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Original Article

Strength and Thermal Analysis in Sulfate Attack of Ordinary and Pozzolanic Rice Husk Ash Concrete

Catherine Mayowa Ikumapayi[a]* and Ponmile Ayanwale Taiwo[b]

[a] *Department of Civil Engineering, Federal University of Technology, P.M.B 704, Akure, Nigeria*
[b] *Department of Civil Engineering, Federal University of Technology, P.M.B 704, Akure, Nigeria*

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ABSTRACT

Durability is one of the unanswered questions limiting the wide acceptability of pozzolanic cement in the world today despite its numerous advantages. Resistance to sulfate attack is one of the durability properties of concrete. Ordinary and 10% Rice husk ash (RHA) pozzolanic cement concrete, with a mix ratio of 1:2:4 were produced. These specimens were subjected to both magnesium and sulfate attacks at 7, 28, 56 and 90 days hydration and their thermal analysis conducted. Information such as complex thermos, thermal effects (DTA) and thermogravimetric analysis (TGA) were collected in Nitrogen gas atmosphere. Hydration behaviour and state of the specimens with and without RHA were analysed. The experiment was conducted at a temperature between 30°C to 980°C and the heating speed was 10°C/min. The results of all the thermal analyses for concrete with 10%RHA (i.e the control), Na_2SO_4 concrete and $MgSO_4$ were obtained and analysed. The result of the compressive strength when Ordinary Portland cement was partially substituted for RHA was also presented. The results showed RHA to be capable of enhancing the compressive strength, sulfate resistance and thermal resistance of concrete if used in the right proportion.

1. Introduction

Concrete and reinforced concrete are vital construction materials that is widely acceptable worldwide because of their various advantages over other construction materials. Despite these advantages they can undergo what is termed as concrete disease [1,2]. Sulfate attack is one of the great enemies in concrete world and could be very destructive in concrete if left uncontrolled [3]. Sulfate can be present in the concrete components such as aggregate and service environment like groundwater, sea-water [4]. It exists in various form like sodium sulfate, magnesium sulfate and other alkali sulfates [5,6,7,8]. Past works on concrete revealed some of the danger that concrete could suffer from sulfate attack. Hekal *et al*, [6] reported that sulfate attack in concrete results into loss of adhesion and strength of such concrete. Likewise Abdul-Rahman *et al*.[3] in their review paper reported that sulfate attack is dangerous to concrete and may cause severe damage to concrete if not mitigated and therefore advocate for its mitigation.

Various researches have been conducted on the way to minimize or eliminate the effects of sulfate attack on concrete as well as increasing the compressive strength and other mechanical properties. Some levels of success were achieved. Abdul-Rahman *et al* [3] research combated sulfate attacked by the use of inhibitors like bio-waste [9] and showed that Bambusa Arundinacea has ability to combat sulfate attack with improved compressive strength when compare with some other inorganic inhibitors. Some other researchers discovered that the use of fly ash, slag, silica fume, sand of dune, saw dusk ash, aloe vera stabilized coconut fibers, goepolymers and metakaolin can be effective in mitigating sulfate attack, enhancing concrete durability and concrete mechanical properties, if used in the right proportion [10-17]. You *et al.* [18] used macro synthetic

* *Corresponding author*
E-mail address: *mayowaik@yahoo.com*
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steel to combat sulfate attack and discovered that the degree of sulfate degradation was reduced. They reported that there was better compactness of the concrete components as a result of the treatment.

Despite all these outputs, there is still a wide margin in achieving durable concrete or sulfate mitigating concrete especially from the angles of sustainability and eco-friendly materials. The use of Rice Husk Ask (RHA) fits into the gap and therefore should be deeply investigated.

Concrete. Past and recent studies reported that Pozzolanic RHA concrete shows better compressive strength, permeability resistance, chloride ion penetration resistance among others [19-21] Different methods of analysis has been previously used to analysed in sulfate attack and other durability tests. This research seeks to analyse the effect of different sulfate attack in concrete and the ability of RHA in mitigating such effects as well as its thermal resistance using Thermal Analyses.

