

DEVELOPMENT OF GEOPOLYMER COMPOSITES INTEGRATED WITH PHASE CHANGE MATERIAL FOR THERMAL ENERGY STORAGE APPLICATIONS

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Engineering degree in
Mechanical Engineering

By

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DEPARTMENT OF MECHANICAL ENGINEERING

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SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with grade "A++" by NAAC

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of Mugilan M (Reg. No. 40150027) and Gopinath G (Reg. No. 40150008) who carried out the project entitled **"DEVELOPMENT OF GEOPOLYMER COMPOSITES INTEGRATED WITH PHASE CHANGE MATERIAL FOR THERMAL ENERGY STORAGE APPLICATIONS"** under our supervision from December 2023 to April 2024.

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ABSTRACT

Innovative materials and systems are the focus of study due to the growing need for sustainable and effective thermal energy storage (TES) solutions. One viable approach to meeting this demand is through the integration of phase change materials (PCMs) with geopolymer composites. Geopolymers are made from natural minerals or industrial waste products and have good mechanical and chemical stability as well as minimal environmental impact. PCMs, on the other hand, are excellent choices for TES applications because of their capacity to store and release significant amounts of thermal energy during phase transitions. The creation and characterisation of geopolymer composites combined with PCMs for TES applications are examined in this work. PCM particles are dispersed throughout the geopolymer matrix during the production process to create a composite material that has the latent heat of fusion for thermal energy storage. During the manufacturing process, PCM particles are distributed throughout the geopolymer matrix to produce a composite material with the latent heat of fusion for thermal energy storage.

The structural, morphological, and thermal properties of the composites are examined using a variety of methods, including differential scanning calorimetry (DSC), scanning electron microscopy (SEM), X-ray diffraction (XRD), and thermal conductivity tests. The objective of this study is to improve the thermal performance, stability, and durability of the geopolymer composite PCM systems by optimizing the composition and processing parameters. These materials have the potential to be used in waste heat recovery, solar thermal energy storage, and building insulation, all of which promote environmental sustainability and energy efficiency. The results of this study have the potential to greatly influence the creation of cutting-edge TES technologies with enhanced functionality and cost-effectiveness.

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LIST OF ABBREVIATIONS

Co ₂	Carbon dioxide
PCM	Phase change material
TES	Thermal energy storage
GHGs	Greatest greenhouse gases
GGBFS	Granulated blast furnace slag
FA	Fly Ash
MK	Metakaolin
NaOH and KOH	Sodium hydroxide, and Potassium hydroxide
Na ₂ SiO ₃	Sodium silicate
OPC	Portland cement concrete
MPCM	Microencapsulated phase change materials
SEM	Scanning electron microscopy
XRD	X-ray diffraction
FT-IR	Fourier transform infrared spectroscopy
GPC	Geopolymer concrete
OPC	Ordinary Portland cement
AAS	Alkali Activator Solution
FFA	Free Fatty Acids
FABGC	Fly ash-based geopolymer concrete

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

This chapter describes the introduction of energies and emissions in the construction industry as well as thermal energy storage. Energy is an important factor in determining the world society's success within the regions such as technical advancement, environmental protection, and economic growth. The ongoing depletion of non-renewable energy supplies leads to the primary problem associated with global warming. With rising living standards and technological advancements, power demand is increasing every day of their life, resulting in excessive fossil fuel usage. Minimizing energy use can help to minimize pollution while also having a positive impact on the energy market [1].

In 2015, The construction industry was responsible for huge energy consumption, estimated at 40% globally, with residential buildings consuming 27% of energy and emitting 17% of CO₂ emissions into the environment [1]. The environment is greatly affected by the construction industry. This is mainly because the life cycle of buildings (such as construction, design, demolition, operation and waste disposal) contributes a significant portion to CO₂ emissions. The majority of CO₂ emissions come mainly from the production of construction materials as well as electricity to bring modern facility to buildings.

Energy consumption of buildings is expected to increase by approximately 48% between 2012 and 2040 [2]. Due to rapid population growth, people's living standards are increasing and the need for heating and air conditioning for thermal comfort is increasing. But in 2018, China (+1.9%), the United States (+2.7%), and India (+5.5%) noticed among the largest increases in greenhouse gas (GHG) emissions [2]. In 2022, the overall emission in China (26%), the United States (US) (13%), the European Union (EU) (above 8%), India (7%), the Russian Federation (5%), and Japan (close to 3%) are the six countries that generate the greatest greenhouse gases (GHGs), collectively making up 62% of the world's emissions[3].

Thermal energy storage (TES) has become widely recognized as a viable option to achieve a low-carbon future within the past ten years. In reality, it is widely accepted that phase-change materials (PCM) may be used to improve the TES for thermal comfort [4]. It is recognized that PCM has the potential to transform the field of sustainable infrastructure development and that it is a viable solution for the built environment.

1.2 GEOPOLYMER

Joseph Davidovitz first used the word "geopolymer" in 1978. An aluminosilicate substance and an alkaline activator undergo a chemical process to generate an inorganic polymer known as a geopolymer. In contrast to Portland cement, which uses limestone and clay as the primary raw material, geopolymers use natural materials (rich in silicon and aluminium) or industrial byproducts [3].

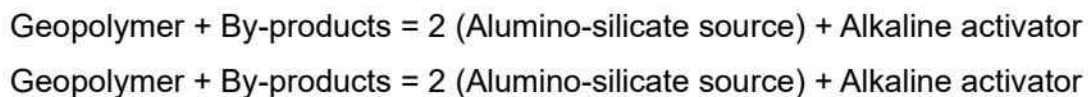
An eco-friendly substitute for Portland cement concrete, another cutting-edge building material, is geopolymer concrete. Geopolymer cement is created by dissolving aluminosilicate materials in a strong alkaline solution. The resulting paste can be mixed with aggregate and allowed to harden to form concrete with strength and elastic properties similar to portland cement concrete. The geopolymer has high initial strength due to its very short curing time. It hardens quickly at room temperature, providing mechanical strength and excellent durability. Traditional concrete has been replaced by alkali-activated binder/geopolymer cement and concrete in recent times.

Alkalizing aluminosilicate raw materials with a variety of base materials—such as metakaolin (MK), fly ash (FA), granulated blast furnace slag (GGBFS), etc.—is necessary to create geopolymers. Sodium silicate (water glass) or sodium silicate (Na_2SiO_3), sodium hydroxide, and potassium hydroxide are frequently included in the alkaline solution needed to make geopolymers (NaOH and KOH) [4].

1.3 GEOPOLYMER CHEMISTRY

The study of inorganic polymers, or geopolymers materials created by polymerizing precursors of aluminosilicate is known as geopolymer chemistry. Geopolymers, in contrast to conventional polymers, are made of inorganic substances, primarily silicon, oxygen, and aluminum. Although the chemical processes that occur during geopolymerization can be intricate, they usually result in the development of a three-dimensional network structure. The production of polymeric structures, the solidification of the geopolymer, and the dissolution of silica and alumina from the source materials are the main reactions.

Using fly ash as an example, the general chemical reaction for geopolymerization can be expressed as follows:



The resulting geopolymer has strength and durability characteristics found in conventional cement-based products. Because they may employ industrial waste materials and frequently have lower carbon footprints than Portland cement, geopolymers have received interest as environmentally acceptable alternatives to cement.

1.4 GEOPOLYMER ADVANTAGES

Advantages in a variety of sectors, including environmental remediation to construction. Their sustainability and environmental friendliness is a significant advantage. When geopolymers are manufactured, significantly less greenhouse gas is released into the atmosphere than when traditional cement production is carried out, which results in significant emissions of carbon dioxide. This decrease in carbon emissions is in line with international efforts to mitigate climate change and encourages more ecologically conscious building methods. Geopolymers are perfect for structural applications because they have high compressive and flexural strength, durability, and other excellent mechanical qualities. They provide durable solutions for infrastructure projects since they are more resistant to corrosion and chemical deterioration than traditional materials. Additionally, geopolymers have quick curing times, which together shorten project timelines and speed up construction, saving money.

The adaptability of geopolymers is an additional advantage. They can be customized based on project needs by changing the composition and curing conditions to meet specific requirements. Their versatility allows them to be applied in a number of industries, such as wastewater treatment to aerospace.

Additionally, by using industrial by-products like fly ash and slag as precursors, geopolymers help reduce waste by keeping these materials out of landfills and lowering the need for virgin resources. This improves economic viability by reducing production costs and minimizing the negative effects on the environment. In summary, the advantages of geopolymers include reduced waste, improved mechanical qualities, environmental sustainability, and versatility. Geopolymers show promise as a replacement for conventional building materials as industries place a greater emphasis on efficiency and sustainability, opening the door to a more resilient and environmentally friendly future.

1.5 GEOPOLYMER APPLICATIONS

A class of inorganic polymers known as geopolymers has drawn a lot of interest due to its wide range of industrial applications. Building materials are one of the well-known uses for geopolymers. Compared to conventional materials like concrete, geopolymer-based materials have greater strength, durability, and resistance to chemical corrosion. These materials can be utilized for a variety of construction projects, such as paving, roads, and bridges in addition to buildings.

Geopolymers exceed expectations in environmental sustainability because they can be made from industrial byproducts like slag, fly ash, and mining wastes, which lessens the need for conventional cement production, which is a major contributor to carbon dioxide emissions. Through the utilization of these waste materials, geopolymers offer a sustainable solution that also assists in reducing landfill waste.

The field of infrastructure rehabilitation and repair is another important application for geopolymers. Steel reinforcements can be shielded from corrosion and utilized for structural reinforcement as well as the repair of deteriorating concrete structures using geopolymer composites. They are suitable for filling in cracks and preserving

the structural integrity of deteriorating infrastructure because of their superior adhesive qualities and strong bond.

Moreover, geopolymers are used in the aerospace and automotive sectors, where strong, lightweight materials are needed. Components for cars, spacecraft, and airplanes can be made using geopolymer-based composites, which reduce weight without sacrificing functionality or safety.

1.6 PCMS WITH GEOPOLYMER

Phase change materials (PCMs) frequently have the capability of absorbing or releasing huge quantities of high latent heat storage density in the conditions of solid-gas, liquid-gas, solid-solid, and solid-liquid phase transitions [5], while at reduced temperatures (at night) it solidifies and releases thermal energy back into the environment. Phase change materials (PCMs) are one way to guarantee a building's thermal performance by adding them to the building envelope [6]. When PCM is added, construction materials like gypsum and concrete gain a large amount of thermal mass, which helps regulate interior building temperatures. In addition to having an effect on a building component's energy efficiency, PCM can enhance aspects of the material's mechanical qualities and durability. As a result, using thermal energy storage (TES) to lower building energy usage becomes more practical. Through the reduction of temperature fluctuations, peak heat flux, and CO₂ emissions, PCM integration enhances both sustainability and energy efficiency [6]. Owing to its special qualities, PCM is a practical, long-lasting material that is utilized to promote thermal comfort in buildings.

To increase a building's thermal performance, Ramakrishnan et al. expanded the use of PCM to cementitious building materials. Peak temperature can be lowered by 4.43 °C when PCM is used in cement materials applied to the building's outer envelope [7]. Preventing PCM from leaking into the building fabric during the phase transition became a significant problem with its incorporation into the structure [7,8].

There are three methods to incorporate PCM into construction materials. Adding it directly into the mixing process is the first way. Submerging the building structure in liquid PCM is a further approach. The most sophisticated and widely used approach, the third one, encapsulates micro or macro PCM. Better particle dispersion is made possible, external volume changes are decreased but contact within the phase

change material and the beginning of the material is also eliminated. Phase change material microencapsulation is now an industrialized process, which makes it highly costly and restricts the product's availability to a few numbers of companies all around the world [8,9].

Improved thermal comfort inside the construction is one of the benefits of adding phase change materials (PCM) to the partition's middle layer [9, 10, 11, 12]. Furthermore, PCM evens out the temperature inside the concrete mixture and lessens thermal gradients, which may lessen the likelihood of cracking. Mixing PCM or paraffin with the other chemical substances will introduce it into the concrete. In between the pores, aggregate, and cement slurry in concrete, paraffin can be added. Paraffin can still leak to the concrete's surface after several cycles of temperature fluctuations. To avoid leaking, paraffin is now kept in encapsulated spheres as a solution to this issue. To address the leaking issue, the encapsulation performs effectively. The smooth surface can often result in insufficient interaction between the material and the cement slurry, adding to the process substantial expense. Macroscopic and microencapsulated hermitization are two categories [9].

The solidification of PCM at its outermost layer during the recovery of heat from the liquid phase, which obstructs effective heat transfer, is one of macroencapsulation's many disadvantages. Because of their lower interior temperature and superior thermal inertia, walls with encapsulated PCM have been demonstrated to have greater thermal storage than conventional concrete walls without PCM in microencapsulation experiments [8].

If using microencapsulated PCMs is not feasible due to cost or other problems, there are alternative options available, but they are not simple. Our observations demonstrate the addition of liquid PCM to a geopolymer matrix [9].

Another type of PCM material used like bio-glycerol, two phases are formed during the production of biodiesel. The lower phase is known as crude glycerol, and the upper phase is known as biodiesel (methyl or ethyl esters). These phases can be easily separated after the reaction mixture is rested at a neutral pH for a few hours [13]. In a well-established biodiesel industry, nearly equal volumes of biodiesel and roughly one-tenth of glycerol are obtained following the transesterification reaction.

Glycerol is a resource and a compound used in the oleo-chemical industry. It is also referred to as 1,2,3-propanetriol. Glycerine is another name for glycerol. It is the main byproduct of the synthesis of biodiesel, and the amount of crude glycerol produced per kilogram of biodiesel is approximately 10% of the total amount of biodiesel produced. [14] Approximately 41.9 billion liters of glycerol will be produced globally [15].

In some papers, Research is also conducted on the use of PCM in multilayer walls with varying configurations of air cavities and insulating material layers. According to the investigation, there was a 44% decrease in heat transfer indoors and a 2.6-hour shift in peak temperature [13,14,15].

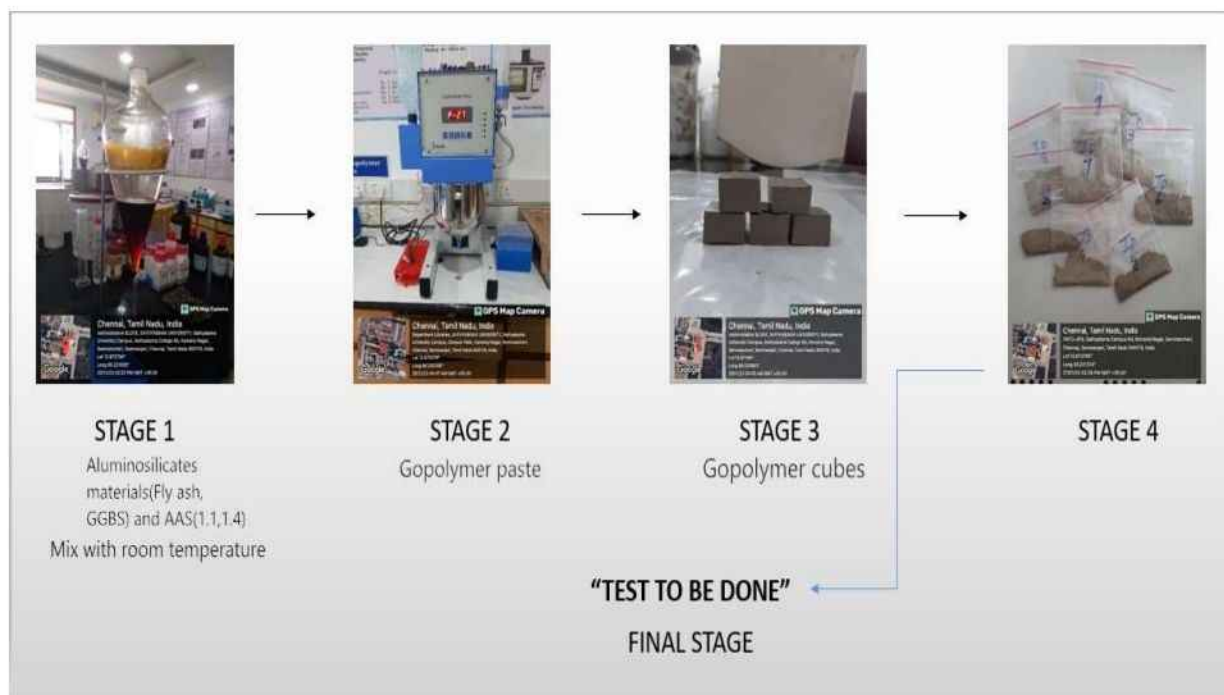


Fig1.1: Geopolymer Additives Making Process

In conclusion, In order to optimize energy usage and promote sustainable heating or cooling systems, thermal energy storage includes storing and retrieving heat for the future use. Geopolymer with Phase Change Material (PCM) include better insulation, higher energy storage capacity, improved thermal conductivity, and environmentally

friendly building options for effective temperature control [13]. There are many types of PCM available in the market, but only some of them only produces the better results when integrated with the geopolymer. Some of the PCM which gives good results are organic PCMs which are paraffin, fatty acids, alcohol, esters. These type of PCM are used because of the advantage of excellent chemical stability, self-nucleation, no phase separation, low steam pressure during melting, nearly no supercooling, high latent heat of phase transition, and corrosivity. Overall all researchers are used majorly paraffin based PCM, so to the addition of this introduction of the pure Bio-glycerol used as a PCM of about to 5 to 15% in the geopolymer concrete for the better thermal energy storage is discussed in the following

CHAPTER 2

LITERATURE SURVEY

This chapter describes the literature survey and explores the integration of geopolymer with various types of PCM materials.

