DEVELOPMENT OF SELF-SENSING CEMENT-BASED COMPOSITE USING RICE-HUSK AND COAL BLEND BIOCHAR

FINAL YEAR PROJECT UG 19

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Submitted to
NUST Institute of Civil Engineering, NICE
School of Civil and Environmental Engineering, SCEE
National University of Sciences and Technology, Islamabad, Pakistan
2023

This is to certify that the

Final Year Project Titled

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has been accepted towards the requirements for the undergraduate degree

in

CIVIL ENGINEERING

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ACKNOWLEDGEMENTS

In the name of Allah, the most Beneficent, and the most Merciful. We are exceptionally grateful to Allah to be able to complete our research project. Without Whose will, we would not have been able to culminate this huge task.

The efforts and sacrifices made by our parents and teachers during our studies and over our lives which has enabled us to be where we are today are greatly acknowledged. Special commendation of our siblings who remained a cause of constant motivation and stood by us during challenging times.

We specially acknowledge and commend the efforts that our supervisor Dr. Rao Arsalan Khushnood, Head of Department (Research) has made for us. His beneficial advice and research commitments to us were a source of motivation. Throughout the project his persistent assistance and him taking time out of his busy schedule for our guidance kept us spearheaded for success. Moreover, his professional counseling of the group in thesis writing and presentation is worthy and something that will help us forever in our practical lives.

We would like to express our gratitude to Dr. Zeeshan Ali Khan, IESE, NUST for providing expertise and permitting us to use the pyrolysis setup. We are thankful to the lab staff of mentioned departments for their assistance in performing the lab tests.

We value the assistance provided by the entire staff (Mr. Riasat, Fawad, and Ismail) of Structures Lab, NICE. Special thanks to Lab Engr. Mati Ullah Shah, Ibrahim Sarwar and Atif Mehmood in coordinating for our testing, and always guiding us during our research.

Finally, we would like to expand our appreciation towards our friends and colleagues who kept encouraging us, remained a constant emotional and physical support, and provided necessary assistance for completion of this project.

Dedicated

To

Our Families and our Supervisor Dr. Rao Arsalan Khushnood

ABSTRACT

Pakistan being an agricultural country produces annually 7.2 million tons of rice which leads to the generation of an annual 2.3 million tons of rice-husk that goes to waste and is either burned away or dumped in the rivers. And biochar is a carbonaceous substance that has the potential to be used in developing multifunctional composites. Various researches have been conducted using different biomasses to produce biochars that alter the properties of cement composites in different ways such as improving its mechanical, physical, sensing, insulation, and many more properties. In this research study, three parts rice husk with one part coal was chosen as the biomass for biochar to develop a multifunctional cement composite that has a self-sensing ability. Contemporary methods of developing self-sensing composites involve the use of carbon fibers and carbon nano particles, which are quite expensive in Pakistan, rendering structural health monitoring an expensive option to adopt. Since biochar utilizes agro-waste, it is consequently cheaper and is an environmentally friendly alternative.

The biochar was characterized using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Laser Particle Size Analysis (LPSA) and Fourier Transform Infrared (FT-IR) spectroscopy. And it investigates the multifunctional attributes of the developed cement composites from the inclusion of the biochar in cement mortar which includes the effect on the compressive and flexural strength, electrical conductivity, carbon sequestration and the self-sensing ability of the composite. And as a result it was found that with the addition of 5% biochar compressive strength improved by 9.91%, with 10% biochar it improved by 36.44% and with 15% biochar it improved by 36.26%, however with increasing biochar, a reduction in ductility was observed. Flexural strength remained relatively same for all the formulations, while electrical conductivity increased by 9.7% for samples with 10% biochar addition. And the water absorption was also found for the same formulation to be 7.55%. Additionally, a strong linear correlation between fractional change in resistivity and strain was found with the coefficient of determination value for two of the data sets being $R^2 = 0.95702$ and $R^2 = 0.96084$ respectively. Proving this composite's potential for usage in structural health monitoring applications as a multifunctional and ecofriendly material.

CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ACRONYMS

AASHTO	American Association of Highway and Transportation Officials
ASTM	American Society for Testing and Materials
EMA	Effective Medium Approximation
FCR	Fractional Change in Resistivity
FTIR	Fourier Transform Infrared Spectroscopy
LPSA	Laser Particle Size Analyzer
MS	Mild Steel
NET	Negative Emission Technology
OH	Hydroxyl Group
PAC	Portland Cement Association
PAHs	Polycyclic Aromatic Hydrocarbons
SEM	Scanning Electron Microscopy
TDAP	Trade Development Authority of Pakistan
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 General

The cement industry produces about 0.9 pounds of CO_2 for every pound of cement made (PCA, 2012) and concrete is one of the most used materials in the world for construction. The annual rice husk production in Pakistan is about 2.3 million tonnes (TDAP,2022), of which most is burnt and disposed of as waste. This contributes to a great amount of air pollution, and it is well known how most of Pakistan is engulfed in smog in the winter season.

Biochar is a carbon-rich material produced by heating organic matter in the absence of oxygen. Coal is a traditional fuel source along with rice husk which is a by-product of rice production during milling, both are quite abundantly available in Pakistan. Using biochar made from the blend of rice husk and part coal as the carbonaceous filler reinforcement material in the cement-based composites may improve their mechanical properties and reduce its overall environmental impact. Cement-based composites are widely used in the construction industry due to their versatility and durability. However, normal cement has limitations, including its high carbon footprint and as well as lack of self-sensing capabilities. Cement based sensors are used nowadays but they make use of carbon nanotubes or carbon fibers, which are both expensive and not easily available.

There is potential in the development of a self-sensing cement-based composite made using biochar from rice husk and coal. The self-sensing mechanism is developed by monitoring changes in its electrical resistivity as a load is applied to the composite and a relation between the two properties can be determined, that can further be exploited to develop sensors and utilized in sensing applications for structural health monitoring. The use of biochar will make the self-sensing composite much more affordable and make use of waste material that would otherwise cause harm to the environment. The production of concrete alone accounts for 8% of global carbon emissions (Pearce, 2021) for which the usage of biochar will help in countering that problem through the process of Carbon Sequestration.

The usage of waste materials to obtain the biochar will make use of waste utilization principle. This has an overall positive impact on the environment. In conclusion, the development of a more sustainable and durable cement-based composite with self-sensing capabilities will have significant benefits for the construction industry.

1.2 Carbon Sequestration

Carbon Sequestration is a process of absorbing & storing carbon from the atmosphere. It lowers the overall carbon footprint and helps in reducing global warming. It is a naturally occurring process that can be enhanced or achieved with technology. Through carbon sequestration we can work with the natural environment to tackle the climate crisis.

1.3 Environmental Impact

The construction sector accounts for a considerable contribution towards the carbon footprint. Biochar inclusive cementitious mortar has properties that absorbs carbon dioxide from the atmosphere. As well as the agricultural waste utilization principle helps in the carbon footprint reduction.

1.4 Problem Statement

Cement based sensors are widely used but they make use of carbon nanotubes or carbon fibers, graphene etc., which are both expensive and not readily easily available in Pakistan. They are both synthetic and do not occur naturally, so they have to be made specifically, which adds to their cost. Overall, these make structural health monitoring (S.H.M) expensive and unviable for some small structures and frequent applications.

Using biochar obtained from the blend of rice husk and coal, we can make a much cheaper and environmentally friendly cement-based sensor. That results in a more affordable and viable alternative.

The carbon sequestration properties of biochar also add to the eco friendliness of this approach as concrete production contributes to about 8% global carbon emissions and the effects of global warming are becoming more serious as the years pass.

1.5 Objectives

To investigate the mechanical & chemical properties of biochar.

To develop self-sensing capabilities of cement-based composite using biochar as a conductive filler.

To investigate the multifunctional attributes and properties of biochar infused cement composites.

To investigate the performance of biochar-infused cement composite with respect to its carbon sequestration potential.

1.6 Thesis Structure

Started with an introduction in chapter 1 and followed with an in depth literature review in chapter 2 relating to biochar, self-sensing composites, and carbon sequestration within the concrete.

Chapter 3 explains the material classification used in this project and a detailed experimental design adopted for this research study consisting of the casting procedure and sampling.

The results of the tests conducted, and their interpretation and inferences have been presented in chapter 4 for self -sensing, biochar, and CO_2 sequestration ability.

