EVALUATION OF IMPACT OF CARBONIZED NANO/MICRO PARTICLES ON THE CONTRACTION AND EXPANSION OF CEMENTITIOUS COMPOSITES

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Certification

This is to certify that **Waqar Khan**, **BCE192001** and **Umar Farooq**, **BCE192009** have successfully completed the final project **EVALUATION OF IMPACT OF CARBONIZED NANO/MICRO PARTICLES ON THE CONTRACTION AND EXPANSION OF CEMENTITIOUS COMPOSITES,**
at the **MYUniversity**, to fulfill the partial requirement of the degree **Civil Engineering.**

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EVALUATION OF IMPACT OF CARBONIZED NANO/MICRO PARTICLES ON THE CONTRACTION AND EXPANSION OF CEMENTITIOUS COMPOSITES

Sustainable Development Goals

SDG No	Description of SDG	SDG No	Description of SDG
SDG 1	No Poverty	SDG 9	Industry, Innovation, and Infrastructure
SDG 2	Zero Hunger	SDG 10	Reduced Inequalities
SDG 3	Good Health and Well Being	SDG 11	Sustainable Cities and Communities
SDG 4	Quality Education	SDG 12	Responsible Consumption and Production
SDG 5	Gender Equality	SDG 13	Climate Change
SDG 6	Clean Water and Sanitation	SDG 14	Life Below Water
SDG 7	Affordable and Clean Energy	SDG 15	Life on Land
SDG 8	Decent Work and Economic Growth	SDG 16	Peace, Justice and Strong Institutions
		SDG 17	Partnerships for the Goals

Range of Complex Problem Solving			
	Attribute	**Complex Problem**	
1	Range of conflicting requirements	Involve wide-ranging or conflicting technical, engineering and other issues.	
2	Depth of analysis required	Have no obvious solution and require abstract thinking, originality in analysis to formulate suitable models.	

3	Depth of knowledge required	Requires research-based knowledge much of which is at, or informed by, the forefront of the professional discipline and which allows a fundamentals-based, first principles analytical approach.	
4	Familiarity of issues	Involve infrequently encountered issues	
5	Extent of applicable codes	Are outside problems encompassed by standards and codes of practice for professional engineering.	
6	Extent of stakeholder involvement and level of conflicting requirements	Involve diverse groups of stakeholders with widely varying needs.	
7	Consequences	Have significant consequences in a range of contexts.	
8	Interdependence	Are high level problems including many component parts or sub-problems	
Range of Complex Problem Activities			
	Attribute	**Complex Activities**	
1	Range of resources	Involve the use of diverse resources (and for this purpose, resources include people, money, equipment, materials, information and technologies).	
2	Level of interaction	Require resolution of significant problems arising from interactions between wide ranging and conflicting technical, engineering or other issues.	
3	Innovation	Involve creative use of engineering principles and research-based knowledge in novel ways.	
4	Consequences to society and the environment	Have significant consequences in a range of contexts, characterized by difficulty of prediction and mitigation.	
5	Familiarity	Can extend beyond previous experiences by applying principles-based approaches.	

Abstract

The utilization of carbon-based additives in cementitious materials has gained significant attention due to their potential to enhance mechanical properties and mitigate environmental impacts. This research endeavours to comprehensively assess the influence of carbonized nano/micro particles on the contraction and expansion behaviour of cementitious composites. In Construction work we provide expansion and contraction joints during concrete pouring which is followed by joint fillers but after some course of time these fillers make problems and leads to leakage. In this research we will be using Bagasse Fiber and pine needles to check its impact on concrete expansion and contraction. Carbonaceous inerts of Bagasse Fiber and pine needles have been transformed via pyrolysis at 500 °C and 700 °C, respectively, in an inert atmosphere and utilized in the preparation of cementitious composites, to improve their performance against expansion and contraction. Carbonized material was characterized through scanning electron microscopy, energy dispersive x-ray spectroscopy, and particle size distribution. Samples of cement mortar with cement to the sand ratio of 1:1.5 were prepared with the addition of 0.025%, 0.05%, 0.08%, 0.20%, 0.50%, and 1.00% pyrolyzed nano-inert by weight of cement after the effective dispersion in water. The uniform dispersion of carbonized particles in the cementitious matrix was evidenced through the forensic analysis. The shrinkage and expansion characteristics of the modified cementitious composites are assessed using strain gauges and other specialized measurement techniques. This research holds implications for the development of sustainable construction materials that exhibit reduced cracking, improved durability, and enhanced overall structural integrity. In conclusion, the investigation into the impact of carbonized nano/micro particles on the contraction and expansion behaviour of cementitious composites represents a vital step towards advancing the field of cement-based materials engineering. By elucidating the complex relationships between carbon additives and dimensional stability, this study strives to pave the way for the innovative design and construction of more resilient and environmentally conscious infrastructure.

Keywords: Bagasse Fiber, Pine Needles, Carbonaceous Inerts

Undertaking

I certify that the project **EVALUATION OF IMPACT OF CARBONIZED NANO/MICRO PARTICLES ON THE CONTRACTION AND EXPANSION OF CEMENTITIOUS COMPOSITES** is our own work. The work has not, in whole or in part, been presented elsewhere for assessment. Where material has been used from other sources it has been properly acknowledged/ referred.

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List of Acronyms

BF	Bagasse Fibers
CM	Cement Mortar
CM-PBF	Cement Mortar Induced with PBF
CM-PPN	Cement Mortar Induced with PPN
CNIs	Carbonized Nano Inerts
CNMPs	Carbonized Nano Micro Particles
EP	End Point
LOI	Loss on Ignition
PBF	Pyrolyzed Bagasse Fibers
PN	Pine Needles
PNMPs	Pyrolyzed Nano Micro Particles
PPN	Pyrolyzed Pine Needles
PV	Peak Value
TGA	Thermogravimetric Analysis

Chapter 1

INTRODUCTION

1.1 Concrete

A hard, chemically inert particulate known as aggregate—typically sand and gravel—is combined with water and a binder, such as cement, to form the structural material known as concrete. The second most used material is concrete.

Concrete was once bound together using gypsum (sulphate) and lime (calcium oxide). Then, in the 1800s, cement was created, and it has since continued to be the most widely utilized binding agent in concrete building.

Concrete is composed of aggregates that are often categorized as Fine (having a size range of 0.025 to 0.65 mm) or Coarse (having a size range of 6.5 to 38 mm), binder materials such as Portland cement or lime, and occasionally admixtures are added to improve specific qualities. The typical components of concrete are displayed in.

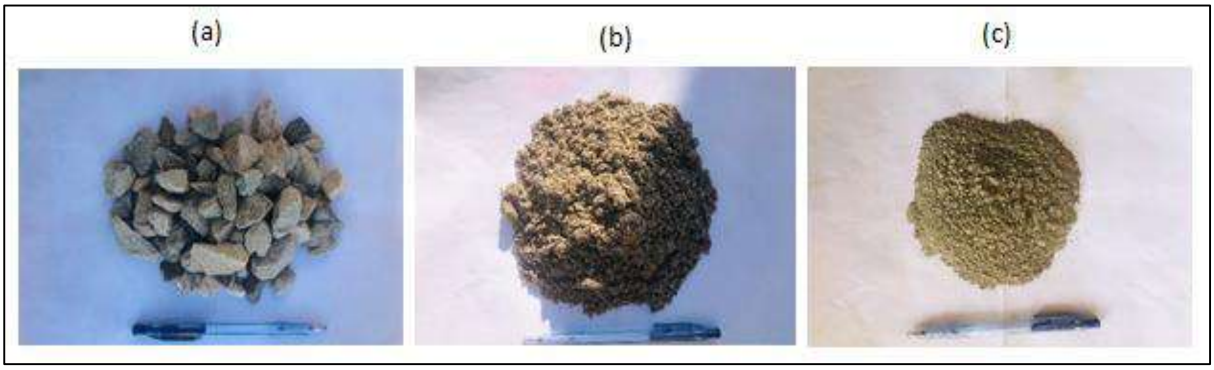

Fig 1.1 (a) Coarse Aggregate, (b) Sand & (c) Ordinary Portland Cement

1.2 Cementitious Materials

Cementitious materials are widely used in infrastructure across the globe because of their affordability, advanced production techniques, and ability to respond to a wide range of climatic conditions. The three primary categories of cementitious composites are concrete, mortar, and paste. Their strength and durability are significantly compromised by their tendency to crack due to their quasi-brittle nature [1]. In the past, adequate attention has been paid to reducing the brittle behavior of cementitious composites. A number of studies have been conducted with the goal of raising the cementitious materials' tensile strain capacity. Numerous studies have continued to focus on the incorporation of fibers in addition to traditional steel reinforcing. Fiber reinforced cementitious materials consist of synthetic or natural materials. The fibers are intended to provide resistance against cracking both in plastic and hardened form of the composite materials. The synthetic fibers comprise of metals like steel, aluminum; polymers like polypropylene, polyester, nylon, acrylic, aramid, and carbon [2]. Concrete with steel fibers is shown in Fig 1.2 and 1.3. The natural fibers include, coir, jute, pineapple leaf, kenaf bast, bamboo, palm, hemp, sugarcane bagasse, and many more [3]-[4].

Fig 1.2 Crack arresting of steel fibers, reprinted with permission from Han,Baoguo Yu, Xun Ou, Jinping. Elsevier [5].

Fig 1.3 Concrete reinforced with steel and polypropylene fibers,Vahid.,Ozbakkaloglu,Tobgay. Elsevier [6].

1.3 Applications of Concrete

Concrete has been used for construction since ancient times. Modern day applications include dams, bridges, homes, streets, patios, swimming polls, balustrades, plain cement tiles, mosaic tiles, pavement blocks, kerbs, drain covers and so on.

1.4 Drawbacks of Concrete

1.4.1 Environmental Drawbacks

In manufacturing process of cement, we require raw materials for this purpose we use mining and transportation which is fuel dependent and directly affect environment. After that in the kiln we need a lot of energy with is obtained from coal, coal directly affect our environment by emitting CO_2, nitrous oxide, Sulphur and other pollutants. Cement is finely grinded into powdered form which produces dust that can harm environment [7].

1.4.2 Structural Drawbacks

Although, Concrete is one of the most widely used construction material, but there are many drawbacks associated with it. Some of these drawbacks are related to the structural behavior of concrete. Structural drawbacks associated with concrete are low tensile strength, toughness and specific strength. Some other issues while working with concrete are creep, shrinkage, fatigue, susceptible to cracking and brittle behavior. Some of these can be modified by adding admixtures, or modifying the concrete structure and ingredients [8].

1.5 Reinforced Concrete

Researchers are using different materials to improve the performance of concrete. One of the most widely used construction material is reinforced concrete [9]. In the recent years, research has seen to improve the physical, mechanical and electrical properties of concrete with the use of carbon based micro/nano materials such as nanotubes, nano fibers and waste steel wires [10]. Other carbon based materials include nano particles(Fe2O3;CuO) [11], [12], [13], Carbon nanotubes(SWCNT and MWCNT) [14], [15], [16], graphene oxide(GO) [17], [18], [19], graphene nano platelets(GNP) [20], and waste steel wires [21]. Composites formed while using these materials not only have high strength, durability and dimension stability but also are cost effective [22].

1.6 Sustainability

Sustainability can be defined as meeting the present needs without compromising the future generation to meet their own needs. Three main pillars of sustainability which are economic, social and environmental. Sustainability is important for existence of human and nature. Concrete is second widely used material all over the world. The production of cement for concrete production produces CO_2 and GHGs. Almost 8-10% of CO_2 is due to production of cement [23]. Sources of CO_2 and GHG production for production of cement are; from combustion of limestone: 50-55%, from fuel combustion: 40-55% and from use of electric power: 0-10% [24]: This CO_2 and GHG degrade environment and cause global warming. Also, for acquiring of concrete constituent, we destruct our natural environment and natural resources are depleting. So, it is necessary to construct a sustainable construction so that total environmental impact during its design life have minimum. It is also necessary to maintain a balance between human and nature, that construction is suggested that maintain a balance between construction and ecology [24].

Briefly, Sustainable construction is that construction that use waste material for construction, renewable resources (infinite resources), mass with thermal property (ability to absorb energy, beneficial for cold environment), use clean energy (solar or wind energy).

1.7 Expansion & Contraction Joints

Joints on concrete are categorized according to function, construction process, building life cycle stages, appearance on concrete and used of tool for their execution. The classification tries to analyze joints not only from a technical point of view, but also with a special focus on the aesthetical side. Taking into account the INTEMAC report “Joints in concrete constructions” by José Calavera Ruiz, joints are classified into four types:

– Expansion joints
− Contraction joints
− Settlement joints
– Construction joints

Shrinkage and expansion movements due to thermal effects, moisture, as well as loads could lead to undesirable cracks, thus leaving the building in an unsafe state, if there does not exist any joints. Contraction and expansion joints are those that are left between building elements at regular intervals to allow the dimensional change of material due to temperature variations. As we can see from the names, contraction joints are to avoid those cracks caused by material shrinkage. And expansion joints are those that are intended to absorb the expansions caused by temperature increases. Since the purpose of these joints is to define the location of cracks in advance, we can also call them planned cracks, in order to intervene aesthetically. The goal of these kind of joints is to allow the slab to crack, during expansion and contraction, in a known location and a straight line[25]. These Joints are helpful in Concrete buildings but the main issue is leakage so to avoid these joints or to minimize them we will be using biochar of bagasse fiber and pine needles to check its impact.

1.8 Bagasse Fiber & Pine Needles

Bagasse fiber is a byproduct of the sugar industry and is a type of natural fiber that can be used as a reinforcement in concrete. It is lightweight, strong, and has good thermal and acoustic insulation properties. When added to concrete, bagasse fiber can improve its tensile and flexural strength, reduce shrinkage and cracking, and increase its durability. Bagasse fiber can also reduce the overall weight of the concrete, making it more cost-effective and easier to handle.

Pine needles are another natural material that can be used as an additive in concrete. They are renewable and readily available, making them a sustainable and eco-friendly option. When added to concrete, pine needles can increase its compressive strength and reduce its water absorption. Pine needles can also improve the workability of the concrete, making it easier to pour and spread, and can improve its insulation properties.

Both bagasse fiber and pine needles can be added to the concrete mix in varying proportions depending on the desired properties and performance of the concrete. However, it is important to ensure that the fibers are evenly distributed throughout the concrete mixture to avoid the formation of weak spots or clumps. Proper mixing and placement techniques should be followed to achieve the desired benefits of these natural additives.

1.8.1 Size and Aspect ratio

Nano fibers or micro fibers are now-a-days used in concrete vastly all over the world. Nano fibers and micro fibers means fibers have at least one dimension 10^{-9}m and 10^{-6}m respectively. The aspect ratio of the fiber is the ratio of its length to its diameter. Typical aspect ratio ranges from 30 to 150. The purpose for the addition of short fibers is to improve the tensile strength and impact-resistance and reduce the brittleness of concrete. Indeed, some fibers actually reduce the strength of concrete. The number of fibers added to a concrete mix is expressed as a percentage of the total volume of the composite (concrete and fibers), termed "volume fraction" (V_f). V_f typically ranges from 0.1 to 3%.

