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# SYNTHESIS AND CHARACTERIZATION OF OPTIMIZED PHASE CHANGE MATERIAL BASED GEOPOLYMERIC CONCRETE FOR THERMAL STORGE

*Ishtiaq Ahmad [a],Muhammad Ilyas [b],Ashraf ilyas Khattak [c], Amir Naveed [a]*
*[a] Department of Chemical Engineering, University of Engineering & Technology Peshawar, Pakistan.*
*MOL-Karak Pakistan*
*Department of Civil Engineering, IQRA National University*
Corresponding author: ishu_ktk@yahoo.com

## Highlights

- Optimization of PCM in the geopolymeric concrete.
- Ability of thermal conductance in PCM based geopolymeric concrete.
- Effect of PCM on the compressive strength of geopolymeric concrete.

## ABSTRACT

To adventure the thermal energy storage properties of phase change materials (PCM), it is necessary to investigate its retention in the pores of concrete. This research work is focused on sensible thermal energy storage composite material system using aluminosilicate rich mud and milled glass powder as source material with phase change material PCM was incorporated in the proportion of 0 %, 5 %, 10 % and 15 by weight with aggregate of geopolymeric mixture composed of red mud and milled glass powder. Red mud and milled glass powder are the source materials which contain optimum ratio of silica and alumina which are basic ingredients for geopolymeric reaction. The ratio of chemical activators ($Na_2SiO_3$/NaOH=2.5, 15M) and source materials were kept constant while the ratio of PCM were changed by different weight percent. PCM based geopolymeric slurry was obtained after 30 min mixing with the speed of 120rpm. PCM based geopolymeric paste was pressed in the mould of 50 $mm^2$ at 10 MPa. The prepared mixed geopolymeric concrete was tested for compressive strength, thermal conductance and porosity through universal testing machine (UTM), conductive meter and water absorption method respectively to achieve the acceptable level for applications in buildings for enhancement of their energy. Addition of 10 % by wt PCM provide optimum compressive strength of 28.64MPa, thermal conductance of 2.811 W/m.K, porosity of 3.7 % and the latest heat of 179.2 KJ/kg. Different characterization techniques i.e: XRD, XRF, SEM and flam test were conducted for PCM based geopolymeric material investigation.

**Keywords**: Milled glass powder; Geopolymerization; Phase change material; Conductance.

## 1. Introduction

Energy is the prime factor for social and economic development, while its crisis due to its large application is the bottleneck for both national and international progress [1]. This time, the large proportions are supplied from the fossil natural energy resources [2]. Applications of fossil fuel not only bring harsh environmental impacts but also give birth to energy crisis [3].To reduce the primary energy consumption from non-renewable energy sources and improvement in the integration of renewable energy sources is the need of hour [4]. Solar energy contributes a lot in overcoming of these problems, as solar energy is now utilizing in commercial, utility and somehow in industrial area also. But solar energy has also its limitation in cold areas where sun heat is not enough and in the night time as there are no sun rays available [5]. In modern era energy storage becomes the most favorable. Where solar energy fails to work thermal storage brings the solution and fulfilled the energy crises [6]. Therefore, energy storage in construction was the major focus of researchers from the last decades. Thermal energy storage systems are there tangible solution due to its low costs, energy saving and eco-friendly to eradicate the energy crisis [7].Improving in construction and material techniques can significantly reduce the routine consumption of indoor heat. Thermal energy storage system, conserve and save both sensible and latent heat energy. On the other hand, Thermal energy storage (TES) concrete are after ceramic in nature which need a calcination (1250 °C) and sintering temperature (900 °C) [8].To construct the sintering and free calcine concrete a novel technique is used which is called geopolymerization [9].

