

# Lime-Rich Sludge Catalysis of Powdered Blast-Furnace Slag with and Without Rice Husk Ash

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## ABSTRACT

More and more people are concerned about environmental cleanliness and preserving the biosphere's delicate eco-system. Despite the widespread belief that economic expansion and risk-free environmental preservation cannot coexist, achieving sustainable growth without compromising environmental quality is not an impossible task. The purpose of this research is to analyse how lime rich sludge (LRS), a waste product from the acetylene industry, affects the activation of granulated blast-furnace slag (GBFS). After 4 hours of calcining at 850 degrees Celsius, the LRS was allowed to cool in the furnace before being slaked with water and dried at 80 degrees Celsius for 24 hours. Ten, twenty, and thirty percent LRS were used in lieu of GBFS. The findings demonstrated an increase in combined water and free lime with increasing LRS concentrations. Compressive strength values for GBFS-LRS blends rise up to 20 weight percent LRS, but drop after reaching 30 weight percent LRS. Variable quantities of rice husk ash (RHA) were added to a mixture comprising 30 wt., percent of LRS in order to increase its compressive strength. Compressive strength was shown to improve with increasing RHA concentration. Results from XRD, FTIR, and TG/DTG tests corroborate the chemical and mechanical characteristics of GBFS-LRS blends in the presence and absence of RHA.

**Keywords:** Granulated blast-furnace slag, lime-rich sludge, rice husk ash, compressive strength.

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## INTRODUCTION

A waste disposal dilemma, impacting both the economy and the environment, has arisen as a consequence of the increased production and use of waste materials. Hundreds of years from now, the ecosystem will still be contaminated by today's garbage. Wastes cause significant air, surface, and ground water pollution as well as economic concerns including the expensive cost of landfill management. This ever-growing waste stream is still a major environmental problem that must be handled in a way that is both cost-effective and kind to the environment. The economic and environmental challenge of waste disposal and the reduction of the depletion of natural resources may both be helped by recycling garbage into usable items to replace the natural/commercial

products whenever feasible [1]. Sludge is generated in massive amounts all around the world and often buried in landfills. Because of the scarcity of available landfill space, scientists have been looking at various methods of sludge recycling and disposal. Using them in civil engineering builds is the most efficient approach to recycle these materials.

Because of the disparity in supply and demand for housing and the high cost of conventional construction materials, as well as the depletion of traditional building materials, the importance of locally created building materials is often understated. In addition, there will be increasing pressure to cut down on cement use what with the pressing need to cut back on energy use and CO<sub>2</sub> emissions and the anticipated growth in cement output to fulfil the requirement of a continually expanding global population. Alternative construction materials that don't break the bank have been studied as a possible solution [2]. Several different types of industry may benefit from repurposing trash since it increases product variety, lowers production costs, and offers a new source of raw materials [3]. Egypt According to environmental principles, the government's management plan promotes byproduct disposal via meaningful reuse. As a byproduct of the production of iron, granulated blast-furnace slag (GBFS) is created when limestone, coke ash, and the siliceous and aluminous residue left over from the reduction of iron ore to iron metal are fused together. Slag, a nonmetallic liquid, develops during this process and floats on the molten iron. After being extracted from the molten metal, it is cooled. There are three distinct forms of slag that may be created, all of which are dependent on the method of cooling: air-cooled slag (ACS), expanded or foamed slag, and granulated slag [4]. Since big crystals cannot form in molten slag due to the quick cooling by water, the resultant slag often consists of more than 95% glass (amorphous calcium aluminosilicates). Slag that has been cooled by water is known as GBFS. LRS, or lime-rich sludge, is a mud that has a high concentration of limewater. Calcium carbide (CaC<sub>2</sub>) and water undergo an exothermic reaction to create acetylene and calcium hydroxide (Ca(OH)<sub>2</sub>) and 27 kcal of energy.

The manufacturing of paper, acetylene, sugar, fertiliser, sodium chromate, and soda ash all result in the production of lime sludge. Sludge is produced by these sectors at an annual rate of around 4.5 million tonnes [5]. Except for carbide sludge, every kind of sludge contains lime (in the form of calcium carbonate). Carbide sludge produced by the acetylene industry is rich in lime, present mostly in the form of Ca(OH)<sub>2</sub>. Even though lime is a primary component of this sludge, its chemical composition varies greatly depending on the kind of limestone utilised in the original procedure. The phospho-chalk from the fertiliser industry, for example, has harmful pollutants including 5–9 percent SO<sub>3</sub>, up to 1.5 percent P<sub>2</sub>O<sub>5</sub>, and up to 2 percent fluoride.