Peiliang Cong et al., (2021)-- The development of geopolymer, a new environmentally friendly cementitious material, can lessen the carbon dioxide emissions brought on by the expansion of the cement industry. In addition to their mechanical properties and quality of material, geopolymer materials also have a number of other like resistance to corrosion and fire. The paper covered preparation techniques, activator types and sources, geopolymerization, and the various fields in which geopolymer materials are utilized[16].

Y.H.Mugahed Amran et al., (2019)-- The Advantage of GeoPC concrete is that it can replace cement by using cement-based materials in combination with alkali-activated solutions. Compared to regular concrete, GeoPC is a relatively new, innovative, and sustainable engineering material that offers several advantages. This paper provides a thorough analysis of GeoPC material, including information on its components, manufacturing processes, curing times, properties, and possible uses in the building sector[17].

Xiaohong Zhang et al.,(2021)-- Porous geopolymers have emerged as one of the most promising inorganic porous materials in the last 10 years due to their wide range of applications, suitable characteristics, affordability, ease of production, and chemical and thermal stability. This review article highlights current state-of-the-art techniques for producing porous geopolymer composites, including direct foaming, embedding lightweight, porous fillers, additive manufacturing, and other approaches, as well as their attributes (mechanical, thermal, adsorption, and other)[18].

ZakkaWyom Paul et al., (2020)-- When making geopolymer concrete, it can be advantageous to use waste materials from industry and agriculture, such as fly ash, slag, rice husk ash, and naturally occurring pozzolanas with a high aluminosilicate concentration. Because of its improved strength, temperature stability, denser microstructure, higher bond strength, and chemical resistance, this form of concrete can replace regular Portland cement (OPC) concrete. The areas where the literature

review was identified in this paper were the geopolymer type, materials, mix design, mechanical qualities, durability properties, microstructure, and adoption and application[19].

Azad A. Mohammed et al.,(2021)-- The mechanical characteristics of geopolymer concrete (GPC) and the data analytic methods utilized to forecast these characteristics were the main subjects of the work. An extensive amount of test data from 126 prior studies was collected, analyzed, and the correlation between different mechanical properties and compressive strength was looked at. Equations were provided for the strain corresponding to peak compressive strength, modulus of elasticity, splitting tensile strength, flexural strength, and Poisson's ratio[20].

Vinh Duy Cao et al.,(2019)-- It was discovered that adding pure phase change materials (PCM) to multilayer walls and incorporating microencapsulated phase change materials (MPCM) into geopolymer concrete greatly enhanced thermal performance. Under ideal circumstances (thick PCM layer and thin insulating layer), there was a 28–30% annual energy reduction. It was discovered that PCM performed better when it was situated near an outdoor area. A heating and cooling system's energy consumption can be greatly reduced by increasing the insulation layer's thickness and decreasing its thermal conductivity, but doing so lessens the MPCM/PCM's high heat storage capacity's effectiveness[21].

Sarra Drissi et al., (2019)-- Given the differences in mix designs and the variety of materials employed in each study, it makes natural that the literature evaluation shows that the compressive strength decrease varies significantly with no obvious pattern. Current PCM case studies, gaps, and future directions will provide a strong foundation and valuable reference for the development of eco-friendly and energy-efficient building materials including PCM [22].

T. Salgueiro et al, (2021) -- The mechanical behavior, modulus of elasticity, specific heat, diffusivity, and shrinkage and capillarity of the new mortars were all examined, along with their thermal properties, which included specific heat, thermal conductivity, and thermal inertia. To measure the impact of the mixing process on the PCM performance, a second analysis was conducted to compare the thermal characteristics of the mortar. By using PCM, the specific heat capacity was raised by 17% and the thermal conductivity was reduced by 24% [23].

Peng Zhang et al., (2018)-- Because of its benefits for sustainability, cement-based enterprises and researchers are becoming more and more interested in the features and applications of geopolymer mortar. In addition to microstructure analysis, this study reviews the mechanical, physical, and durability properties of geopolymer mortars, such as their tensile strength, compressive strength, elastic properties, flexural performance, water absorption, and shrinkage properties. It also examines the properties of fresh geopolymer mortars, such as workability, setting time, and temperature. The characteristics of several geopolymer mortar types that are made with diverse source materials as base materials are also reviewed in this study[24].

Qiang Fu et al., (2021)-- The remarkable mechanical properties and durability of fly ash-based geopolymer concrete (FABGC) make it an eco-friendly building material. The goal of the investigation into the microstructure and durability of FABGC is to meticulously gather data on the material's pore structure, interface characteristics, and alkali-activated reaction. The mechanisms of FABGC degradation under various corrosive environments are analyzed, and the factors affecting its durability and microstructure are also covered. It is observed that the aluminosilicate gel produced by the alkali-activated method of FABGC optimizes the pore structure and interfacial transition zone. It is possible to somewhat increase the durability of FABGC by forming microstructures efficiently. There are currently differing views on the durability and microstructure of FABGC [25].

Frantisek Skvara et al.,(2006)-- The primary focus of the published works to date has been on the polymer character (NMR in solid phase) of geopolymers, their mechanical properties, the impact of aggressive environments, their leaching ability, and other factors. There aren't many details about the other material properties of geopolymers, such as porosity, aggregate-border geopolymer qualities, or micromechanical qualities, in the literature. These parameters are the focus of this study[26].

A.M.Mustafa Al Bakri et al.,(2013)-- The microstructure of the various geopolymer raw material types was examined in this study. Various materials, including fly ash (class F) and kaolin, were combined with an alkaline solution that contained sodium hydroxide and silicate in the right ratios to the alkaline activator and sodium silicate to sodium hydroxide. After being cured for 24 hours at 70°C using various raw

material types, the geopolymer samples were kept at room temperature until the testing was carried out. Testing was done seven days after the geopolymers were aged[27].

Aissa Bouaissi et al., (2019)-- In this study, it was found that the 28-day splitting tensile strength and the 28-day compressive strength of GP (geopolymer) concrete increased by 58% and 100%, respectively, when 10% HMNS (high-magnesium nickel slag) and 20% GGBS were used in place of fly ash (FA). Microstructure investigation of the GP concrete using SEM, XRD, and FTIR revealed the emergence of an aluminosilicate amorphous phase in a three-dimensional network. The SEM pictures reveal a totally compact and cohesive geopolymer matrix, which explains why the mechanical properties of the FA-based GP concrete with both GGBS and HMNS are improved [28].

Pratyush Kumar et al.,(2018)-- This study examined three different weight percentages of GGBS and metakaolin clay: 80%-20%, 50%-50%, and 20%-80%, respectively. As an alkaline activator solution, a 10M solution of sodium hydroxide and sodium silicate was employed. Ambient curing of the geopolymer samples has been chosen for practical reasons. After seven days of curing, the degree of response was assessed for every percentage fluctuation in the geopolymer concrete. The compressive, split tensile, and four-point loading tests have all been used to examine the mechanical characteristics of geopolymer concrete. Using an ultrasonic pulse velocity test, the structural integrity of the geopolymer specimen casting was investigated. The technology of Fourier transform infrared spectroscopy (FTIR) was utilized to investigate the microstructure of geopolymer concrete [29].

TABLE 2.1: LITERATURE SURVEY TABLE

No:	Authors	Title	Studies	Referen ce
1	P Cong, Y Cheng	Geopolymer material advancements: An extensive overview	The article included preparation techniques, activator types and sources, geopolymerization, and the various domains in which geopolymer materials are applied.	30
2	X Zhang, C Bai	Porous geopolymer composites: A review	The state-of-the-art in the creation of porous geopolymer composites is highlighted in this review article along with its properties (mechanical, thermal, adsorption, and other), applications, and manufacturing techniques (direct foaming, embedding lightweight, porous fillers, additive manufacturing, etc.).	31
3	WP Zakka, NHA S Lim	A scientometric review of geopolymer concrete	The study analysed related bibliographic data mined from the Scopus database. The research gaps identified were in the areas of geopolymer type, materials, mix design, mechanical properties, durability properties, microstructure, and adoption and application.	32

4	AA Mohammed, HU Ahmed	Examination of Geopolymer Concrete's Mechanical Properties: A Thorough Examination and Data Interpretation	geopolymer concrete; mechanical properties; data analysis; compressive strength; flexural strength; splitting tensile strength; elastic modulus; review; state of the art; survey.	33
5	HU Ahmed, AA Mohammed,	Sustainable Geopolymer Concrete Composites' Compressive Strength: A State-of-the-Art Analysis	Mixture Proportion Parameters, Methodology, fly ash; compressive strength; curing; alkaline solution; sustainable concrete; sustainability; circular economy; composite materials design	34
6	L Imtiaz, SKU Rehman	An Overview of Current Progress and Developments in Green Geopolymer Concrete	This article provides a critical review of the components of geopolymer concrete (GPC), their effects on the material's fresh and hardened properties, factors influencing those properties, reinforced GPC, geopolymer OPC concrete, application of GPC, and geopolymer concrete mortar (GCM).	35
7	K Kishore, N Gupta	Mechanical characterization and assessment of composite geopolymer concrete	This study uses a rich aluminosilicate source of ground granulated blast furnace slag (GGBS) that has been cured at room temperature for 7, 28, and	36

			56 days to access the design factors for the GPC manufacturing.	
8	J Zhao, L Tong,	Eco-friendly geopolymer materials: An examination of enhanced performance, possible uses, and sustainability evaluation	Preparation process of geopolymers, Reaction mechanism of geopolymers, Rheological property and optimization of geopolymer materials. Hardening characteristics and optimization of geopolymer materials. Property of porous geopolymer as building insulation materials.	37
9	M Wasim, TD Ngo	A cutting-edge analysis of geopolymer concrete's resilience for environmentally friendly infrastructure and buildings	Introduction Methodology, Development of microstructure in GPCs-recent studies, GPC for repairs of reinforced concrete structures. Durability and corrosion of GPC.	38
10	TH Tan, KH Mo	Geopolymer is now being developed as a substitute adsorbent for the removal of heavy metals.	Geopolymer; Adsorption; Heavymetal; Porous Geopolymer; Sustainability	39
11	M Rahjoo, G Goracci	Thermal Energy Storage (TES) Prototype Based on Geopolymer	Concretes based on ordinary Portland cement (OPC) are frequently employed as sensible TES materials in	40

		Concrete for High-Temperature Applications	various applications. However, because to the heat degradation processes in their structure, their performance is restricted to operating temperatures below 400 °C.	
12	M Sivasakthi, R Jeyalakshmi	Flyash geopolymer mortar: Impact of the substitution of river sand by copper slag as a fine aggregate on its thermal resistance properties	In this work, we have replaced river sand in a fly ash geopolymer mortar with copper slag as an aggregate, and we have examined the impact of this substitution on the mortar's thermal resistance. X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and field emission scanning electron spectroscopy (FESEM) were used to study changes caused by heat.	41
13	A Boros, T Korim	Development of Geopolymer Foams for Multifunctional Applications	The current study's objective is to create metakaolin-based geopolymer foams by combining the processes of saponification, peroxide breakdown, and gel casting. The influence of H ₂ O ₂ concentration, the kind and quantity of stabilizer, and the	42

			addition of slag were all studied in relation to physical attributes. To describe the material structure, studies using XRD, FTIR, SEM, CT, and thermogravimetric techniques were carried out.	
14	M Rahjoo, G Goracci	Geopolymer Concrete Performance Study for High-Temperature Thermal Energy Storage (TES) Applications	The terms concentrated solar power, thermal energy storage, geopolymer, TES, and OPC are used.	43
15	A Drabczyk, S Kudłacik-Kramarczyk	Review of Geopolymer Nanocomposites: Novel Materials for Sustainable Development	This article showcases certain nanoparticles, such as carbon nanotubes, graphene, nanosilica, and titanium dioxide, that have been integrated into geopolymer matrices. Using scientific databases, the review was created, paying close attention to works on geopolymer nanocomposites.	44

From the above literature survey, Overall, the world researchers founded many types of PCM's and they also integrated with the geopolymer. They also analysed the various types of PCM materials and finalised the better PCM material which gives the higher thermal storage which is used in the various types of applications. Also, the study involves the microstructure of the various geopolymer, amount of activator solution to be used and weight percentage of the GGBS and fly ash used. About the conclusion from the above all, introduction of the new PCM is very essential from the waste byproducts.

So, to overcome this some process is been done in the below methodology to produce new type of PCM from the waste management. The crude which available during the process of biodiesel production is been used and filtered to form pure form of bio glycerol which is used as PCM for better thermal energy storage. Bio-glycerol is been used in the mixture of about 5 to 15%. After the formation of geopolymer concrete with PCM many tests are been done for the confirmation of thermal storage. Some of the test are XRD, FT-IR, SEM, TGA-DTA and Thermal Conductivity.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The aim is to advance the development of geopolymer composites integrated with phase change materials as high-performance and sustainable solutions for thermal energy storage, contributing to energy efficiency, renewable energy utilization, and environmental protection.

3.2 SCOPE

- Some of the primary goals and components of the development of phase-change material-integrated geopolymer composites for thermal energy storage applications are as follows:
- **Material Composition and Formulation:** To get the required thermal characteristics and energy storage capacity, the composition of geopolymer composites, including the kind and quantity of additives, phase change materials (PCMs), and precursors, is being investigated and optimized.
- **Fabrication Techniques:** Investigating and creating processes for mixing, curing, and shaping geopolymer composite PCM systems in order to guarantee homogeneous dispersion of PCMs within the geopolymer matrix and preserve structural integrity.
- **Comprehensive characterization and testing of the geopolymer composite PCM systems** should be carried out, including thermal analysis (such as differential scanning calorimetry, thermal imaging), structural analysis (such as X-ray diffraction, scanning electron microscopy), and thermal analysis (e.g., differential scanning calorimetry, thermal conductivity measurements), mechanical testing, and durability assessments under thermal cycling conditions.
- **Comprehensive characterization and testing of the geopolymer composite PCM systems** should be carried out, including thermal analysis (such as differential scanning calorimetry, thermal imaging), structural analysis (such as X-ray diffraction, scanning electron microscopy), and thermal analysis (e.g., differential scanning calorimetry, thermal conductivity measurements),

mechanical testing, and durability assessments under thermal cycling conditions.

- **Thermal Performance Optimization:** Assessing and improving the heat storage capacity, thermal conductivity, heat transfer efficiency, thermal stability, and long-term performance under cyclic thermal loads of geopolymer composite PCM systems.
- **Application-specific adaptation** refers to modifying the characteristics and architecture of geopolymer composite PCM systems to meet the needs of particular thermal energy storage applications. Examples of these applications include HVAC systems, solar thermal energy storage, building insulation, electronic device thermal regulation, and industrial waste heat recovery.
- **Environmental Impact and Sustainability:** Evaluating how geopolymer composite PCM systems will affect the environment and be sustainable, taking into account their ability to recycle materials, life cycle analysis, and the reduction of greenhouse gas emissions and improvement of energy efficiency.
- **Cost Analysis and Scalability:** To determine the commercial viability, cost-benefit analyses, economic feasibility studies, and scalability assessments are carried out.