Chapter 5 discusses the conclusions drawn from this research work and suggestions for further study.

CHAPTER 2

LITERATURE REVIEW

2.1 Biochar

As defined by the Intergovernmental Panel on Climate Change (IPCC), biochar, a negative emission technology (NET) is a more stable form of carbon rich material produced by the pyrolysis of biomass [1]

The process is a greener alternative to open burning of biomass and leads to reduced formation of greenhouse gases. This carbonaceous material when added to cement improves its electrical conductivity that in turn improves the self-sensing ability of the cement composite [1].

2.1.1 Environmental Impact of Biochar

Biochar has the potential to not only become an efficient source of bio waste utilization, but also help achieve carbon neutrality by becoming an agent for carbon sequestration in structures. Resulting in an overall reduction in the carbon footprint of the construction industry.

Biochar is a green material that possesses a high specific surface area and a notable attraction towards nonpolar substances. It is proficient in capturing and storing carbon dioxide via pore adsorption. The capacity of CO_2 adsorption differs among several types of biochar, and the adsorption ability can be influenced by the production conditions of biochar material**s**, such as pyrolysis temperature and time [2]

In natural environments, carbon dioxide can penetrate the pore solution of cementitious composites, leading to carbonation that produces carbonate. Subsequently, carbonate ions react with calcium hydroxide in the composites to form stable calcium carbonate. This process enables carbon sequestration by absorbing carbon dioxide from the surroundings and converting it into a stable form of calcium carbonate within the cementitious composites. Passive carbonation of cement substrates in the natural environment is slow due to low atmospheric CO_2 concentration, resulting in inefficient carbon sequestration.

Consequently, several studies have aimed to enhance carbon sequestration efficiency. Including the addition of biochar into cementitious composites [2]

Adding biochar to cementitious composites reduces CO_2 emissions, captures CO_2 through carbonation, and permanently immobilizes CO_2 by forming carbonation products. Consequently, biochar concrete is an effective means of achieving carbon sequestration. And [3] reports that the carbonation and CO_2 uptake of the composite increased by 5 %-7% with the inclusion of biochar in the composite.

Based on estimations, the conversion of agricultural waste to biochar at a rate of 373 million tonnes per year has the potential to sequester roughly 500 million tonnes of carbon dioxide annually. This amount is equivalent to approximately 1.5% of the global CO_2 emissions released each year [4,5].

2.1.2 Multifunctionality of Biochar

The inclusion of biochar as an additive or replacement in cementitious composites results in an improvement in crucial properties such as compressive, tensile, and flexural strength, increased fracture energy and ductility, water tightness and reduction in sorptivity, insulating behavior, resistance to degradation in corrosive environments and electromagnetic interference shielding.

Approximately 20% increase in compressive and tensile strengths, 20-26% increase in flexural strength, up to 41% reduction in water absorption, 8-11% stronger resistance to corrosive degradation, a stated increase in shielding of 335%, 214%, 122% and 76% was achieved at the four specified frequencies(0.94 GHz, 1.56 GHz, 2.46 GHz, and 10 GHz), with a reported approximate increase of maximum 150% in fracture energy is also observed by inclusion of biochar [6–9].

2.1.3 Use of Biochar as an Electrically Conductive Filler in Composites

Having approximately up to 80% carbon content, biochar results in increased electrical conductivity of cement composites. Compared to carbon fibers and nanotubes which are used in lower dosages of up to 1% by weight, biochar is used in a higher dosage of up to 15% by weight to ensure similar increase in electrical conductivity. An approximate increase of 33.6% in conductivity of cement composites is reported [10]. Another study reports a decreased electrical resistivity comparable to that of the addition of graphite particles, by the inclusion of 15% by weight biochar [11].

Additionally, biochar costs less even with higher dosages, as compared to carbon fibers and nanotubes [10]

2.1.4 Biochar Production

To ensure that a biomass is appropriate for producing biochar, it is crucial to examine and analyze its elemental composition, surface functional groups, thermal stability, and structure. Thus, characterizing and evaluating the biomass is essential to determine its suitability for the intended purpose [7]. The moisture content present in biomass is a crucial parameter that influences the biochar formation process. A higher moisture content necessitates a greater amount of energy and elevated temperatures to accomplish pyrolysis. Consequently, the use of biomass with reduced moisture content is more advantageous because it requires less energy and heat, which improves the economic feasibility of the process [12].

Research reports that prior studies utilized a temperature of 500 °C to generate biochar, which resulted in enhanced compressive and flexural strength. These findings could guide subsequent investigations on biochar-based concrete or cement composites, where biochar produced at temperatures close to 500 °C could be used. Raising the temperature further to 700 °C or 800 °C would demand more energy during batch production, hence less practical [7].

For serving the purpose of self-sensing, a biochar with a relatively higher carbon content is required. Rice husk being rich in silica produces biochar with carbon contents of approximately 40-45% at pyrolysis temperature 500°C [13,14]. The temperatures up to 500°C are quite suitable for feedstock containing rice husk as further increase in temperature is reported to result in lesser solid biochar yield and more conversion into biogas and bio-oil(Zhang, Liu, and Liu, 2015). And the reported yield from the feedstock is approximately 35% (Shackley Simon and Sohi, 2013). Hence a requiring a need to make minor changes to the feedstock to improve the carbon content, yield, and keeping the overall required pyrolysis temperature at 500°C.

2.1.5 Biochar Characterization Based on Pyrolysis Temperature and Time

Pyrolysis conducted for biochar can be characterized into two main types namely: fast(flash) pyrolysis and slow pyrolysis, hence the biochar produced from the respective pyrolysis differs in properties that eventually affect the properties of the cementitious composites in which it is included [17]

Fast pyrolysis refers to heating the biomass rapidly up to the required temperature to mainly ease the production of liquid oil from the biomass. The biochar resulting from flash pyrolysis is comparably low in fixed carbon contents and has a relatively lesser yield of solid mass from the biomass. Compared to slow pyrolysis in which the heating rate is low and residence time at required temperature is higher, which leads to the formation of higher carbon content solid mass of greater yield [17]

2.2 Self-Sensing Cement Based Composites

Self-sensing/piezoresistive cement-based composites are cement with added electrically conductive fillers that due to its high electrical conductivity allow real time observing of stress-strains in the structure by measuring the changes in its electrical resistivity. Ordinary Portland Cement has generally low electrical conductivity, hence the addition of a conductive filler such as carbonaceous nano/micro particles improve its sensing properties through processes such as conduction through contact (percolation) or tunnelling effect [18].

It is reported that self-sensing composites have precedence over traditional sensors due to their low authenticity, low durability, high construction requirement and poor compatibility which limit their usage in large structures [19,20].

2.3 Characterization of Self-Sensing Composites Based on Different Fillers

A study pertaining to use of graphene nano platelets as conductive fillers establish a strain sensing ability for various dosages, and a relation between fractional change in resistivity and strain is established [19] Several studies have been conducted reporting the efficiency of carbon fibers as conductive fillers. Having excellent and ideal properties for sensing applications, even smaller dosages (1% by weight) provide excellent electrical conductivity that improves the sensing capability of the composite [21,22]. Mechanical property improvements besides electrical conductivity enhancement has been reported by [23] The only major limitation to its usage is its higher costs as compared to other fillers [24].

Other filler materials typically explored for self-sensing capability of cement composites include carbon nanotubes, carbon black, graphene, and even mixed usage of the fillers. Several research conducted have also explored the effect of filler dosage, filler dispersion, curing age moisture content, and temperatures [24].

2.4 Conduction Theories

To further explain the phenomena of conduction within the cement composite, many theories and models have been proposed which include percolation theory (it considers the conductivities of the fillers and the matrix, as well as the volume fraction of the fillers) [25], tunnel effect theory (some of the parameters from this model include tunneling current density, barrier limit, barrier height, and gap width between fillers) [26], electric field emission theory (the parameters for this theory include current density, field intensity, and tunnel frequency) [27], and effective medium theory (Certain variables of this model are volume fraction, and critical volume fraction of high conductivity phases, and conductivities of high, and low phases and the composite matrix) [28].