2. Materials and Methods

2.1. Materials

Ordinary Portland cement of Dangote *3X* brand conforming with British standards BS 12 [22] was used. River sand and crushed granite of 25 mm maximum nominal size were used as fine and coarse aggregates respectively. RHA was gotten by calcinating Rice husk at 600^{o}C and the product was then sieved using 75 µm (No.70) . Sodium sulfate (Na_2SO_4) and magnesium sulfate (Na_2SO_4) solutions at 23±2 °C were prepared with 30,000mg/L concentration. Concrete cubes of sizes 150 x 150 x 150 mm^3 were prepared for compressive strength test of 1:2:4 mix ratio of cement to sand to coarse aggregates.

2.2. Materials Testing

Chemical test on the RHA was done using Minipal 4 Energy Dispensing X-ray Fluorescence Spectrometer (EDXRF) and the oxide composition was obtained. Fine and coarse Aggregates were tested and graded in accordance with BS EN 933-1 [23]. Setting times for the 10%RHA and OPC were carried out in agreement with the standard specification [24] as well as the compressive strength of the concrete using universal testing machine. The concrete cubes were cured for 28 days in lime water and later immersed inside sulfate solutions of Sodium sulfate (Na_2SO_4) and magnesium sulfate (Na_2SO_4) solutions [25-26] at 23±2 °C both of 30,000mg/L concentration. The compressive strength test was performed on the concrete after 7-, 28-, 56- and 90-days immersion in the sulfate solutions.

2.3. Concrete Mix Proportions

The mix proportions shown in Table 1 was adopted in this study. 10%RHA indicates partial replacement of OPC with RHA of 10% quantity. Concrete was mixed in this designated proportion with Conplast SP430 superplasticizer added in small proportions to improve and keep the slumps range between 50mm-60mm.

. Table 1. Mix proportion of concrete

Mix	**Cement (kg)**	**RHA (kg)**	**Fine aggregate (kg)**	**Coarse aggregate (kg)**	**Water (kg)**	**Water/ Cement binder ratio**
CTL	399.12	0	798.24	1597.00	219.52	0.55
10% RHA	359.21.	39.91	798.24	1597.00	219.52	0.55

2.4. Thermal Analysis and Thermal Gravimetric Analysis

The thermal analyses were carried out for all the mix proportions. Simultaneous TGA/SDTA 851 apparatus was used. This apparatus gathers multiple information from the sample's behaviour during heating. Information such as complex thermos, thermal effects (DTA), thermogravimetric analysis (TGA), differential thermogravimetric weight changes and (DTG) were collected in Nitrogen gas atmosphere. Hydration behaviour and state of the cubes samples were analysed with the use of thermal analysis. A known weight of each of the sample was investigated and the Temperature Program Heat was from 30.00 °C to 980 °C @ 10 °C/minute in Nitrogen atmosphere with a cleaning rate of 20 mL/minute.

3. Results and Discussion

3.1. Chemical Composition of RHA

The results of the chemical test of the RHA is presented in Figure 1. The sum of the three chief oxides (SiO_2 + Al_2O_3 + Fe_2O_3) is 88.04% shows RHA to be a pozzolans of class N [27]. Percentage of SO_3 is 0.36% and is less than 4.0 maximum while the loss on ignition is 3.18% also less than the maximum of 10. The result follows the same trend as past research outcomes [28-29]. This is an indication that RHA is a pozzolan.