There are free alkalis present in concentrations of up to 2% in sludges made from paper, sugar, and chromium. Chromium concentrations of up to 10% and chloride concentrations of up to 2% are found in the chromium and carbide sludges, respectively. Cement and similar construction materials can't be used in large quantities because of the presence of these harmful pollutants . Cement mortars and concretes made using lime sludge (LS) have been the subject of little research. Pastes made from a mixture of waste paper sludge ash (WSA) and ground granulated blast-furnace slag (GGBS) were tested for their compressive strength and hydration properties at a water-to-binder (w/b) ratio of 0.5. Relative strengths are provided, with findings compared to those

of regular Portland cement (PC) paste. The relative strengths of WSA–GGBS pastes were found to be somewhat weak in the beginning (1d), but then grew dramatically by 7d, and again by 28d and 90d. The paste made up of 50% WSA and 50% GGBS had roughly 50% of the strength at 90d as the PC control paste. The mineral components of the WSA and the hydration products of WSA–GGBS pastes were identified via X-ray diffraction (XRD) and thermo-gravimetric (TG) analyses, respectively. Gehlenite, calcium oxide, bredigite, together with minor quantities of anorthite and calcium carbonate, and even smaller amounts of calcium hydroxide and quartz, make up the bulk of the WSA's crystalline components [6]. In underdeveloped nations, much of the rice husk (RH) that is produced is either incinerated at the rice field or mill, or thrown away as trash. Pollutants including carbon dioxide (CO<sub>2</sub>) and carbon monoxide (CO) would be discharged into the air if a pile of rice husk were to be burned without any kind of supplemental oxygen. Methane emissions might also come from the aerobic degradation of rice husks in landfills. One of the most pressing problems we face today is global warming, caused in part by the emission of greenhouse gases into the atmosphere [7]. One viable option for dealing with agricultural trash is recycling the wasted materials. The ash recovered by burning RH contains about 75% amorphous silica, making up roughly 20% of the recoverable weight. By carefully burning RH at temperatures below 800 degrees Celsius, a highly reactive pozzolanic material is created. Although it is a byproduct of the paddy business, RHA may be harmful to the environment if not disposed of correctly. RHA is a renewable supply of silica since it can be harvested every year, unlike natural pozzolana. According to ASTM, a material is considered to be pozzolanic if its chemical examination reveals that more than 70% of the substance consists of iron oxide (Fe<sub>2</sub>O<sub>3</sub>), silicon oxide (SiO<sub>2</sub>), and aluminium oxide (Al<sub>2</sub>O<sub>3</sub>). With RHA, cement concrete has better workability, low heat of hydration, lower permeability, and higher strength. Lime rich sludge (LRS), a byproduct of the acetylene industry, was expected to have an effect on the hydration of GBFS in this study. Furthermore, the water intake

The effects of RHA on the GBFS/LRS blend's properties were investigated.

## **PROCEDURES AND EXPERIMENTAL MATERIALS**

### **MATERIALS**

Lime rich sludge (LRS), granulated blast-furnace slag (GBFS), and rice husk ash were employed in this study (RHA). The LRS was supplied by INSOMAT Company of Tammoh, Giza Governorate, Egypt, while the GBFS was supplied by Iron and Steel Company of Helwan, Egypt. The GBFS was ground mechanically until the Blaine surface area was 3500 cm<sup>2</sup>/g. The paddy fields of Tatoon, Etsa, El-Fayoum, Egypt, were the source of the RH used in this study. The pentosane and lignin were removed by one hour of boiling, followed by washing with distilled water and drying at 110 degrees Celsius for twenty-four hours. After burning dried RH with a particle size of 0.5 mm at 450 °C, RHA with a silica content of over 90% and a surface area of 60 m<sup>2</sup>/g was produced. X-ray diffraction confirms the amorphous nature of BFS and RHA. Results from X-ray fluorescence (XRF) spectrometry examinations of GBFS, LRS, and RHA oxides.