1.9 Carbonaceous inerts

Carbonaceous inerts are defined as the material derived from organic or inorganic feedstock that contain a high amount of Carbon. They include coal, petroleum products, carbon black, tar, and many carbon containing alloys. Pakistan is an agricultural country and abundant quantities of agricultural waste is produced every year: If properly refined and treated, these wastes can be employed for many useful purposes [26]–[28]. Just like many scientific fields, the construction materials technology has also benefited from waste agricultural wastes: These wastes have either been extensively used as natural fibers, as discussed in the introduction section. Additionally, many studies have shown that if these wastes are burnt, their ashes can be used as cementing or pozzolanic materials for enhancing the properties of the cementitious composites [29]–[32]. The SEM images of wheat straw ash burned at different temperatures are shown in Fig 1.4. However, it has been indicated that the direct combustion of crop residues in agricultural fields is harmful for the environment, owing to emission of greenhouse gases [33], [34]. Many researchers have suggested alternate ways for useful application of the agricultural waste, which involves the extraction of carbonaceous inerts via several techniques like, hydrogenation, fermentation, combustion, bio-conversion etc. [22], [35]. According to a number of other researchers, pyrolysis is the most effective technique for extracting useful carbonaceous inerts from agricultural wastes [36]–[38].

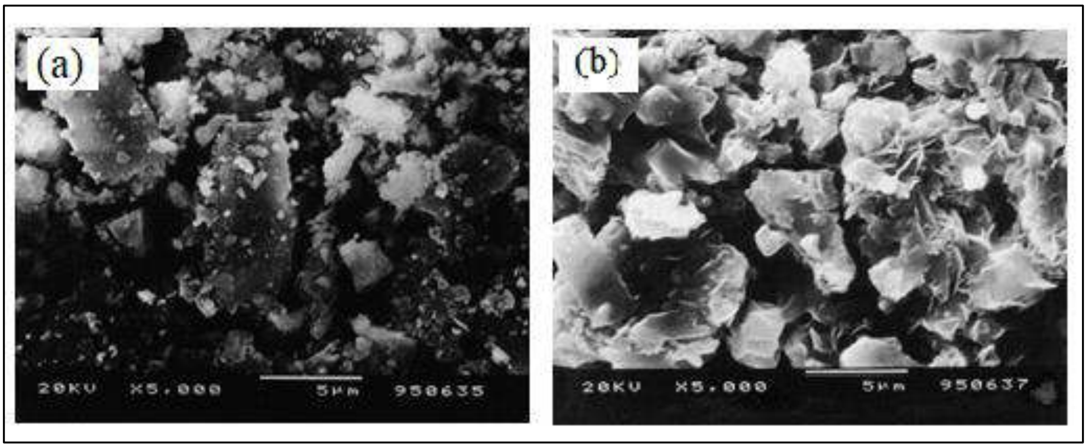

Fig 1.4 SEM image of wheat straw ash burned at 570°C (a) and at 670°C (b) reprinted with permission from Biricik, Hasan Aköz, Fevziye Berktay, I.lhan Tulgar, Ali N. Elsevier [32].

1.9 Pyrolysis

Pyrolysis is an endothermic process that involves the thermo-chemical decomposition of raw bio-mass in the inert environment at high temperature and pressure [39]. This process produces various useful products such as solid bio char, liquid bio-oil and fuel gases as highlighted in the Eq. 1 below [40]:

$$\text{Biomass} \xrightarrow{\text{Heat}} \text{Biochar(s)} + \text{Bio oil(l)} + \text{Fuel gas(g)} \qquad \text{Eq .1}$$

Pyrolysis has the potential to transform environmentally hazardous wastes into stable valuable products, which are less harmful to life and environment [41]. According to Ruan et al., one of the main advantage, pyrolysis offers over other techniques, is that the desired product (liquid, solid or gas) can be produced by adjusting the operational parameters, i.e. temperature, pressure, heating rate and residence time [42]. It has also been reported that biochar produced from slow pyrolysis having low heating ramp with longer residence time exhibit more homogeneous character than those produced by fast pyrolysis [43]: The SEM images of rice husk and wheat straw and their chars after fast pyrolysis in the Drop Tube Furnace at various temperatures are shown in Fig 1.5 & 1.6 respectively. A typical pyrolysis process is shown in Fig 1.5 in the form a flow diagram [44], whereas the general layout of the pyrolysis process is shown in Fig 1.8 [45].

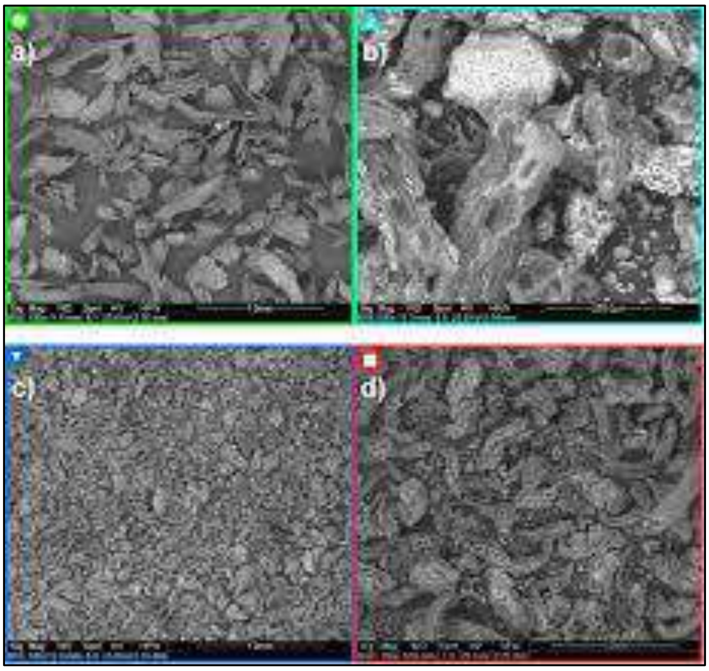

Fig 1.5 SEM images of rice husk: green biomass (a) and chars after fast pyrolysis in the DTF at 900 °C (b), 1100 °C (c), 1300 °C (d), reprinted with permission from Pott Maier, Daphanie Costa, Mário Farrow, Timipere Oliveira, Amir A. M. Alarcon, Orestes Snape, Colin. Elsevier [43].

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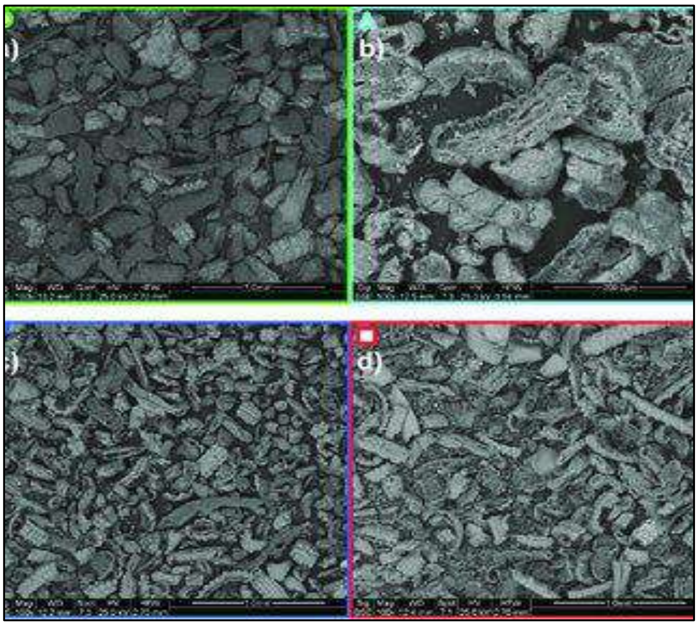


Fig 1.6 SEM images of wheat straw: green biomass (a) and chars after fast pyrolysis in the DTF at 900 °C (b), 1100 °C (c), 1300°C (d), reprinted with permission from Pott Maier, Daphanie Costa, Mário Farrow, Timipere Oliveira, Amir A. M. Alarcon, Orestes Snape, Colin. Elsevier [43].

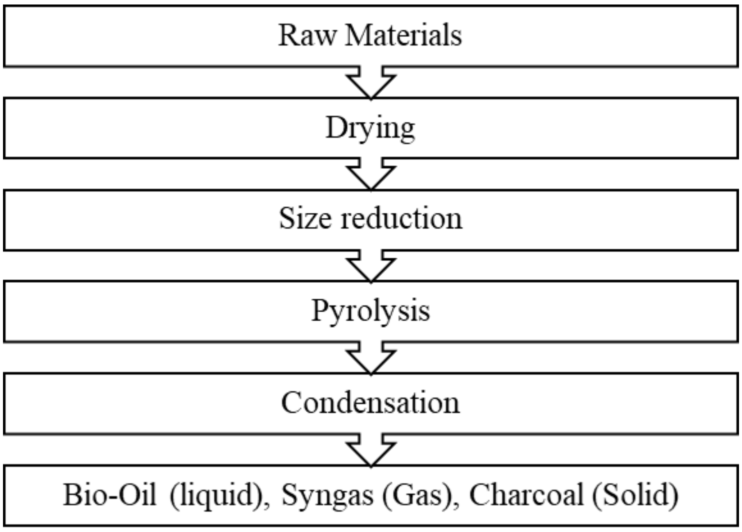


Fig 1.7 Flow diagram of pyrolysis

Fig 1.7 shows the flow diagram of pyrolysis process in which we take raw materials and dry it followed by the size reduction step in which we minimize the size of raw material. Pyrolysis is temperature is provided to the dryed raw material and the temperature at which pyrolysis occurs is noted.

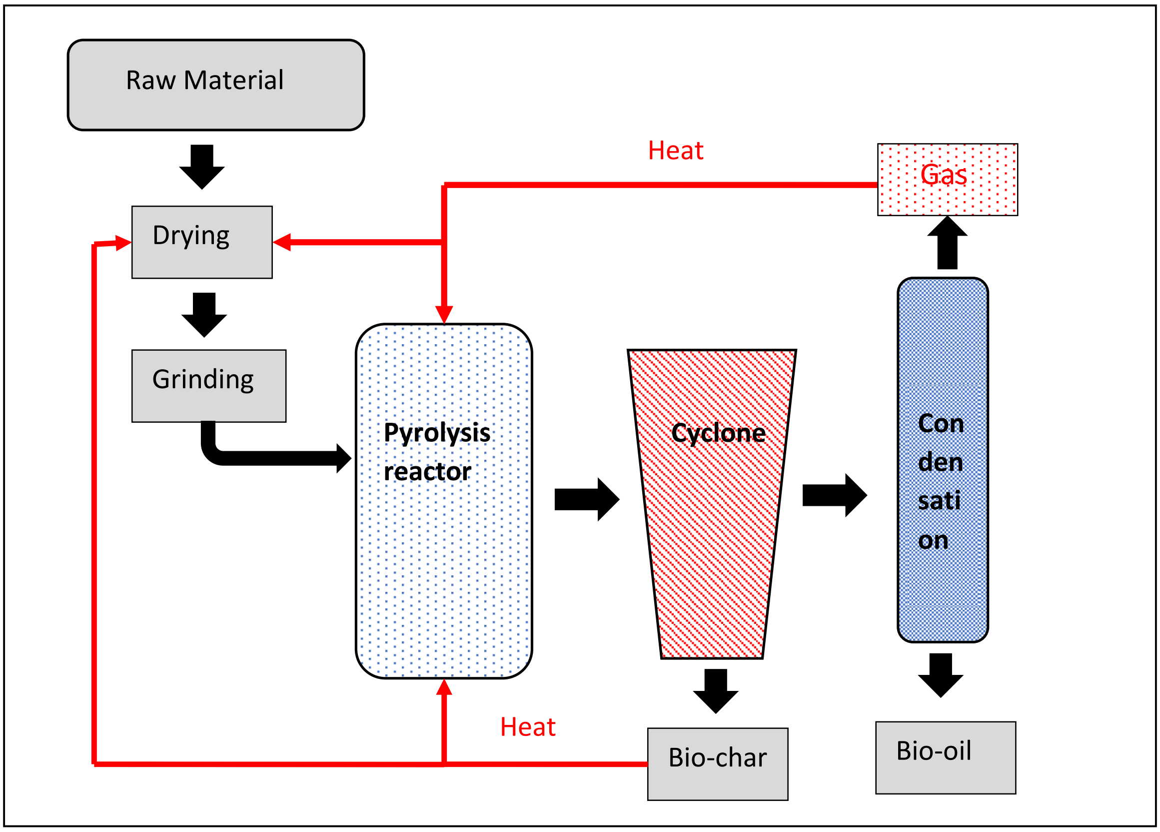


Fig 1.8 General layout of pyrolysis process

Pyrolysis is termed as slow or fast, depending on the heating ramps and the operating temperature: Slow pyrolysis is characterized by low heating ramp (<100K/min) and low temperature (~300ºC), whereas, fast pyrolysis refers to that carried out at high heating ramp (>100K/min) and higher operating temperature (~500ºC or more) [46]. Pyrolysis is reported to lack synergetic effects and it has been indicated that the yield is proportional to the percentage of biomasses in the feedstock [47].

1.11 Research Problem

" In Construction work we provide expansion and contraction joints during concrete pouring which is followed by joint fillers but after some course of time these fillers make problems and leads to leakage. In this research we will be using Baggase fiber and pine needles to check its impact on concrete expansion and contraction.

This research problem focuses on investigating how the incorporation of carbonized nano and micro particles affects the volume changes (contraction and expansion) of cementitious composites.

1.12 Research Objectives

The major objectives of this research

- To assess the effect of carbonized nano/micro particles on the expansion properties of cementitious composites and ascertain if these particles contribute to expansion or contraction.
- To analyze the optimal dosage and characteristics of carbonized nano/micro particles for enhancing the expansion and contraction related issues in cementitious composites.
- To provide recommendations for the industry regarding the practical use of biochar in improving the expansion and contraction behavior of cementitious composites.

Chapter 2

LITERATURE REVIEW

Bio char is a light-weight black carbon residue, obtained after eliminating water and other volatile ingredients, mainly by pyrolysis. Various researchers have used numerous feedstocks for producing bio char: They can be agricultural waste like wheat, rice and sugarcane or forest residues like water hyacinth, beech trunk, hemp herd and sawdust etc., as shown in Fig 2.1. In the following Table 2.1, different raw materials, used for producing bio char by pyrolysis and their results are described

Fig 2.1 (a) Wheat straw (b) Rice straw (c) Sugarcane Bagasse (d) Forest residues

2.1 Raw Materials Based

Table 2.1 Summary of literature on the use of different raw materials for producing Bio char

Sr #	Materials	Procedure	Results	References
1	Water Hyacinth	Prepared bio char briquettes and studied under different heating conditions	At the temperature of 425ºC, the yield of water hyacinth bio char was 55%	[48]

Sr #	Materials	Procedure	Results	References
2	Oriental Beech	Synthesize bio char by pyrolysis at 493, 523 and 593K	High yield of Carbon rich bio char is at lower temperature	[49]
3	Olive Husk	Steam pyrolysis is carried at particle size between 0.34 mm and 0.5 mm	Yield of about 29-40% at a temperature of 900 K	[50]
4	Untreated and Phosphoric Acid treated Corncob	Pyrolysis at Temperatures ranging 400-600ºC at constant heating rate of 16°C/min in a quartz reactor	Yield decreases with increase in temperature; however, Pretreatment increases the yield.	[51]
5	Tea Waste	Done pyrolysis in the temperature range of 300-400ºC	Carbonization and Aromaticity of bio char improves with temperature	[52]
6	Pomegranate Seeds	Conducted pyrolysis at temperatures, ranging 400 to 800 ºC	Bio char contains high carbon content, having high bulk density and calorific value	[53]
7	Soybean oil cake	Pyrolysis of chemically activated soybean oil cake with K_2CO_3 and KO, at 600 ºC and 800 ºC	High yield of bio char with K_2CO_3, having surface area of 1353 m^2/g at 800 ºC	[54]
8	Woody Waste (Pinus Radiata)	Pyrolysis at a temperature range of 500-900 ºC	High yield of bio char at lower temperature Yield decreases with increase in temperature	[55]
9	Rice Husk	Performed pyrolysis at different temperatures on sample (particle size of 0.5-7mm)	35% yield of bio char at 480 ºC	[56]

Sr #	Materials	Procedure	Results	References
10	Coconut Husk	Produce bio char at temperatures ranging from 400 ºC to 1000 ºC	Decrease in yield and increase in aromaticity with increase in temperature	[57]
11	Peanut Shells	Prepared bio char by conventional as well as microwave irradiation pyrolysis	Microwave irradiation followed by pyrolysis produces bio char with enhanced adsorption characteristics	[58]
12	Chickpeas waste and Peanut shells	Carried out pyrolysis for bio-char production, using a temperature range from 350 ºC to 600 ºC	Yield of 28% and 46%, using chickpeas waste and peanut shells respectively, at 350 ºC with heating rate of 15 ºC per minute	[59]
13	Waste Coffee	Pyrolysis at 700 °C for 1 hr. in N_2 atmosphere at a temperature range from 250 °C to 400 °C, using a heating rate of 5 °C/min	Yield of 18%, with the bio char having high porosity and average diameter of pores of about 7 µm	[60]
14	Rice husk and Wheat straw	Produce bio char, using slow and fast pyrolysis	19.4% yield for rice husk and 25.4% yield for wheat straw at 900 °C Yield decreases with increase in pyrolysis temperature	[43]
15	Sawdust	Produce bio char, using temperatures from 250-950 °C	Bio char with the highest calorific value can be produced using temperatures in the range of 450 to 650 °C	[61]

The following Figs 2.2 and 2.3 shows the SEM images of water hyacinth charcoal and effect of temperature on yield of bio char (corncob) respectively.