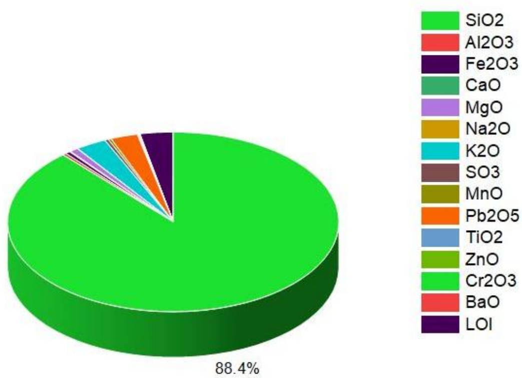


Table 2. Particle size distribution for the constituent materials

Fig 1. (a) Oxide Composition of the RHA.

3.2. Physical Properties of the Research Materials

The natural moisture content and bulk density of the fine aggregates used were 11.34% and 2233.05 kg/m^3. Limestone Portland cement of Dangote brand was used. The particle size distribution of the coarse aggregates and the fine aggregates as well as their hydrometer analysis are shown in Table 2. The river sand is well graded and therefore suitable for use in construction works [30]. Less than 5% RHA was also retained on BS 45 µm sieve as specifies by the standard [27]

3.3 Effect of RHA on setting time of cement paste

Effect of RHA on the setting time of cement was studied by partially replacing OPC with RHA from 0% to 20% at a stepsize of 5%. The results is shown in Figure 2. The presence of RHA reduces the initial and final setting times of the resulting pozzolanic cement. The setting time obtained still falls within the recommended values of 45 minutes minimum and 600 minutes maximum specifies by BSI EN 196 [24]. This is an indication that RHA cement within this percentage replacement meets the setting times criteria of cementitious materials. Furthermore, it also shows that RHA pozzolanic cement takes shorter time to fully hydrate than OPC concrete. This result is also in accordance with the result of some past researchers [29,31]. Base on the result of the initial and final setting times, when using RHA as pozzolans the use of superplasticizer will help in improving the workability especially in self compacting concrete.

Sieve size	**Coarse aggregates% Passing**	**River sand % Passing**	**RHA % Passing**
14.0mm	100.00		
12.0mm	68.42		
10 .0mm	32.70		
6.0 mm		100.00	
5.0 mm	1.96	93.94	
4.0 mm	0.36	92.24	
3.0 mm		90.46	
2.36 mm		88.10	
1.80 mm		86.58	
1.70 mm		83.23	100.00
600 µm		66.59	63.80
500 µm		63.57	55.10
212 µm		22.95	8.20
150 µm		12.25	1.70
0.75 µm		9.60	0.60
pan	0.00	0.00	0.00

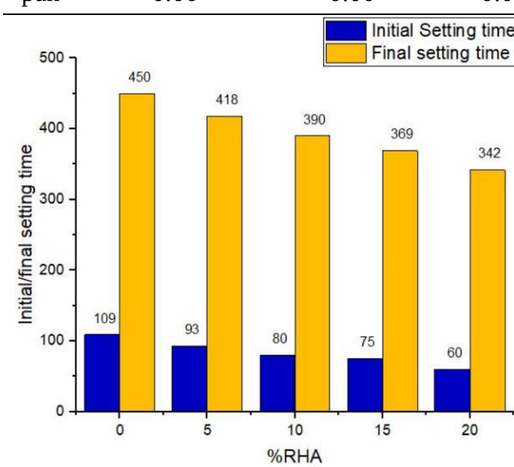


Figure 2: Effect of RHA on setting time of cement paste

3.4 Effect of RHA on the Compressive strength of Concrete subjected to Na_2SO_4 and $MgSO_4$

The result of the resistance of compressive strength of RHA and OPC concrete to Na_2SO_4 and $MgSO_4$ of 30,000 mg/L concentration is as shown in Figure 3 and 4 respectively. From the two Figures the optimum percentage replacement for RHA obtained is 10%, at this percentage, maximum compressive strength was obtained even up to 90days. The results also show that sulfate of low concentration has no significant effect on compressive strength of concrete throughout the 90 days for Na_2SO_4 while for $MgSO_4$ there is a reduction in the compressive strength at 90 days. This is an indication that $MgSO_4$ is more aggressive to concrete than Na_2SO_4 which is in accordance with the result obtained by Coopamotoo and Rughooputh as well Souza *et al.* [26, 32,]. Souza *et al.*