2.5 Theoretical Calculation for Percolation Threshold

Different models have been developed to describe the percolation phenomena, and one such is Bruggeman's effective medium approximation (EMA) model. It is used to estimate the minimum volume fraction of conductive filler needed for a composite material to become conductive. It assumes that the filler is randomly distributed in the material and that the electrical conductivity of the composite is isotropic. The estimated volume fraction depends on the electrical conductivity of the filler and the matrix, as well as the geometry of the sample. The Bruggeman model assumes randomly oriented particles. If the filler has a specific aspect ratio, a different model may need to be used [29].

The model however is still ineffective at calculating the percolation threshold effectively as it does not consider several factors that affect the percolation. And the other models are effective at calculating percolation threshold for conductive fillers which have a greater this aspect ratio. But the biochar particles are quite spherical and irregular in shape, so the theories don’t provide accurate results. Hence the formulations were devised keeping in view the existing literature.

Chapter 3

MATERIAL AND EXPERIMENTAL METHODOLOGY

3.1 Materials

This chapter explains the materials used for the development of self-sensing mortar, the properties they exhibited, and the processes they went through. All the standards and protocols were maintained during the acquisition of the materials as described in the sub-sections below.

3.1.1 Biochar

The high carbon residue that is obtained by a process known as pyrolysis is called biochar. The use of biochar as a filler material enhances multiple material properties of the cement such as conductivity, strength, water absorption, etc [30]. Depending on the parameters it has detailed properties enhancement however, in this project its effect on conductive properties and mechanical properties are under study [31].

The process of obtaining biochar is explained below:

3.1.1.1 Rice Husk and Coal Blend

The rice husk used was from rice farms in Tando Muhammad Khan, Sindh and the coal was obtained from Islamabad.

The aim of the project overlapped with the SDG goal which means our chosen biochar materials must be environmentally sustainable. For that, we chose Rice Husk blended with coal (3:1) as raw materials.

Pakistan is an agricultural country and rice is a major crop of this country. According to an estimate, annually 1828 tons of rice husk is wasted as either burned or disposed of to no use. The idea of using the waste rice husk to produce biochar which would then be used as conductivity enhancer for the cementitious material.

As our main aim is using biochar that is high in carbon content, rice husk which is usually high in silica was blended with coal to improve its carbon content so optimum content of biochar may be used in our product. The materials obtained were then pyrolyzed to obtain the required product i.e., biochar.

3.1.1.2 Pyrolysis

Pyrolysis is the controlled heating of the substance in the absence of oxygen by purging it with the presence of an inert gas at a high temperature to remove any residual bi-products and obtained pure carbon. The temperature ranges from 400-700° C depending on the material pyrolyzed. [32]

The required temperature to pyrolyze rice husk and coal blend is 400-500° C which separates the volatile matter and carbon is left behind. For this process, rich husk was well blended into fine particles and coal was ground into fine particles and then sieved from sieve # 100 (0.149 mm). The obtained materials were mixed in a 3:1 with three parts being rice husk and one part being coal.

Blended rice husk and coal were pyrolyzed in a heating chamber in the presence of nitrogen acting as the inert gas. The whole assembly was heated up to 550° C with a ramp of 5° C per minute and a hold of about 30 minutes at 550° C. During the whole process, volatile materials are evaporated in the form of gas and vapors, which can be condensed to form oil a biproduct of pyrolysis, leaving behind pure carbonaceous residue.

Pyrolysis of this blend in the presence of nitrogen as an inert gas gave about 50 % yield at 550 ° C. Yield of the process depends on the standards followed and the quality of the instrument and the obtained yield being in accordance with the established research speaks to the topnotch quality of the process.

3.1.1.3 Biochar – Pulverized

The pyrolyzed product obtained is rich in carbon content. The obtained biochar needed to be pulverized and sieved to be used as a cement filler.

Biochar was pulverized in mortar and pestle in accordance with the ASTM D420 and AASHTO T87, for mechanical and physical analysis of the selected material. After pulverizing the biochar, it was sieved through sieve # 200 (75 µm). It is to be noted the biochar used was completely dried either through the pyrolysis process or by oven drying to prevent any moisture penetration within.

A finely powdered biochar was obtained which was used for the casting of the project.

3.1.1.4 Characterization of Biochar

The microstructural analysis of biochar was done by conducting SEM, LPSA, FTIR and XRD. The Scanning Electron Microscopy (SEM) is used to analyze the microstructure and surface topography of the biochar. Laser Particle Size Analysis (LPSA) measures the particle distribution of biochar. The Fourier Transform Infrared Spectroscopy (FTIR) is used to identify and characterize the chemical composition and X-Ray Diffraction (XRD) to examine the crystalline structure and phase composition of biochar.

The properties imparted in the cement composites by biochar are mainly dependent on the physiochemical properties of its constituent biomass including the morphology of produced biochar, its particle size, and chemical composition.

3.1.1.4.1 Scanning Electron Microscopy

The scanning electron microscopy (SEM) is the imaging technique used to observe the surface morphology and ultrastructure of materials at high magnification and for biochar, SEM is used to visualize the physical and chemical features of the material, such as the pore structure, particle size, and surface texture. The SEM images were obtained using

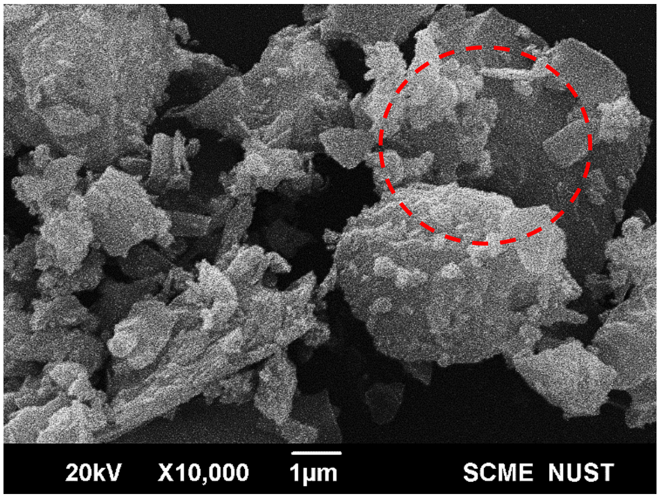


Figure 1. SEM image of biochar at x10000 magnification

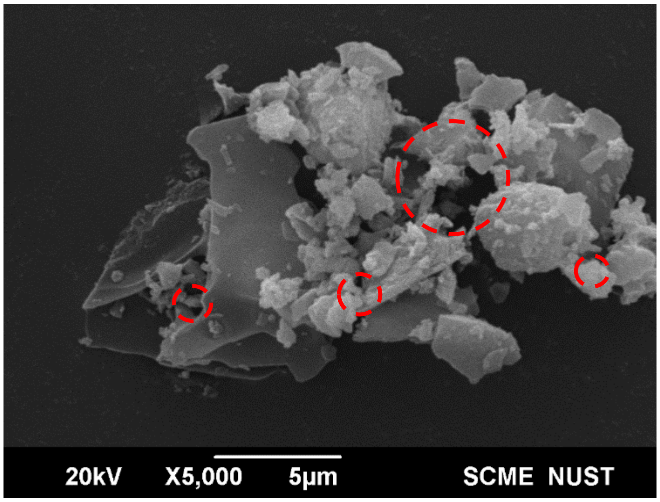


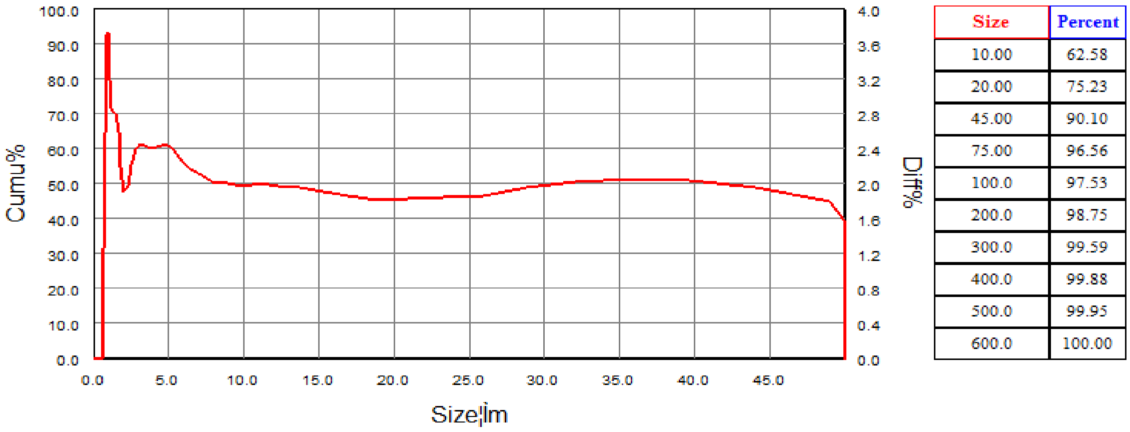
Figure 2. SEM image of biochar at x5000 magnification

JEOL Japan JSM-6490A SEM, operated in high vacuum mode at an accelerating voltage of 20kV.