### **CONVENTIONS OF ACTUAL USE**

XXII. In the first step, hydrated and carbonated components were burned off of the lime rich sludge (LRS) by calcining it at 850oC for 4 hours. It was heated in the furnace, cooled, and slaked

with water to produce hydrated lime before being dried in an oven electric drier at 80 °C for 24 hours. Because of its sensitivity to moisture and carbonation, dry LRS was stored in a desiccator. It was ground down to a consistent 45 m in size. LRS concentrations of 10, 20, and 30% wt. replaced GBFS. Different amounts of RHA (5%, 7.5%, and 10% wt., %) were added to a GBFS-LRS mix that already included 30% wt., % of LRS to evaluate the RHA's pozzolanic activity. Each dry combination (BFS-LRS or BFS-LRS- RHA) was thoroughly homogenised by grinding its constituent components in a porcelain ball mill for 1 hour. After placing the dried samples on a non-absorbent surface, a water to cement (W/C) ratio of

At the middle, a crater was dug out with a diameter of 0.25, and water was poured into it. To help absorb the water, the dry component of the mixture was softly towelled over the wet portion for approximately 1 minute. After that, I used an average measuring trowel to mix everything up vigorously and continuously for approximately three minutes. The pastes were crushed and homogenised by hand in stainless steel cube moulds of one inch thickness. For a few minutes, the moulds were vibrated to aid in compaction. Once the last layer was compacted, a thin-edged trowel was used to smooth up the top surface of the mould. For subsequent examinations at various curing periods, the specimens were cured for 24 hours in a humidity room at 100% RH, then demoded and cured under tap water (1, 28 and 90 days). After the allotted amount of time had passed, the hydration of cement pastes was halted by grinding 10g of representative sample in a beaker containing a 1:1 methanol–acetone combination. and then stirred by machine for a period of one hour. After several washes in the stopping solution and diethyl ether, the mixture was filtered through a G4, dried at 70 degrees Celsius for one hour, collected in polyethylene bags, sealed, and kept in desiccators . The percentage of ignition loss of a dried sample is equivalent to its chemically mixed water content (on the ignited weight basis). About 2 grammes of the dried-out material were heated to 1,000 degrees Fahrenheit for an hour. Each sample's free lime water content was taken into account to adjust the combined water content result . Samples of each hydrated cement paste (0.5 g) were placed into 40 ml of a glycerol-ethanol combination (1:5 v/v), together with a tiny quantity of anhydrous BaCl<sub>2</sub> (0.5 g) as a catalyst and phenolphthalein (0.5 g) as an indicator, and the free lime content was calculated in this way. This was cooked for 30 minutes in a conical flask with an air reflux on a hot plate (the colour becomes pink). Until the last trace of pink gone, the flask's contents were titrated with a 0.1N alcoholic ammonium acetate solution. If the pink colour emerges upon resuming heating, the titration is continued with the ammonium acetate solution until no more pink colour arises upon heating. Percent CaO = [(W1V)/W] 100, where W is the starting weight, W1 is the quantity of CaO in grammes that was added, and V is the volume of alcoholic ammonium acetate in millilitres . Compressive strength was assessed using ASTM C109M-12 at each testing interval. Three cubes were dried in a dehydrator at 105 degrees Celsius for 24 hours. The Seidner, Riedinger, Germany, 600 KN force capacity compressive strength equipment was used for the tests. The XRD, FTIR, TG, and DSC methods were used to examine a subset of the hydration products to confirm the process anticipated by the chemical and mechanical experiments. The current investigation used XRD using the powder technique. This was accomplished using a Philips PW3050/60 diffractometer equipped with a Cu k radiation (=1.5418) X-ray source. The size of the scan's steps was 2. With a collecting time of s, and a 2 range of 5-60, what you get is very impressive. The X-ray tube was set to operate at a constant 40.0 KV and 40.0 mA. For phase identification of a wide range of

crystalline phases in a sample, an online search of a standard database (JCPDS database) for X-ray powder diffraction pattern is useful. The DT-30 Thermal Analyzer was used to perform the DTA, which included heating the sample to 1000 °C in a nitrogen environment (Schimadzu Co-Kyoto, Japan). Amounts of calcined alumina, about 50 mg (76 m), were utilised as filler. A tiny crucible made of platinum and rhodium held the finely powdered moist cement mixture. All studies used a constant heating rate of 20oC/min [8]. Since the alkali halide pressed disc approach further reduces scattering, it was used to record the infrared (IR) spectrum analysis . In an agate mortar, exactly 2.0 mg of the sample was ground with 198 mg of potassium bromide to create a homogenous mixture. The mixture was compressed into a 1 cm diameter translucent disc under vacuum. We used a Mattson Genesis IR to capture infrared spectra of KBr discs in the region of 400 to 4000 cm<sup>-1</sup> for examination.