Overall, the scope of development includes multidisciplinary research and innovation efforts aimed at advancing the design, performance, applicability, and sustainability of geopolymer composites integrated with phase change materials for diverse thermal energy storage applications.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS AND METHODOLOGY USED

This chapter describes the materials and methods like, fly ash (FA), ground granulated blast furnace slag (GGBS), paraffin, and bio-glycerol were the ingredients and techniques used in this work to create a new composite material. The industrial by-products fly ash and GGBS were obtained, and they are known for their respective pozzolanic and latent hydraulic properties. Because paraffin is a readily accessible hydrocarbon and works well with other ingredients, it was selected as a binder. Bio-glycerol was chosen as a possible addition to improve the material's characteristics and sustainability profile because it comes from renewable sources.

Table 4.1 List of Materials used in the Experiments

Materials	Particle Size	Density	Ref
Paraffin-based phase change material	Liquid/solid	0.88 kg/L for solid 0.76 kg/L for liquid	7,8,9
Sodium Hydroxide (NaOH)	Liquid	1.19 kg/L	7,8
Sodium silicate (Na ₂ SiO ₃)	Liquid	1.39 kg/L	7,8
Ground granulated blast furnace slag (GGBS)	(Solid)0.2–70 µm	1236 kg/m ³	7,8
Fly Ash (FA)	3–70 µm	1262 kg/m ³	7,8,9
Bio-Glycerol solutions	Liquid	0.998 kg/L	7,8,9

The general procedure used for the experiment involved multiple stages:

- (1) Creating composite mixtures containing different ratios of FA, GGBS, paraffin, and bio-glycerol;
- (2) Utilising a mechanical mixer to mix the ingredients uniformly.
- (3) Compacting the mixture into standard-diameter moulds.
- (4) Curing the specimens in a controlled environment to promote hydration and binder solidification.

To maximise the appropriate mix design and obtain the required qualities, a variety of reaction parameters have been taken into account. The crucial parameters specifics changed during the preparations and are listed below.

- Blend ratio for fly ash GGBS: 0, 50, and 100%.
- Solutions of sodium silicate with varying silica moduli (M_s 0.6, 0.8, 1.1, 1.2, 1.4, and 1.6) in $\text{SiO}_2/\text{Na}_2\text{O}$.
- Curing conditions: Fly ash geopolymer samples were cured at 80°C for 24 hours, whereas samples made using GGBS were cured at room temperature.
- The supplements: In specifically, a partial replacement using the weight of the precursor was used. To create a workable mixture, the liquid-to-solid ratio for paste creation was 0.4.
- Depending on the mix proportion, the liquid-to-solid ratio for mortar and concrete varies from 0.45 to 0.6. Trials were used to determine these ratios.
- Every test was carried out following a minimum of three days of curing.

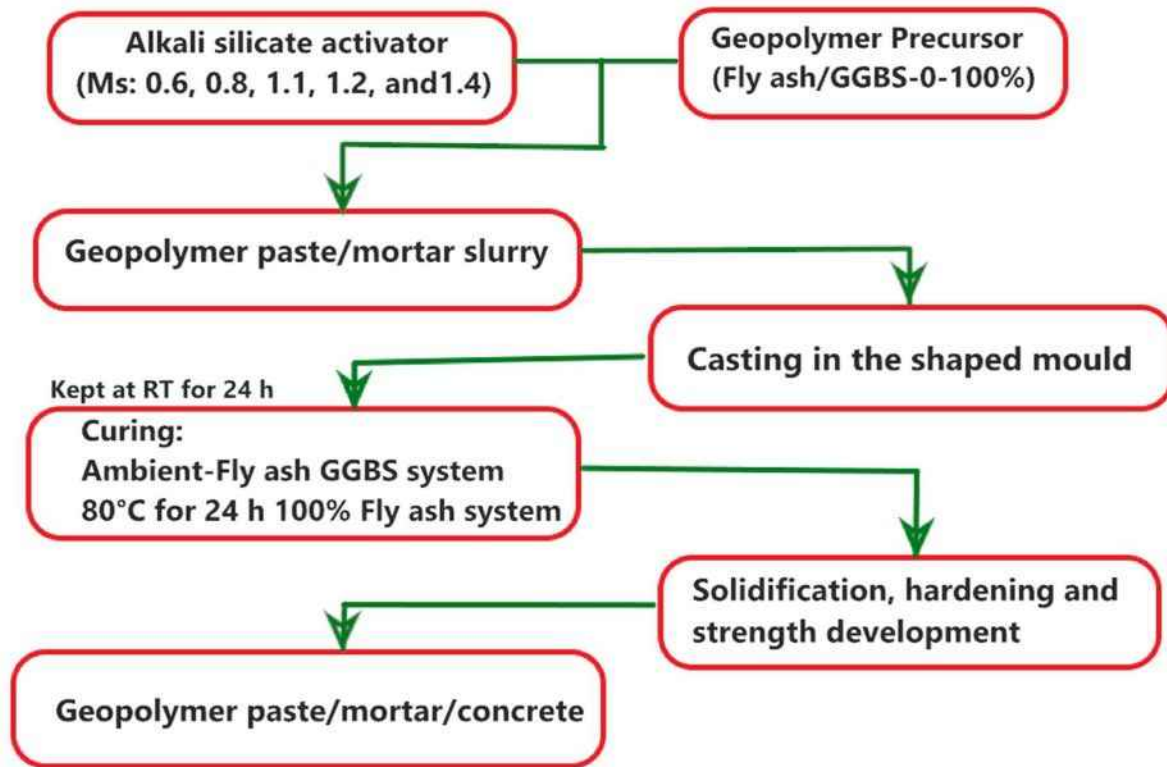


Fig 4.1: The General Procedure of Geopolymer Concrete

Additionally, to evaluate the microstructure and functionality of the created composites, characterisation methods including mechanical testing, scanning electron microscopy (SEM), FT-IR, and X-ray diffraction (XRD) were used. The materials and techniques used in this study were intended to investigate the viability of producing durable and sustainable composite materials appropriate for a range of technical applications by using industrial by-products and renewable additions. It involves thermal conductivity at different temperatures (the material's capacity to transfer heat), thermal shock resistance, and thermal stability is determined by TGA-DTA.

4.2 FLY ASH

Fly ash is been purchased from Ennore Thermal Power Plant, which is located at Chennai, Tamil Nadu. Fly ash is a finely powdered waste produced in power plants when pulverized coal is burned. It is made up of small, circular particles that are abundant in iron, alumina, and silica. Fly ash is a byproduct of burning coal and is thought to be good for the environment because it doesn't need to be disposed of in landfills. Its pozzolanic qualities, which increase strength and durability, have made it

more popular for use in building materials like concrete. By diminishing the need for conventional cement manufacturing, the addition of fly ash also helps to promote sustainable practices by cutting carbon emissions. Furthermore, fly ash can lessen permeability and increase workability in concrete structures, which makes it an important resource for the building sector with advantages for the environment and the economy. Fly ash is used in the process of production of bricks, as a soil stabilizer, and in the construction of roads. By replacing products with larger environmental footprints with its use not only lessens the environmental impact of coal combustion leftovers but also conserves natural resources. The circular economy idea is best shown by the sustainable use of fly ash, which turns waste into a useful resource for a variety of businesses.

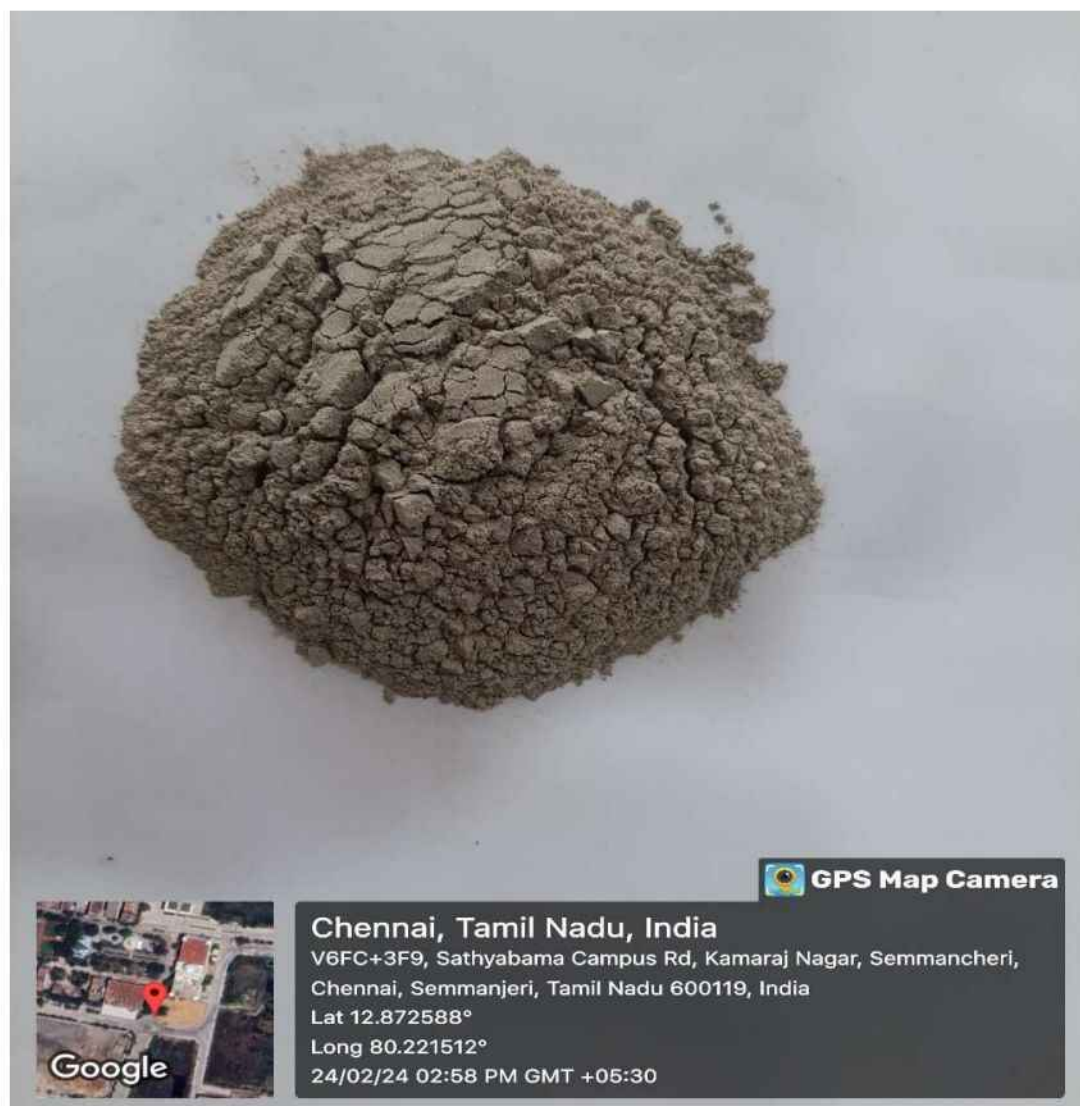


Fig 4.2: Fly ash

4.3 GROUND GRANULATED BLAST FURNACE SLAG

GGBS is known as the Ground Granulated Blast Furnace Slag and it is the byproduct of the manufacturing pig iron and Steel Industry. GGBS is been purchased for JSW Steel Plant which is been located at Bellary District at Karnataka. A ground granulated blast furnace slag (GGBS) is particularly extracted from the blast furnace during the manufacturing of iron. This finely powdered substance is created when water quickly cools molten slag, producing a glassy granular product. Because of these advantageous characteristics, GGBS is frequently employed as an additional cementitious material in the building sector. Portland cement can be partially substituted with GGBS concrete, which increases strength and durability. Because of its strong reactivity with calcium hydroxide, a result of cement hydration, more cementitious compounds are formed, which improves concrete's overall performance.

In addition, GGBS reduces sulphate attack and alkali-silica reaction, which makes it an important part of constructions exposed to harsh environmental conditions. When GGBS is used in buildings, less Portland cement is used, which lowers greenhouse gas emissions related to Portland cement manufacture. Furthermore, GGBS is a recyclable material that provides an environmentally friendly way to handle industrial leftovers. It is frequently used in many different applications, including as marine constructions, precast concrete products, and road building. GGBS implementation in the building industry is consistent with eco-friendly practices that prioritize resource efficiency and the advancement of a circular economy.

Table 4.2: physical characteristics of GGBS and Fly ash

Physical Properties	Bulk Density kg/m ³	Specific Gravity	Fineness (Blaine) m ² /kg	Cementing Efficiency Factor (28 days)	Residue on 45-micron sieve (%)	Loss of Ignition (%)
Fly ash	1005	2.15	330	0.93	35	1.4
GGBS	1360	2.92	425	0.95	ND	1.8



Fig 4.3: Ground Granulated Blast Furnace Slag

4.4 PARAFFIN

Paraffin is a complex mixture of hydrocarbons that is mostly employed in coating and sealing. It is a waxy, crystalline, flammable material that is particularly obtained from distillates of wood, coal, petroleum, or shale oil. It is also used as the phase change material. Paraffin is been purchased commercially from Nice Chemicals private Limited which is in Kochi, Kerala.

A substance known as paraffin phase change material (PCM) has the ability to change phases from solid to liquid at a given temperature, efficiently storing and

releasing thermal energy in the process. Hydrocarbons such as paraffin are frequently utilized as PCMs because of their capacity to change from a solid to a liquid condition across a small temperature range. Paraffin PCMs are frequently used in a variety of applications, such as electronics, construction materials, and textiles when it comes to thermal energy storage. They are especially helpful in controlling and maintaining temperature.

The PCM functions as a thermal buffer by absorbing heat from its surroundings during the solid-to-liquid transition. On the other hand, it releases heat that has been stored during the liquid-to-solid transition, which aids in controlling temperature in a controlled setting. The capacity of paraffin PCMs to store a substantial quantity of latent heat during the phase transition, offering effective thermal energy storage, is one of its main advantages. Because of this characteristic, they are useful in applications where energy efficiency or temperature control are critical, including passive solar heating systems, building climate control, or the creation of smart textiles. Paraffin PCMs optimize thermal energy usage, lessen the need for active heating and cooling systems, and improve overall energy efficiency across a range of industries, all of which support energy conservation initiatives.



Fig 4.4: Paraffin

4.5 ALKALI ACTIVATOR SOLUTION PREPARATION (AAS)

To prepare alkali activator solutions in varying molarities, the alkali material (for example, sodium silicate or hydroxide) is diluted in water to the appropriate concentration. The following general instructions can be used to prepare alkali activator solutions in various molarities:

The molarity selection depends on several factors, including the type of aluminosilicate material being utilized (such as fly ash or metakaolin). The molarity has different values such as 0.6, 0.8, 1.1, and 1.4 and each value has different ratios. The molarity value of 0.6 has (1:1:3) sodium hydroxide, sodium silicate, and water. The molarity value of 0.8 has (1:2:2) sodium hydroxide, sodium silicate, and water. The molarity value of 1.1 has (1:3.5:0.5) sodium hydroxide, sodium silicate and water. The molarity value of 1.4 has (100g:25g) sodium hydroxide and sodium silicate.



Fig 4.5: Alkali Activator Solution

4.5.1 AAS with Molarity 1:1 Solution

Solution of Sodium Hydroxide (NaOH):

An electronic compact scale equipment can be used to measure a NaOH solution directly in 40 ml into an appropriate container (like a beaker).

Solution of Sodium Silicate:

140 ml of the sodium silicate solution is measured into a suitable container (such as a beaker) with an electronic compact scale machine.

Water:

Use to measure 20 ml of water with the help of an electronic compact scale machine.

Mixing:

Combine the indicated amounts of NaOH solution, sodium silicate solution, and water in a suitable container (such as a beaker). To guarantee homogeneity, give the mixture a good stir.