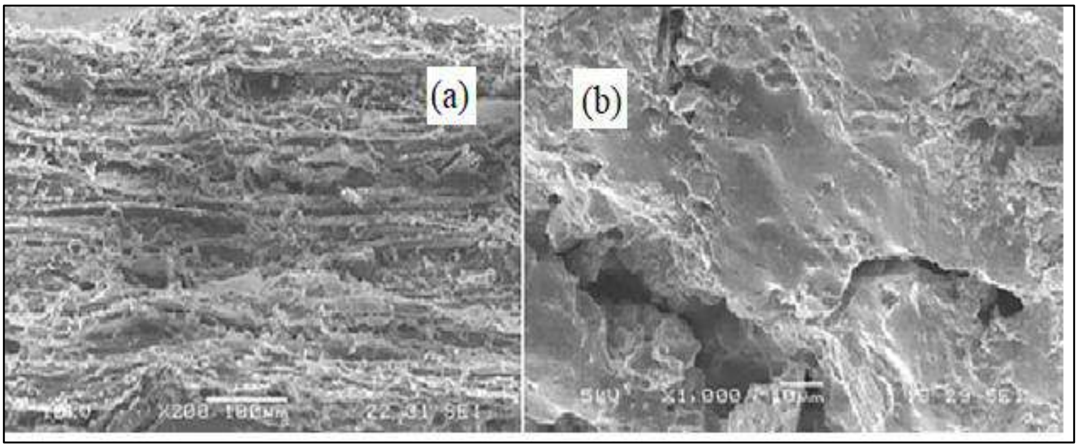


Fig 2.2. SEM images of water hyacinth charcoal (a) and water hyacinth briquette (b), reprinted with permission from Carnage, Naomi P. Tal agon, Romel B. Peralta, Jose P. Shah,Kalpit Paz-Ferreiro, Jorge. Elsevier [48].

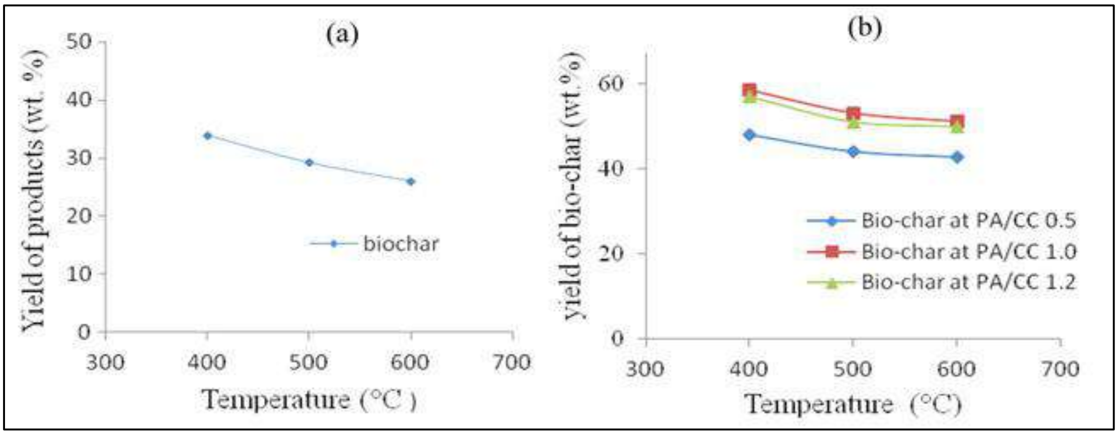


Fig 2.3 Effects of (a) temperature and (b) pretreatment on % yield of biochar from corncob at different temperature and pretreatment ratio, reprinted with permission from G.K Gupta, M.Ram, R. Bala, M. Kapur, M.K. Mandol. Elsevier [51].

2.2 Particle size Based

In the following Table 2.2, different particle size materials, used for producing bio char by pyrolysis and their results are described.

Table 2.2 Summary of literature on the use of materials of different sizes for producing Bio char

Sr #	Materials	Procedure	Results	References
2	Coconut Shells	studied the pyrolysis, with particle size	increase in bio char yield with increase in particle size	[63]

Sr #	Materials	Procedure	Results	References
3	Olive Waste, Straw and Birch (hard wood)	studied the fast pyrolysis temperatures 800-1000°C, using particles sizes from 0.5 to 0.8 mm and from 0.8 to 1.0 mm	increase in bio char yield with increase in particle size	[64]
4	Municipal Solid Waste	effect of particle size (0–5 mm, 5–10 mm and 10–20 mm) using 800°C temperature	smaller particle size resulted in higher syngas yield with less bio char, smaller size also leads to lesser carbon content in biochar	[65]

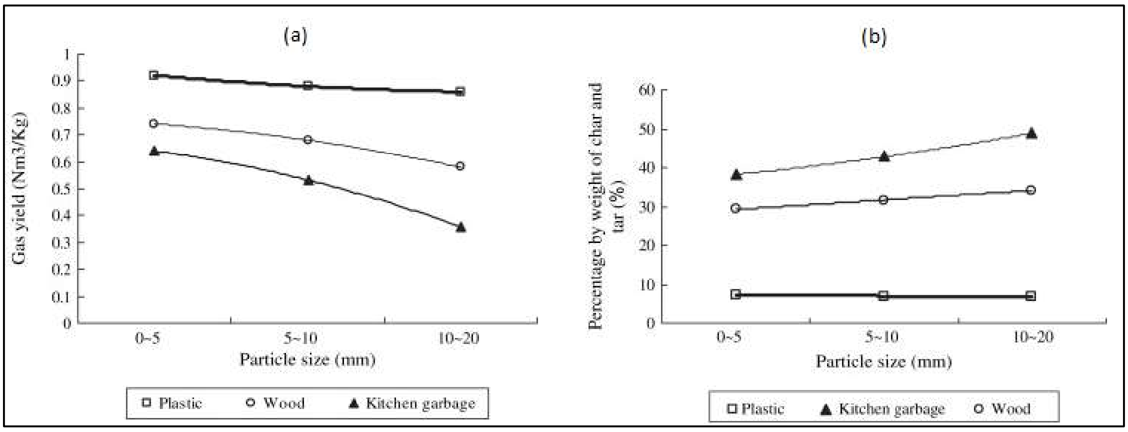


Fig 2.4 Gas yield of plastic, wood and kitchen garbage as a function of particle size (a) and weight percentages of char and tar as a function of particle size (b), reprinted with permission from S. Luo, B. Xiao, Z. Hu, S. Liu. Elsevier [65].

The gas yield of plastic, wood and kitchen garbage as a function of particle size and Weight percentages of char and tar as a function of particle size are shown in Fig 2.4.

2.3 Temperature Based

Temperature has the most significant effect on the outcomes of a pyrolysis process, as pointed out by numerous researchers. In the following Table 2.3, different temperatures used for producing bio char by pyrolysis and their results are described.

Table 2.3 Summary of literature on the use of materials at different pyrolysis Temperature for producing Bio char

Sr #	Materials	Procedure	Results	References
1	Wheat Straw and Lingo Sulfonate	studied the pyrolysis at different temperatures	the yield of decreased with increase in temperature, highly porous and aromatic bio char can be produced at higher temperatures	[66]
3	Bamboo	studied pyrolysis of bamboo at temperature ranging from 300 to 600°C	Bio char yield of 80% at 300°C, as compared to 30% at 600°C	[68]
4	Miscanthus	Studied pyrolysis at temperatures between 350 and 450°C	higher temperatures result in more stable bio char materials	[69]
5	Wheat straw and Woodchips	carried out slow pyrolysis in the temperature range of 400-525°C	surface area of biochar particles increased with increase in temperature	[70]
6	Hickory wood (HW), Bagasse (BG), and Bamboo (BB) feed stocks	studied the effect of temperature on the outcome of pyrolysis, under different temperatures (300°C, 450°C, 600°C) respectively	lower temperatures increased the yield, while higher temperatures increased the carbon content and produced thermally stable products	[71]
7	Soybean Stover and Peanut shell	studied characteristics of bio char at 300°C and 700°C	higher temperature enhanced the surface area and aromaticity of bio char	[33]
8	Oil distillation residue	effect of temperature on pyrolysis by fixing 400, 600, and 800°C	higher temperatures lead to higher carbon content,	[72]
9	Rapeseed stem	studied the effect of temperature (200-700°C in 50°C increment), heating	temperature as the most effective parameter, influencing the properties; higher temperatures enhance	[34]

Sr #	Materials	Procedure	Results	References
10	Safflower seed press cake	investigated the effect of temperature on pyrolysis	higher temperatures (600°C) lead to higher carbon content (80.7%), and aromaticity, and lower surface area	[73]

The following fig 2.5 shows the production (%) of different bio char samples under different temperatures. The effect of pyrolysis temperature and heating rate on safflower seed cake bio char yields at different temperatures and heating rate are shown in Fig 2.6.

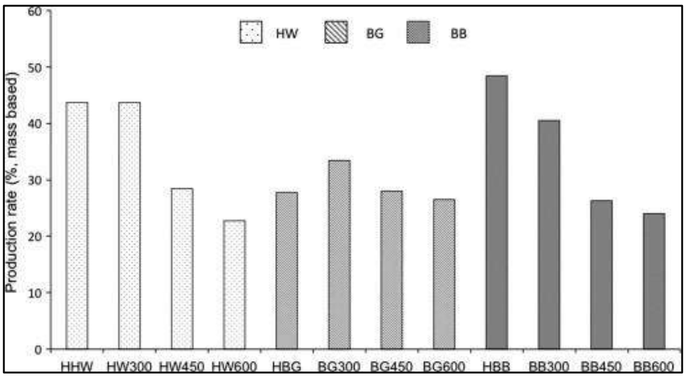


Fig 2.5 Production of different bio char samples, HHW, HW300, HW450, HW600, HBG, BG300, BG450, BG600, HBB, BB300, BB450, and BB600 are hydro chars and/or bio chars produced from hickory wood (HW), bagasse (BG), and bamboo (BB) feed stocks under different temperatures (300°C, 450°C, 600°C), respectively, reprinted with permission from Y. Sun, B. Gao, Y. Yao, J. Fang, M. Zhang, Y. Zhou, H. Chen, L. Elsevier [71].

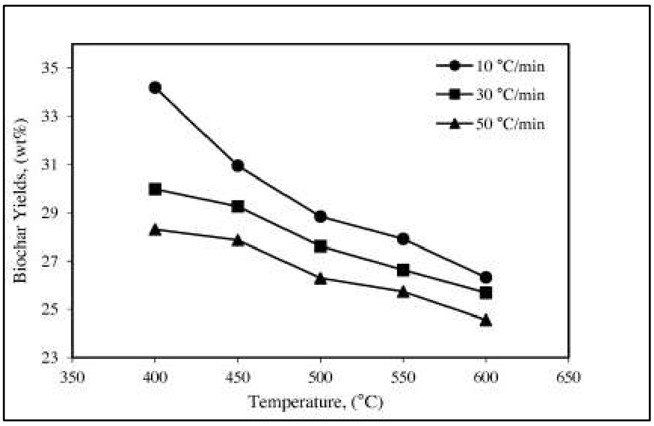


Fig 2.6 Effect of pyrolysis temperature and heating rate on safflower seed cake bio char yields, reprinted with permission from D. Angın. Elsevier [73].

Based on the studies, intended to evaluate the effect of pyrolysis temperature on the production of bio char, it is concluded that regardless of material: (1) Temperature is the most influential parameter, controlling pyrolysis, (2) Higher temperatures decrease the yield of bio char, (2) Higher temperatures enhance the carbon content, (3) Higher temperatures (600°C and above) significantly enhance porosity, pore volume, aromaticity, and alkalinity.

2.4 Heating Rate Based

Following Table 2.4 shows the use and results of bio char obtained by pyrolysis under different heating rates.

Table 2.4 Summary of literature on the use of materials at different pyrolysis Heating rate for producing Bio char

Sr #	Materials	Procedure	Results	References
1	Rapeseed	studied the pyrolysis using heating ramps of 5, 10, 20, 30, 40, and 50K/min	higher rates of mass losses at higher heating ramps	[74]
2	Radiata pine (softwood)	Investigated the effect of heating ramp on the pyrolysis	higher heating ramps resulted in melting of the particles and imparted smoother surfaces and induced large cavities	[75]
3	Moso bamboo (Phyllostachys edulis)	studied the effects of heating rates of 5, 10, 20 and 30°C/min on characteristics of the products	higher rates lead to lower liquid and solid contents, and enhance the specific area of the bio char	[76]
4	Scrap tires	investigated the effect of heating rates (5-80°C/min) on pyrolysis	enhancement of surface area with increase in heating temperature	[67]
5	Rice straw	Pyrolysis of rice straw at 600-1000°C temperature range	increase in pore size and surface area of bio char with increase in heating rate	[77]

Sr #	Materials	Procedure	Results	References
6	Pine chips (Pinus Brutia)	studied the pyrolysis using temperature (300-550°C) and heating rate of 7 and 40°C/min	highest yield of 36% of bio char at a temperature of 300 °C with 7C/min heating rate, increase in heating rate decreases yield	[78]
7	Wheat straw	. investigated the effect of heating rate from 5 to 20 °C/min	increase in bio char yield with increase in heating rate	[79]

The previous studies show that heating rate has both positive and negative effect on the production of bio char via pyrolysis. According to Mani et al., at lower heating rate, most of the materials show effective heat transfer, leading to more efficient cracking, and more weight loss in the form of volatiles: This leads to an increase in biochar yield with increase in heating rate [79].

2.5 Pressure Based

Following Table 2.5 shows the use and results of bio char obtained by pyrolysis under different pressure.

