineralogy results showed a high presence of Brucite, Gypsum and Ettringite which behaves in a similar way as in acidic medium.

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