Final Solution:

With the appropriate amounts of NaOH, sodium silicate, and water, the resulting combination may be regarded as your alkali activator solution.

Setting time:

After preparation of an alkali activator solution (AAS) in 1.1 and it becomes hot, it may be due to an exothermic reaction. Additionally, if the solution needs to be poured into a tube for a specific duration (i.e. one day) before preparing control samples.

4.5.2 AAS with Molarity 1:4 Solution

Solution of Sodium Hydroxide (NaOH)

A NaOH solution can be measured directly in 100 ml into a suitable container (such as a beaker) with an electronic compact scale machine.

Solution of Sodium Silicate:

25 ml of the sodium silicate solution is measured into a suitable container (such as a beaker) with an electronic compact scale machine.

Mixing:

In a sufficient container (such as a beaker), combine the specified amounts of sodium silicate and NaOH solution. Stir the mixture thoroughly to ensure homogeneity.

Final Solution:

The mixture that results may be thought of as your alkali activator solution if suitable proportions of sodium silicate and NaOH are added. Generally, the alkali activator solutions (AAS) should be prepared one day or 24 hours before the control sample preparation.

Setting time:

If an alkali activator solution (AAS) is prepared and it gets heated, an exothermic reaction can be the cause. Furthermore, if the solution must be put into a tube and left there for a certain amount of time(say, a day) before creating control samples.

4.5.3 Control Sample Preparation

Ground granulated blast furnace slag (GGBS) and fly ash (FA) waste materials are investigated and found to form a GP by activating polymerization using a mixed solution of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH).

There are two sample preparations one is 100% fly ash and another one is 70% fly ash plus 30% ground granulated blast-furnace slag (GGBS or GGBFS). 100% fly ash is concentrated with 1.1 and 1.4 AAS (alkali activator solutions) and another 70% fly ash plus 30% ground granulated blast-furnace slag (GGBS or GGBFS) is also concentrated with 1.1 and 1.4 AAS (alkali activator solutions).

Sample 1 preparation:

Take 100g of fly ash directly and measure with the help of an electronic compact scale machine. Then, take the already-prepared 1.1 AAS up to a maximum of 60 ml measure with the help of an electronic compact scale machine. After all the

measurements, pour the correct proportion of 1.1 AAS slowly into the 100g of fly ash beaker. Then, Stir the mixture thoroughly to ensure homogeneity. After the homogeneity form came pour it into the moulded pattern. Then place the moulded pattern at room temperature for one day or 24 hours and the next day place the moulded pattern in a hot air oven at 80° c for one day or 24 hours.



Fig 4.6: The Moulded Pattern Box

The preparation of the 1.4 AAS sample is also the same as the 1.1 AAS sample except that it incorporates the 1.4 AAS.



Fig 4.7: Geopolymer concrete/mortar (100% FA) with 1.1. and 1.4 AAS.

Sample 2 preparation:

Take 70g of fly ash plus 30g of GGBS directly and measure with the help of an electronic compact scale machine. Then, take the already-prepared 1.1 AAS up to a maximum of 60 ml measure with the help of an electronic compact scale machine. After all the measurements, pour the correct proportion of 1.1 AAS slowly into the 70g of fly ash plus 30g of GGBS container. Then, Stir the mixture thoroughly to ensure homogeneity. After the homogeneity form came pour it into the moulded pattern. Then place the moulded pattern at room temperature for one day or 24 hours. The preparation of the 1.4 AAS sample is also the same as the 1.1 AAS sample except that it incorporates the 1.4 AAS.

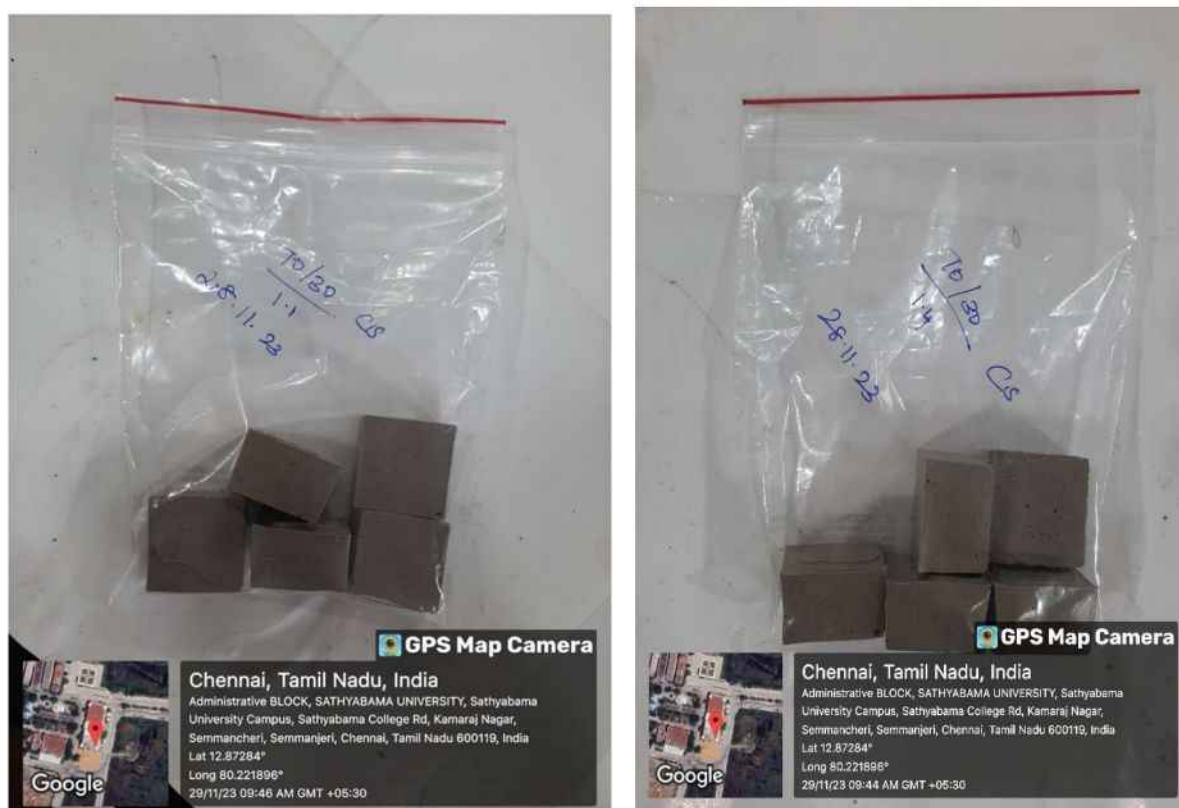


Fig 4.8: Geopolymer concrete (70% FA and 30% GGBS) with 1.1 and 1.4 AAS

4.6 BIO-GLYCEROL

Bio-glycerol most likely refers to glycerol or glycerine derived from bio-based or renewable sources during the production of biodiesel. Bio-glycerol is been extracted through the biodiesel production process from the Centre of Waste Management at Sathyabama University, Chennai. It undergoes so many characterization processes to become the pure glycerol form.

Bio-glycerol can be produced through the transesterification of triglycerides found in plant oils or animal fats. Glycerol is produced as a byproduct of this process in addition to biodiesel. Because bio-glycerol is made from renewable feedstocks and lessens dependency on non-renewable resources, its use is consistent with sustainable practices.

Bio-glycerol is also used as a phase-changing material. Phase change materials are substances that, when they go through a phase transition that is, when they shift from a solid to a liquid or vice versa have the ability to absorb or release a sizable quantity of heat.

The melting point of glycerol is approximately 18.2 degrees Celsius, or 64.8 degrees Fahrenheit. Because of this characteristic, it can be used in situations where a PCM is required to absorb and release thermal energy within a particular temperature range.

Glycerol takes in heat from its environment when it melts, or changes from a solid to a liquid, and releases heat when it freezes or changes back to a liquid. Glycerol is used as a PCM in thermal energy storage systems, passive temperature regulation building materials, and other applications where accurate temperature control is crucial.

In a variety of sectors, the use of glycerol as a PCM can enhance temperature control and energy efficiency.

4.6.1 Crude Glycerol Purification Procedure

Crude glycerol, a byproduct of biodiesel production, often needs purification to meet the desired quality standards for various applications. Methanol, salts, fatty acids, and water are among the contaminants that must be eliminated during the purification process. In a methodical procedure for separating refined glycerol from crude glycerol produced during the manufacturing of biodiesel:

Crude Glycerol Source:

The crude glycerol used in this process is obtained as a byproduct of biodiesel production. Biodiesel production typically involves the transesterification of triglycerides (fats and oils) with methanol or ethanol to produce biodiesel and crude glycerol.

Removal of Unreacted Methanol:

The first step involves heating 100 ml of crude glycerol to 80°C in a 500 ml beaker. This process helps remove any unreacted methanol that might still be present in the crude glycerol.

Acidification:

The next step is to adjust the pH of the crude glycerol to a desired value of 1. This is achieved by adding either phosphoric acid or sulfuric acid (99%). Acidification is a

common step in the purification process of glycerol, and it helps in breaking down impurities.

Stirring and Phase Separation:

The acidified solution is then stirred at a constant rate of 200 rpm for one hour at a temperature of 35°C. After stirring, the solution is left overnight to allow for phase separation. A separating funnel is then used to separate the solution into two layers. The top layer is rich in Free Fatty Acids (FFA), while the bottom layer is rich in glycerol.

Neutralization:

The glycerol-rich bottom layer is further processed by neutralizing it to a pH of 7 using a sodium hydroxide (NaOH) base solution. This neutralization step helps to remove excess acidity and stabilize the glycerol.

Heating and Inorganic Salt Formation:

The neutralized glycerol-rich solution is then heated to 80°C with continuous stirring at 200 rpm. During this process, inorganic salts are formed. These salts are then filtered out using Whatman filter paper, separating them from the glycerol solution.

Activated Charcoal Purification:

The filtrate obtained from the previous step, which still contains impurities, undergoes purification using activated charcoal. The ratio of activated charcoal to the solution is 5:1, and the mixture is stirred for an hour. Activated charcoal is effective at adsorbing impurities and colourants.

Final Filtration:

The last step involves filtering the solution through the Whatman filter paper once again. This step helps remove any remaining impurities and particles, resulting in the desired purified glycerol.

Acidic Solution preparation:

In 10 ml of Concentrated sulfuric or phosphoric acid mixed with 40 ml of (reverse osmosis) RO water.

Base Solution Preparation:

In 20 grams of NaOH mixed with 100 ml of (reverse osmosis) RO water.

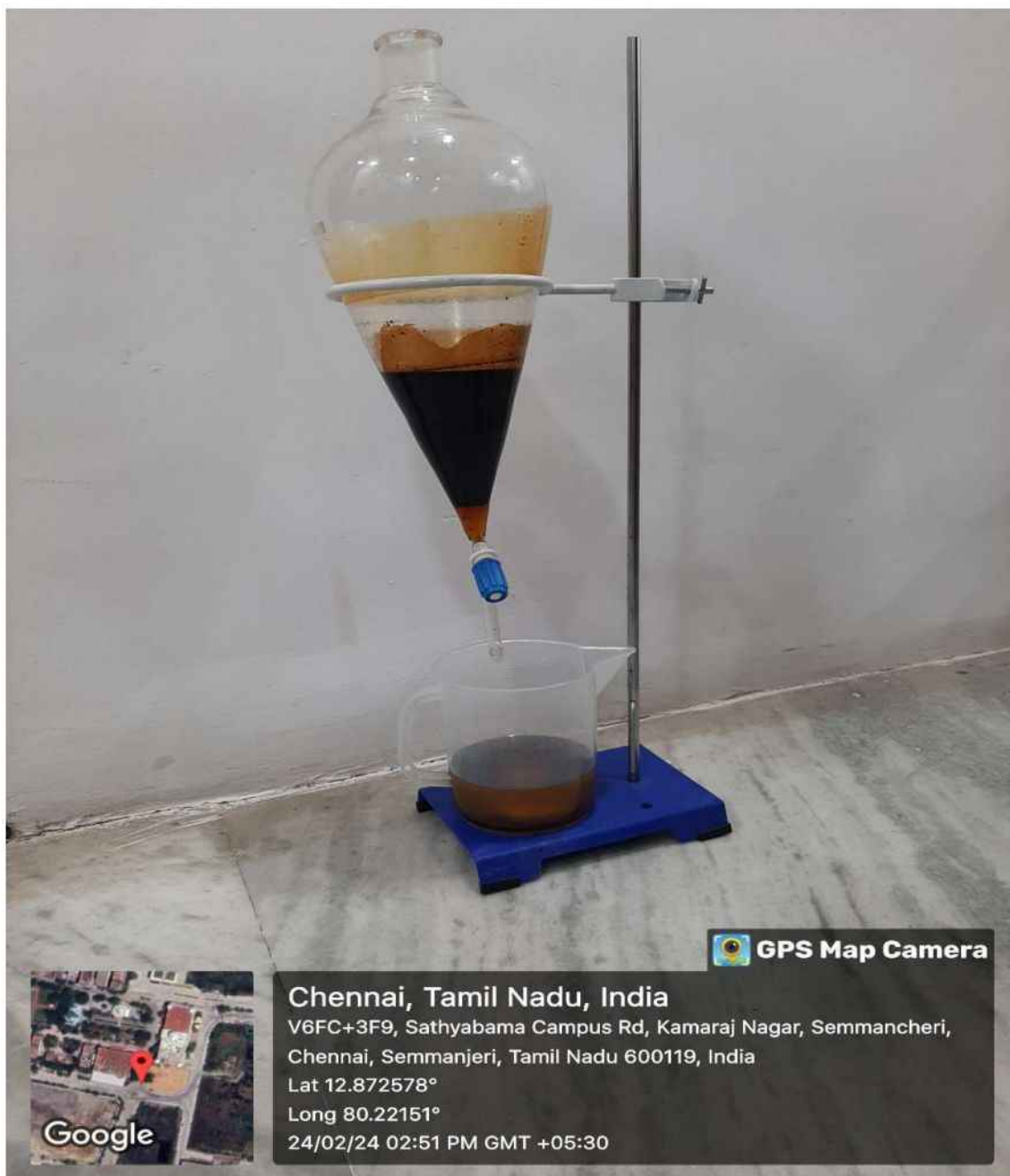


Fig 4.9: Crude Glycerol

Table 4.3: Overview of several binder types used in mortar containing PCM integrated polymer matrix (PCM) geopolymer composites summation.