The images show that rice-husk particles are diffused on the coal particles such that their interfacial interactions suggest both the particles have physically bonded. The biochar exhibits flakey, and irregular surface morphology, with evenly distributed particles of rice husk and coal. The flakiness can be attributed to the particles of biochar while a crystalline formation can also be observed indicating a combined solid residue of rice husk and coal blend pyrolysis. Some micro pores can be observed in **Error! Reference source not found.** that are considered important as they can play a role as adsorption sites for CO_2 as well as sites to store water that promotes secondary hydration of the composite. And the rough irregular surface makes it suitable as an interlocking agent.

3.1.1.4.2 Laser Particle Size Analysis

This technique is used to determine the size distribution of particles in a sample. It provides information about the particle size distribution, which is an important factor that influences the physical and chemical properties of biochar. The particle sizes were observed using Laser particle size analyzer BT-9300ST with a particle size observing range of 0.1µm to 1000µm. In this study the values for D10, D75, and D90 were found to be D10 = 0.982 µm, D75 = 19.73 µm, and D90 = 44.76 µm.



Size	Percent
10.00	62.58
20.00	75.23
45.00	90.10
75.00	96.56
100.0	97.53
200.0	98.75
300.0	99.59
400.0	99.88
500.0	99.95
600.0	100.00

Figure 3. Particle size distribution of Biochar particles

3.1.2 Cement

The cement used for casting throughout the project was Bestway grade 53. The properties of the cement are listed below:

Table no. 1: Properties of Cement

Mean Size of Particles (μm)	**9.5**
Initial Setting Time (min)	>30
Final Setting Time (min)	>600
Specific Gravity	3.14
Standard Consistency (%)	30-35
Specific Density (kg/m^3)	2200

3.1.3 Sand

Lawrencepur sand was used as a filler throughout the project. The fineness modulus of the sand was 2.55. Mean particle size, specific gravity, and water absorptions are listed below:

Table no. 2: The Properties of Sand

Fineness modulus	**2.55**
Mean Particle Size (mm)	0.62
Specific Gravity	2.67
Water Absorption (%)	1.6

The sand properties are all according to the ASTM standards. Consistency was maintained throughout the testing for more uniform properties and results.

3.1.4 Superplasticizer

The superplasticizer used was Sika Plastiment – 100, density of the chemical is 1160 kg/m^3. The selected dosage of superplasticizer was 0.75 % by wt. of the cement. Protocol and instructions according to ASTM C494 standards were followed, the proper dosage was added, and mixing was done accordingly.

The use of a superplasticizer is to achieve the required conditions without changing the mix design ratios. Biochar has a water sequestering property that means when added into the cement mix it tends to absorb the water hence making the mix dry and nonworkable. The use of the superplasticizer resulted in increased workability and strength without the addition of water.

3.1.5 Wire mesh

In our testing procedures, the core installed was MS Steel wire mesh with a 0.5” opening. Four wire meshes were installed per sample to measure its resistivity using four probe method. Installation and testing were done following the ASTM C1876.

According to ASTM C1876 the bulk electrical resistivity of cored mortar samples can be measured by applying an electrical field. Resistivity is the amount of resistance faced by the ions when moving across the applied field through the installed cores i.e., the wire mesh.

3.2 Experimental Methodology

To conduct experimentation on our concept we devised an experimental methodology to perform tests on 70 mortar cubes. In the first phase, casting was done to find the optimum biochar percentage required by performing electrical conductivity tests using four-probe method and compressive and flexural strength tests to investigate the mechanical properties of the mortar. Compressive tests were performed at 7, 14 and 28 days of curing to investigate the changes in initial and final compressive strengths of the mortar. Flexure and electrical conductivity tests were performed on 28 days of curing. For phase II, 20 samples with optimum biochar content were cast to investigate the mortars self-sensing abilities, water absorption, carbon sequestration and carbon dioxide inhaling properties. All tests were performed on prisms of 40 mm x 40 mm x 160 mm at 28 days of curing.

Table No. 3: The Formulations of Composites

	Test	Samples	Formulations	Quantity	Total
Phase I	Compressive Testing	Mortar Cubes (2×2×2 in)	4	9	36
	Flexural Testing	Mortar Prism (1.58×1.58×6.3 in)	4	3	12
	Electrical Conductivity	Mortar Prism (1.58×1.58×6.3 in)	4	3	12
Phase II	Stress-Strain Sensing	Mortar Prism (1.58×1.58×6.3 in)	1	18	18
	Water Absorption	Mortar Prism (1.58×1.58×6.3 in)	1	4	4
	Carbon Sequestering	Mortar Cubes (6x6x6 in)	1	8	8
		Total			90

The following contains the details of the experimental methodology that was opted to proceed with the research.

3.2.1 Mix Design

In order to formulate the mix design, literature was referred but no substantial information could be extracted. Therefore, keeping in view the properties of material and atmospheric condition e.g. humidity, temperature a mix design was devised. The cement to sand ratio was kept, 1 to 2.75 and water to cement ratio, 0.4 for the four formulations devised and is selected after a series of trial casting. The superplasticizer is 0.75% by weight of cement and kept constant for all samples.

To find the optimum percentage of biochar a total of four cement composite formulations were developed including three formulations with different concentrations of biochar and one control sample for comparison. The three biochar concentrations are chosen based on the existing research corresponding to biochar inclusion.

Therefore the percentages of biochar used to find the optimum amount through experimentations are 5, 10 and 15% and control sample with 0% biochar for comparison.

Table No. 4: The Mix Proportions

Mix ID	**Mix Descriptions**	**Mix Proportions (kg/m^3)**				
		Cement	**Sand**	**Biochar**	**Water**	**Superplasticizer**
PC (Control)	Plain mortar sample	586.62	1613.45	-	234.71	2.93
BC5	Mortar sample with 5% biochar	586.62	1613.45	29.33	234.71	2.93
BC10	Mortar sample with 10% biochar	586.62	1613.45	58.59	234.71	2.93
BC15	Mortar sample with 15% biochar	586.62	1613.45	87.99	234.71	2.93

3.2.2 Mixing Regime

The mix design was prepared using Hobart mixer in accordance with ASTM C-305. Biochar was sonicated in water using a sonicator for effective dispersion. Sonication was done at 25°C for 15 minutes. Cement and sand were weighted according to the formulation and dry mixed for 2 minutes. The sonicated solution along with the superplasticizer were added to the dry mix and further mixed for 5 minutes. The mix was poured in molds in 3 parts while tamping it 25 times with a steel rod each time for proper compaction.

3.2.3 Curing

After casting of the specimens demolding was done after 24 hrs. After that, the samples were cured at room temperature (25° C) for 7, 14 and 28 days. The testing was performed on the samples at 7, 14 and 28 days respectively. For electrical conductivity, samples were cured for 28 days, air dried and tested for the most accurate results.

3.3 Testing Methodology

3.3.1 Compressive Strength Testing

The compressive strength test is performed on 2x2x2 in. cubes of all 4 formulations (PC, BC5, BC10 and BC15) as per guidelines of ASTM C109. A total of 9 samples were cast for each formulation and were cured for 7, 14 and 28 days. Universal Testing Machine (UTM) is used to perform the test and stress strain graphs were obtained to analyze the results.

3.3.2 Center Point Loading Flexure Testing

According to ASTM C348 specifications, the center point loading flexure test is carried out on 1.58×1.58×6.3 in. prisms. Three samples for each of the formulations were cast and tested at 28 days of curing using UTM. The three-point bending test is performed and the mean value of the three samples is used for flexure strength calculations for each formulation.