Table 2.5 Summary of literature on the use of materials at different pyrolysis Pressure for producing Bio char

Sr #	Materials	Procedure	Results	References
1	Pine	studied the effect of pressure (5-20 bar) on the pyrolysis at 800°C	pressure affects the physical and chemical properties of bio char	[80]
2	Coal	studied the pyrolysis of coal using steam under high pressure	reaction rate decreases with increase in pressure	[81]
3	Rice husk and Saw dust	studied the effect of pressure (4, 7 and 10 bars) on the pyrolysis	increase in yield from 4 to 7 bar pressure and an overall increase in liquid oil yield with increase in pressure	[82]

4	Acacia wood (hard wood native in Australia)	studied the effect of pressure on pyrolysis in the range of 1-6 bar	increased bio char yield and reactivity with increase in pressure. Optimum temperature is 617°C and optimum pressure is 6 bars	[83]
5	Pine sawdust	studied the effect of pressure on pyrolysis using high pressure (0-50 bar)	high pressures promoted more yield by secondary cracking of oil; enhances the structures and compactness	[84]

According to Basile et al., the enhanced pressure increases the retention time of the volatiles; the volatiles react further with the pyrolysis products, making secondary bio char. The secondary reactions are of exothermic nature that decrease the heating demand of pyrolysis and enrich secondary products with additional carbon content. Also the overall yield of bio char increases with increase in pressure [85].

2.6 Resident Time Based

The effect of residence time on the characteristics of bio char is often neglected; however, some researchers have reported its significance given in Table 2.6.

Table 2.6 Summary of literature on the use of materials at different pyrolysis Resident Time for producing Bio char

Sr #	Materials	Procedure	Results	References
1	Rapeseed stem	Pyrolysis	increase in residence time enhances the surface area and morphology of the bio char	[34]
2	Cornstalk, Rice husk, Peanut husk and Tobacco stalk	Pyrolysis	decreased yield, enhanced pH and carbon content of the bio char product with increase in temperature and residence time	[86]
3	Different raw materials	studied the synergetic effect of residence time (0.5, 1, 2, 4, 8	at lower temperature (300°C), the bio char yield decreased while its pH	[87]

		and 24 h) and temperature (300, 400, 500 and 600°C) on the pyrolysis	increased with increase in residence time; higher temperature, residence time had negligible effects on yield and pH of bio char	
4	Palm kernel shell	studied the effect of residence time (30-75 min) on bio char	yield decreased and the pH of the bio char increased with increase in residence time	[88]

From the above tabular discussions, it is concluded that lower temperature (< 400°C), slower heating rate, shorter residence times, larger particles and higher pressures enhance the yield, while higher temperatures, longer residence time, slow heating ramps, and high pressures lead to stable and aromatic bio chars. According to Rasul et al., different reactions take place at different temperatures, and at high temperatures, the liquid and solid molecules break down enriching the gas phase [89].

2.7 Selected Method Based

According to Simmons et al., several methods of biomass pyrolysis have been developed by the researchers and the industrial experts [86]: They include, basic or conventional pyrolysis, fast pyrolysis, catalytic pyrolysis and thermal plasma pyrolysis.

Table 2.7 Summary of literature on the use of Different Pyrolysis Methods for producing Bio char

Sr #	Method	Description	References
1	Conventional pyrolysis	carried out at low temperatures and low heating ramps, and as such favors high yield of bio char	[90]
2	Fast pyrolysis	characterized by high temperature and heating rate, short vapor residence time, rapid cooling of vapors; all favor high yield of bio-oil Fast pyrolysis liquefies solid biomass into liquid bio-oil [91]	[90]
3	Fast Pyrolysis	required amount of bio-oil can be extracted in fast pyrolysis, by controlling operating	[92]

		conditions, most importantly by quick condensation and by limiting the vapors residence time to less than 2s	
4	Fast Pyrolysis	reduces bio-oil yield, but enhances its calorific value and water content	[93]
5	Catalytic pyrolysis	the most commonly applied catalysts for pyrolyzing plastic waste are silica, alumina and zeolites	[94]
6	Thermal plasma pyrolysis	feedstock is inserted into a plasma; This heats up the raw material very rapidly. High temperature and heating rate quickly destroy the waste, producing gas and solid residue	[95]

2.8 Use of bio char in the cement and concrete

Use of biochar for enhancing soil fertility and sequestration of carbon is a not a new topic [96]–[99]: However, its use for enhancing concrete properties is quite new. It is expected that the use of bio-char in building materials might reduce as much as 25% emission of greenhouse gases into the atmosphere [100]. The following table 2.7 describes how the use of bio char affect the activity and performance of cementitious composites.

Table 2.8 Summary of literature on the use of Bio char in Cementitious composites

Sr #	Pyrolysis Materials	Procedure	Results	References
1	biochar and MgO	studied the effect of bio-char on the properties of cement paste	Biochar exhibited a positive effect on mitigating the autogenous shrinkage of cement pastes owing to its internal curing.	[101]
2	Bio char powder	added powder @ 2.5-10% as mass of cement in concrete, using accelerated curing and heat treatment	lightweight concrete with uniform porosity and considerable saving in overall cost of the material	[102]

3	Municipal solid waste	studied the effect of partial replacement (5-45%) of sand by bio char; in preparing cementitious mortars	bio char particles absorb water, which is available for internal curing of the material, absorption from micro pores leads to the densification of ITZ around bio char	[103]
4	Sawdust	Use bio char as additive in cementitious mortars @ 2% by mass of cement	enhances early compressive strength, imparts ductility, density and imperviousness to the specimens	[100]
5	hazelnut shells	3 g of feedstock was inserted, heating rate of 6ºC/min, with final temperature as 800ºC. ranging from few nm to 10µm were added to mortar @ 0, 0.5, 0.8, and 1.0% by mass of cement	highest flexural strength and the best post-cracking behavior with 0.8% carbonaceous materials	[104]
6	Application of rice husk biochar and thermally treated low silica rice husk ash	study aims to investigate the influence of TRHA, and blend of RHA and RHB on shrinkage , strength and ductility	Thermally RHA leads to higher silica content and specific surface area, which improve pozzolanicity of RHA as mortar admixture	[105]
7	Dry distillers' grains from bio ethanol industry	Studied the effect of bio char as filler in concrete	enhanced sound absorption, reduced thermal conductivity, material density and compressive strength; due to the porous structure of the bio char	[106]
8	Bamboo	investigated the effect on characteristics of	reduced strength, increased porosity and pore volume. The strength decreases with	[107]

Sr #	Materials	Procedure	Results	References
9	Nano softwood	studied the effect of bio char, obtained by pyrolysis at 700ºC, and added @ 0.8 and 1% by mass of cement	enhancement in flexural strength, fracture energy, toughness, and ductility	[108]
10	Timber	studied the effect of partial replacement of sand by bio char on the properties of cementitious mortars with fixed flow-ability	exponential decrease in strength with increase in bio char content	[109]
11	Sawdust charcoal	studied the influence of bio char, prepared at 300 and 500ºC; on the characteristics of cementitious mortars	enhanced early compressive strength (1-2% addition), enhanced water tightness, and no effect on flexural strength and drying shrinkage	[110]

Bio char is highly porous material, as such it leads to the reduction of workability, nevertheless, the adsorbed water is not chemically bound and is released with the passage of hydration reactions: This makes the bio char an internal curing agent that assist developing the micro pores and pore structure of the parent materials. Additionally, bio char decreases water evaporation, which is the cause of plastic and drying shrinkages in cementitious materials [103], [105].

Carbonaceous inerts have also found another way for enhancing the cracking resistance, toughness and energy absorbing capacity of cementitious materials. As indicated in the introduction part of this review, polypropylene (PP) fibers are added to cementing materials for many benefits, however, it has been pointed out that smooth surface of PP fibers hinders the formation of bond with the surrounding cement matrix [111]. Also, the hydrophobic surface of fibers leads to the formation of a thin water layer on PP fibers, which weakens its ITZ: It is known as wall effect [112]. Gupta et al. studied the effect of sawdust bio char prepared at

300ºC, as coating material of PP fibers, reinforced in cementitious mortars [113]: They have reported reduced workability, enhanced strength, post-cracking ductility, and impermeability.

According to Gupta et al., the high water absorbing capacity of bio-char hold significant amount of water during mixing phase of cementitious composites, which adversely affects the flow-ability [113]. According to Ghani et al., bio-char are hydrophilic in nature and as such have the capacity to adsorb water in the early stages of hardening: This in turn hinders the formation of capillary pores, enhancing the density and early compressive strength [114]. The reduced plastic shrinkage can also be addressed by using bio-chars, owing to their hydrophilic properties. Another potential advantage with these particles might be the reduction of bleeding as pointed out by Ely many et al., while working on the use of non-pozzolanic fillers in self-compacting concrete [115].

Chapter 03

RESEARCH METHODOLOGY

This research involved the use of nano sized carbonized inert materials synthesized by the pyrolysis of pine needles and bagasse fibers under controlled temperature and pressure. Different materials and methods were employed for the expansion and contraction of cementitious composites which are discussed below throughout this chapter.

3.1. Collection of Materials

The materials involved some of the common components of cementitious matrix and some specified synthesized carbonaceous materials from raw agricultural wastes.

3.1.1 Cement

Type-I ordinary Portland cement was used conforming to ASTM C-150. The chemical composition of cement was revealed with the help of X-ray Fluorescence (XRF) as shown in table 3.1.

Table 3.1. Chemical Composition of OPC

Parameter	Cao	SiO2	MgO	Al2O3	Fe2O3	LOI
Content (%)	3.84	65.00	19.19	2.23	4.97	3.27

3.1.2 Sand

Locally available Lawrencepur sand confirming to ASTM C-33 was used as filler material in mortar. The properties of sand used in this research are shown in table 1.3.

Table 3.2. Properties of Sand

Property	Fineness Modulus	Specific Gravity	Bulk Density (Kg/m3)	Water Absorption (%)
Content	3.15	2.69	1585	2.76

3.1.3 Agricultural waste materials

Two agricultural waste materials were used for the production of bio-char. These two agro-waste materials were chosen based on their abundance in raw form and their negative impacts on the environment. One of the selected materials was Pine Needles and the other was Bagasse Fiber.

• **Pine Needles:**

The genus Pines of kingdom Plantae is most widely occurring class of trees with 90 plus classes among which 24 are of Asian origin. Pine trees have clusters of needled shaped leaves that are responsible for the production of food by the process of photosynthesis. The green needle like leaves remain there for almost two years and start falling between the month of March and July by changing the color to brown [116].These brown colored fallen pine needles do not decompose for almost three years and remain there as a waste. So, these are selected to improve some properties of cementitious composites. The unprocessed and processed pine needles are shown in Fig 3.1

(a) Unprocessed PN (b) Processed PN

Fig 3.1 Processed and unprocessed pine needles

• **Bagasse Fiber:**

Bagasse is the pulpy fibrous material which is left after the extraction of juices from sugarcane. It is basically the solid by-product of sugarcane after the separation of liquid component. Sugarcane has an annual production of 1.89 billion tons around the world in almost 100 countries and 674 million tons in Asia [117]. Brazil has the maximum annual production of sugarcane followed by India, China, Thailand, and Pakistan (Nune set al., 2020). The chemical composition of washed and dried bagasse is shown in Table 3.3. The unprocessed and processed bagasse fibers are shown in Fig 3.2.

Table 3.3. Composition of Bagasse

Component	Percentage
Cellulose	45-55%
Hemicellulose	20-25&
Lignin	18-24%
Ash	1-4%
waxes	<1%

3.1.4 Superplasticizer

High range water reducing admixture (HRWRA) was added in mortar so that the flowability might not be compromised. In our research polycarboxylate ether Based superplasticizer was employed in mortar which not only retained the workability of mix close to the normal standard but also had a positive impact on the strength of the matrix.

(a) Processed BF (b) Unprocessed BF

Fig 3.2 Processed and Unprocessed BF

The dosage of the superplasticizer at W/C of 0.35 was set to 1% by weight of cement The properties of HRWRA are displayed in Table 3.4

Table 3.4. Properties of HRWRA

State	Density Kg/l	Color	pH	Chloride Content
Liquid	1.08	Light Brown	4.3	Nill

3.2 Methods

The process of Expansion & contraction Eco-friendly and sustainable cementitious composites with enhanced strength and Thermal conductivity involved the production of Nano/Micro sized carbonaceous inert by implying pyrolysis of the two selected Agro-waste materials. After the production of biochar, micro-analysis was carried out to investigate the properties of biochar at micro and nano scale. At last, the cement mortar samples were casted by adding different percentages of biochar followed by their mechanical and micro-structural analysis to verify the achievement of our objectives. The details of the procedures mentioned above are discussed in coming sections.

3.2.1 Production of Bio-Char

Production of Bio-char involved various steps starting from the collection of waste materials to the final grinding of bio-char to micro/nano scale. All these steps are discussed one by one below.

3.2.1.1 Collection of Agro-Waste Materials

Two Raw agricultural wastes were first collected from region of Pakistan Swabi where they were present in abundance. Pine Needles were brought from pine forests of District Swabi Village Kalu Khan in raw and Bulk form. Whereas, the bagasse was collected from the fields of sugarcane of Kalu Khan District Swabi.

3.2.1.2 Pre-processing of waste material

Both the pine needles and bagasse fibers were first cut into small pieces of length approximately 2-3cm. PNs and BFs were then thoroughly washed by distilled water to remove any attached dust particles on the surface. Washed material was dispersed in large trays and kept at room temperature for 12 hours for moisture removal from surface. After that the partially dried material was kept in oven at a temperature of 105±5◦C for 24 hours to completely remove any moisture present in small pieces. The processed material was then stored in air tight bags for further use. The final form of agro-waste material after pre-treatment is shown in fig 3.3.

3.2.1.3 Thermogravimetric Analysis (TGA)

Thermogravimetric Analyzer was used to study the decomposition regime of PNs and BFs under inert atmosphere (N2 atmosphere) to pick up a temperature for pyrolysis. TGA was also performed in the presence of oxygen to Fig out the amount of carbon present in both the agricultural wastes before pyrolysis. The TGA curves obtained at the end were then analyzed to get our desired output i.e., finalized temperature for pyrolysis and carbon content.

(a) Processed BF (b) Processed PN

Fig 3.3. Agro-wastes after pre-processing

Almost 15-20mg of PN and BF sample was first placed in ceramic crucibles to start the TGA process. The whole process was carried out up to a temperature of 1000◦ C with the heating ramp of 20◦ C/min. For providing inert atmosphere in the chamber Nitrogen gas was induced as a purge gas at a flow rate of 80ml/min and for performing TGA in the presence of oxygen, O2 was performed in the same conditions up to a temperature of 800◦. After the completion of the process a curve between percentage mass loss and temperature was obtained in inbuilt software of analyzer which was further analyzed by Differential Thermogravimetry (DTG) to find out the temperature range of maximum weight loss and a temperature after which no considerable mass loss has occurred. The temperature after which no or very minute mass loss was observed was finalized for further process of pyrolysis.

3.2.1.4 Pyrolysis of Agro-waste materials

A pyrolysis setup was developed comprising of a tube furnace with quartz tube and an assembly for the provision of inert gas for the carbonization of the collected waste materials as shown in Fig3.4a. The gas used for provision of inert atmosphere. Was Argon (AR). The quartz was first sweeped with argon gas to remove any other gases present in the tube. Then the pressure of 0.02MPa of inert gas was maintained in the quartz tube. Bio-gas and bio-oils produced during the process were allowed to escape from the outlet of the tube from time to time. 50 gm of pre-processed agro-waste material was induced in the tube each time and heated to a temperature predefined by TGA. The heating ramp was kept same as that in TGA i.e., 20◦C/min. Pine needles were pyrolyzed up to a temperature of 700◦C and bagasse fiber was pyrolyzed up to 500◦C. After the achievement of desired temperature, the produced bio-char was allowed to cool at the same rate as that of heating ramp and a pure black colored carbonaceous material was obtained as shown in Fig 3.4b

(a) Tube Furnace

(b) Produced Bio-char

Fig 3.4 Pyrolysis Setup (a) Tube Furnace (b) Produced Bio-char

(a) Processed PN

(b) Processed BF

Fig 3.5 Processed PN and BF biochar

3.2.1.5 Ball Milling of Bio-Char

The pyrolyzed bio-char was grounded to micro/nano sized particles by using a ball mill apparatus. The ball milling operation was performed for 30 minutes at a frequency of 35 Hz.