The term geopolymerization was first coined by J. Davidovits in St. Quentin, France in 1970s [10]. Geopolymerization is a sustainable development of free sintering with low cost [11]. Mechanism of geopolymerization involves a heterogeneous chemical reaction between alumina-silicate materials with alkali of silicates [12].Geopolymer concrete is a new development in concrete technology in which cementitious material, rich in silica and alumina, is activated using alkaline solution. Sodium silicate and sodium hydroxide was used as alkaline liquid with the ratio of 2.5. Effects of sodium hydroxide concentration on the compressive strength, higher concentration of sodium hydroxide solution result in a higher compressive strength. Higher concentration of sodium hydroxide solution provides better dissolving ability to Meta kaolinite and produces more reactive bond for the monomer, consequently increase inter-molecular bonding strength of the geo-polymer. The compressive strength, flexural strength and apparent density of the resulting geo-polymer were increased. Hydroxide and silicates of sodium or potassium are use as chemical activators for dissolution of fly ash [13].Overall, free sintering geopolymer base concrete has many advantages over other the ceramic concrete such as two steps simple synthesis process with low cost, eco-friendly, high compressive strength and easy addition of agents [14]. Liquid Phase change materials (PCM) are used as matrix agent in the geopolymeric mixture for both storage and release of energy in the form of heat [15].

Phase change materials are existing in organic, inorganic and eutectics []. Phase change materials storage energy and use at in a later time. Initially, heat from external atmosphere is absorbed by the PCM material to certain temperatures which melt and store the energy []. After certain time when the external temperature decrease the melted PCM solidified and release heat [16]. Thermal storage ability of PCM is dependent on the melting point, thermal conductance, outdoor environment and specific heat of the PCM. Building materials especially concrete, high volume, surface and mechanical strength are also the factor of consideration [17]. Therefore, synthesis of sintering-free building materials with direct incorporation of phase change materials (PCM) could reduce energy consumption the system of heating and cooling [18].

In this research work, PCM based geopolymeric building material were synthesized and characterized used for thermal energy storage (TS). Organic affine (C8) were used as phase change material for thermal storage and release in different proportion by weight.

## 2. Material and methods

### 2.1 Materials



Red mud and milled glass pieces obtained from local market (Peshawar, Pakistan) were used as a raw material for geopolymerization reaction. Milled glass pieces are freely available as waste material. Red mud is a residue coming from the metallurgical treatment of bauxite with the Bayer process. Millions of tons of red mud are produced annually worldwide and disposed of on land, degrading vast areas. Therefore, red mud utilization is a first-priority issue for any alumina plant, while milled glass is another source raw material for geopolymerization in this research work. Normally, milled glasses are obtained by thermal activation of broken pieces materials in the range of 600 °C to 1000 °C. Composition analysis of both the source material was done through X-ray Fluorescence analysis as show in table 1. Both red mud and milled glass pieces were grinded and the sized was reduced to 20 µm using ball mill. Slurry paste was obtained by mixing with sodium silicate and sodium hydroxide with the source material at 120 rpm for 30 minutes. Concentrated solution of sodium hydroxide (Nobel chemical limited, Pakistan) 15 M was prepared in deionized water (98 % purity). M.S. S. Salwa et al.,[19] found that porosity and compressive strength are molarity dependent factors. Sodium silicate [Nobel Chemical Limited, Pakistan] of 15.8 % of Na2O 55.0 %, SiO2=30.4% and 60.0 % of water by mass were mixed with sodium hydroxide solution. Ratio of chemical activators were kept 2.5 while the ratio of source material and chemical activators were 2.0/. Para-affine (C8) were added to the paste in the weight ratio of 5 %, 10 % and 15 % at 120 rpm as shown in figure 1. Prepared samples were mould in the 50 $mm^2$ at 10 MPa and placed in the muffle furnace at 60 °C for 1 hour.