Method	PCM material	Base material	Mechanical properties	Conclusion	Ref
Micro encapsulated	paraffin	Fly ash-based geopolymer mortar	20%Improved thermal efficiency	The interior temperature of the cubicles containing 10% and 20% phase change material (PCM) experienced a reduction of 4.4°C and 5.5°C, respectively. The addition of phase change material (PCM) up to a concentration of 20% resulted in a loss in compressive strength. Enhanced+++ ++ thermal efficiency and energy conservation.	45
Encapsulated by melamine - formaldehyde.	Paraffin	1. Aerial lime based mortar 2. Gypsum-based 3. Cement mortar 4. Hydraulic lime mortar	The mechanical features changed. Gypsum-based mortar performed better in heat	The inclusion of phase change material (PCM) resulted in a reduction in workability.	46

Encapsulated by calcium silicate.	Paraffin-impregnated expanded perlite.	Clay-based geopolymer mortar.	The impact of temperature on mechanical properties is negligible. The geopolymer mortar specimens, both subjected to high and low temperatures, exhibited a 28-day compressive strength of 8 MPa.	The heat storage ability of geopolymer mortar has been enhanced.	47
Encapsulated by melamine – formaldehyde	Paraffin	Cement paste and mortar.	The heat conductivity is increased. The thermal conductivity exhibited consistent values throughout the temperature range of 10 to 40	The thermal conductivity of cement composites PCM-cement mortar specimens was seen to decrease as the volume percentage of PCM increased from 0% to 30%..	48

			degrees Celsius.		
Micro encapsulated	paraffin wax	Cement mortar	Enhancements in the specific heat capacity and latent heat.	The development of phase change material (PCM) in cement mortar resulted in a reduction in thermal conductivity.	49
Micro encapsulated	paraffin wax doped with graphite flakes	Cement mortar	Increased thermal inertia The magnitude of temperature variations was decreased. The structural properties undergo a modification.	The heat storage capacity of mortar based on PCM/granite flakes has been enhanced.	50
Micro encapsulated	paraffin wax	Portland cement mortar	There has been a reduction in the mechanical strength.	Addition of PCM The thermal inertia went up. The thermal reaction was the same for all ages.	51
Non encapsulated	pure paraffin	Cementitious mortar containing fly ash	The use of phase change material (PCM) resulted in a	The presence of non-encapsulated phase change material (PCM) inside the cement matrix has been shown to result in interference	52

			reduction in both compressive strength and flexural strength.	and subsequent delays in the hardening process. This interference has been found to cause a loss of up to 30% in PCM content when incorporating up to 20% PCM in mortar.	
Encapsulated by calcium silicate	Paraffin is impregnated and expanded into graphite .	Red mud geopolymer mortar.	The thermal properties of both hot and cold-treated geopolymer were shown to be unaffected by the phase transition of phase change material (PCM). However, the encapsulated geopolymer composite wall surfaces containing PCM exhibited greater	The compressive strength of red mud geopolymer exhibited a positive correlation with the increase in particulate matter content (PCM) and the duration of the cure period.	53

			absorptivity, reduced heat losses, and the capability to provide thermal heating and cooling.		
Micro encapsulated	Paraffin mixed with expanded perlite	Cement mortar	The thermal conductivity of paraffin was improved by the addition of graphite to the mixture.	To obtain enough strength, it was determined that an optimal weight percentage of 20% wt. of phase change material (PCM) was necessary.	54
Encapsulated	Paraffin melamine-formaldehyde	Aerial lime based plaster based on gypsum	The subject of interest pertains to the mechanical properties. The gypsum mortar based on PCM demonstrated a greater energy-saving potential of up to 21%.	The heat storage capacity of gypsum-based mortar is improved by its high micro porosity. The higher performance seen in aerial lime-based mortar may be attributed to its low micro porosity.	55

4.7 TRAIL SAMPLE MIXTURE RATIO

The trail sample mixture ratio of 100% Flyash is defined as M1.70% and 30% of Flyash and GGBS mixture is defined as M2. The 1.1 AAS solution is defined as A. 1.4 AAS solution is defined as B. BG5 represents about 5% of Bio-glycerol. P5 represents about 5% of Paraffin. 15% of Bio glycerol is defined as BG15. 15% of Paraffin is defined as P15. M1B represents the combination of both 100% Flyash and 1.4 AAS solution. M2A represents the combination of both 70%-30% of Flyash and GGBS and 1.1 AAS solution. The mixture ratio is ID is listed in the Table 2.

Table 4.4: Trial Sample Ratios

Sample ID:	Fly ash (g)	GGBS (g)	1.1AAS (ml)	1.4AAS (ml)	Paraffin (ml)	Bio-glycerol (ml)
Control sample ID's						
M1A	25	--	9.35	--	---	--
M1B	25	--	--	9.35	---	--
M2A	17.5	7.5	9.35	--	--	--
M2B	17.5	7.5	--	9.35	--	--
Trial sample mix ID's						
M1AP5	25	--	10.45	--	0.55	--
M1BP5	25	--	--	10.45	0.55	--
M1AP15	25	--	9.35	--	1.65	--
M1BP15	25	--	--	9.35	1.65	--
M1ABG5	25	--	10.45	--	--	0.55
M1BBG5	25	--	--	10.45	--	0.55
M1ABG15	25	--	9.35	--	--	1.45
M1BBG15	25	--	--	9.35	--	1.45
M2AP5	17.5	7.5	10.45	--	0.55	--

M2BP5	17.5	7.5	--	10.45	0.55	--
M2AP15	17.5	7.5	9.35	--	1.45	--
M2BP15	17.5	7.5	--	9.35	1.45	--
M2ABG5	17.5	7.5	10.45	--	--	0.55
M2BBG5	17.5	7.5	--	10.45		
M2ABG15	17.5	7.5	9.35	--	--	1.45

CHAPTER-5

RESULTS AND DISCUSSION

5.1 MECHANICAL PROPERTIES

5.1.1 Compressive Strength

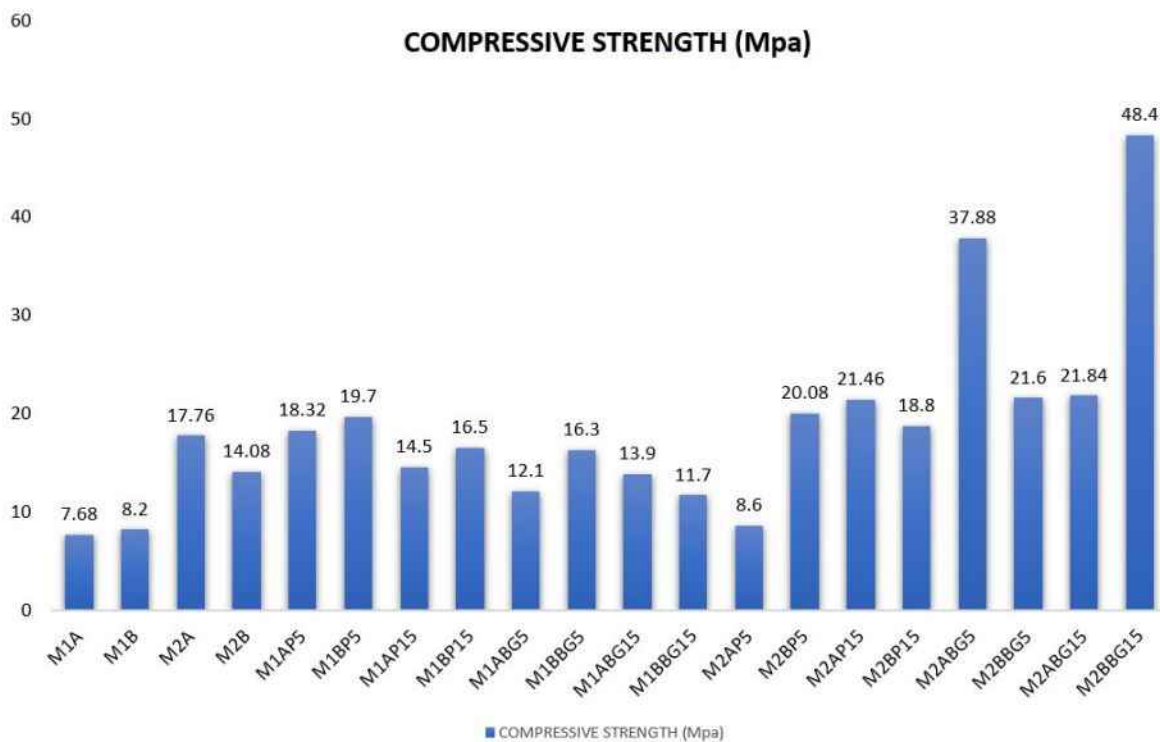


Fig. 5.1 Compressive strength graph of 18 samples

Using compressive testing equipment with a loading rate of 2 KN/s, the composite's compressive strength was assessed at 7 days of age. Strength retention following heat treatment at high temperatures was computed. To understand the effect of the addition of bioglycerol as PCM on composites strength at elevated temperatures studied. The compressive strength is tested in 18 samples and withstands the compressive strength of up to 8 samples like M1AP15, M1BP15, M1ABG15, M1BBG15, M2AP15, M2BP15, M2ABG15, and M2BBG15. By analyzing the various samples 8 samples gives out the better result. M2BBG15 gives out the high compressive strength compared with the remaining 7 samples. According to the

below graph sample M2BBG15 can resist about 48.4MPa of compression compared to remaining 7 samples which has only the compressive resist ranges from 14MPa to 22MPa . From the above test results the sample mixture M2BBG15 has the highest amount of compressive strength. Higher amount of compressive strength leads to the increased stability of the buildings to withstand long life time. From this above study, by adding of bio-glycerol pcm does not leads to affect the compressive strength of the geopolymer concrete.

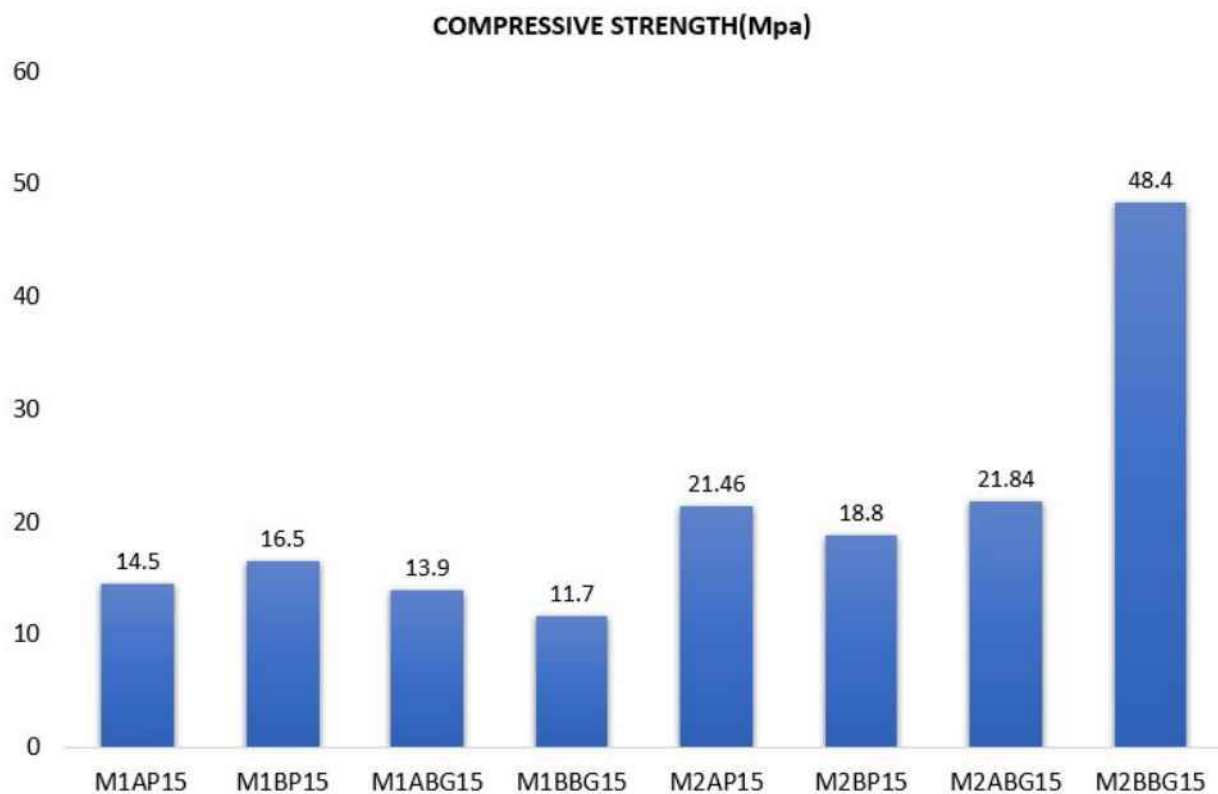


Fig. 5.2: Graph of Good Compressive Strength Samples

5.1.2 XRD analysis of Geopolymer Composites

The XRD method provides insight into potential changes that might have arisen between the initial material and the final geopolymer that was created. The XRD pattern of the geopolymer samples made with 1.1, and 1.4 is shown in figures. There were not many variations in the XRD pattern between the activator's higher and lower concentrations. The XRD pattern of the geopolymer derived from the 100% FA with 1.1 and 1.4 AAS and the PCM of 15% Paraffin or 15% bio-glycerol samples are like M1AP15, M1BP15, M1ABG15, and M1BBG15 is given in the figure.

The geopolymer binder gel formation has been confirmed by the presence of the amorphous N-A-S-H (sodium aluminosilicate hydrate) type gel hump in the range of $26-30^\circ$ in 2θ and the hydroxy sodalite ($\text{Na}_4\text{Al}_3\text{Si}_3\text{O}_{12}\text{OH}$) peak at 2θ value of 24. In addition, other unreactive crystalline peaks are also present such as Mullite(Al_2O_3), Quartz(SiO_2), and Hematite(Fe_2O_3). These phases have weight percentages of approximately ~6% (Quartz), 16.52% (Mullite), and 2.98 % (Hematite), respectively, according to the Rietveld method [23]. The Mullite appeared at 2θ of 12,21,23,24,31,33.7,34.5,35,41,43,48,49 and 50.7.

The Quartz has appeared at 2θ of 21,27,31,36,39,41.3,45.5,50.54,55 and 57. The Hematite has appeared at 2θ of 31,35.5 and 36.5. The aluminosilicate structure of this material is unaffected by these phase shifts, contributing to its dimensional stability. The XRD-6000, an X-ray diffractometer from Shimadzu, Japan, was used for the test. Small fly ash geopolymer paste particles were cut into pieces for the XRD analysis sample, which was then ground into a powder that met the test requirements. The operating parameters for the test were 40 kV and the Cu K-alpha wavelengths were 1.54060 and 1.54439 Å.

M1AP15:

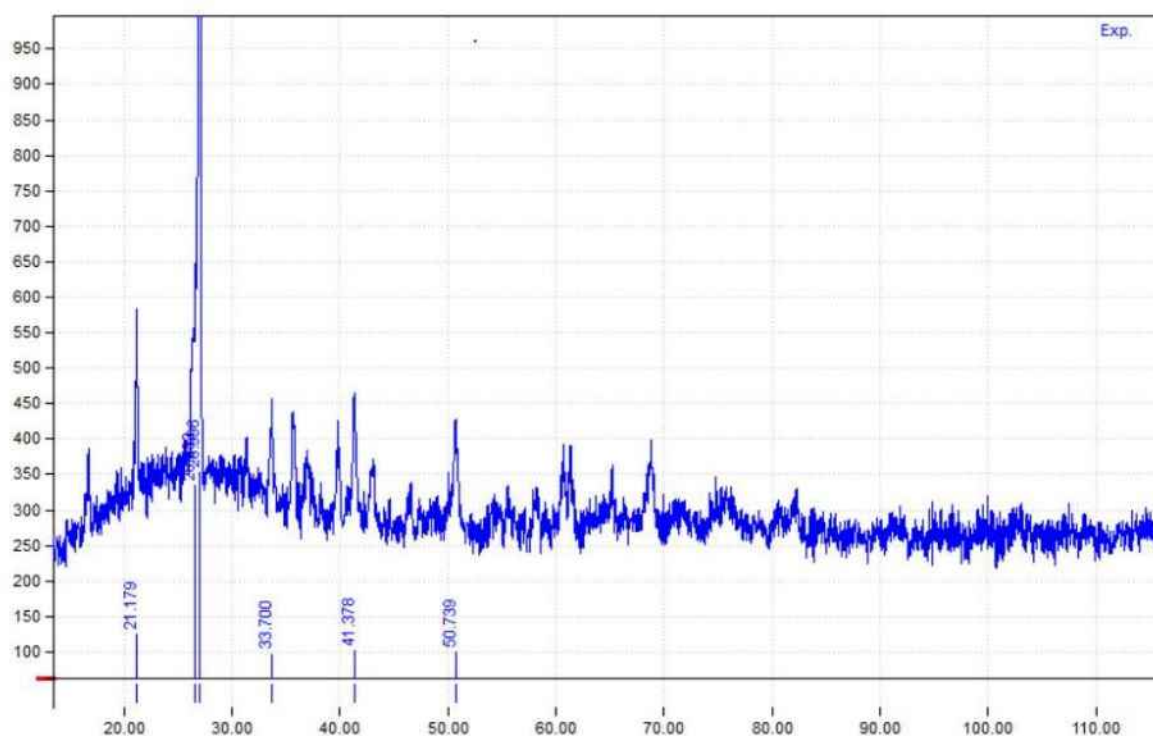


Figure 5.3: Xrd graph for M1AP15

M1BP15:

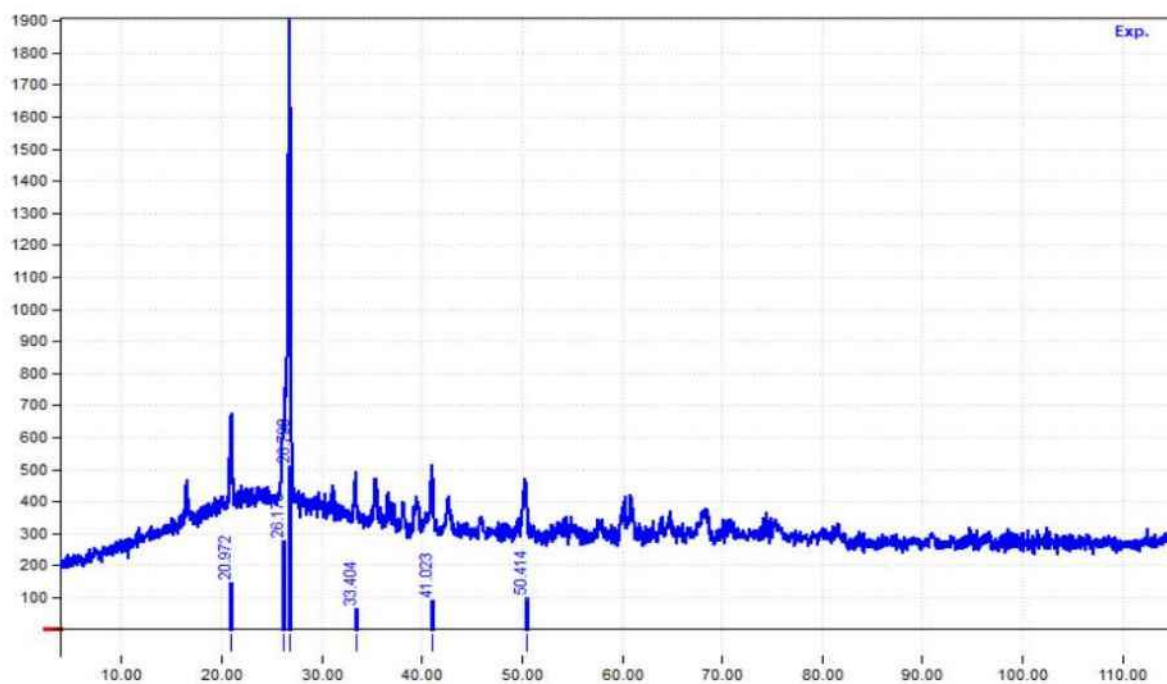


Fig 5.4: Xrd graph for M1BP15

M1ABG15:

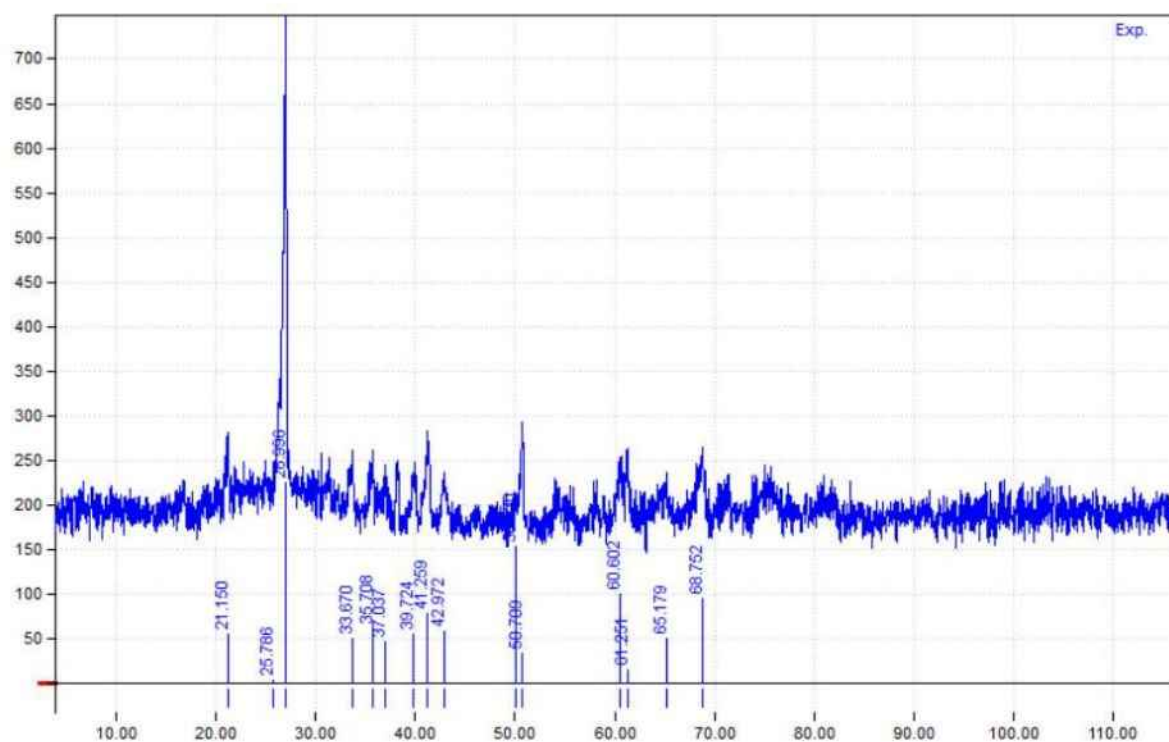


Fig. 5.5: Xrd graph for M1ABG15

M1BBG15:

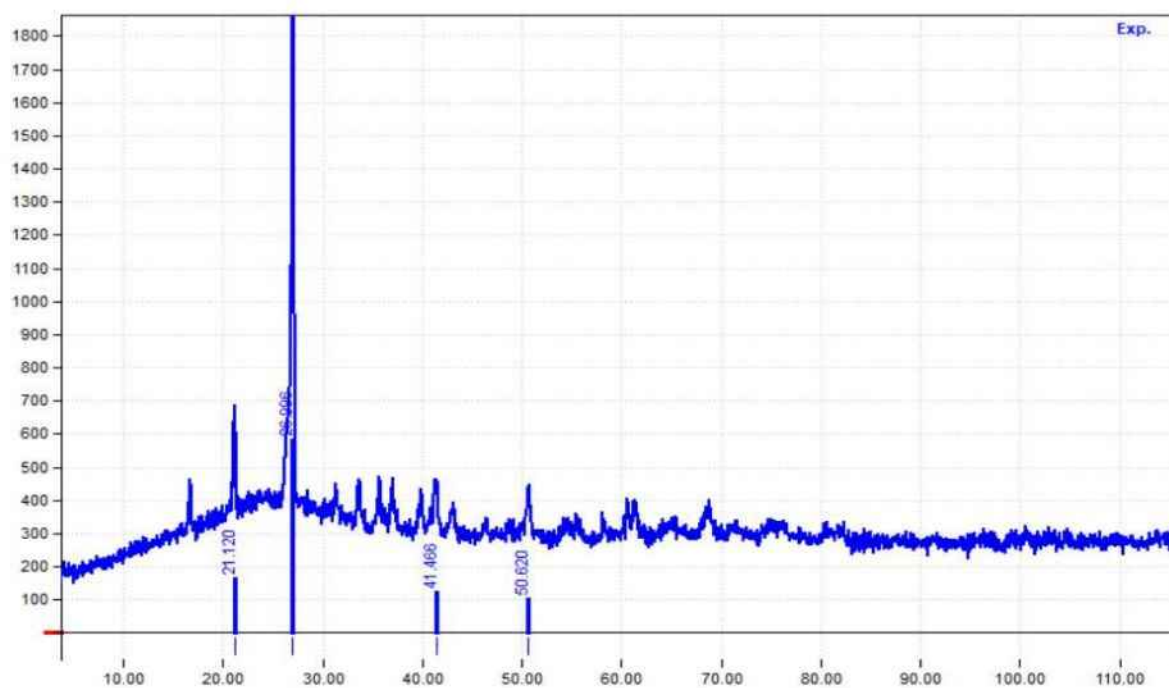


Fig. 5.6: Xrd graph for M1BBG15

The XRD pattern of the geopolymer derived from the 70% FA and 30% GGBS with 1.1 and 1.4 AAS and the PCM of 15% Paraffin or 15% bio-glycerol samples are like M2AP15, M2BP15, M2ABG15, and M2BBG15 is given in the Figures. The Figures display the fly ash and GGBS XRD patterns. After alkali activation, GGBS and fly ash can generate a complicated reaction product that consists of a combination of binding gels, including (N,C)-A-S-H gels (which include Ca and contain N-A-S-H gels) and C-A-S-H gels (which contain Na).

The unreactive crystalline phases are represented by the peaks in the raw fly ash that have been identified as quartz (SiO_2), mullite (Al_2O_3), and hematite (Fe_2O_3). Additionally, the amorphous alumina silicate hump is present in the raw fly ash at around $22\text{--}30^\circ$ 2θ . Akermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$), Gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$), and Anhydrite (CaSO_4) all show peaks in the raw GGBS. These minerals also display a diffused phase at $24\text{--}35^\circ$ 2θ , which is caused by the existence of a glassy phase. The Anhydrite has appeared at 16.8, 21.3, 26.7, 31.9, 34.4, 36.5, 37.6, 38, 40.2, 41, 42, 43.2 and 44. The Akermanite and Gehlenite has appeared at 28.4.

M2AP15:

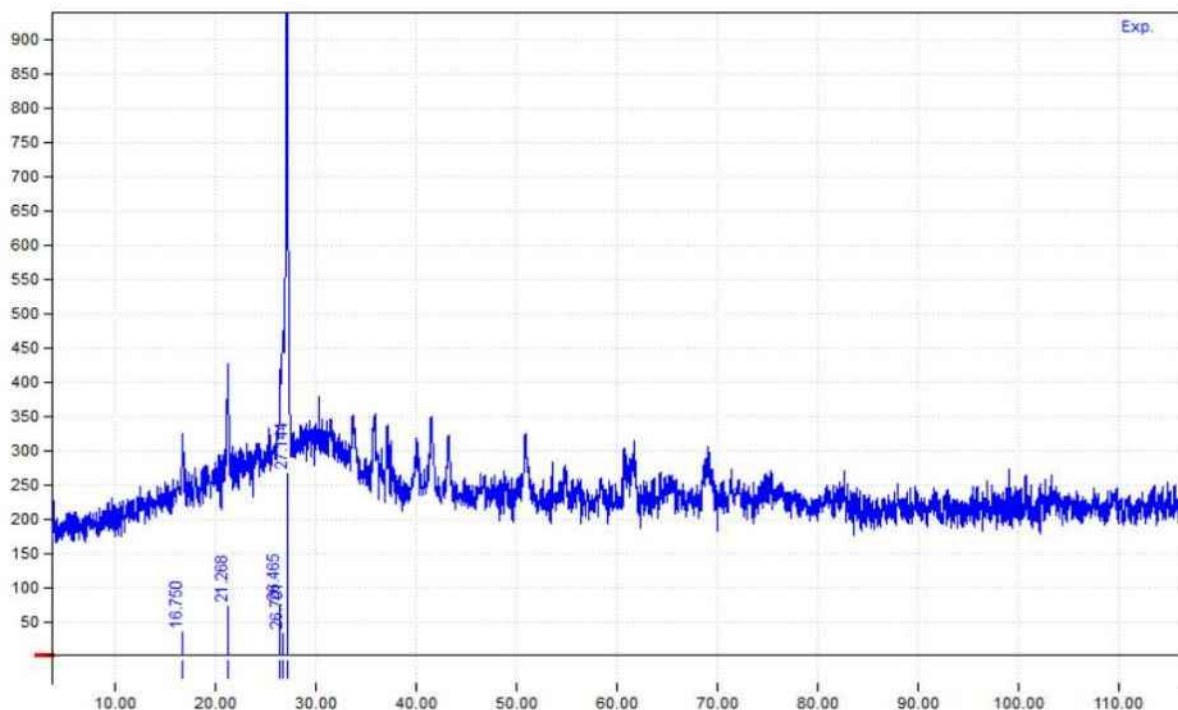


Fig. 5.7: Xrd graoh for M2AP15

M2BP15:

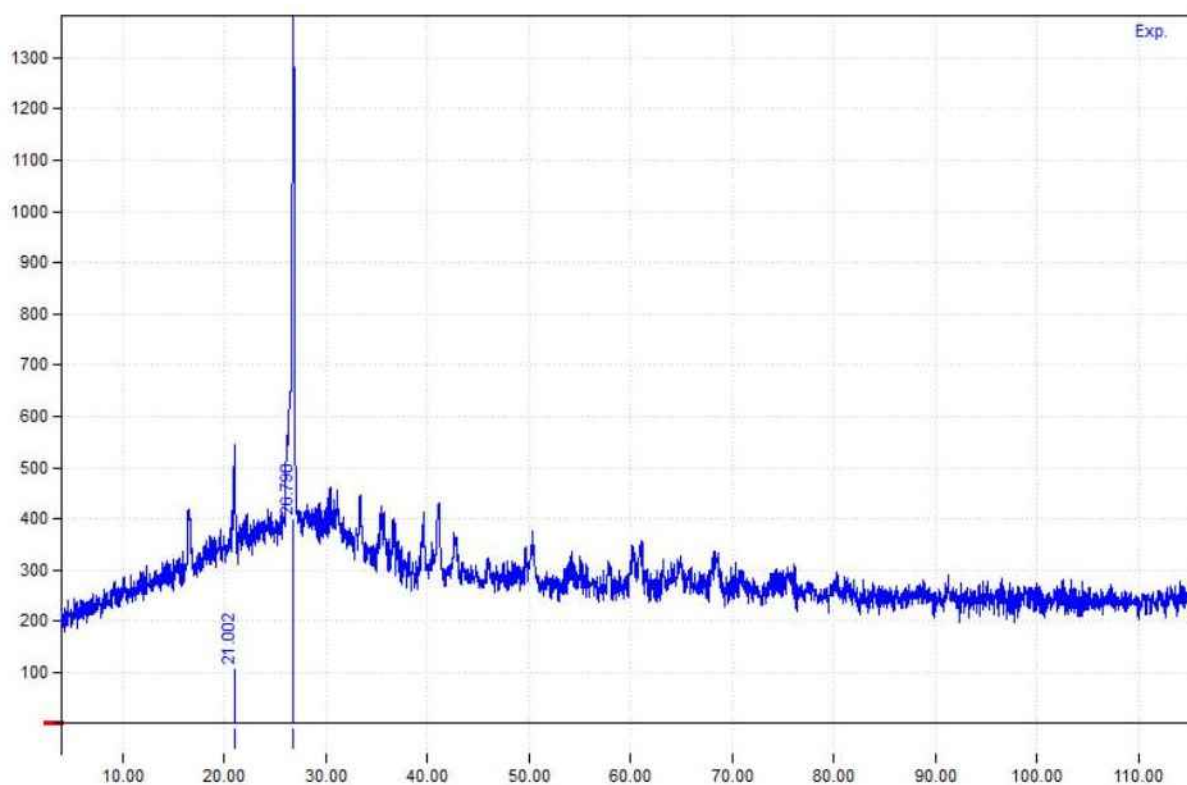


Fig. 5.8: Xrd graph for M2BP15

M2ABG15:

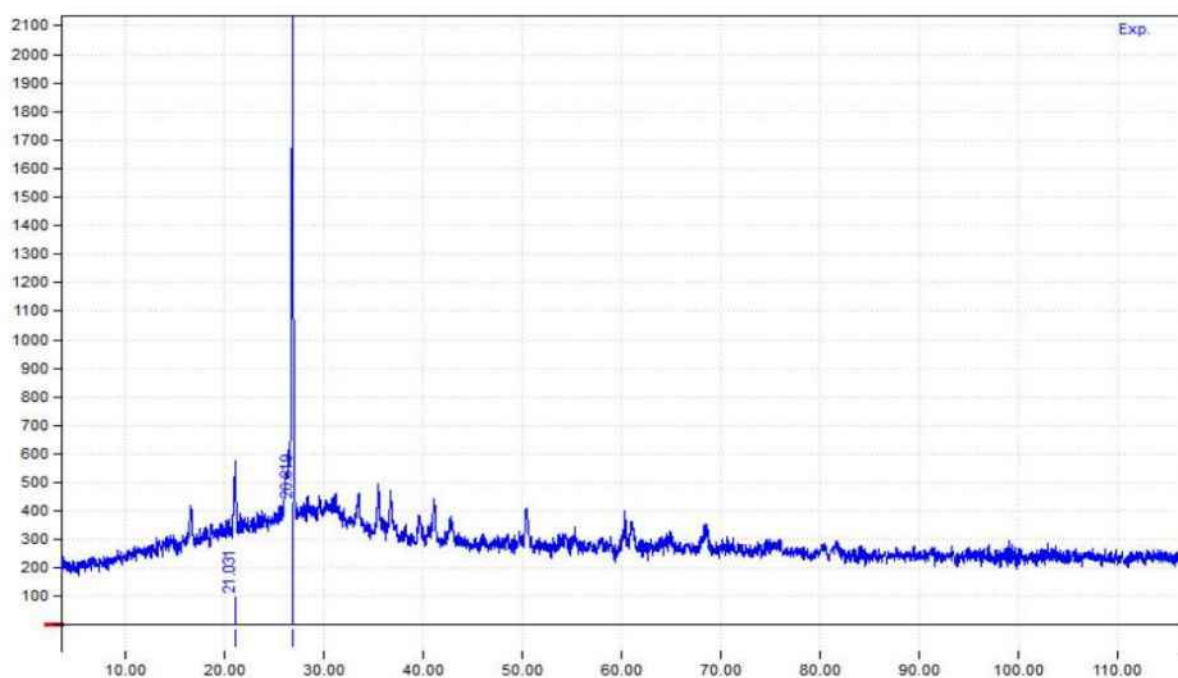


Fig. 5.9: Xrd graph for M2ABG15

M2BBG15:

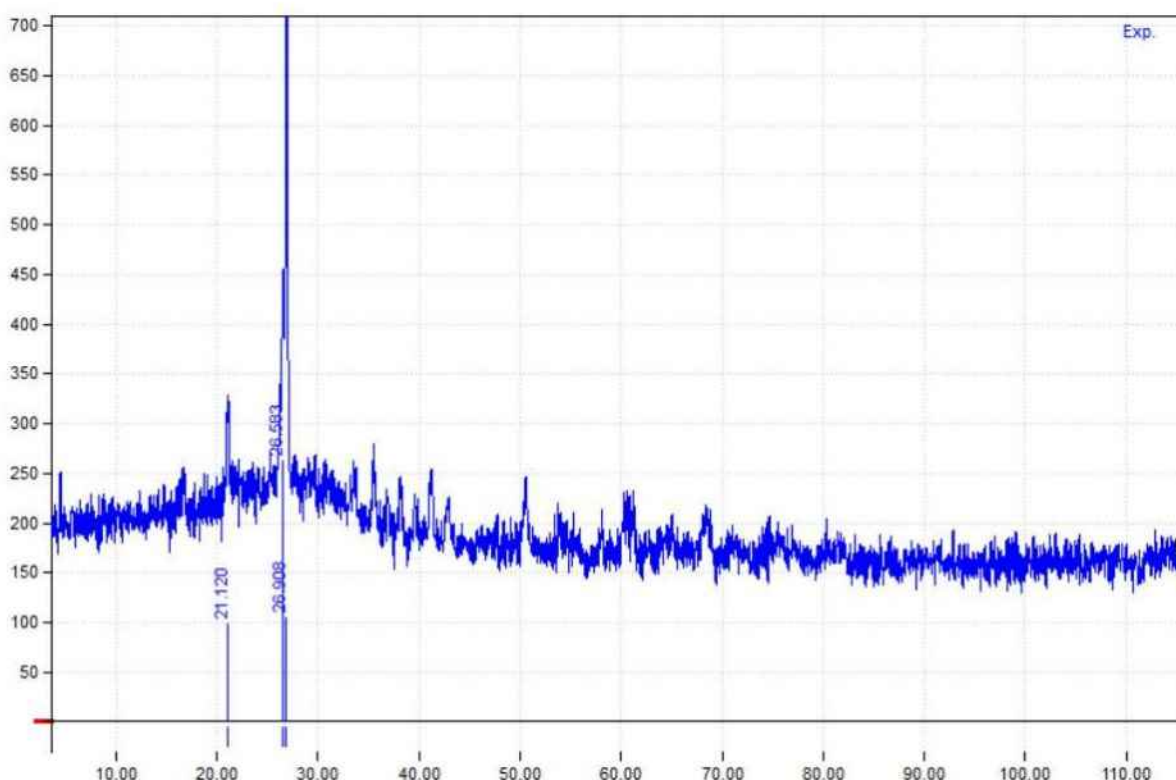


Fig. 5.10: Xrd graph for M2BBG15

5.1.3 ATR FT-IR

In the context of geopolymer characterization, the use of ATR FT-IR spectroscopy plays an important role in determining the reactive nature of the source material and validating the development of the aluminosilicate network structure.

Fly ash geopolymer's vibrational frequency assignment may be adjusted by adjusting the SiO₂/Na₂O by 1.1 and 1.4. The Figure appropriate FT-IR spectra. The Si-O-T (T=Si/Al) stretching band, or fingerprint band for the geopolymer, is caused by an externally asymmetric stretching vibration of the silicate network and emerges in the 950–1250 cm⁻¹ range. A lengthening of the Si-O-T bond, a reduction in the bond angle, and a consequent drop in the molecular vibrational force constant are indicated by a shift of the major Si-O-T asymmetric stretching band to lower wavenumbers. A silicate network may experience this change for a variety of reasons, such as an increase in the percentage of silicon sites with non-bridging oxygen (NBO) charge stabilisation by sodium cations or an increase in tetrahedral Al substitution [24,25,26].

The wave number ranges from 600-650 represents the Network Si-O-Si(or Si-O-Al) symmetric stretching. 730-790 represents the Network Si-O-Si(or Si-O-Al)

stretching. 830-890 represents the O-C-O bending(carbonates). 950-1250 represents Asymmetric Si-O-Si and or Al-o-Si stretching. 1400-1480 represents c-o Stretching(carbonates). The wave numbers are been found in the below graphs peaks represents the different stretching obtained in the variety of samples. A new peak is founded in the samples containing bio-glycerol as pcm at 500-500 range. Same wise the sample containing both flyasd and ggbs indicates some new peak compared to sample containing only flyash. A new peak is obtained at the range of 3500 at this sample mixutres

M1AP15:

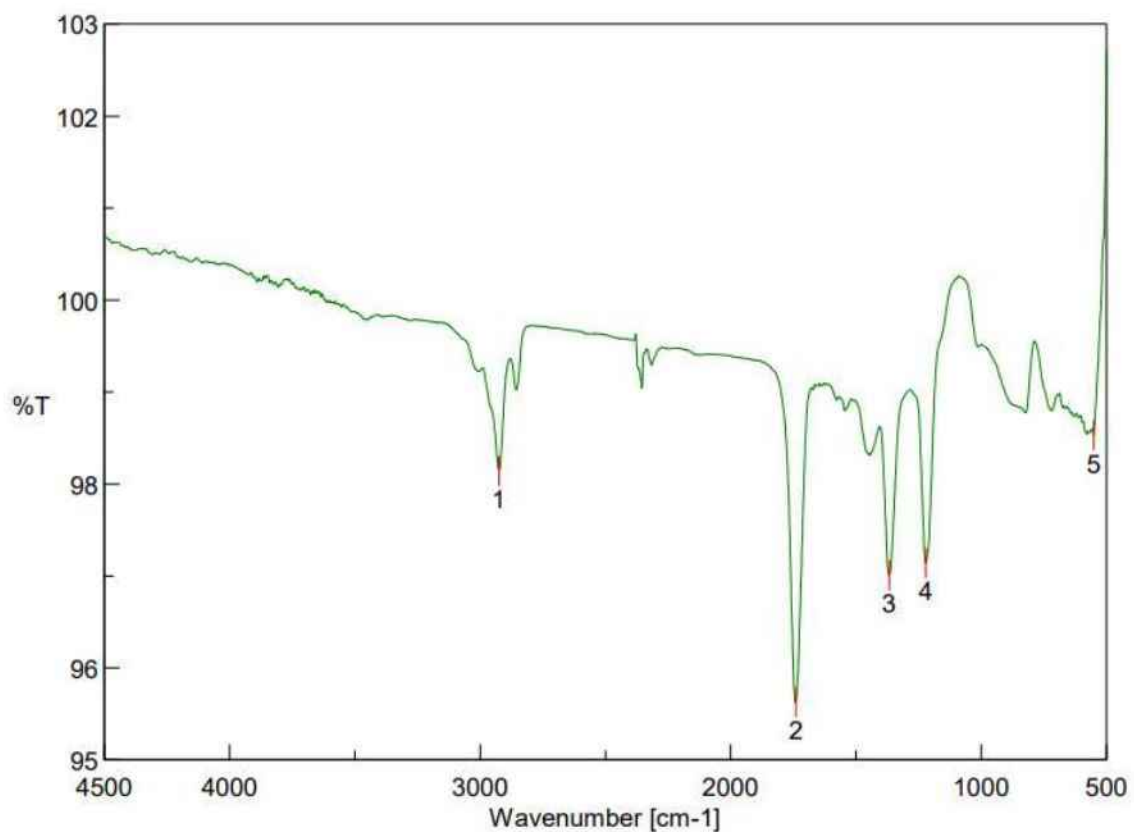


Fig. 5.11: FT-IR graph for M1AP15

M1BP15:

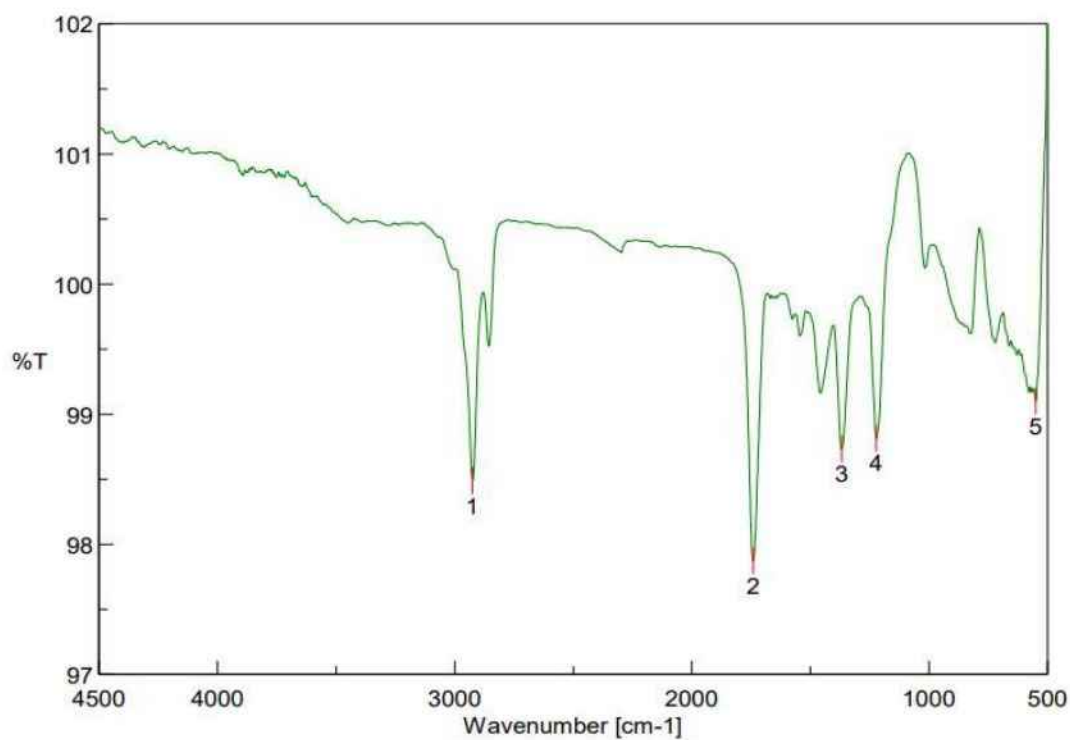


Fig. 5.12: FT-IR graph for M1BP15

M1ABG15:

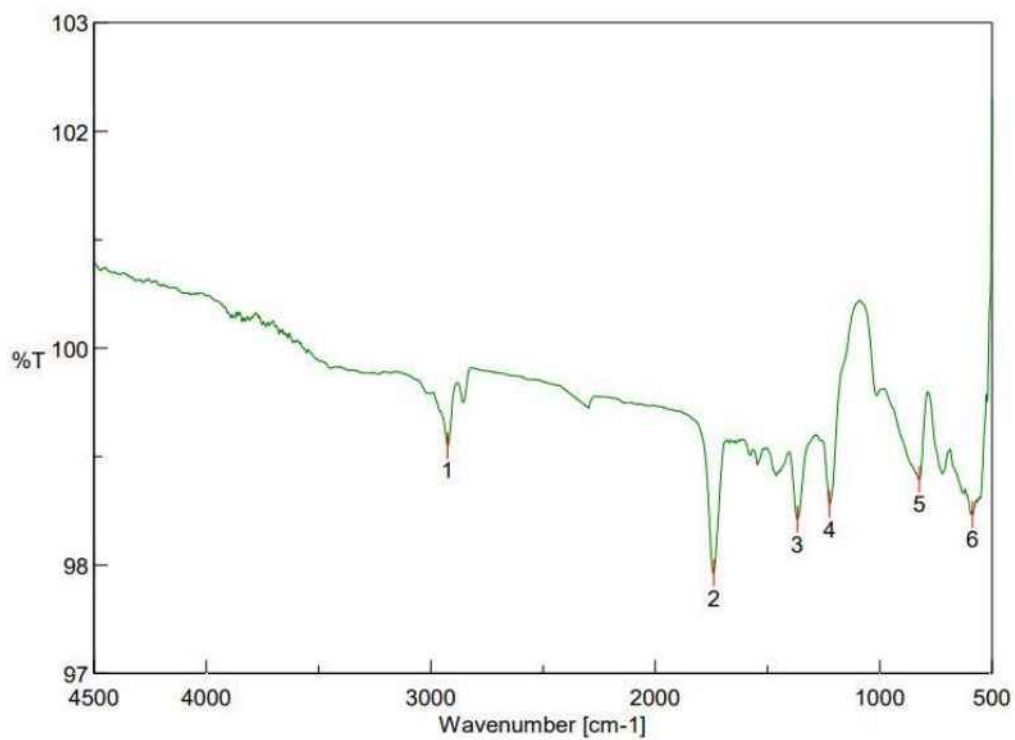


Fig. 5.13: FT-IR graph for M1ABG15

M1BBG15:

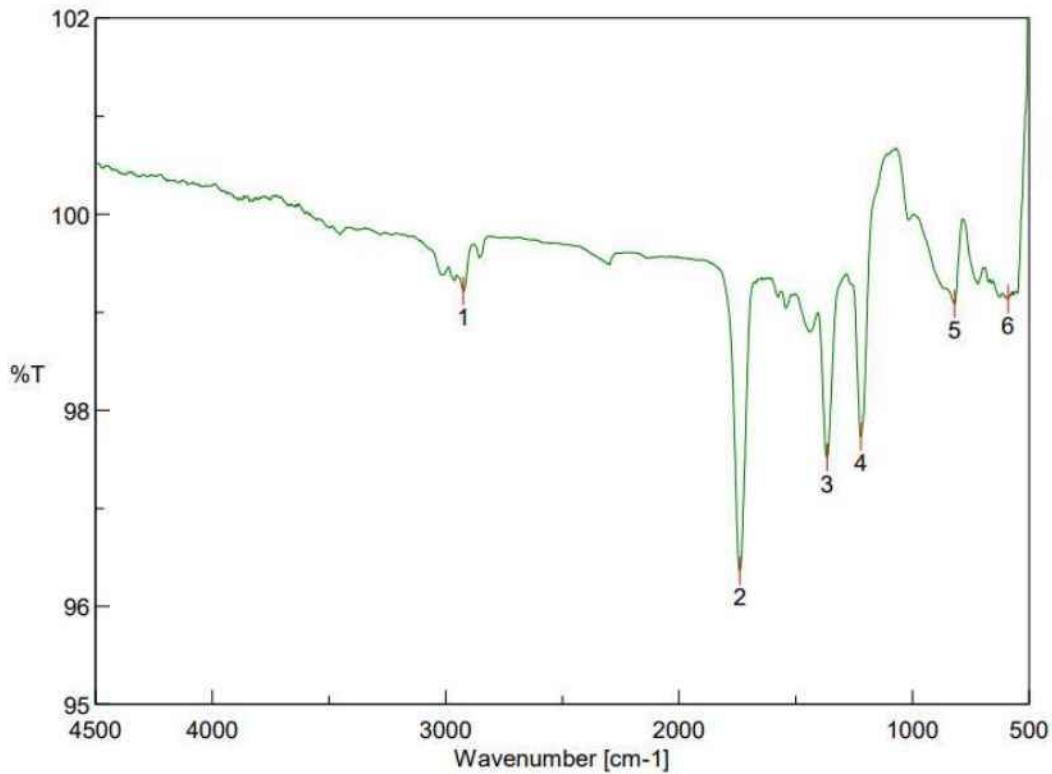


Fig. 5.14: FT-IR graph for M1BBG15

The wave number ranges from 600-650 represents the Network Si-O-Si(or Si-O-A1) symmetric stretching. 730-790 represents the Network Si-O-Si(or Si-O-A1) stretching. 830-890 represents the O-C-O bending(carbonates).950-1250 represents Asymmetric Si-O-Si and /or A1-o-Si stretching. 1400-1480 represents c-o Stretching(carbonates). A new type of peak is obtained in the below graphs. The sample mixture is (flyash+ggbs), at the range of 3250-3500 a new peak is obtained. This peak represents the indication of bio-glycerol pcm mixing. From this it indicates there is some change compared with the two different pcms.