3.2.2 Characterization of Bio-Char

Fine Black pyrolyzed powder was then analyzed using various techniques in order to investigate the particle size, shape, texture and elemental analysis. The crystalline structure was also analyzed for better understanding of crystalline and amorphous phases of bio-char. Furthermore, the dispersion of bio-char in water was also analyzed after sonication at different intervals.

3.2.2.1 Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) was performed to study the particle shape, size and texture. The purpose of running this analysis was to confirm the presence of micro and nano sized particles in bio-char before its use in cementitious composites.

3.2.2.2 Energy Dispersive X-Ray Spectroscopy (EDX)

Energy Dispersive X-Ray Spectroscopy (EDX) was carried out to examine the elemental composition of bio-char. The main objective of this test was to Fig out the total carbon content present in the pyrolyzed pine needles and pyrolyzed bagasse fibers.

3.2.2.3 Particle Size Distribution

Particle size distribution was studied by making suspension of carbonized particles in water. For this purpose, 25mg of PBF and PPN was mixed in 100ml of water and the mixture was sonicated for 30 minutes. The suspension was then analyzed with the help of a Zetasizer Nano ZSP for revealing the sizes of all the particles present in the mixture.

3.2.3 Casting of Cementitious Composites

After the confirmation of presence of high carbon content and nano sized particles in bio-char, mortar samples were prepared. Two types of samples i.e., mortar prisms (40x40x160 mm3) and mortar tiles (2"x2"x2") were prepared by intrusion of carbonaceous nano/micro inert of PBFs and PPNs.

3.2.3.1 Mix Proportions

Total thirteen different formulations were prepared by the incorporation of both pyrolyzed bagasse fibers (PBF) and pyrolyzed pine needles (PPN) at different percentages i.e., 0, 0.025, 0.05, 0.08, 0.2, 0.5 and 1.0% by weight of cement. 21 mortar prisms (40x40x160 mm3) were casted for each formation for mechanical testing and EMI-SE respectively. 1:1.5 cement sand mortar was used with water to cement ratio of 0.35 because it is an ideal recipe for mortar samples (Basheer-u-Deen and Anandan, 2014). It was observed that the workability started reducing on the addition of carbonized nano materials. In order to avoid the reduction in flow-ability of mortar polycarboxylate ether-based superplasticizer was added in a quantity of 1% by weight of cement. The detailed mix proportions of all the formations are shown in the table 3.5.

Table 3.5 Mix proportions of various mortar formulations

Sr No.	Sample ID Bagasse Fiber and PPN	C/S Ratio	W/C	Biochar %age	Cement (gm)	Sand (gm)	Water (gm)	HRWRA (gm)
1	- - - - - -	01:01.5	0.35	0%	420	733.3	147	4.2
2	CM-0.025%	01:01.5	0.35	0.03%	420	733.3	147	4.2
3	CM-0.05%	01:01.5	0.35	0.05%	420	733.3	147	4.2
4	CM-0.08%	01:01.5	0.35	0.08%	420	733.3	147	4.2
5	CM-0.2%	01:01.5	0.35	0.20%	420	733.3	147	4.2
6	CM-0.5%	01:01.5	0.35	0.50%	420	733.3	147	4.2
7	CM-1%	01:01.5	0.35	1%	420	733.3	147	4.2

3.2.3.2 Dispersion of Carbonaceous Inerts

Carbonized nano/micro particles were sonicated in water using a bath So nicator. Different pre-decided percentages of carbonized particles were added into 80% of the water required to prepare the mortar samples. The mixture of water and carbonized material was thoroughly mixed with the help of a stirrer followed by sonication. The time of the sonication was finalized based on the UV-Visible Spectroscopy that came out to be 30 minutes. After effective dispersion the solution of Carbonized particles was ready to be used in the preparation of mortar samples.

3.2.3.3 Mixing Regime

Carbonized nano/micro inert of bagasse fibers and pine needles were dispersed in 80% of the total required water and HRWRA was dissolved in remaining 20% of water. The mortar mixing was done in accordance with the British standard i.e., EN 196-1. The whole process involved the following steps:

• Measured quantity of ordinary Portland cement and 80% of water containing CNIs were put into the bowl of mortar mixer.

• Mixture of cement and water was mixed at low speed for 30s and remaining 20% water containing HRWRA was added.

• In next 30s the required amount of sand was added gradually at slow speed mixing. The speed of mixer was changed to high and mixing was continued for 30 more seconds.

• The mixer was then stopped for 90s. During this time the mix adhered to the sides was scrapped and brought to the middle of the bowl with help of rubber scrappers.

• After scrapping the sides mixing was done at high speed for 60s which resulted into the completion of mixing process.

3.2.3.4 Casting of samples

Fig 3.6 Casting of Controlled Samples

Immediately after the mixing of mortar it was poured into oiled molds for making desired samples. For prisms (40x40x160 mm3) freshly prepared mortar was poured into 3-gang molds in two layers. Each layer was given 60 jolts on mortar jolting machine for perfect compaction of composites. For tiles, mortar was poured into water tight oiled molds and compacted by hand for removing any type of voids. Both prisms and tiles were kept in molds for 24 hours at room temperature by covering with wet cotton clothes for avoiding any rapid moisture loss. Samples were then de-molded and stored in curing tank at room temperature i.e., 20 ±2◦C until the time of testing.

3.3 Mechanical Testing

Various tests were performed on mortar prisms to evaluate different mechanical properties of cementitious composites intruded with CNIs. Expansion and Contraction, Flexural strength, compressive strength and Density test were some of the characteristics investigated during this research.

(A) Test for Expansion & Contraction

This test method covers the determination of length changes of expansive cement mortar, while under restraint, due to the increasing of temperature according to ASTM designation C806-95

We Calculated the expansion or shrinkage at 28 days age as follows:

$$Ex = \frac{Lx - Li}{10 \times 250} \times 100 \qquad \text{Eq 3.1}$$

where:

Ex = expansion at x age, %,

Lx = comparator reading of specimen at x age – reference bar comparator reading, in. (mm), and

Li = initial comparator reading of restraining rod − reference bar comparator reading, in. (mm).

(B) Flexural Strength

Flexural strength was determined on the basis of three point bending test in accordance with ASTM C348-21 as shown in Fig 3.7. The loading rate was set to 0.05KN/s as mentioned in the code. The formula used for the calculation of flexural strength is as follows ;

$$\sigma_f = 3PL/2bd^2 \qquad \text{Eq 3.2}$$

Where,

σ_f = flexural strength,

MPa,P = maximum load, N,

L = length between the supports, mm,

b = width of the prism, mm and

d = depth of the prism, mm.

(C) Compressive Strength

Compressive strength was determined in compression testing machine in ac- cordance with ASTM C349-18 as shown in Fig 3.8. The loading rate was set to 1.8KN/s as mentioned in the code. The formula used for the calculation of compressive strength is as follows ;

$$\sigma_c = P/A \qquad \text{Eq 3.3}$$

Where,

σ_c = compressive strength,

MPa,P = maximum load, N,

A = contact area of sample, mm^2,

(D) Dry Density Test

(a) Sample Preparation

After the curing period, the concrete specimens were carefully demolded and allowed to air-dry to a constant weight.

(b) Dry Density Measurement

The dry density of each concrete specimen was determined using the following formula:

$$Dry\ Density(\rho d)= \frac{\text{Mass of OvenDried Specimen}}{\text{Volume of Specimen}} \qquad \text{Eq 3.4}$$

where:

- Mass of Oven-Dried Specimen Mass of Oven-Dried Specimen is the mass of the concrete specimen after oven-drying to a constant weight, and
- Volume of Specimen is the volume of the concrete specimen calculated using its dimensions.

3.4 Micro-structural analysis of cementitious composites

After all the investigation of mechanical properties and having Figd out theformations with enhanced strength and ductility, a micro-analysis of mortar sampleswith higher credentials was carried out. Scanning Electron Microscopy (SEM) was conducted in order to observe the micro-structure of cement matrix with CNMPs and to observe the reason of increment in strength.

Fig 3.7 Electromagnetic interference shielding testing setup

and ductility of composites. Alongsidewith SEM, energy dispersive x-ray spectroscopy was conducted at different spots ofthe mortar specimen induced with PBF and PPN for the confirmation of presence ofcarbonized particles uniformly throughout the matrix. The reactivity of PBF and PPN in cementitious composites was checked withthe help of x-ray diffraction analysis of mortar samples. XRD peaks of control samples and samples induced with pyrolyzed materials was compared to Fig out the formation of any chemical compound due to addition of PBF and PPN.

Chapter 4

RESULTS AND DISCUSSION

4.1 General

In this section the results of different tests performed during the research arepresented and discussed in detail. The results are broadly divided into two sections i.e., Characterization of Bio-char and Enhanced Mechanical properties of cementitious composites. Characterization of bio-char involves thermogravimetric analysis (TGA), scanning electron microscopy(SEM) and particle size distribution. Whereas, the mechanical properties of cementitious composites involve compressive strength, flexural strength and Electromagnetic Interference Shielding Effectiveness (EMI-SE).

4.2 Characterization of Bio-Char

Before usage in cementitious composites the biochar was first characterized with the help of various techniques to investigate its suitability and confirm the achievement of our objectives i.e. production of micro/nano sized carbonized inert.

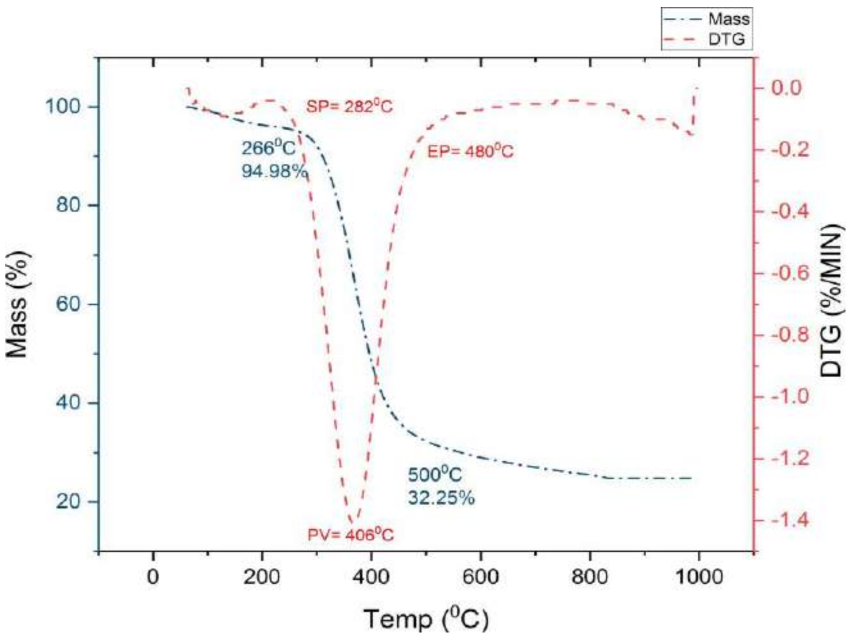
4.2.1 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was performed to analyze the decomposition regime of raw agro-wastes utilized for the production of carbonized nano inert and also to investigate the amount of carbon content. TGA was executed in two different atmospheres i.e. in the presence of oxygen and in the absence of oxygen.

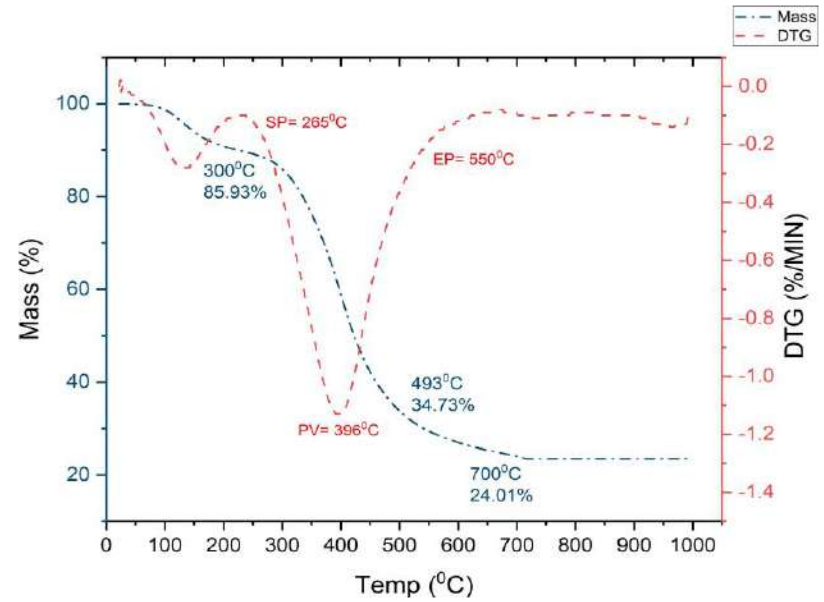
(A)Thermogravimetric Analysis in the absence of Oxygen

TGA in the absence of oxygen was conducted in order to pick up a temperature at which pyrolysis of agro-wastes can be accomplished. The results of TGA in N_2 atmosphere are presented in the form of graphs in Fig 4.1. Thermogravimetric (TG) and differential thermogravimetric (DTG) curves of bagasse fibers in N_2 atmosphereare shown in Fig 4.1a. TG curve of bagasse in the absence of O_2 revealed that initially 6-7% mass was reduced upto a temperature of 266°C which might be referred to some moisture loss. 68% mass was reduced at 500˚C and no considerable mass was lost beyond 500˚C which implies to the complete decomposition of bagasse fibers. Moreover, the DTG curve of bagasse in N_2 atmosphere depicted that the maximum mass loss was occurred within the temperature range of 282˚C (SP) to 480˚C (EP) and the temperature at which the rate of mass loss was maximum was 406˚C as shown in Fig 4.1a.

Thermogravimetric (TG) and differential thermogravimetric (DTG) curves of pine needles in N_2 atmosphere are shown in Fig 4.1b. TG curve of pine needles in the absence of O_2 revealed that initially 15-16% mass was reduced up to a temperature of 300˚C which might be referred to some moisture loss. 76% mass was reduced at 700˚C and no considerable mass was lost beyond 700˚C which implies to the complete decomposition of pine needles. Moreover, the DTG curve of pine needlesin N_2 atmosphere depicted that the maximum mass loss was occurred within the temperature range of 265˚C (SP) to 550˚C (EP) and the temperature at which the rate of mass loss was maximum was 396˚C as shown in Fig 4.1b. So, according to TGA in the absence of oxygen shown in Fig 4.1 the finalzed temperature for the cabonization of bagasse fibers and pine needles came out tobe 500˚C and 700˚C respectively.