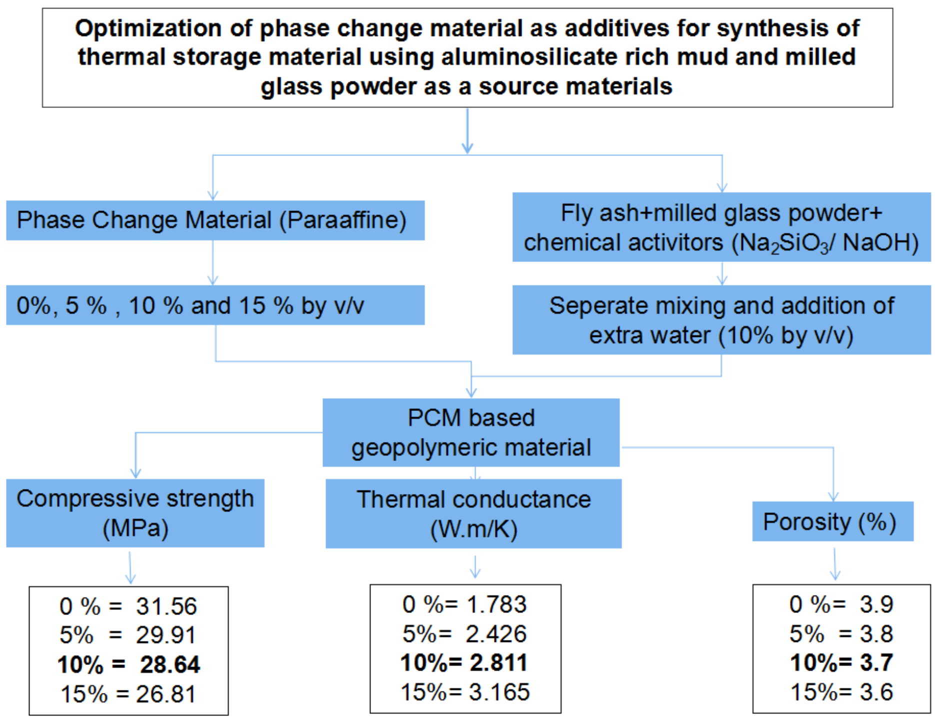


*Figure 1: synthesis and chacterization of PCM based geopolymeric materials for thermal energy storage.*

## 3. Results and discussion

### 3.1 XRF analysis

X-ray Fluorescence analysis (Model: XRF-1800, Manufacturer: Shimadzu) was done for percent composition. The aggregate percentage of silica and alumina oxide in milled glass was found 85 precent while in red mud the percentage was found 51 percent which was considered an optimum ration for development of geopolymeric substrate as shown in table 1. Combination of red mud and milled glass in the 50 by weight ratio needs no additives for silica and alumina oxide for ratio adjustment. After mixing the ratio of $SiO_2/Al_2O_3$ was recorded 2.8 after XRF analysis. Wang et al., [13] reported in his research work that high ratio of aluminosilicate give high compressive strength of resulting material.

*Table 1: XRF (mass %) assurance of composition of milled glass and red mud.*

| Oxides | Composition (Milled glass) | Composition (Red mud) |
|---|---|---|
| $Al_2O_3$ | 28 | 59 |
| $SiO_2$ | 50 | 21 |
| CaO | 3 | 0.004 |
| MnO | 0.03 | 0.32 |
| $TiO_2$ | 3.2 | 0 |
| $Fe_2O_3$ | 12 | 1.42 |
| $SO_3$ | 0.12 | 2.31 |
| $K_2O$ | 0.7 | 1.8 |
| $Na_2O$ | 0.2 | 1.21 |
| LOI | 0.53 | 0.82 |
| MgO | 1.3 | 0 |
| $P_2O_5$ | 0.42 | 0 |



### 3.2 EDX analysis

EDX is a qualitative analysis employed for the chemical composition or elemental analysis of sample. The arrangement consist of characteristics X-ray detector in order to detect and separate the characteristics X-rays of elements present in the sample, liquid nitrogen dewar that is used for cooling and an EDX software to analyze energy spectra of various elements. Silicon and lithium crystals are the two commonly used detectors that operate at low voltages. Crystals of samples absorb the energy of the incident x-rays thus producing free electrons and produces an electrical charge bias. The energy of individual X-rays is thus converted into electrical voltages of proportional sizes. EDX analysis shows that milled glass have high ratio of iron (Fe) , calcium (Ca), magnesium (Mg) and silica (Si). The same compositional analysis was done for red mud as shown in figure 2.