reported that for both RHA and Silica fume pozzolanic concrete subjected to $MgSO_4$ and Na_2SO_4 attack, higher compressive strength loss was experienced in MgSO4 than Na2SO4. This also further implies that effect of sulfate attack is a function of the sulfate concentration as well as the particular type of sulfate present in the service environment this is in conformity with the research report of Premlall *et al*. and Souza *et al.* [32,33]. The results in Figure 3 and 4 further showed that RHA concrete specimens have better resistance to any type of sulfate attack than OPC concrete for all the days because it experienced lower compressive strength loss at the two type of sulfate media. Souza *et al*.[32] also obtained similar result.

3.5 Thermal analysis of the concrete specimen

The descending TGA thermal curve in Figure 5 indicates the rate of weight loss of the sample of concrete with 10% RHA replacement level (OPC), concrete subjected to Na_2SO_4 (OPC/ Na_2SO_4), and concrete subjected to $MgSO_4$ (OPC/ $MgSO_4$) . The figure shows three steps of weight loss. For the concrete with 10% RHA replacement level (OPC), the first step weight loss which can be traced to moisture or water [34-36] was about 2.73 % or 0.22 mg between 25.5^{o}C and 261.11°C. The second step weight loss, at 383.33°C, resulted from loss of [34-36] with the mass loss of 72.73 % or 5.97 mg. The origin of the third step, a weight loss of 3.1 % or 0.2448 mg due to carbon dioxide [34-36] was observed at 884.8^{o}C.

Similarly, for OPC/ Na_2SO_4 shown in Figure 6, the first step weight lost was 4.208 % or 0.415 mg between 27.99^{o}C and 252.94°C and the second step weight loss, at 541.16°C, was 73.688 % or 7.275 mg. The third step weight loss of 3.68 % or 0.363 mg was observed at 887.4^{o}C.

For OPC/ $MgSO_4$ shown in Figure 7, the first step lost about 1.053 % or 0.1 mg between 25.56^{o}C and 241.16°C while the second step at 600.00°C lost 82.632 % or 7.908 mg and a weight loss of 2.629 % or 0.252 mg was observed at 883.6^{o}C for the third step.

These results shows that greater percentage of the weight loss in all these specimens can be traced to the second step weight loss due to carbon monoxide. The mechanism behind the behaviour and greater aggressiveness obtained in $MgSO_4$ when compared with Na_2SO_4 can be explained in this same second step. $MgSO_4$ has a weight loss of 82.632 % due to carbon monoxide while Na_2SO_4 has a weight loss of 73.688 % due to carbon monoxide. The control samples without any sulfate attack exhibits the least weight loss of 72.73 % at this same second step. All these further shows that sulfate attack is not concrete friendly especially $MgSO_4$ attack.

3.5 Comparative analysis of the thermal results

The plot of combined TGA Thermograms of OPC, $MgSO_4$, and Na_2SO_4 concrete shown in Figure 8 further revealed the effect of temperature on the weight loss of the three specimens. The three results of the thermogravimetric analysis (TGA) revealed that concrete with 10%RHA (i.e the control) has the best maximum thermal degradation temperature of 461.02.73 ◦ C and a stable weight of 27.03%. Na_2SO_4 concrete has a maximum thermal degradation temperature of 490.7 ◦ C and a stable weight of 22.272%. $MgSO_4$ has the worst maximum thermal degradation temperature of 553.7 ◦ C and a stable weight of 18.97%.

The percentage char at 886.75 ◦C which is the residue/left over of the materials are 23.468%, 16.556% and 17.82% respectively as shown in Figure 9. This further explains the mechanism of strength and other properties deterioration in the various specimens. All these indicate that sulfate is not concrete friendly which agrees with the report of Rahman *et al*.[3] and therefore can be mitigated using RHA [19-21].