(a) TG and DTG of Bagasse Fibers



(b) TG and DTG of Pine Needles

Fig 4.1 TG and DTG in N_2 atmosphere (a)Bagasse Fibers (b)Pine Needles

(B) Thermogravimetric Analysis in the presence of Oxygen

TGA in the presence of oxygen was conducted in order to Fig out the amount of carbon present in agro-waste material. The results of TGA in O_2 atmosphere are presented in the form of graphs in Fig 4.2. In light of the TG curves of bagasse fibers and pine needles it can be concluded that the amount of carbon contentin bagasse and pine needles is 87.12% and 82.03% respectively.

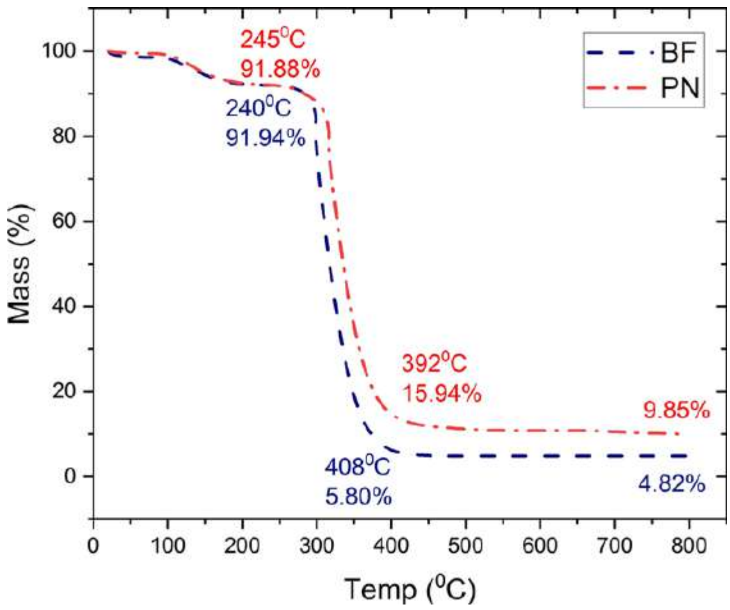
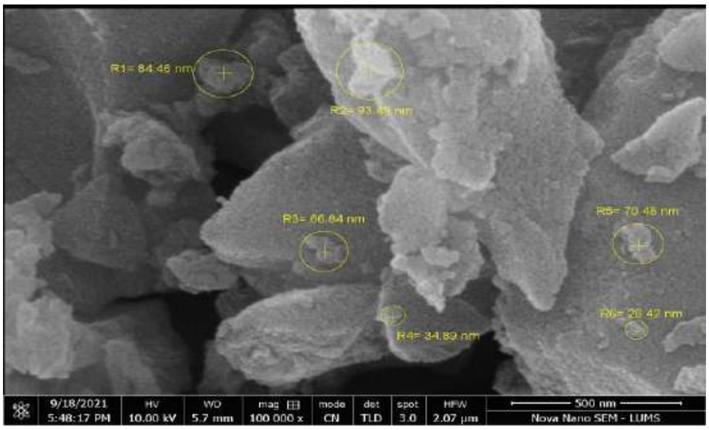


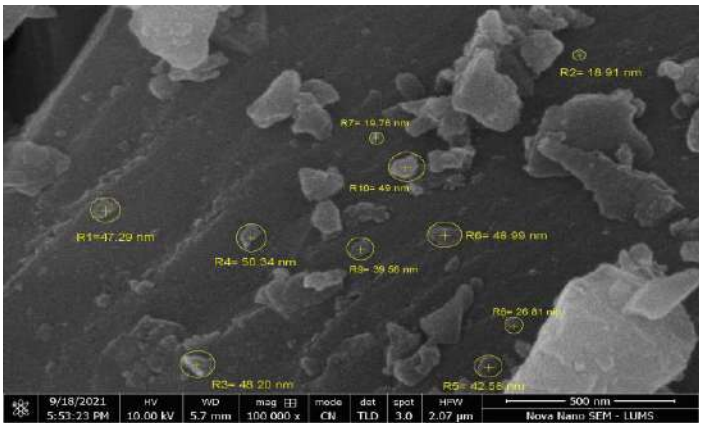
Fig 4.2 TGA of BF and PN in Oxygen atmosphere

4.2.2 Scanning Electron Microscopy of Bio-char

Scanning electron microscopy of pyrolized bagasse fiber and pine needles revealed that both the pyrolyzed products have nano and micro sized particles with irregular angular shapes and rough texture as shown in Fig 4.4. The minimum particle size of grounded PBF observed in SEM images at 100,000x magnification is26.42nm as shown in Fig 4.4a. Similarly, the minimum particle size of groundedPPN observed in SEM images at 100,000x magnification is 18.91nm as shown in Fig 4.4b. The rough texture and irregular shapes of particle are helpful for makinga stronger bond with cementitious composites These micro-analysis results confirmed the achievement of one of our goals i.e., grinding of pyrolyzed media to nano sized particles.



(a) SEM of PBF

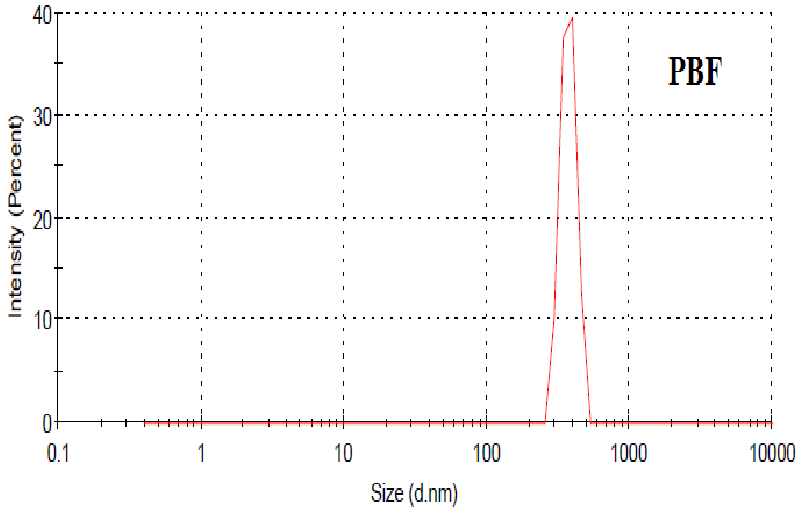


(b) SEM of PPN

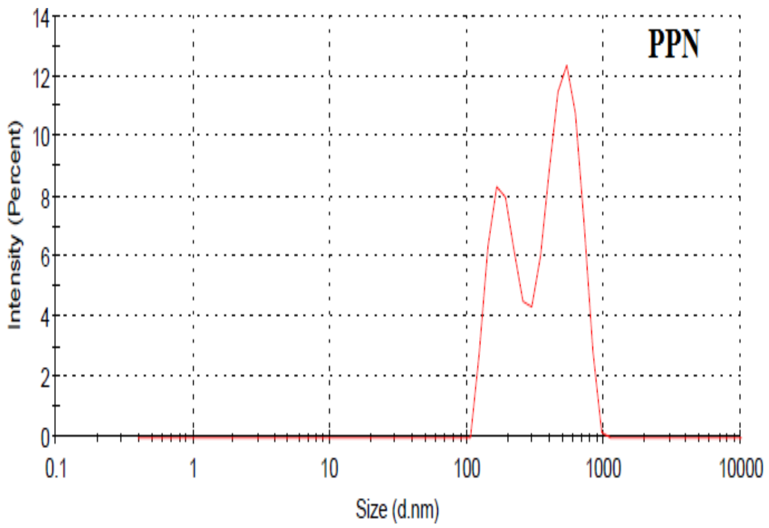
Fig 4.3 Scanning electron microscopy of bio-char

4.2.3 Particle Size Distribution

The particle size distribution of PBF and PPN revealed the range of the size present in a bulk as presented in Fig 4.7. It can be observed from that the most ofthe particles of PBF and PPN lies within the range of 100 nm to 1000 nm. In case ofPBF the range of the particle size was observed to be 250-550 nm as shown in Fig 4.7a. Whereas in case of PPN the major representation of particles was in the range of 100-1000 nm 4.7b



(a) PSD of PBF



(b) PSD of PPN

Fig 4.4 Particle Size Distribution Curves

4.3 Mechanical Evaluation of Cementitious Composites

After the characterization of bio-char and confirmation of its suitability tobe used in cementitious composites it was used in cement mortar and mechanical testing was carried out. The mechanical evaluation involved compressive strength, flexural strength , Expansion & Contraction and Density Test.

4.3.1 Compressive Strength

The results of compressive strength tests on cement mortar samples having various percentages of carbon nano inerts of bagasse fibers and pine needles at various ages are displayed in Figs 4.10 and 4.11. It is evident from the results that the peak values of compressive strength at 28-days are in line with the results of ultrasonic pulse velocity.

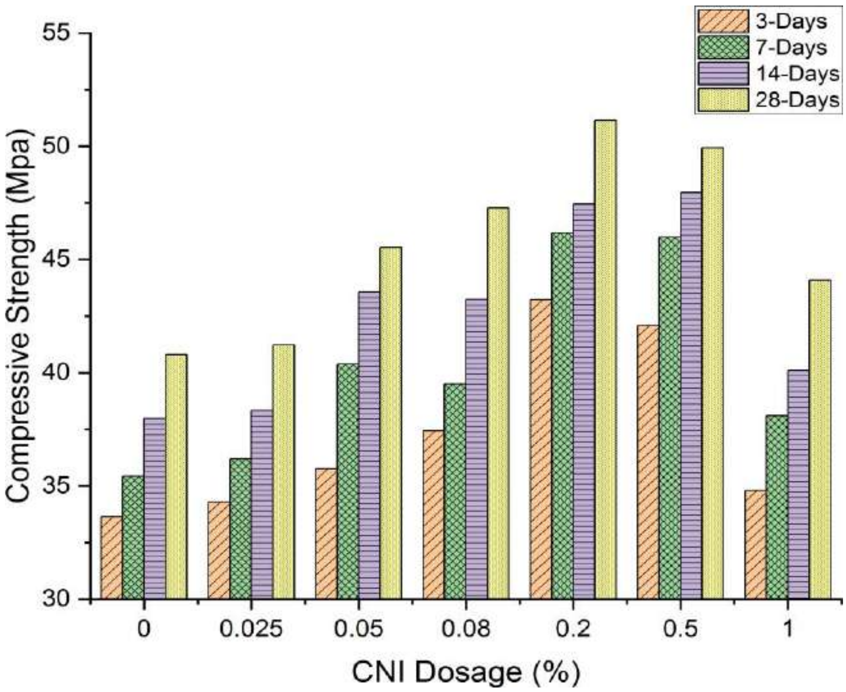
Mortar specimen with PBF showed maximum 28-days strength at 0.2% dosage whereas samples with PPN showed maximum 28-days strength at 1% addition. An increment of 25.36% and 23.84% as compared to control was observed by 0.2% CM-PBF and 1% CM-PPN respectively, as shown in Fig 4.11. The improved strength due to addition of CNIs as compared to control samples was most probably due to the refinement of micro-structure of cement mortar. The micro-structural refinement might be due to pore filling by nano/micro sized carbon particles. Furthermore, the strength increment could also be attributed to crack prevention because of PBF and PPN because carbon nano particles have the tendency to either stop the crack or delay its propagation.

Table 4.1 Compressive Strength at 28-days

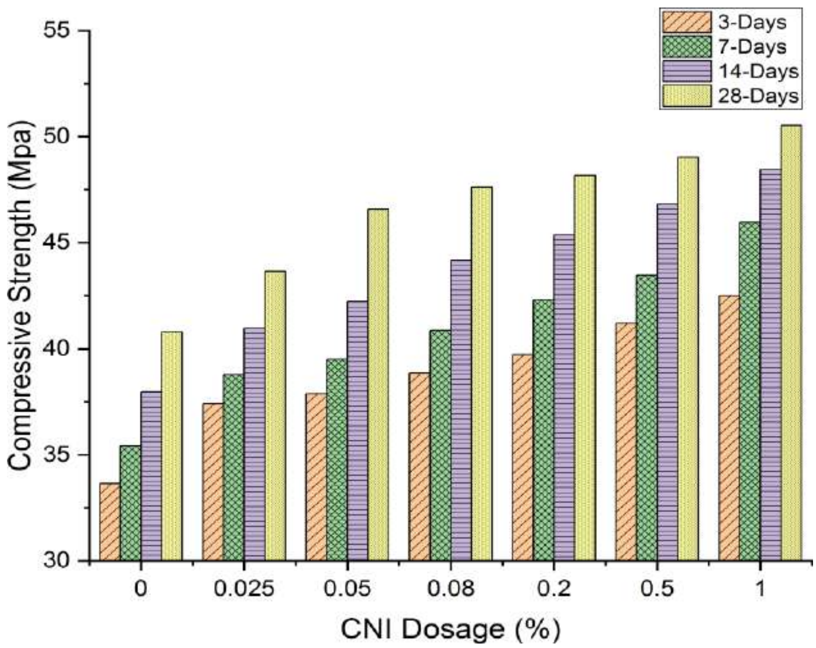
Formulation	Compressive Strength (MPa)	Formulation	Compressive Strength (MPa)
CM	40.7	CM	40.7
CM-0.025%PBF	41.22	CM-0.025%PPN	43.63
CM-0.05%PBF	45.5	CM-0.05%PPN	46.57
CM-0.08%PBF	47.25	CM-0.08%PPN	47.63
CM-0.2%PBF	51.14	CM-0.2%PPN	48.17
CM-0.5%PBF	49.92	CM-0.5%PPN	49.04
CM-1%PBF	44.09	CM-1%PPN	50.52

The strength development at 3,7,14 and 28 days of control mortar samples and samples with different dosages are shown in Fig 4.10. The strength development CM-PBF and CM-PPN with respect to time can be seen in Fig 4.10a and 4.10b respectively. In both CM-PBF and CM-PPN the compressive strength has gradually increased with passage of time for each formulation. The strength development withtime can be attributed to the hydration process of cement paste during the curing period.

The results of flexural strength tests on cement mortar samples having various percentages of carbon nano inerts of bagasse fibers and pine needles at various ages



(a) Compressive strength of CM-PBF at 3,7,14 and 28 days



(b) Compressive strength of CM-PPN at 3,7,14 and 28 days

Fig 4.5 Compressive strength of cementitious composites with different CNIs dosages

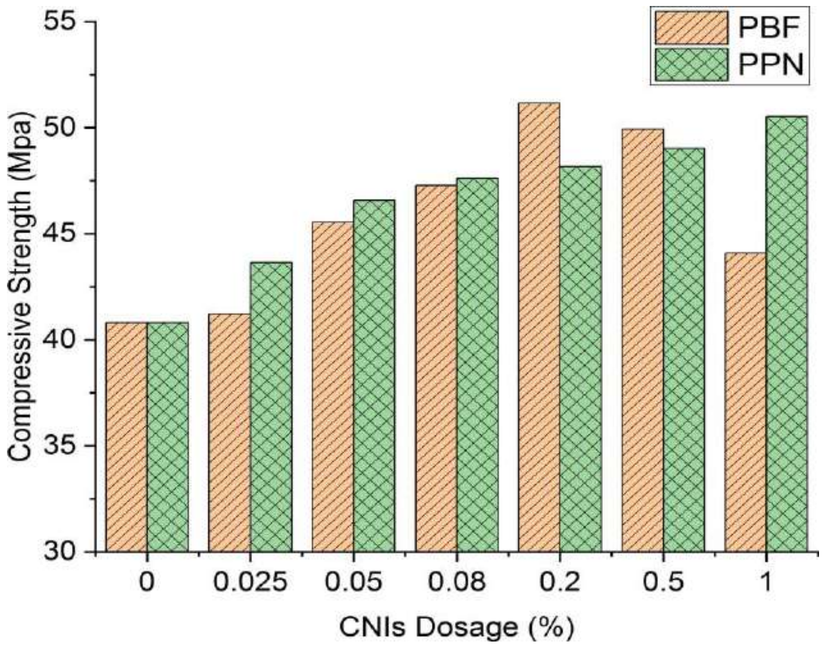


Fig 4.6 Compressive strength at 28-days

are displayed in Figs 4.12 and 4.13. The trend in strength development with age and the formulations with peak flexural strength values are very similar to that of compressive strength discussed earlier.