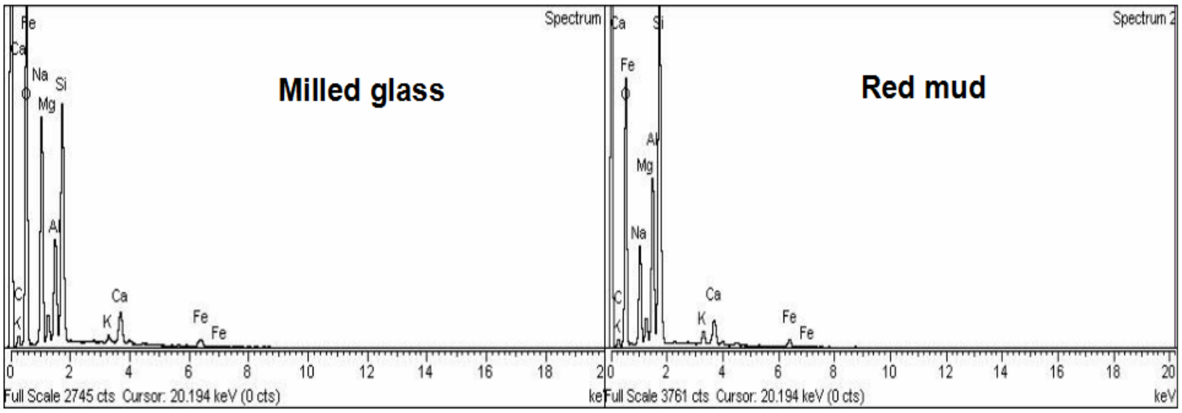


*Figure 2: EDX analysis of Red mud and Milled glass.*

**3.3 XRD analysis**

XRD analysis was done to investigate the nature of source material. XRD spectrum of milled glass shows that the material is semi-amorphous nature after thermal activation and suitable for chemical activation. Peaks for silica and alumina are semi-amorphous; however there are some peaks for semi-crystalline in between 20°-27° at 2θ as shown in figure 3.

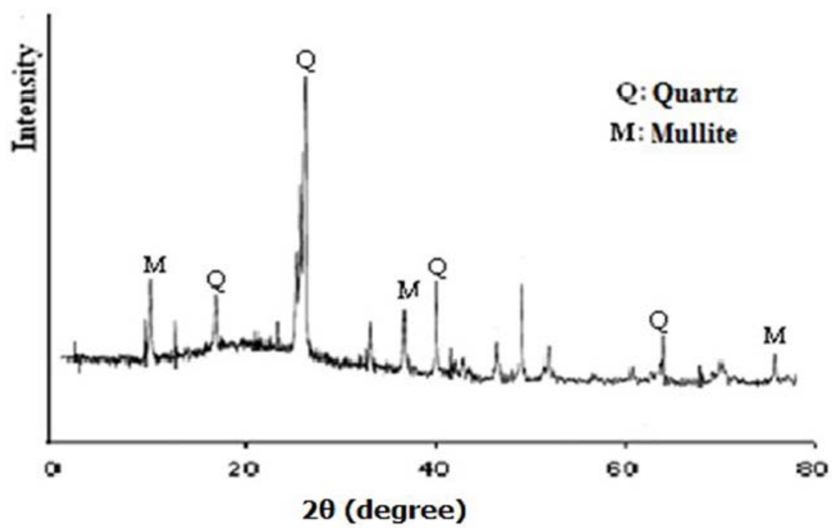


*Figure 3: XRD pattern of milled glass.*

XRD analysis shows that red muds have also amorphous nature. Broad and hump peaks in the range of 15 to 20° shows the amorphous phase of silica in rice husk ash as shown in figure 5. XRD analysis was done to determine the binding nature of elements and hydroxyl ions (OH) which was bound with silica oxide.