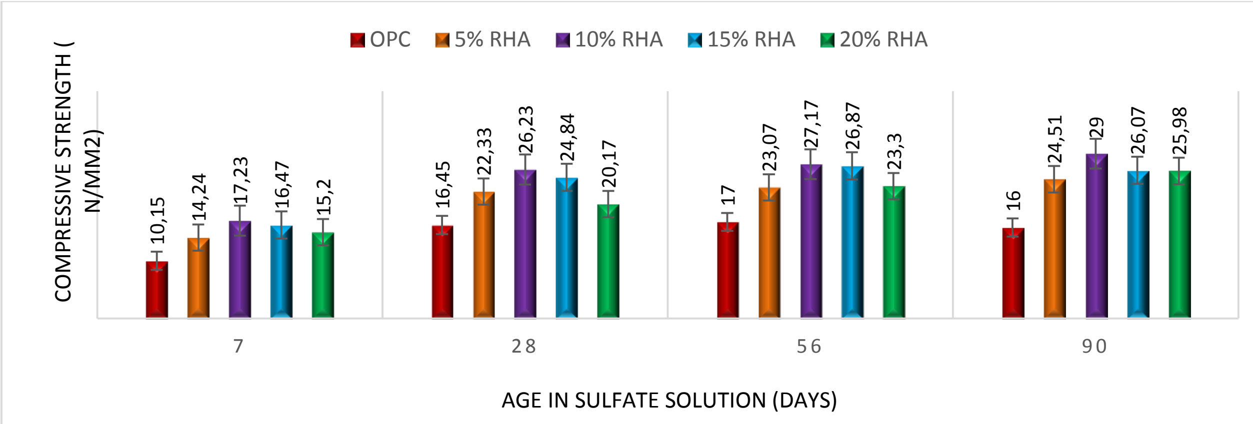


Figure 3: Compressive Strength of Concrete Immersed in Na_2SO_4 of 30,000 mg/L concentration

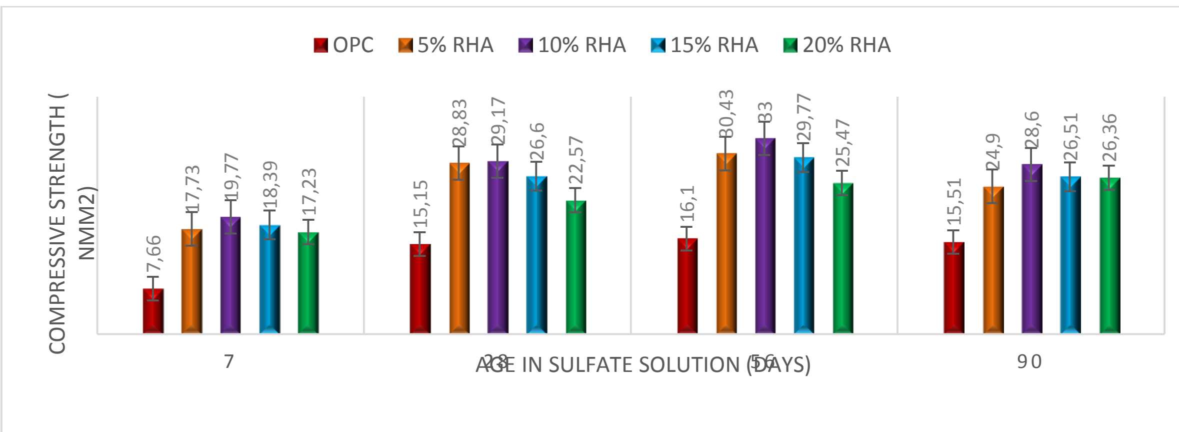


Figure 4: Compressive Strength of Concrete Immersed in $MgSO_4$ of 30,000 mg/L concentration