3.3.3 Four Probe Electrical Resistivity Testing

For electrical resistivity test, four wire meshes, which will act as electrodes, are imbedded at equal distances in each of the sample cubes during the casting process. The four probe Wenner array setup is made in which direct current (DC) is passed through the outer electrodes while measuring the voltage difference between the inner probes. The measured potential difference is then used to calculate the electrical resistivity of the cement-composite samples.

3.3.4 Water Absorption Testing

Water absorption test is performed according to ASTM C642 to evaluate the quality and durability of the samples by providing information on the porosity and permeability of the cement composite. For the test, the cast samples are oven dried at 110 ± 5°C for at least 24 hours and until the mass change is less than 0.5%. Then the samples are immersed in water at approximately 25°C for not less than 48 hours and until the mass change is less than 0.5%. The water absorption of the samples are determined by using the following equations:

Absorption after immersion, % = $\frac{(W_{sat} - W_{dry})}{W_{sat}} \times 100$

where W_{sat} is the saturated weight in air (kg); W_{dry} is the oven-dried weight (kg) of the cement composites.

3.3.5 Self-Sensing Ability Testing

To analyze the self-sensing properties of samples, prisms are casted with 4 wire meshes imbedded at equal distance in each. A setup is made in which uniaxial load is applied through UTM at a constant loading rate while measuring strain through strain gauge and resistivity simultaneously through four probe Wenner array setup.

The cement-composite's self-sensing ability is determined by establishing a relationship between the fractional change in resistance and strain through regression analysis. To determine the sensitivity of the sensing ability of the samples, the gauge factor is utilized, which represents the fractional change in resistance per unit strain. The fractional change in resistivity (FCR) is calculated using the following formula:

$$FCR = \frac{\Delta\rho}{\rho_o}$$

Where, $\Delta\rho$, and ρ_o are change in resistivity and initial resistivity respectively.

3.3.6 Carbon dioxide Monitoring Test

CO2 monitoring test is performed to measure the affect of biochar on the carbon sequestering capability of the cement-composites. This test is carried out on 6x6x6 in. cube samples cured for 28 days. The samples are placed in sealed climate-controlled chamber with known initial CO2 and EXTECH sensor is used to measure CO2 with time.

Chapter 4

EXPERIMENTAL TESTS AND RESULTS

4.1 Elemental and Crystallinity Analysis, and Chemical Composition of Biochar

4.1.1 Fourier Transform Infrared Spectroscopy

It is a technique used to identify and analyze the chemical composition of a material. It reveals information about the functional groups and chemical bonds present in the material. Through FTIR spectrum of biochar, identification of chemical functional groups provide insight into the chemical properties and reactivity of biochar. It is crucial to ascertain because the feedstock used for this study's biochar is a unique blend, and hence information regarding its stability is found using the FTIR Spectrum.

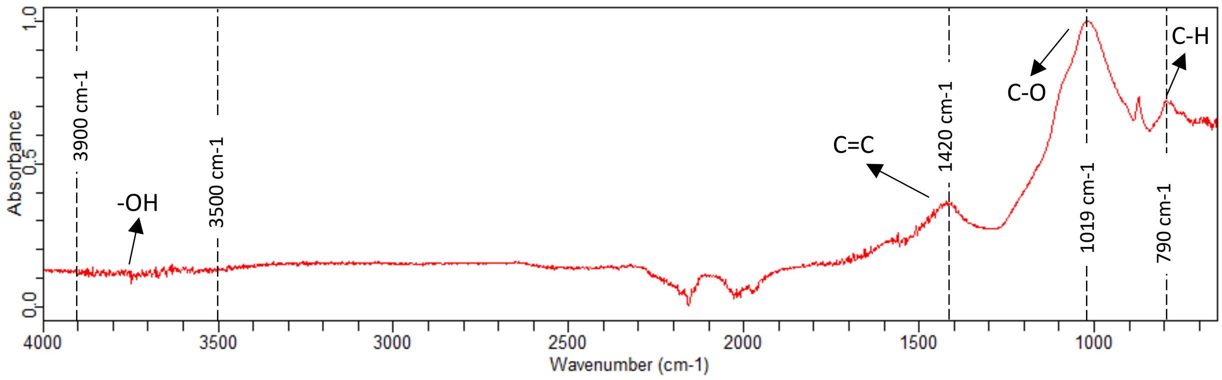


Figure 4. FTIR spectrum of rice-husk and coal (3:1) blend biochar.

The presence of minor peaks in the 3500 cm-1 to 3900 cm-1 range with mean absorbance of 12% is consistent with the presence of hydroxyl (-OH) groups, which are commonly found in lignocellulosic biomass such as rice husk. The coal component of the sample likely contains a small amount of moisture, which could also contribute to the presence of the -OH peaks. The depressions of 0% absorbance in the 2000 cm-1 to 2300 cm-1 range suggest the absence of triple bonds (C≡C) or nitriles (C≡N), which are not typically found in lignocellulosic biomass or coal. The steady peak at 1400 cm-1 with approximately 40% absorbance is consistent with the C=C stretching vibrations in an unsaturated compound, such as an alkene or an aromatic compound. This peak could be attributed to the presence of polycyclic aromatic hydrocarbons (PAHs) that are commonly found in coal. The huge peak at 1000 cm-1 with 100% absorbance is likely due to the presence of a C-O bond, which could be attributed to the presence of silica. Rice husk is known to contain high amounts of silica, which is typically present as amorphous silica or crystalline silica. The lesser peaks in the 700 cm-1 to 900 cm-1 range with a mean absorbance of 70% could be due to C-H bending vibrations in an alkane or an aromatic compound. These peaks could be attributed to the presence of both rice husk and coal components.

To improve the carbon content of the biochar, coal was introduced. It can be identified from the spectrum that silica is in higher amounts, for which the introduced coal has provided adequate amounts of carbon to raise the overall carbon content of the biochar. Making it suitable for self-sensing applications.

4.1.2 X-Ray Diffraction Analysis

For biochar analysis, XRD is used as a powerful technique to determine the crystalline phases and mineral content of the samples. The mineral content and crystalline structure of biochar can affect its reactivity with cement, which can in turn impact the strength and durability of the resulting composite material.

An assessment of crystallinity is done by comparing the peaks such as narrow high intensity peaks around $2\theta = 26°$ correspond to that of crystalline quartz (SiO_2) and similarly the broad peaks correspond to that of amorphous silica presence around $2\theta = 22°$, this is

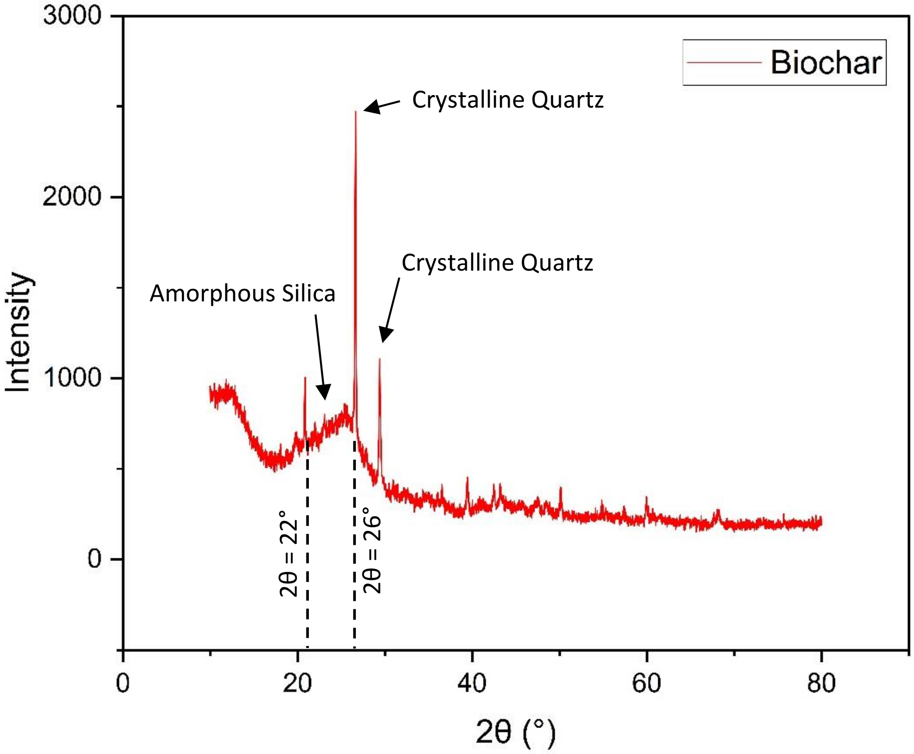


Figure 5. X-Ray Diffraction Spectrum of Biochar

based on comparison with similar peaks reported by [33]. The amorphous phase of the biochar ensures the pozzolanic action of the silica present in the biochar while the crystalline phase remains inert.