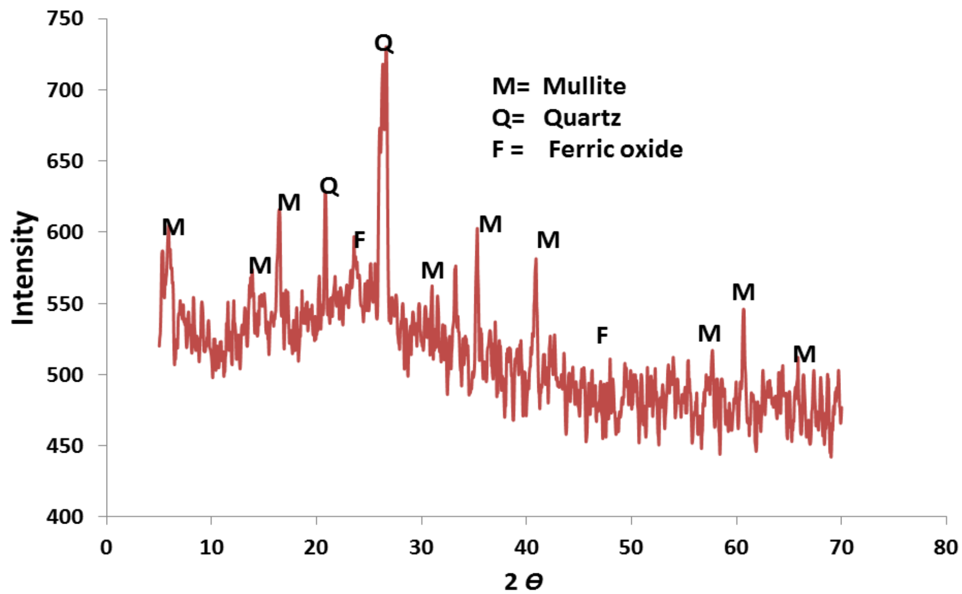


*Figure 4: XRD pattern of red mud.*

**3.4 Compressive strength and porosity analysis**

For compressive strength analysis all the prepared samples was subjected to Universal testing machine (UTM). The PCM based geopolymeric concrete were placed in a universal testing machine and specific load were applied until the sample was breakdown. This procedure was also applied for all the specimens and their respective breakage points were recorded as shown in Figure 1. Thermal conductance and porosity was measured through thermal conductive meter and water immersed methodology. Porosity and thermal conductance of connecter is inversely related to each other. Higher the pores in the concrete lower will be the compressive strength. K. Ram [20], investigated that pores in the concrete resist the in the way of conductance. Other researcher [21], reported that organic agents reduced thermal conductance, compressive strength and increase the porosity during hydrothermal treatment.

### 3.5 Thermal storage capacity of PCM based geopolymeric materials

The heating and cooling of phase change material based geopolymeric concrete was done through a differential scanning calorimeter (DSC) based on nine thermal conductive meters (Material Resource Laboratory, UOP) in the range of 60 to 40 °C. All the measures were repeated at least five times for each substrate of different ratio of phase materials to evaluate the phase change latent heat. All the prepared samples of different PCM based additives was subjected to the differential scanning calorimeter (DSC) at initial temperature of 45 °C and increase up to 50 °C, after 5 mins the sample temperature was reduced to 4 °C. The absorb and release latent heat was recorded as shown in table 2. Results show that geopolymeric concrete of 10 percent by weight store latent heat of 179.2kj/kg**.** G. Kastiukas et al., reported in his research work that phase change materials up to a specific limit increase the thermal conduce latent heat storage and release [22].

*Table 2: Latent heat storage and released capacity of PCM based geopolymeric concrete.*

| Sample | Additive concentration | Process | Phase Temperature | | Latent Heat (kJ/kg) |
|---|---|---|---|---|---|
| Paraffin based Geopolymeric concrete | 5 % wt | Charging | 45 | 50 | 171.4 |
| | | Discharging | 50 | 46 | 153.2 |
| Paraffin based Geopolymeric concrete | 10 % wt | Charging | 45 | 50 | 179.2 |
| | | Discharging | 50 | 46 | 150.4 |
| Paraffin based Geopolymeric concrete | 15 % wt | Charging | 45 | 50 | 174.2 |
| | | Discharging | 50 | 46 | 151.1 |

### 3.6 SEM analysis

In present study, the surface morphology of 10 % by weight geopolymeric concrete was examined using a JEOL 5910 SEM with operating voltage of 20kV at Centralized Resource Laboratory (CRL), Department of Physics, University of Peshawar. Surface SEM of prepared sample was done to find pore size distribution, pore size and any pine holes cracks. It was observed that prepared 10 % by weight geopolymeric concrete surface have no majors and minors cracks as shown in figure.