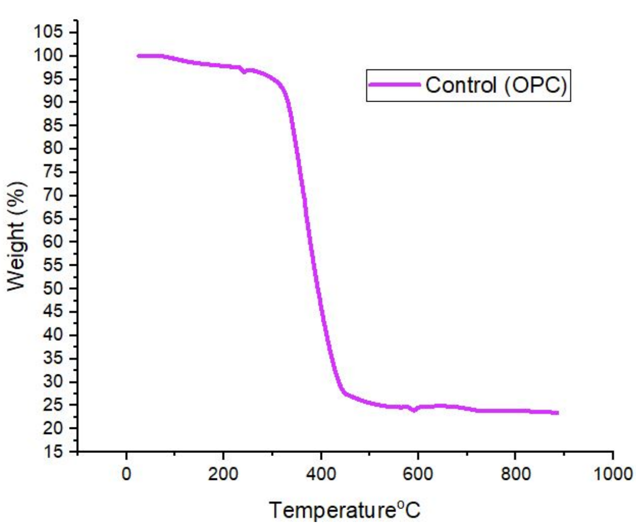


Figure 5: TGA Analysis for the control specimen (OPC)

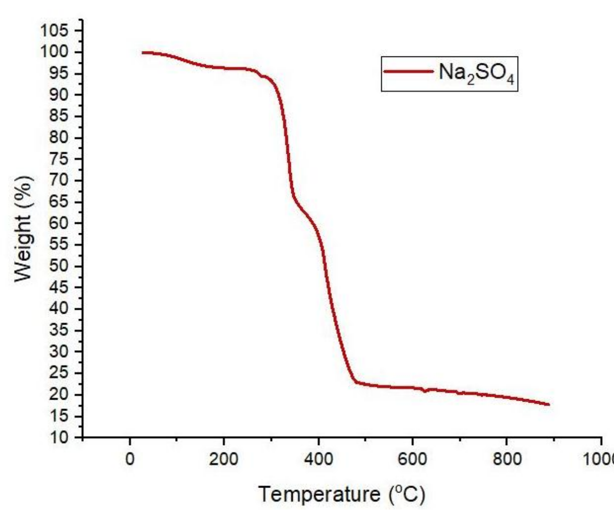


Figure 6: TGA Analysis for the OPC/ Na_2SO_4

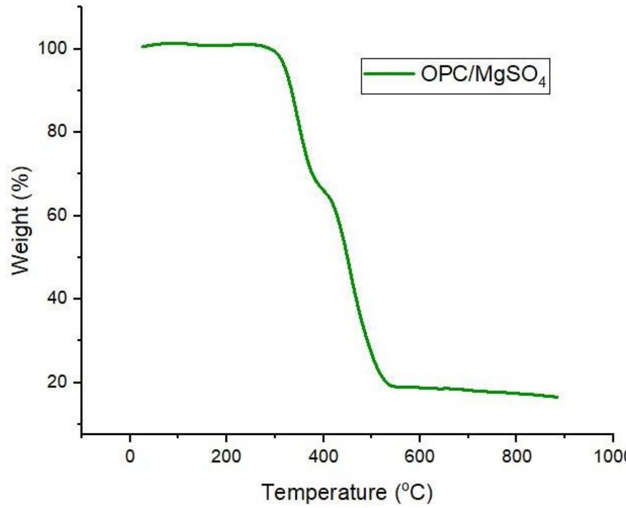
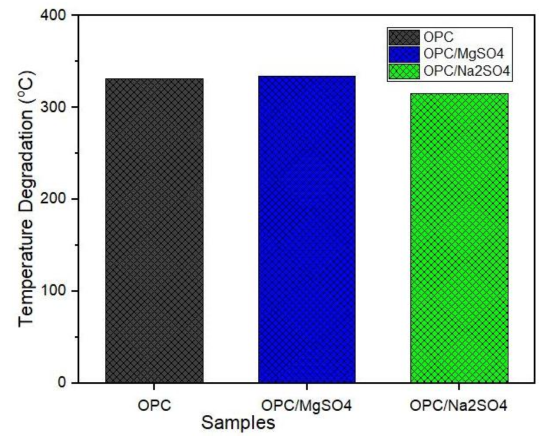