Mortar specimen with PBF showed maximum 28-days strength at 0.2% dosage whereas samples with PPN showed maximum 28-days strength at 1% addition. An increment of 15.61% and 15.83% as compared to control was observed by 0.2% CM-PBF and 1% CM-PPN respectively, as shown in Fig 4.12. Strength improvement in mortar samples with CNIs as compared to control samples was most probably due to the crack bridging, crack contouring and crack branching because of the presence of PBF and PPN. Presence of carbon nano/micro particle at any location divides a major crack into two or three cracks thereby dissipating the energy and delaying its growth.In crack contouring the crack contour around a carbonized particle and follows a long route, thus requires more energy to propagate. In some cases crack may pass straight from a location but carbonized particle has the tendency to keep the two cracked parts together by act as a bridge or pin between them. So, all these mechanisms either stops

the crack growth at nano level or delay its propagation thereby increasing the flexure strength of cement mortar up to some extent.

The strength development at 3,7,14 and 28 days of control mortar samples and samples with different dosages are shown in Fig 4.13. The strength development CM-PBF and CM-

PPN with respect to time can be seen in Fig 4.13a and 4.13b respectively. In both CM-PBF and CM-PPN the flexural strength has graduallyincreased with passage of time for each formulation. The strength development withtime can be attributed to the hydration process of cement paste during the curing period.

4.3.2 Flexural Strength

The results of flexural strength tests on cement mortar samples having various percentages of carbon nano inerts of bagasse fibers and pine needles at various ages.

The decrement in strength at some higher dosages compared to their corresponding lower dosages might be due to the creation of weaker zones because of theagglomeration of carbonized particle. These weaker zones might have facilitated theeasy passage of crack thereby decreasing the strength to some extent.

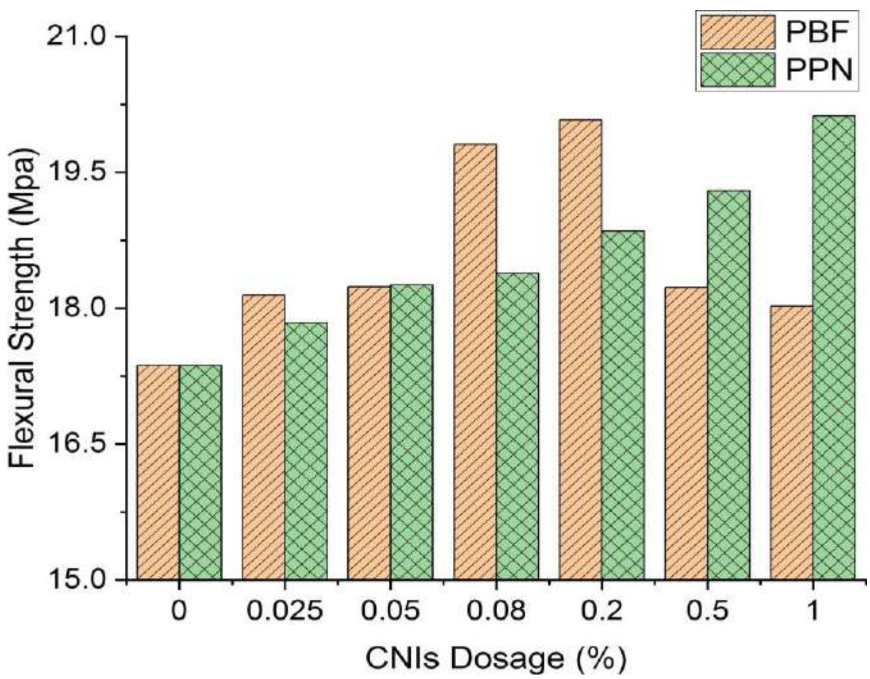
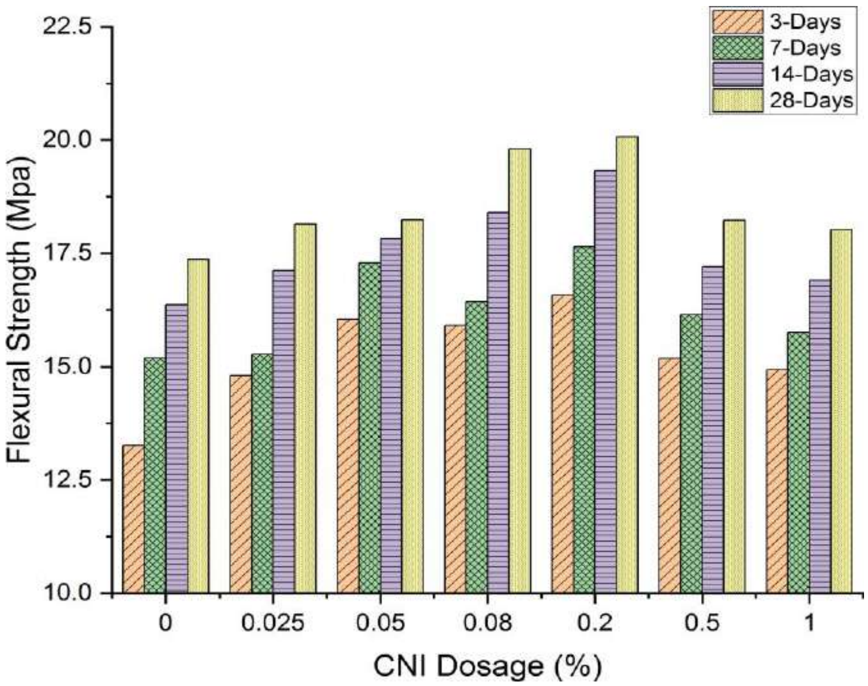
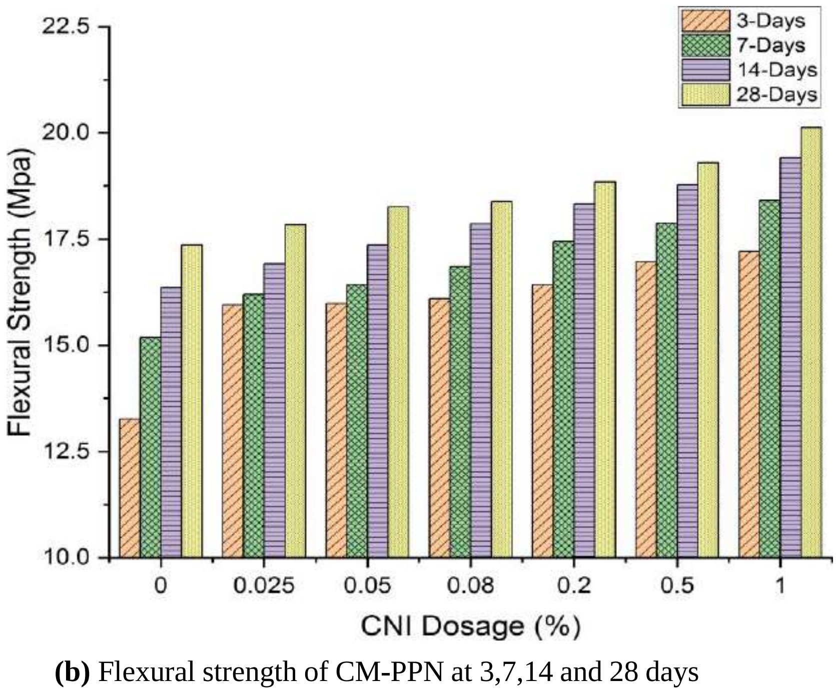


Fig 4.7 Flexural strength at 28-days

The strength development at 3,7,14 and 28 days of control mortar samples and samples with different dosages are shown in Fig 4.13. The strength development CM-PBF and CM-PPN with respect to time can be seen in Fig 4.13a and 4.13b respectively. The strength development withtime can be attributed to the hydration process of cement paste during the curing period.



(a) Flexural strength of CM-PBF at 3,7,14 and 28 days



(b) Flexural strength of CM-PPN at 3,7,14 and 28 days

Fig 4.8 Flexural strength of cementitious composites with different CNIs dosages at different ages

Table 4.2 Flexural Strength at 28-days

Formulation	Flexural Strength (MPa)	Formulation	Flexural Strength (MPa)
CM	17.36	CM	17.36
CM-0.025%PBF	18.14	CM-0.025%PPN	17.84
CM-0.05%PBF	18.23	CM-0.05%PPN	18.25
CM-0.08%PBF	19.8	CM-0.08%PPN	18.38
CM-0.2%PBF	20.07	CM-0.2%PPN	18.84
CM-0.5%PBF	18.22	CM-0.5%PPN	19.29
CM-1%PBF	18.02	CM-1%PPN	20.11

4.3.3 Expansion & Contraction Test

The mortar samples after 28 days strength was placed in oven for 24 hours at 100 ℃ to check the effect of biochar used in concrete on expansion.

Table 4.3 Percentage expansion at 100 ℃

Carbonized Nano Inerts %age	Initial Reading(mm)	After Heating at 100℃	Expansion (mm)	Percentage
0%	9.881	10.101	0.22	0.087%
0.025%	9.744	9.98	0.236	0.073%
0.05%	10.108	10.201	0.093	0.037%
0.08%	9.813	9.895	0.083	0.033%
0.20%	9.712	9.78	0.068	0.027%
0.5%	9.955	10.03	0.075	0.022%
1%	10.041	10.056	0.015	0.006%

Concrete expansion, often referred to as alkali-silica reaction (ASR), is a chemical reaction that occurs between alkaline substances in the concrete (e.g., sodium and potassium) and certain types of reactive silica present in aggregates. This reaction can cause the concrete to crack and degrade over time, compromising its structural integrity.

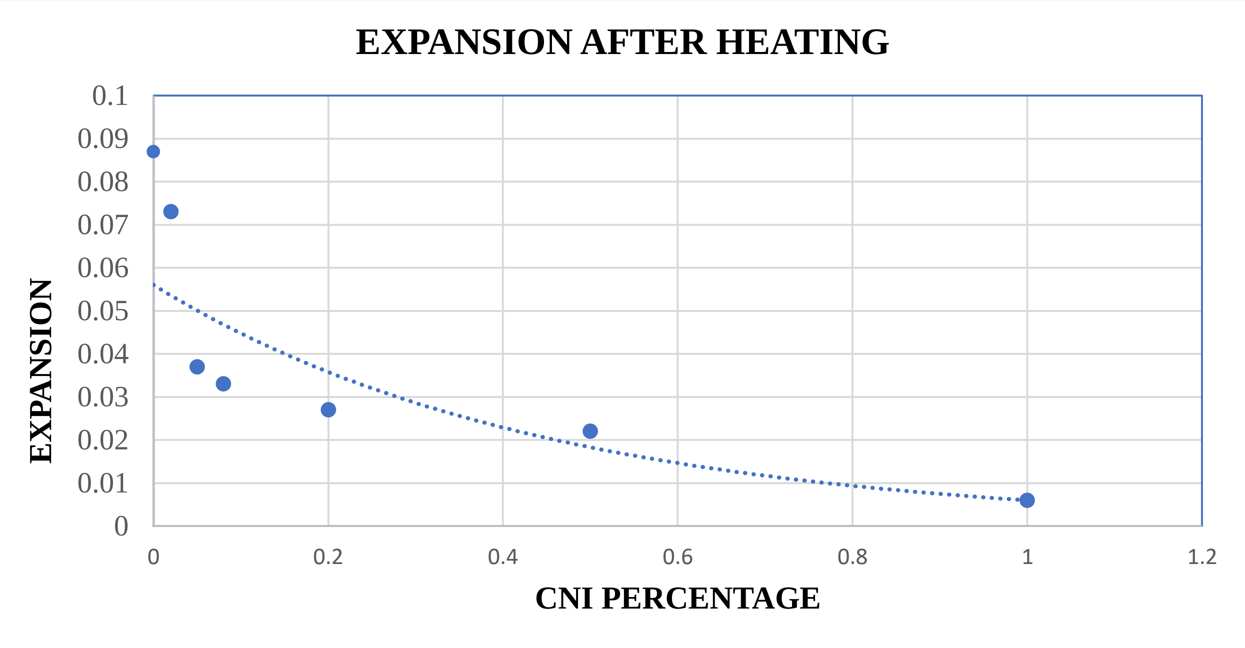


Fig 4.9 Expansion at different dosages of Biochar

Fig 4.9 shows that with initial dose of CNI rate of decrease of expansion is higher. While with high dose of CNI rate of decrease of expansion is low.

The potential benefits of incorporating biochar of bagasse and pine into concrete to reduce expansion are attributed to its unique properties:

1. **Pozzolanic Activity**:

Biochar of bagasse fiber and pine needles contains amorphous carbon and reactive components that exhibit pozzolanic activity. Pozzolanic materials react with calcium hydroxide in the presence of moisture to form additional cementitious compounds, improving the strength and durability of concrete.

2. **Dilution of Reactive Aggregates**:

By incorporating biochar into the concrete mix, it can dilute the content of reactive aggregates, such as certain types of silica, that are prone to the alkali-silica reaction. This reduction in the concentration of reactive silica can help mitigate expansion.

3. **Physical Barrier**:

Biochar can act as a physical barrier, hindering the migration of alkaline ions within the concrete. This can help reduce the likelihood of these ions coming into contact with reactive silica, thereby minimizing expansion.

4. **Water Absorption**:

Biochar has a high surface area and can absorb water. By absorbing excess moisture within the concrete, biochar may help in controlling the availability of water for the alkali-silica reaction.

Here the question arises that why concrete expands? There are several reasons why concrete can expand. The primary reason is due to the presence of moisture. When moisture is present, it can cause the concrete to absorb water. As the water evaporates, the concrete will shrink.

However, if the concrete was already at its maximum shrinkage level and there remains water present, the concrete can begin to expand. Additionally, the heat generated during cement hydration can also cause the concrete to expand

4.3.3.1 Types of Concrete Expansion

There are two main types of concrete expansion: drying shrinkage and thermal expansion. Drying shrinkage is caused by the evaporation of water within the concrete. As the water leaves the pores within the concrete, the material will shrink, which can result in cracks.

The second type, thermal expansion, is due to exposure to temperature changes. As temperatures increase, the particles within the concrete will expand, leading to possible cracks and movements.

4.3.3.2 How to control Concrete Expansion?

We can control Concrete thermal expansion by using biochar of pine needles and bagasse fiber as primary additive. This can be said by using the knowledge gained from data above that with increasing the dosage of biochar the value of concrete thermal expansion decreases.

4.3.3.3 Contraction Test

Similarly, after performing thermal expansion test mortar samples were cooled at room temperature and again measured so that if any change in length occurred can be measured. The graph below shows the gauge readings before freezing.