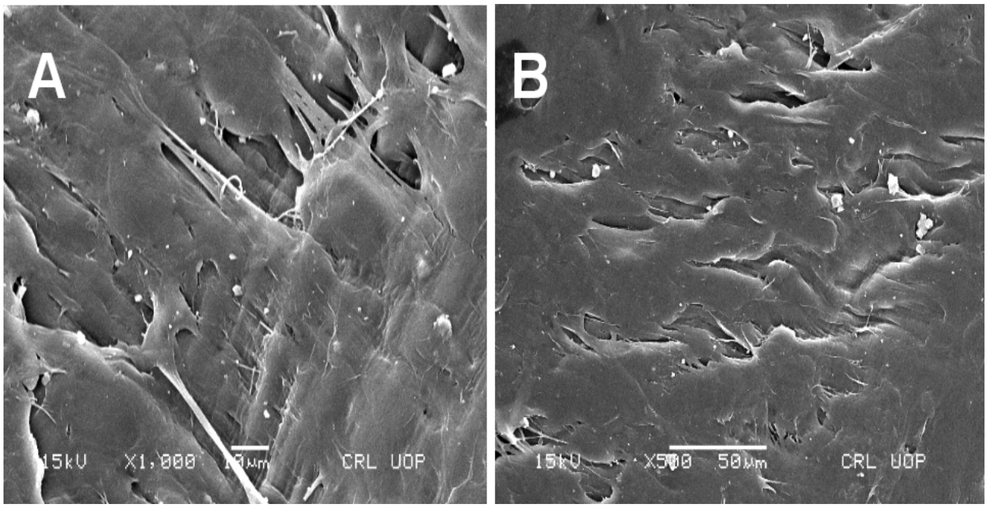


Figure 6: SEM analysis of 10 percent PCM based geopolymeric substrate at different magnification. **(A)**. 10um **(B)**. 50 um.

SEM analysis also conform the compacted monographs of prepared sample after its subjection to thermal shocks. It was observed that there was no minor and major cracks appear after the experimental results. SEM micrograph also shows that no void holes were found in the surface of PCM based geopolymeric concrete.

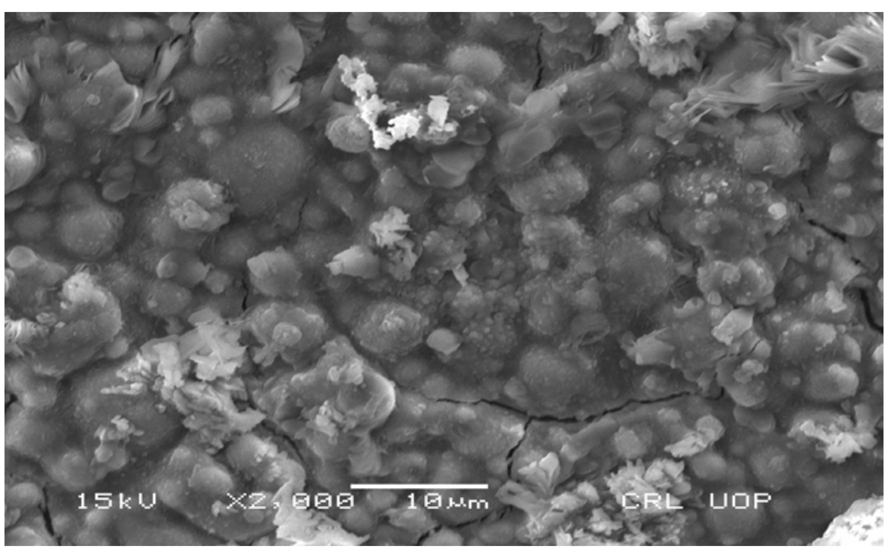


Figure 7: SME analysis of 10 percent PCM based geopolymeric substrate after five cycles.

## 4. Conclusion

Geopolymerization is green technology of sintering-free synthesis inorganic ceramic concrete has many advantages and potential of waste and mineral utilization as raw materials. In this study, transformation of red mud and milled glass to ceramic concrete thermal energy storage was done through geopolymerization reaction. Through experimental results it has been confirmed that using geopolymeric concrete as substrate phase change materials have economic and high thermal storage capacity as compare to other concrete. Results show that latent heat storage (kJ/kg), compressive strength (MPa) and thermal conduction (W/m.k) of phase change material was maximum at the addition of 10 percent by weight. Expensive nature of ceramic concrete in the measures of sintering temperature and high calcination temperature and low thermal conductivity issue can be addressed by using geopolymerization techniques.

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