4.2 Electrical Conductivity Test for Biochar

The conductivity for biochar was measured using a conductivity meter. Standard conductivity was measured of deionized water sample, which was used as a baseline conductivity. Then 5g of biochar was dispersed in 50ml of the water sample and again the conductivity was measured. This value was subtracted from the initial value to get the conductivity of biochar. This method has been referred in various researches including (Zimmerman, 2010).

Rice husk biochar conductivity was found to be 21 **μS/m.** This corresponds to optimum value for the particle to be able to cross the percolation threshold to be used as a filler in cement composites compared to existing researches. For better understanding a comparison of electrical conductivity of different carbon based particle has been provided:

Material	Conductivity (µS/m)
Rice Husk Biochar	21
Carbon Black	17.5
Corncob Biochar	8.54
Thermoplastic Biochar	11.9

4.3 Characterization of Biochar Infused Cement Composite

4.3.1 Morphology

4.3.1.1 Scanning Electron Microscopy

Scanning electron microscopy is employed as a high-magnification visualization technique, that allows us to observe the surface morphology and internal structure. Provides sufficient insight into the pore structure and distribution as well as biochar-cement interaction and bonding. Which explains any enhancement of property that is observed in a biochar infused cement composite.

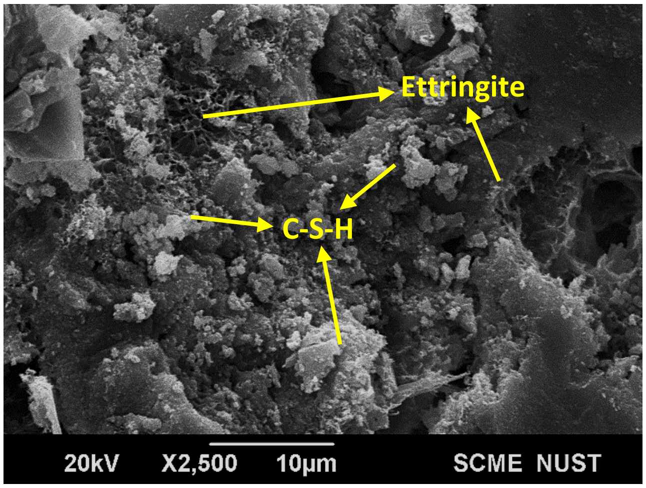


Figure 7. SEM image of control sample

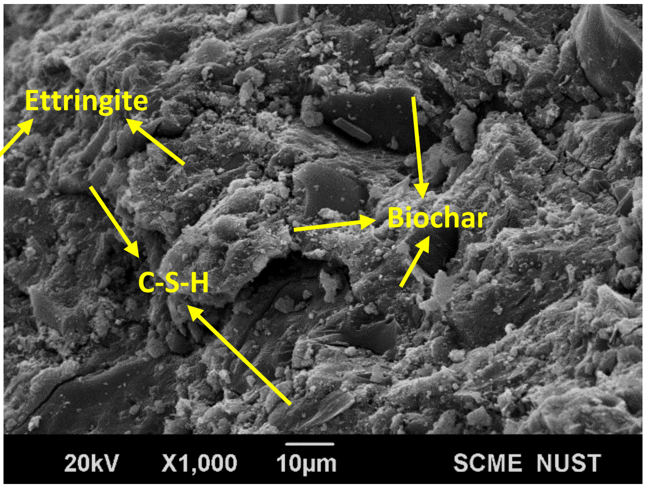


Figure 6. SEM image of cement composite sample with 15% biochar content

The typical micrograph of control sample in Figure 7. shows a dense cement matrix with some irregularities and voids. A clear formation of ettringite and calcium hydroxide is visible. While in Figure 6. a visible presence of solid biochar particles is there around which cement matrix has densely formed. The biochar particles pack the matrix densely and a very significant reduction in visible voids can be observed. Additionally, the ettringite and calcium hydroxide formation around the biochar particles is similar to typical cement matrix indicating the relatively inert behavior of biochar. Also the close formation of hydration products surrounding the biochar particles may indicate its internal curing due to the absorbed moisture by biochar.

4.4 Physical Characteristics and Properties Analysis

4.4.1 Compressive Strength Testing

To observe the effect of biochar addition on compressive strength of cement composite, four recipes were made with varying biochar percentages. A control sample was cast with 0% biochar, while 3 recipes of 5%, 10% and 15% biochar addition were cast and tested. 6 samples of each recipe were cast and tested at 7 days, 14, days and 28 days of curing.

The results show that the addition of biochar has caused an increase in the compressive strength. With 5% addition resulting in an increase by 9.91% at 28 days of curing age, 10% resulting in an improvement by 36.44% at 28 days of curing age, and 15% addition resulting in an increase by 36.26% at 28 days of curing. Although significant improvement in strengths is observed, it is pertinent to note that by increasing the amount of biochar from 10% the strength improvement is no different than that of 10% increase, so it can be inferred that biochar after a certain dosage dose not further cause any improvement in strength.

Figure 8. Cement composite samples with 5%, 10%, and 15% biochar content for compressive strength testing

Figure 7. Cement composite sample in the Universal Testing Machine (UTM).

the improvement in compressive strengths of the composite due to increasing addition of biochar. And at the same time it can be observed with the increasing strength, there’s a reduction in ductility, which is due to the porous nature of biochar, that in higher quantities creates a network of interconnected pores, reduces the overall bulk density for higher strengths can potentially reduce the self-weight and lead to reduced costs.

The increase in the strengths can be explained by the phenomena of the reservoir effect of biochar, as it absorbs the mixing water initially and densifies the surrounding mix, and later that absorbed water is diffused back, causing secondary hydration, and improving strengths

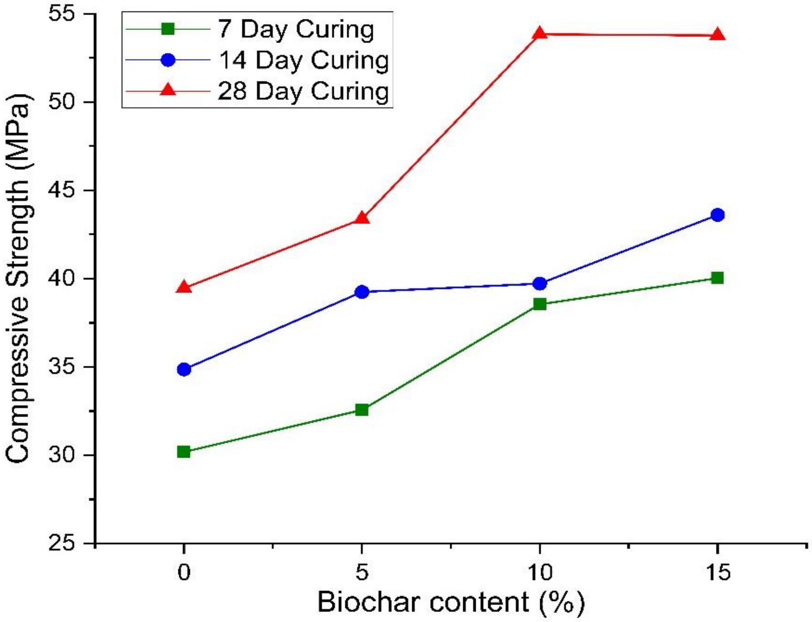


Figure 10. Compressive strength of 0%, 5%, 10% and 15% biochar cement composite samples at the curing age of 7, 14, and 28 days.