Figure 7: TGA Analysis for the OPC/ $MgSO_4$

3.6: Comparative analysis of the thermal results

The plot of combined TGA Thermograms of OPC, $MgSO_4$, and Na_2SO_4 concrete shown in Figure 8 further revealed the effect of temperature on the weight loss of the three specimens. The three results of the thermogravimetric analysis (TGA) revealed that concrete with 10%RHA (i.e the control) has the best maximum thermal degradation temperature of 461.02.73 ◦ C and a stable weight of 27.03%. Na_2SO_4 concrete has a maximum thermal degradation temperature of 490.7 ◦ C and a stable weight of 22.272%. $MgSO_4$ has the worst maximum thermal degradation temperature of 553.7 ◦ C and a stable weight of 18.97%.

The percentage char at 886.75 ◦C which is the residue/left over of the materials are 23.468%, 17.82% and 16.556% respectively as shown in Figure 9. This further explains the mechanism of strength and other properties deterioration in the various specimens. This strengthened the need for eliminating or reducing sulfate attack in concrete and the fact that thermal resistance also depends on the type of sulfate attack which is line with the compressive strength results.



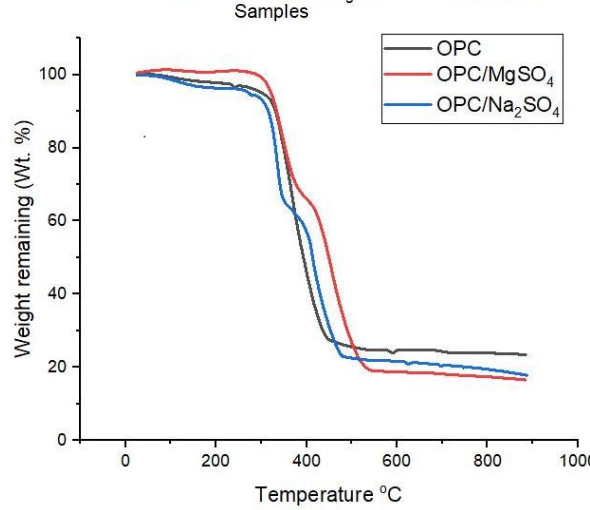


Figure 8: Plot of TGA Thermograms of OPC, OPC/$MgSO_4$, and OPC/Na_2SO4

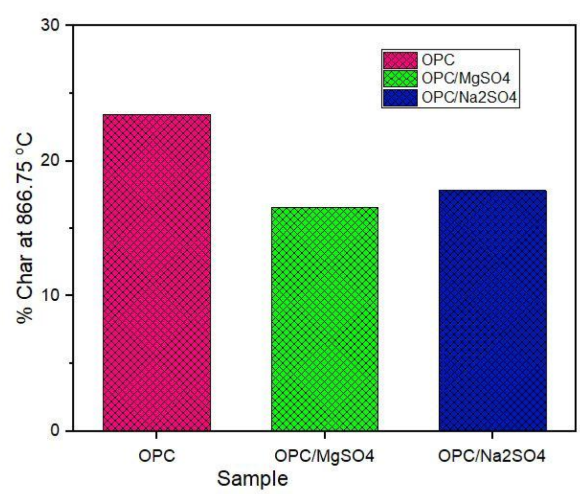


Figure 9: Plot of percentage % char of OPC, OPC/$MgSO_4$ and OPC/Na_2SO_4

Thermo-oxidative parameters of all samples calculated from TGA and presented as a function of % $MgSO_4$ and % Na_2SO_4 are shown in Table 3. Degradation Temperature of 5%, 10% and 50% of OPC, OPC/$MgSO_4$ and OPC/Na_2SO_4

are also shown in Figures 10 to12. The trend of the results shows that the degradation temperature of OPC/$MgSO_4$ at all these temperatures is the highest.