Table 4.4 Percentage Contraction at -20℃

Carbonized Nano Inerts %age	Initial Reading(mm)	After Freezing at -20℃	Contraction (mm)	Percentage
0%	9.891	9.623	0.268	0.105%
0.025%	9.748	9.538	0.210	0.077%
0.05%	10.131	9.998	0.133	0.06%
0.08%	9.822	9.731	0.091	0.055%
0.20%	9.727	9.672	0.055	0.038%
0.5%	9.963	9.925	0.038	0.015%
1%	10.025	10.012	0.013	0.005%

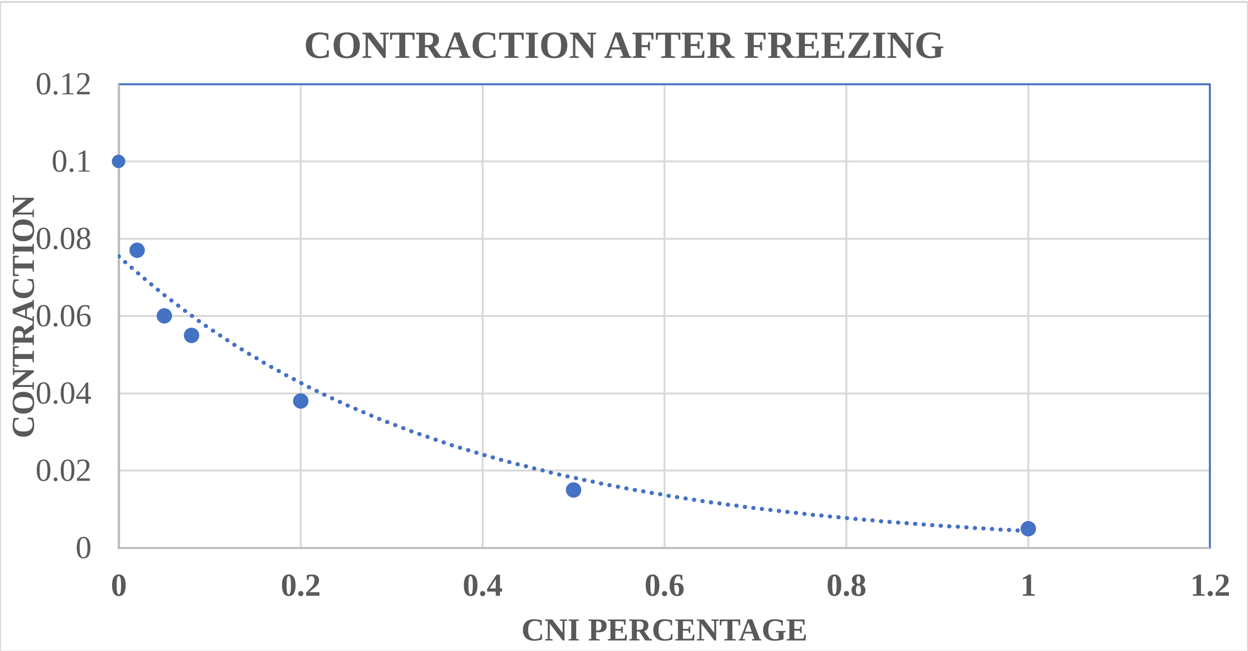


Fig 4.10 Freezing at -20℃ Temp

After 24 hours of freezing at -20℃ the samples were removed from freezer and measured again.

With initial dose of CNI the rate of contraction is higher & with higher dose of CNI the rate of decrease of contraction reduces this is due to carbon rich behavior of biochar produced from bagasse fiber and pine needles through pyrolysis . Concrete can experience contraction or cracking after freezing at low temperatures due to the expansion of water during freezing and subsequent contraction upon thawing. Biochar may help mitigate these issues through several mechanisms:

1. **Pore Structure Modification**

Incorporating biochar into the concrete mix can alter the pore structure, leading to a more refined and interconnected pore network. This modified pore structure can reduce the volume available for water to expand during freezing, thereby reducing the potential for internal stresses and cracking upon thawing.

2. **Water Absorption and Retention**

Biochar's porous structure allows it to absorb and retain water. During freeze-thaw cycles, this water retention can help maintain a more stable internal moisture level in the concrete, reducing the potential for excessive expansion and contraction that may cause damage.

3. **Insulation Properties**

Biochar has some insulative properties due to its carbon-rich structure. This can help moderate the temperature changes within the concrete during freeze-thaw cycles, minimizing the extreme temperature variations that could exacerbate contraction and cracking.

4. **Minimizing Ice Crystal Formation**

Biochar may interfere with ice crystal formation within the concrete by providing nucleation sites that encourage smaller, more evenly distributed ice crystals. This can reduce the pressure exerted on the concrete matrix, mitigating potential damage.

5. **Enhanced Mechanical Properties**

The addition of biochar can enhance the mechanical properties of concrete, such as strength and toughness. A more robust concrete structure can better withstand the stresses associated with freeze-thaw cycles, reducing the likelihood of contraction and cracking.

4.4 Density Test

Density test was carried out after putting the samples in oven for 24 hours at 100℃ temperature following table 4.4 shows the dry density of all mortar samples at different dosages of biochar.

The density tests were conducted in triplicate for each concrete mixture composition, and the average density was calculated. Data obtained from the tests were analyzed statistically to determine any significant variations in density due to the inclusion of biochar derived from bagasse fiber and pine needles. Graph below shows the data obtained from density test.

Table 4.5 Density at different dosages of biochar

Formulation		
Biochar PPN-PBF		Density(kg/ft3)
CM		67.6765
CM-0.025%		67.5265
CM-0.05%		67.417
CM-0.08%		67.22
CM-0.2%		66.3435
CM-0.5%		66.107
CM-1%		65.5155

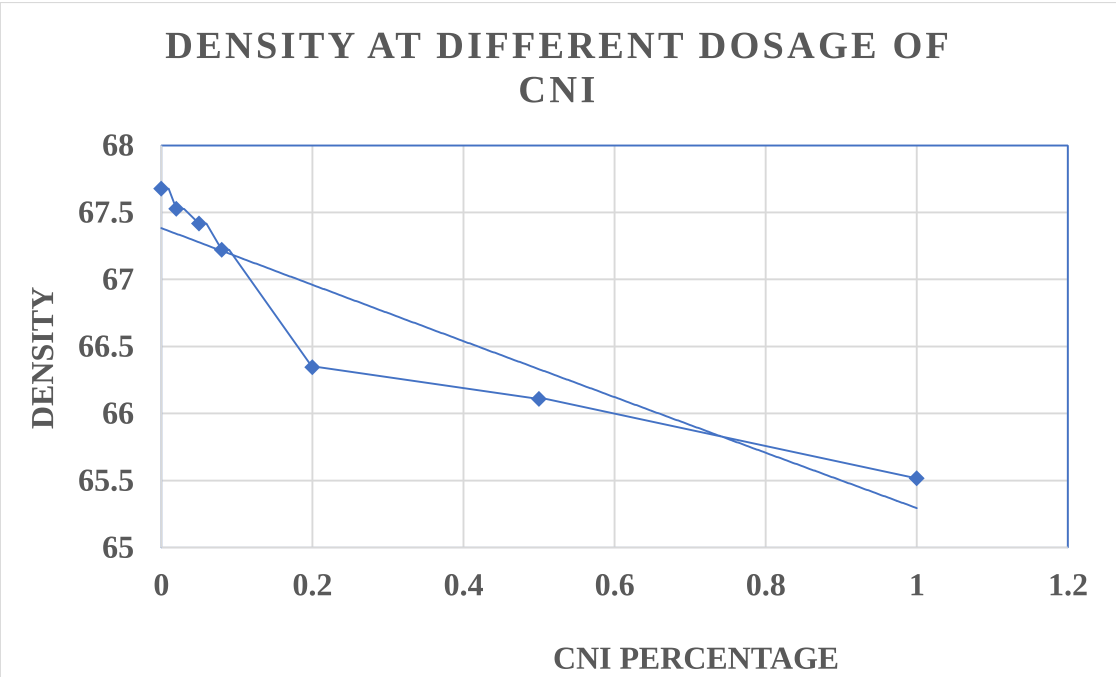


Fig 4.11 Density of concrete mortars at different %ages of biochar

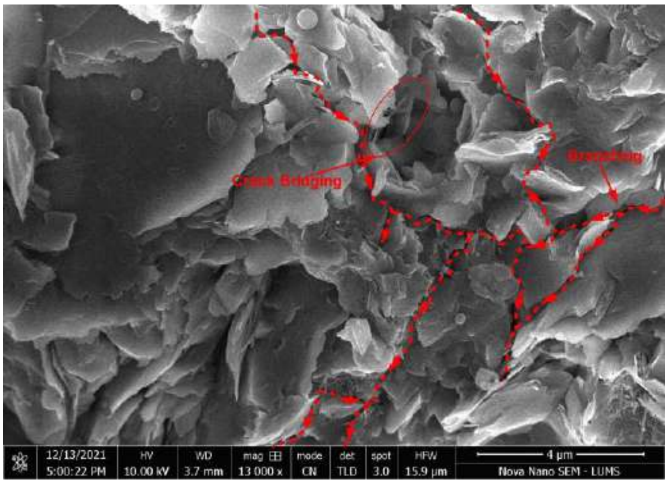
We can clearly see that with increasing the percentage of biochar the density of concrete is decreasing this is due to biochar obtained from bagasse fiber and pine needles is generally lightweight and porous, which means that when added to a concrete mixture, it is likely to decrease the density of the concrete. Pine needle and bagasse fiber biochar typically has a lower bulk density compared to traditional concrete components like cement, sand, and aggregates.

Similar to other lightweight biochar materials, the addition of pine needle and bagasse fiber biochar to a concrete mix displaces some of the heavier components, resulting in a lower overall density for the concrete mixture.

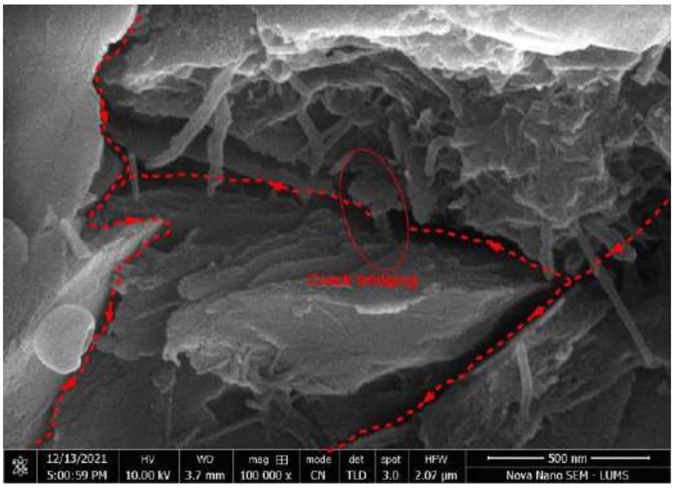
4.5 Microscopic Analysis of Cementitious composites

The microscopic images of fractured cementitious composites have revealed the reasons behind the enhancement of mechanical properties i.e., enhanced strength, ductility and fracture toughness. SEM micro-graphs showed crack bridging, crack branching and crack contouring mechanisms in cementitious matrix. These crack resisting phenomena are considered as the major cause of improvement in mechanical properties as discussed in previous sections. It is evident from Fig 4.16 that at some locations crack either got divided into branches or contoured around the carbonized particles. In both the cases the fracture response of the matrix gets enhanced by eitherdissipating energy while branching or taking long propagation path due to contouringaround the carbonized particles.

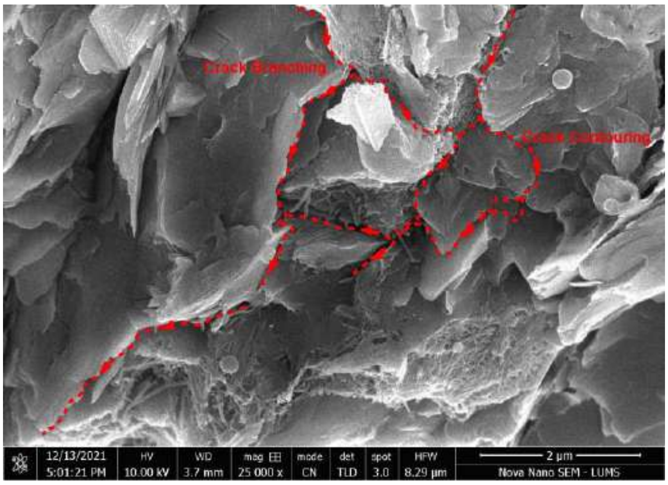
The inertness of carbonized nano particles of bagasse fiber and pine needles in cementitious composites was analyzed with the help of x-ray diffraction analysis.It can be observed from Fig 4.17 that the major portion of cement mortar induced with PBF and PPN is composed of calcium silicates, calcium silicate hydrates and tricalcium aluminates, that are the major hydration constituents in ordinary cement sand mortar. On comparison of cement mortar samples induced with PBF and PPN with control samples, it was evident that there was no significant alteration in chemical and crystalline composition of mortar due to intrusion of pyrolyzed nano particles. So, it can be concluded that the PNMPs of bagasse and pine needles behaved as inert material in cementitious composites without showing any reactivity with the constituents of cement mortar.



(a)



(b)



(c)

Fig 4.12 SEM micro-graphs of fracture surfaces

Chapter 5

CONCLUSIONS AND FUTURE PERSPECTIVES

5.1 Conclusions

The investigation into the impact of various dosages of bagasse fiber and pine needles biochar on the expansion and contraction of cementitious composites has yielded significant findings. The results consistently demonstrate a reduction in both expansion and contraction characteristics of the cementitious composites with increasing dosages of these materials.. The important outcomes of this research are as follows;

- Both the bagasse and pine needles have the potential to be transformed into micro/nano carbonaceous inert with carbon content of 86.06% and 85.91% respectively. Use of bio-char not only influence the properties of cementitious composites but also reduces the environmental issues associated with them.
- At dosage levels of 0.025% to 0.5% for both bagasse fiber and pine needles biochar, a noticeable decrease in expansion and contraction has been observed. This reduction is attributed to the reinforcing and stabilizing effects of these additives within the cementitious matrix. The improvement in dimensional stability suggests that these materials can be effectively used to enhance the performance of cementitious composites.
- Thermal Expansion and contraction of cementitious composites were reduced by the incorporation of pyrolized bagasse fiber and pine needles.
- Reducing the thermal expansion and contraction will help us to reduce the use of expansion and contraction joints. These joints in buildings are filled with filler but after some time of construction there starts leakage. So, by using pyrolized bagasse fiber and pine needles in concrete we can control expansion and contraction.
- Density of cementitious composites induced with pyrolized bagasse fiber and pine needles were calculated which showed decrease in density.

5.2 Future Perspectives

Based on the outcomes of this research, the following recommendations can be made:

Optimal Dosage Selection:

Further research should explore the exact dosage of bagasse fiber and pine needles biochar that provides the best balance between reduced expansion and contraction and cost-effectiveness. This would enable more precise recommendations for practical applications.

Performance in Real-world Scenarios:

Extensive field tests and evaluations in real construction projects should be conducted to validate the effectiveness of bagasse fiber and pine needles biochar in enhancing the durability of cementitious composites. These tests should consider various environmental and load conditions.

Environmental Impact:

Assess the environmental impact of using these materials, including the sustainability of sourcing bagasse fiber and pine needles biochar, and whether they align with eco-friendly construction practices.

Standardization and Guidelines:

Collaborate with industry and regulatory bodies to establish standards and guidelines for incorporating bagasse fiber and pine needles biochar in cementitious composites, ensuring safe and consistent application.

Economic Feasibility:

Investigate the economic feasibility of large-scale production and utilization of these materials, taking into account cost, availability, and market competitiveness.

In conclusion, the reduction in expansion and contraction observed by incorporating bagasse fiber and pine needles biochar in cementitious composites presents a promising avenue for improving the performance and longevity of construction materials. Further research and practical implementation are essential to harness the full potential of these materials for the construction industry.

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