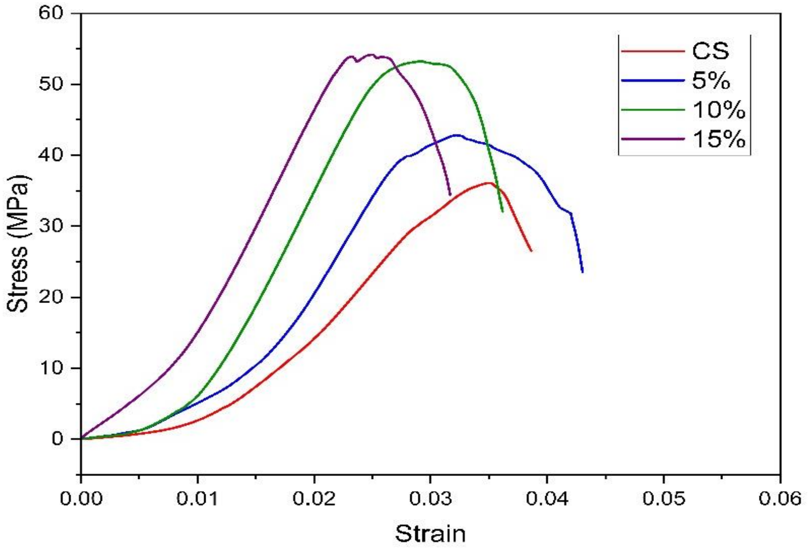


Figure 9. Stress strain curves of cement composite samples with 0%, 5%, 10% and 15% biochar content.

over time. This is consistent with existing findings (Gupta & Kua, 2018; Muthukrishnan et al., 2019).

4.4.2 Center Point Loading Flexural Strength Testing

Improvement in flexural strength is an important parameter since cement composite is weak in flexure. Our tests performed showed a very slight improvement in the composite's flexural strength with the addition of biochar in the composites. For the test, three 40 mm x 40 mm x 160 mm prisms were cast for each biochar dosage with the additions of biochar being 0%, 5% 10% and 15% at the water curing age of 28 days.

It was observed that the addition of biochar at different dosages had little to no effect on the flexural strength. A slight increase of about 5.04% strength was observed in the 10% biochar sample in comparison with the control sample, while both 5% and 15% had no difference in strength as compared to the control sample. The slight increase can be attributed to the filler effect of the biochar particles. And the lack of Improvement in the strength overall can be due to the biochar particles being round, irregular in shape, had they been irregular and elongated, then the effect observed would have been different, as observed in previously conducted researches on biochar made of other materials.

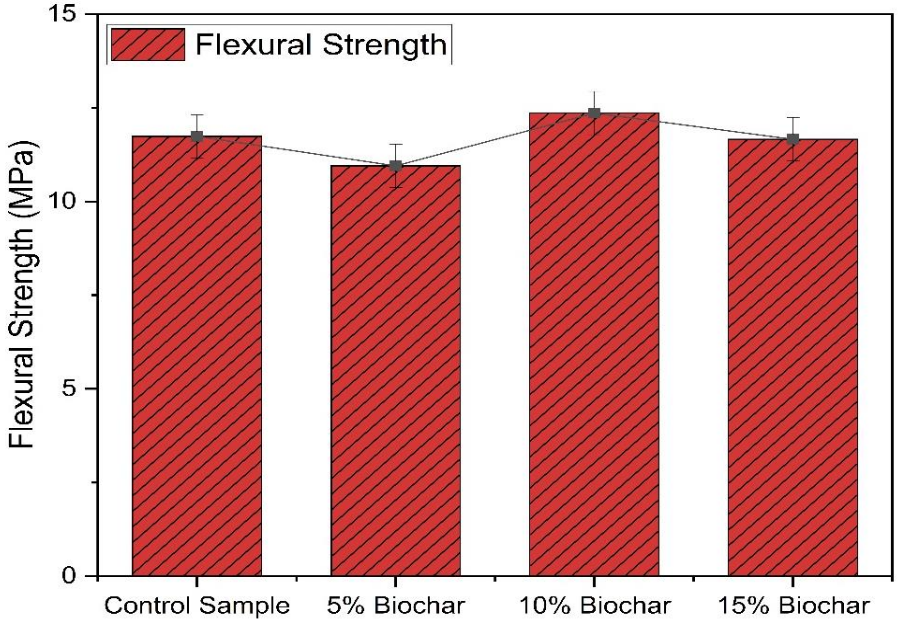


Figure 11. Flexural strength of 0%, 5%, 10% and 15% biochar cement composite samples at the curing age of 28 days.

4.4.3 Four Probe Electrical Resistivity Testing

The effect of different percentages of biochar content inclusion was tested using the four probe Wenner Array setup, with four wire mesh electrodes placed at equal distances in the composite samples cubes. It was observed that with increasing biochar contents, the electrical resistivity decreased consequently the electrical conductivity improved, which is a reciprocal of resistivity. The conductivity was observed to be the highest for 10% biochar content among all the dosages. This test was conducted to determine the optimum biochar content for the self-sensing phase of the project.

The testing resulted in the following resistivities, and the subsequent conductivities of the samples:

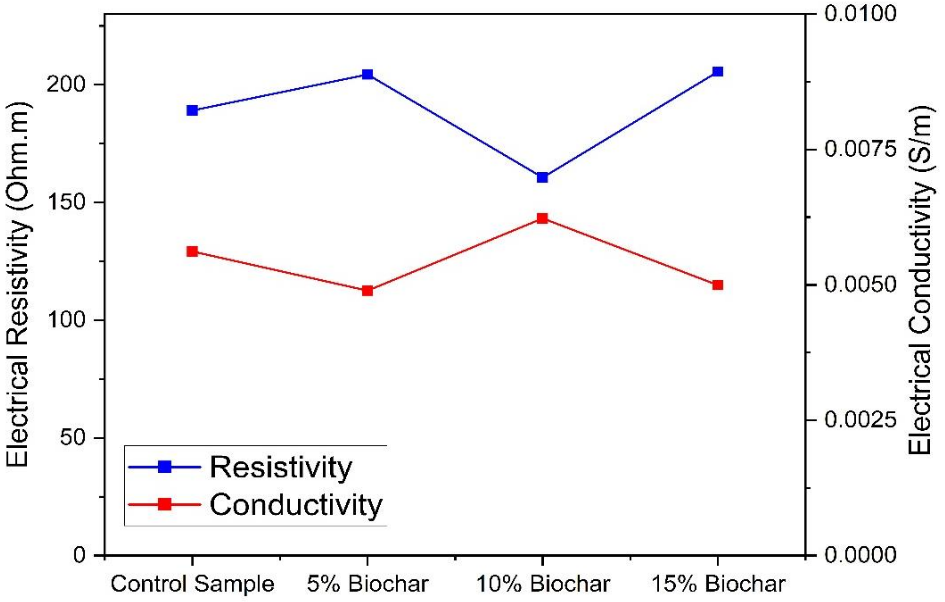


Figure 12 Electrical Resistivity and conductivity of cement composite samples with different biochar contents at the curing age of 28 days.

4.4.4 Water Absorption Testing

This test is used to determine the amount of water that the cement composite absorbs in a specific period of time. The water absorption test is an important indicator of the quality and durability of cement composite. A higher water absorption rate indicates that the composite is more porous and can absorb more moisture, which can lead to problems such as cracking, erosion, and the growth of mold and other fungi. This can ultimately lead to a reduction in the overall durability and performance of the composite.

Similarly, a lower water absorption rate indicates that the mortar is less porous and can better resist the damaging effects of moisture. This can help to ensure that the composite remains strong and stable over time against weathering and deterioration.

The water absorption of the samples are determined by using the following equations:

Absorption after immersion, % = $\frac{(W_{sat}- W_{dry})}{W_{sat}} \times 100$

Absorption after immersion% = $\mathbf{7.55\%}$

where W_{sat} is the saturated weight in air (kg) and W_{dry} is the oven-dried weight (kg) of the cement composites.

The absorption percentage indicates a compact and packed matrix and a durable composition, and this absorption is sufficiently lesser than that of a good normal mortar as reported in studies. [35]

4.5 Self-Sensing Ability Analysis

4.5.1 Self-Sensing Ability Testing

The sensing capability of the cement composite is assessed using the properties such as applied stress, strain, and electrical resistance of the sample. In theory and existing researches, the test is performed by applying uniaxial load on the sample while measuring the strain and changes in resistivity. The strain sensing property of the composite is determined by developing a relation between the fractional change in resistance and strain using regression analysis, and gauge factor is used to determine the sensitivity of the sensing ability of the composite which is the fractional change in resistance per unit strain.