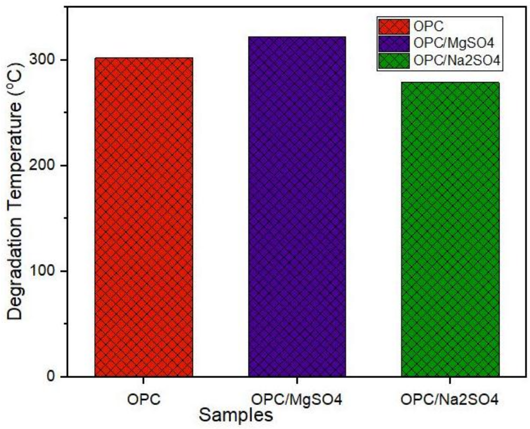


Figure10: Plot of 5% Degradation Temperature of OPC, OPC/$MgSO_4$ and OPC/Na_2SO_4

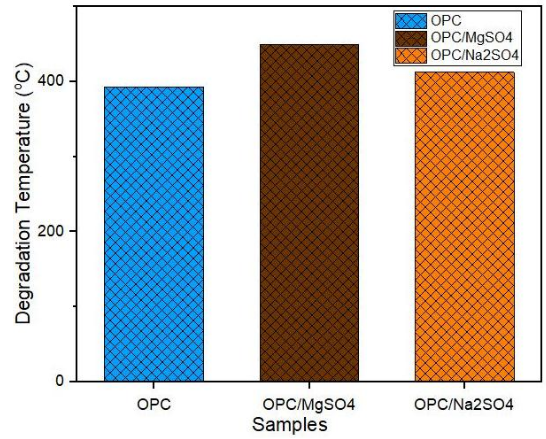


Figure 12: Plot of 50% Degradation Temperature of OPC, OPC/$MgSO_4$ and OPC/Na_2SO_4

Figure 11: Plot of 10% Degradation Temperature of OPC, OPC/$MgSO_4$ and OPC/Na_2SO_4

Table 3: Thermo-oxidative parameters of the concrete samples

Sample designation	***T_5(°C)***	***T_{10}(°C)***	***T_{Max}(°C)***
OPC	*302.288*	*331.756*	*393.413*
OPC/$MgSO_4$	*322.093*	*333.947*	*449.161*
OPC/Na_2SO_4	*279.161*	*315.503*	*412.894*

4.0 Conclusions

From the study conducted on mitigating sulfate attack in concrete using rice husk ash, it can be concluded that;

i. The RHA is further confirmed to be a pozzolan.
ii. Na_2SO_4 attack of 30,000 mg/L concentration (low concentration) has no considerable effect on the compressive strength of concrete up to 90 days while $MgSO_4$ attack of 30,000 mg/L concentration showed compressive strength reduction at 90 days.
iii. The results of the TG and DTA showed that greater percentage of the weight loss in all these specimens could be traced to the second step weight loss, which is due to carbon monoxide.
iv. Sulfate attack reduces the thermal resistance of concrete.
v. RHA has a great prospectus in enhancing the sulfate and thermal resistance of concrete.
vi. The replacement level of RHA up to 20% is recommended with the optimum percentage at 10%.

5.0 Recommendations

On the basis of this research findings, the potential of Rice Husk Ash to mitigate sulfate attack in concrete is high and thermal stability or resistance of concrete can also be enhanced through its use. Therefore, RHA should be adopted as cement admixture for sulfate resistance in concrete especially in aggressive areas. Its use can be enhanced by employing the use of superplasticiser to improve the workability.

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