## **RESULTS**

The water has been chemically mixed. Displays the change in W<sub>n</sub> percent, of hydrated GL10, GL20, and GL30 blends throughout the course of various curing durations. W<sub>n</sub>, percent measurements clearly rise dramatically with the hydration time up to 28 days; this is due to direct interaction between water and the solid grains of the BFS/LRS mix and deposition of hydrated products in certain of the pores; resulting to an increase in W<sub>n</sub>, percent. Nonetheless, after 90 days, the W<sub>n</sub> progressively rises, since water is less likely to diffuse through the coating of hydration products into the unhydrated regions of BFS and LRS grains. Hardened pastes prepared with GL30 have a greater W<sub>n</sub>, percent than those made with GL10 or GL20 mixes at all ages of curing. High lime concentration in the cured GL30 paste is responsible for this property. The W<sub>n</sub> concentrations of cured BFS-LRS pastes (GL30) at 5, 7.5, and 10 weight percent RHA, respectively. It is clear that after one day of curing, the GL30R10 combination yields the lowest W<sub>n</sub> level in the hardened paste. Since RHA's active silica forms a gel at a slower pace than water, it slows down the hydration rate of GBFS-LRS pastes while they are young. However, the combined water values at a later age of hydration are greater if the RHA content is higher. Because the active silica in RHA combines with free lime to create extra CSH with high combined water content, this is the case. After 28 days of curing, the W<sub>n</sub> value of GL30R10 mix is very similar to that of GL30R7.5. Thus, it may be concluded that the addition of 7.5 wt. % RHA completely consumes the free lime content.

### **Unrestricted Lime Content**

Free lime values of hydrated GL10, GL20, and GL30 mixtures at various curing durations. For all mixtures, free lime concentrations (FL, percent) clearly decrease as cure time increases. Calcium silicate hydrate (CSH) and calcium alumino-silicate hydrate (CASH) are constantly being formed as a result of the pozzolanic interaction between LRS and BFS (CASH). However, M10 has a lower free lime concentration than either M20 or M30. It can be shown that as the percentage of LRS rises in BFS-LRS blends, so does the amount of free lime. Monitoring the decline in FL, percent with curing time and RHA, percent enables one to infer the Pozzolanic reaction rate of RHA with the freed lime during the hydration of GBFS-LRS-RHA mixtures. Varied curing durations result in different amounts of free lime in GL30, GL30R5, GL30R7.5, and GL30R10. The active silica in RHA combines with free lime to generate CSH, which explains why free lime concentrations drop as RHA percent rises. Further, as can be shown in the TGA/DTG and XRD

tests, the increase in CSH comes at the price of FL. At later hydration ages, GL30R10's free lime concentration is quite similar to that of GL30R7.5, showing that the addition of 7.5 wt., percent RHA consumes the most FL. The combined water findings are consistent with the free lime results for all mixtures.

### **Power to withstand compression**

The compressive strength values of hydrated GL10, GL20, and GL30 mixtures at various curing durations. All hardened specimens show a definite upward trend in compressive strength values up to 90 days of hydration. Because of the constant synthesis of CSH during extended hydration of BFS/LRS pastes, the remaining unhydrated residual sections of GBFS are held together. Compressive strength values are shown to rise up to 20 wt., % (GL20) of LRS in a mix, then fall off when 30 wt., % LRS is added (GL30). This is because the activation or dissolution of GBFS improves up to 20 wt., percent LRS, which in turn increases the compressive strength; however, in the case of GL30, the excess lime content does not react with GBFS, leading to an increase in free lime content that has a detrimental effect on the mechanical properties of the hardened paste. Moreover, the GL30 paste's generated CSH seems to have a larger lime content than the CSH of the paste created from the GL20 or GL10 mixes, which both have a lower lime concentration; lime-rich CSH has weaker hydraulic capabilities than lime-poor CSH. Compressive strength values for GL30, GL30R5, GL30R7.5, and GL30R10 at various curing durations. When we compared the compressive strength values of samples with and without RHA, we found that the RHA had a negative effect on the compressive strength of samples at early hydration ages, especially at 1 day, with the compressive strength value subsequently increasing observably starting at 28 days. After 90 days of hydration, the compressive strength value of the mix containing 7.5% RHA is > 63% greater than the value of the mix containing 0% RHA. Furthermore, the increase in compressive strength of the hardened samples is very little affected by the addition of RHA (10 percent). The increased strength qualities may be attributed to the use of 7.5% RHA, which works out better than using free lime in GL30 since it produces a greater quantity of CSH.