M2AP15:

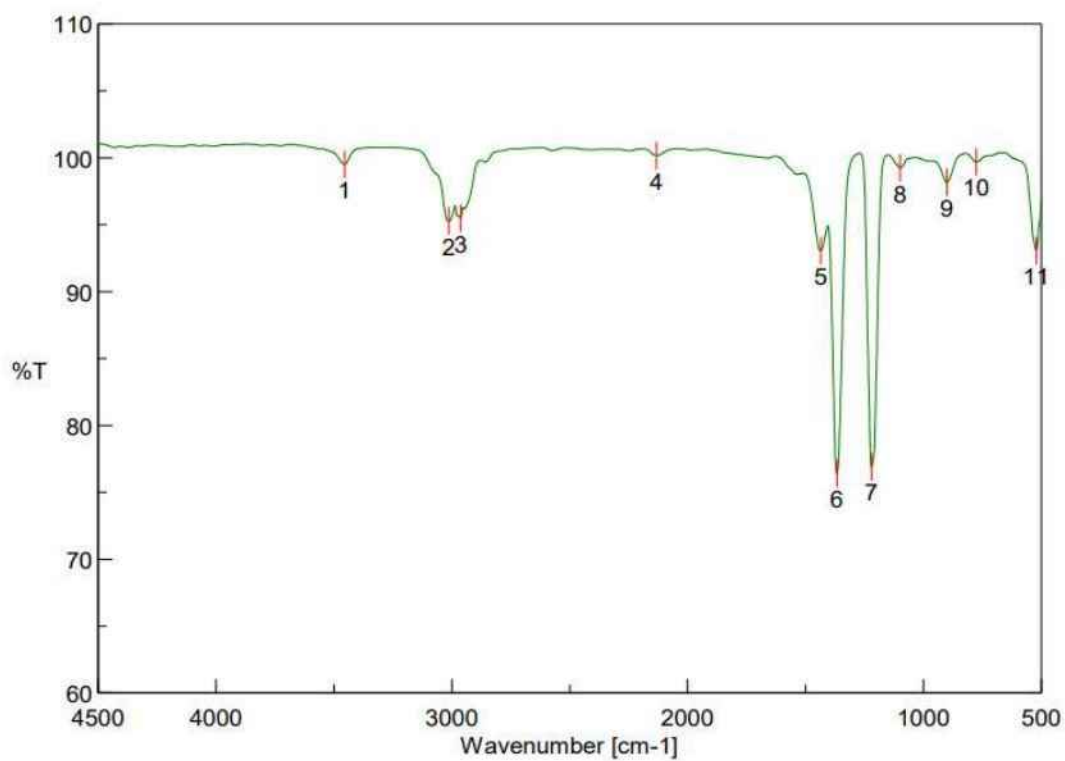


Fig. 5.15: FT-IR graph for M2AP15

M2BP15:

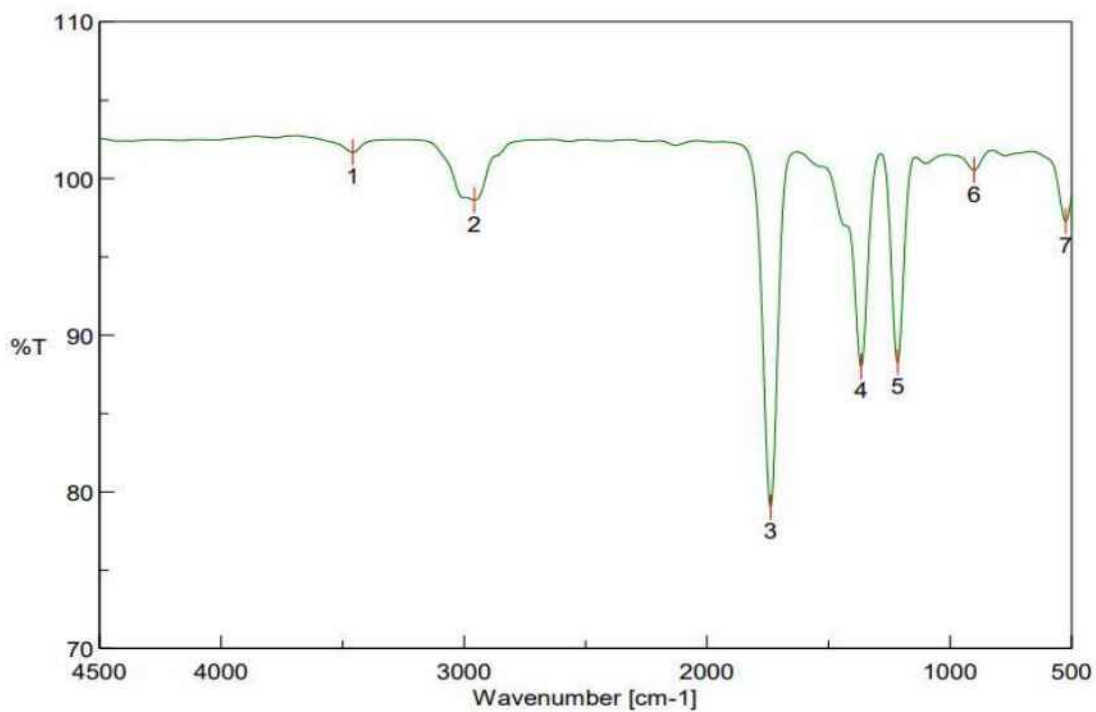


Fig. 5.16: FT-IR graph for M2BP15

M2ABG15:

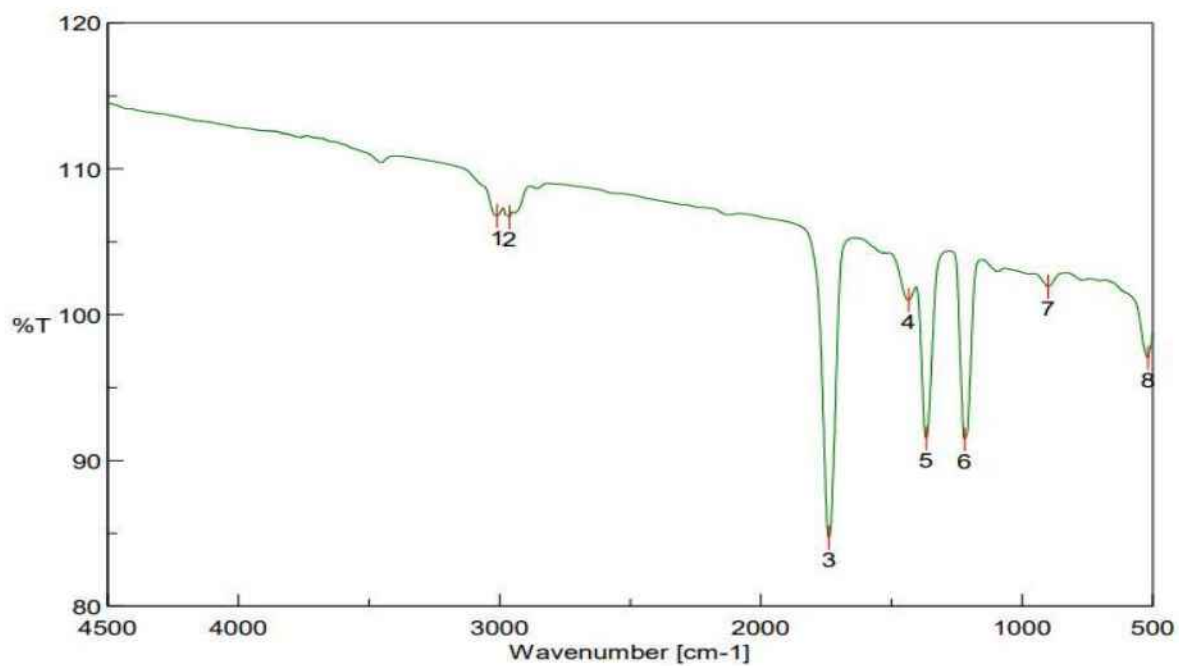


Fig. 5.17: FT-IR graph for M2ABG15

M2BBG15:

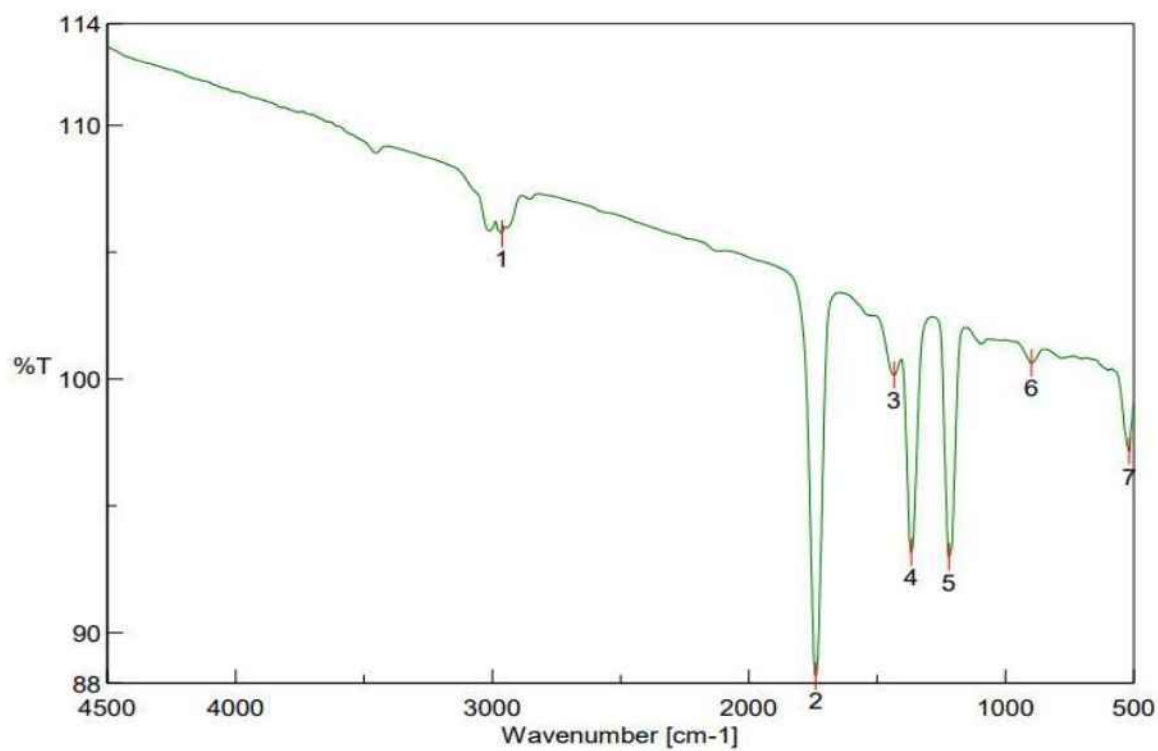


Fig. 5.18: FT-IR graph for M2BBG15

5.2 THERMAL PROPERTIES

5.2.1 Thermal Conductivity

The variations in thermal conductivity of geopolymer mortars are shown with the sample id in the below table. As it can be seen that sample id M2BP15 possesses with the lower thermal conductivity with 0.435 W/mK corresponding to the other sample id's. Variation of the thermal conductivity against temperature studies revealed that thermal conductivity values are declining mainly with increasing temperature. Reduction in thermal conductivity value at higher temperatures may be partly due to the change in the bulk density. Thermal conductivity is directly proportional to the density of the bulk samples. The sample id M2BBG15 possesses higher thermal conductivity compared to the other sample id with 1.1 W/mK. While heating the brick size (21 cm x 11 cm x 7 cm) sample, no visible cracks are observed. The BG addition impacts the thermal conductivity to 1.1 W/m.K positively by increasing the moisture content it is ultimately confirmed via FT-IR studies. The Thermal conductivity results for the various sample are listed in the below table.

Table 5.1: Thermal Conductivity Results of Samples

Sample ID	Thermal conductivity (W/m.K)
M1AP15	0.66
M1BP15	0.545
M1ABG15	0.75
M1BBG15	0.725
M2AP15	0.55
M2BP15	0.435
M2ABG15	0.91
M2BBG15	1.1

SUMMARY AND CONCLUSION

In the realm of sustainable energy technologies, the incorporation of phase change material (PCM) into geopolymer composites for thermal energy storage (TES) applications is a promising development. This evolution allows for the derivation of various important conclusions about the effectiveness, efficiency, and possible uses of these kinds of systems. First off, there are a lot of benefits when it comes to thermal energy storage capacity when geopolymer composites and PCM are combined. As inorganic polymers made from industrial waste, geopolymers offer a strong and chemically robust substrate into which PCM can be embedded. This matrix improves the composite's mechanical strength while also making it easier for thermal energy to be transferred and stored effectively.

Furthermore, a major factor in deciding the composite's overall performance is the choice of PCM. PCM materials can optimize energy storage capacity while guaranteeing quick heat transfer during charging and discharging cycles if their phase change temperatures match the intended operating range. Fatty acids, salt hydrates, and paraffin wax are common PCM options; each has unique benefits with regard to temperature stability and phase change characteristics.

Excellent heat storage and release properties are demonstrated by geopolymer composites coupled with PCM in terms of thermal performance. As thermal energy is applied to the composite during the charging phase, the PCM changes phases from solid to liquid and absorbs a sizable quantity of latent heat. A stable storage environment is ensured by this process, which takes place at a nearly constant temperature. On the other hand, when the PCM is discharging, the heat it releases can be used for a number of purposes, including industrial activities, hot water production, and space heating. The capacity of geopolymer-PCM composites to reduce temperature swings in buildings and other structures is one of their major benefits. Thermal energy can be stored during off-peak hours and released when needed by inserting these materials into construction features like walls, floors, or ceilings. This reduces the demand for traditional heating and cooling systems. This enhances environmental sustainability and overall cost savings in addition to increasing energy efficiency.

Moreover, a broad variety of TES applications can benefit from the long-term stability and endurance of geopolymer composites. Reliability is ensured throughout the operating lifespan of these materials, since they demonstrate negligible degradation even in difficult environmental conditions over time. They also make it easier to integrate them seamlessly into new construction projects and into the retrofitting of existing buildings for energy efficiency upgrades because they are compatible with existing building materials and construction procedures.

Alongside the more general objectives of carbon footprint reduction and the integration of renewable energy, geopolymer composites combined with PCM are being developed. Such systems facilitate the grid integration of variable renewable energy sources, such wind and solar power, by providing effective thermal energy storage and management. Conclusively, the incorporation of phase change material into geopolymer composites for thermal energy storage applications is an extremely promising technology that holds great potential for extensive implementation in diverse industries. These cutting-edge technologies can be crucial in moving us closer to a future that is more sustainable and energy-efficient through continuous research and development activities focused on improving material qualities, boosting system performance, and extending application domains.

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APPENDIX



Image taken during the sample making process