The fractional change in resistivity (FCR) is defined as:

$$FCR = \frac{\Delta\rho}{\rho_o}$$

Where, $\Delta\rho$, and ρ_o are change in resistivity and initial resistivity respectively.

The piezoresistive testing resulted in the following calculated values of fractional change in resistivity with applied strain, and two such data sets have been shown below. A good correlation between FCR and strain shows a good self-sensing ability of the composite. The data shows a high coefficient of determination value (**R^2 = 0.96084** and **R^2 = 0.95702**) which means there is a strong linear correlation between FCR and the strain value. The results suggest that biochar has the potential to serve as a cost-effective substitute for expensive carbon nanomaterials in the production of cement-based composites that exhibit strong electrical conductivity and can function as self-sensing materials.

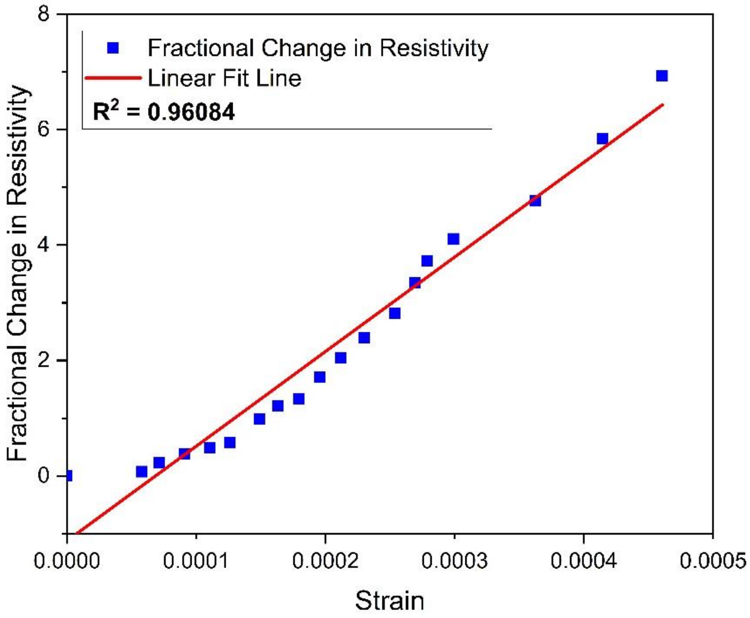


*Figure 14. Fractional Change in Resistivity for 10% Biochar content cement-based composite sample with **R^2 = 0.96084***

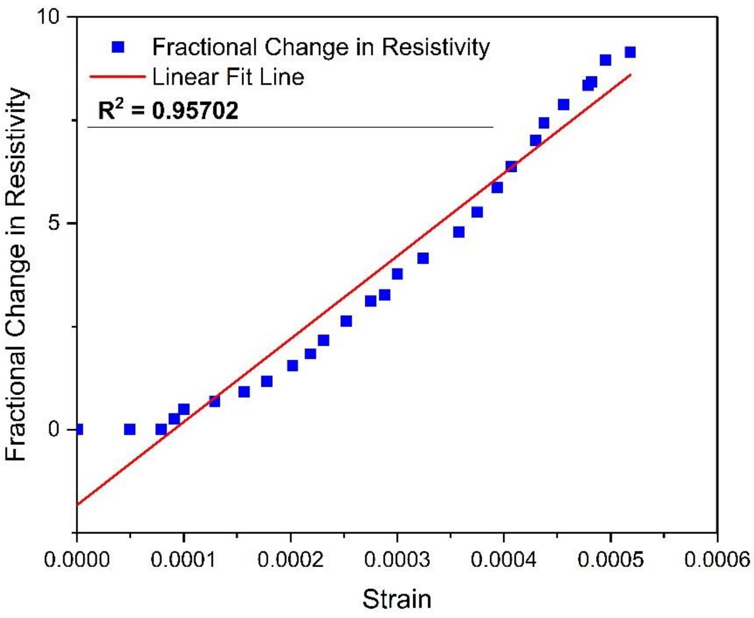


*Figure 15. Fractional Change in Resistivity for 10% Biochar content cement-based composite sample with **R^2 = 0.95702***

4.6 Carbon Neutrality and Environmental Impact

One major benefit of this multifunctional composite is the carbon sequestration and inhaling property of biochar and with it the composite. Cement composites on their own absorbs CO_2 but that results in carbonation of it, resulting in the acidity increase and further problems.

While with biochar, within the micropores of the biochar, CO_2 is stored and hence is removed from the environment in a way that doesn’t harm the cement composite itself also.

This removal results in a net offsetting of the greenhouse gas from the environmental system.

4.6.1 Carbon dioxide Monitoring Test

To measure the biochar's effect on carbon sequestration capability of the cement composite, a climate-controlled chamber was utilized to measure the amount of CO_2 absorbed by the composite. The amounts of CO_2 were measured in parts per million(ppm), and a specific concentration of 4000 ppm was maintained, while the samples were observed overtime using EXTECH sensor that records CO_2 with time. The sensor probe was fitted inside the chamber, while through an inlet a concentration of CO_2 was also provided initially.

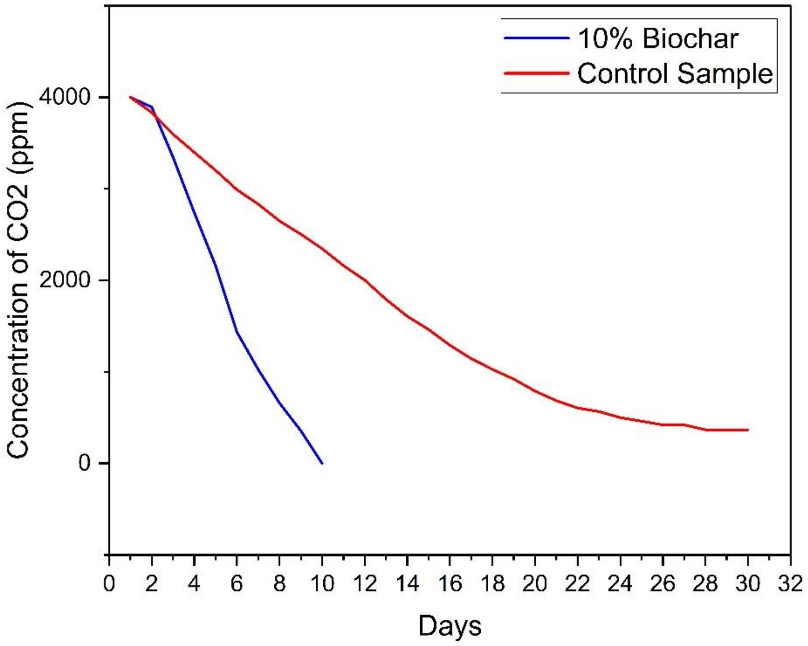


Figure 16. Concentration of CO_2 absorbed by cement composite samples

The test was conducted to determine the changes in concentration from a maximum value of 4000 ppm to a minimum value. In the results it can be observed that the control sample took around 30 days to reach its saturation of CO_2 absorption while the sample with biochar reaches the saturation point, much faster and around 10 days absorbs most of the CO_2 in the chamber, indicating a promising potential of biochar infused cement composite to be used as carbon sequestering material.

Chapter 5

CONCLUSIONS

5.1 Discussions and Future Recommendations

The addition of biochar to cement-based composites has been shown to improve their strength and self-sensing properties, making them a promising material for use in a variety of applications.

Biochar serves as an effective electrically conductive filler at an optimum concentration of 10 wt.% for sensing purposes, corroborated by theoretical percolation as well as by experimentation. Which has resulted in the production of a multifunctional material that has strong sensing ability due to its electrical conductivity.

The use of biochar produced from rice husk and coal waste can also help to reduce the environmental impact of these materials by providing a sustainable alternative with waste management and utilization. Reducing the net carbon footprint of the composite with increased carbon sequestration attribute.

The self-sensing properties of the biochar-cement composite offer potential for use in smart infrastructure, where early detection of damage and monitoring of structural health is critical for safety and rapid maintenance. And its further development in the form of cement based sensors can be done with future researches. The researches can also be conducted to optimize the biochar content even more, while long term durability can also be researched upon. And the performance of this material under cyclic loading can also be studied in future researches to further develop upon this materials multifunctionality.

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