### **X-ray diffraction analysis**

XRD patterns of GL20 at 1, 28, and 90 days of hydration. Peaks for calcium hydroxide (CH), calcium silicate hydrate (CSH), and calcium carbonate (CaCO<sub>3</sub>) may be seen in most XRD diffractograms (CC). As expected, the data demonstrate that with time, the strength of the CH peak decreases while that of the CSH and CC peaks increases. This seems to be because CH, found in LRS, perpetually activates GBFS to produce CSH. Also, during the curing process, part of the CH gets carbonated by the atmosphere. Hydrated samples of GL10, GL20, and GL30 are shown as XRD diffractograms after 90 days of cure. Results clearly demonstrate that the M30 mix, when compared to the GL20 and GL10 mixes, yields the largest quantity of residual CH. This fits well with findings from studies using free lime. Furthermore, GL20's CSH peak is more prominent than GL10's and GL30's. This proves that the mixture with 20% LRS had the maximum compressive strength compared to the others tested. Additionally, there is insufficient CH in the GL10 mixture for full GBFS- dissolution. On the other hand, the GL30 blend has a lot of high concentrations of LRS, which lead to GBFS's full disintegration in the mixture. Due to the presence of sufficient LRS in the GL20 blend, GBFS may be activated and dissolved, leading to the development of an

abundance of hydration products. The compressive strength findings correspond well with the XRD findings. XRD patterns of GL30, GL30R5, GL30R7.5, and GL30R10 hardened pastes after 90 days of hydration. Similar peaks of CSH, CH, and CC may be seen in all patterns, however their relative intensities vary. As the proportion of RHA rises from 5 to 10, the peak intensity of CH drops. All RHA-containing samples also had weaker CC and CH peak intensities compared to their RHA-free counterparts. This is because the strong pozzolanic interaction with RHA consumes free CH, resulting in the synthesis of more CSH. To add insult to injury, RHA lowers the system's odds of being carbonated by the air. With 10% RHA present, there is just a little increase in CH consumption compared to when 7.5% III was included in the same matrix (E). Spectroscopy using the Far-Infrared (FTIR)

The FTIR-spectra of GL20 after 1, 28, and 90 days after hydration. The O, Si, O bending vibration is associated with the 476 cm<sup>-1</sup> absorption band. The Si-O-Al molecule's symmetric stretching vibrations are attributed to the short absorption bands at 689 and 715 cm<sup>-1</sup>. Carbonate-containing phases are associated with the absorption bands at 876 and 1459-1492 cm<sup>-1</sup>. The 969-991 cm<sup>-1</sup> strong absorption band is due to the Si-O-T molecule's asymmetric stretching vibration (T: Si or Al). Specifically, the absorption bands at 1665 and 1661 cm<sup>-1</sup> are associated with the bending vibration of the H-OH group, while the absorption bands at 3439 and 3548 cm<sup>-1</sup> are associated with the symmetric stretching vibration of the OH group. This band weakens as curing progresses. Absorption band intensity associated to Si-O-T increases with curing time, suggesting that this is because LRS is being consumed as a consequence of GBFS activation with time, leading to the formation of additional hydration products. Additionally, this band's historically large FWHM narrows with time. With longer cure times, up to 90 days, this band moves to a lower wavenumber. The structural ordering of the silicate network is responsible for the rise in CSH crystallinity, which in turn causes the shift to low wavenumber and the reduction in FWHM [9]. The absorption band associated with air carbonation, on the other hand, grows in strength with time, indicating that carbonation of hydrated lime continues during the course of curing. The FTIR-spectra of cured GL10, GL20, and GL30 pastes after 90 days. Compared to GL20 and GL10, GL30 has stronger absorption bands at 876 and 1489-1496 cm<sup>-1</sup>, which are associated with atmospheric carbonation. This is because GL30 has a high percentage of free lime. Compared to GL10 and GL30, the FWHM of the highly resolved absorption band corresponding to Si-O-T at 967 cm<sup>-1</sup> is lower in GL20, suggesting the high structural ordering of the silicate network generated in GL20. After 90 days of hydration, the FTIR spectra of GL30, GL30R5, GL30R7.5, and GL30R10 Absorption bands at 1648-1659 cm<sup>-1</sup> and 3534-3584 cm<sup>-1</sup>, respectively, have weaker intensities due to increasing RHA concentration. These results demonstrate that RHA % correlates positively with the free lime intake. When compared to the other mixes we tested, the GL30R10 had the least carbonation (GL30, GL30R5 and GL30R7.5). The findings of the XRD study corroborate this observation (F). Comparing TG and DTG

TGA and DTG thermograms of hydrated GL20 at 1, 28, and 90 days. From the TGA graphs, we could calculate the amounts of water that were lost throughout the hydration process. It can be shown that the weight loss of GL20 at 1000°C rises from 1 to 90 days as the curing period increases.

The DTG thermograms demonstrate that the strength of the CSH peak at 50-200°C has grown as a consequence of the continual activation and dissolution of the GGBFS section, which has led to the creation and accumulation of extra quantity of CSH. Curing time decreases the endothermic peak associated with calcium hydroxide (CH) at 481°C. This is because as the LRS ages, the CH reacts with the GBFS, producing CSH and CASH, and the strength of the CSH endothermic peak rises at the cost of the CH peak. Endothermic peak associated with calcium carbonate becomes more intense with time. This is because the carbonation level of the atmosphere increases as the beer ages. Thermograms (TG and DTG) of 90-day-old hardened pastes composed of GL10, GL20, and GL30. To put it simply, GL30 paste loses more mass once it hardens than GL10 or GL20. As can be seen in DTG thermograms, the quantity of CH and CC in GL30 hardened paste is much higher than that of GL10 and GL20. When comparing GL10 and GL20, it is evident that GL20 produces more CSH, showing that the level of pozzolanic activity rises in tandem with the LRS content. On the other hand, the quantity of CSH created in GL30 is virtually the same as that formed in GL20, indicating that, the 20 percent LRS is adequate for the activation of GBFS-hydration. TG/DTG thermograms of hardened pastes at 90 days post-hydration. with a range of RHA concentrations from 0% (GL30) to 10% (GL30R10). It has been shown that, as RHA concentration rises, less CH is produced because the RHA interacts with free lime, producing more CSH. As the RHA level increases, the calcium carbonate content falls. The fact that people are using more limes without having to pay for them is evidence of this. Up to 7.5 percent, the quantity of CSH rises precipitously with RHA, before levelling out at 10 percent.

## **CONCLUSIONS**

The following is a conclusion that may be derived from this study:

1 The ratio of water and free lime that has been chemically coupled to LRS increases as LRS concentration rises. Compressive strengths of GBFS-LRS mixtures are shown to rise up to 20 percent LRS, then decline to 30 percent LRS. Three, the combined water during one day of curing is lower the greater the RHA concentration. While at higher hydration ages, the total water content rises as RHA % increases. RHA percent increases are accompanied by a fall in free lime levels throughout the board. Since the combined water and free lime contents and compressive strength values of the GBFS-LRS mix with 10% RHA are very similar to the equivalent values of the GBFS-LRS mix with 7.5 percent RHA, this suggests that the addition of 7.5 percent RHA to GBFS-LRS is the optimal addition level. Compressive strength values rise with increasing RHA concentration, while XRD, FTIR, and DSC analyses reveal that free lime content and the intensity of CH characteristic peaks decrease with RHA addition.

Five, low-cost temporary buildings may benefit from using the GBFS-LRS-RHA composite, which comprises 30% LRS and 7.5% RHA. It may be utilised in the low range of compressive strength for the preparation of non-structural parts when strength is not needed. This composite might also be utilised in mortar for brickwork, increasing its durability over time.

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