

Experimental Study on Mechanical Properties and Micro- Structure of Perlite Powder Concrete Subjected to Elevated Temperatures

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Abstract: *Now-a-days fire accidents are occurring more in number. To tackle that a suitable concrete should be made which contains property of fire resistivity. In this research, a material namely Perlite Powder is used, the purpose of PP is to replace with cement for different percentages of PP subjecting to elevated temperatures in concrete. For this purpose, PP with cement replacement of 1, 3, 5, 7% by weight has been made, those specimens kept in a furnace for 1 hr after reaching the target temperature. Experiments conducted in this study include compressive strength, flexural strength, tensile strength at 28 days. Micro- structural analysis such as X-Ray Diffractometer (XRD), Scanning Electron Microscope (SEM) were conducted. The researches have focused that the PP with cement replacement of 5% by weight achieved better results at elevated temperatures when compared to normal concrete.*

Index Terms: *Compressive strength, Elevated temperature, Fire resistance, Perlite powder, Micro-structural analysis*

I. INTRODUCTION

Perlite is a type of natural volcanic glass form of rhyolitic magma. The name perlite was given to it in 1822. Although many experiments were done on perlite in 1929, the real breakthrough came in the 1930s, expanded perlite being invented in 1938. When Perlite was heated to 900-1100 °C, the surface of the grains softens and in the pores the sealed water changes into steam that causes increase in the volume then it transforms in to a light weight cellular material [1]. This expanded perlite is used in several applications like in construction, industry, horticulture [2]. Use of natural PP in concrete has the following advantages: reduction of Portland Cement consumption, improved workability, lower permeability, higher strength and durability [3].

Perlite is a glassy volcanic rock obtained from Pumice. It contains 70-75% SiO₂, 12-18% Al₂O₃ and 2-5% artificially consolidated water. Due to the presence of high SiO₂ and Al₂O₃ contents perlite shows pozzolanic characteristics. Upon rapid warming, water held in the perlite vaporizes and forms bubbles in the softened rock, this causes perlite expansion up

to fifteen to twenty times of its volume. This microstructure gives perlite several favorable properties such as great insulation properties, low density and high porosity making the expanded perlite a lightweight mineral filler. Perlite concrete can also be sprayed as a fire retardant backup for metal curtain walls [4]. Most of the Perlite reserves are in Turkey approximately 4.5 billion tonnes out of 6.6 billion tonnes in the world. Other reserves are located in Greece, USA, Japan, Philippines, Russia, Hungary, Mexico and Italy [1]. The benefits of light-weight concrete are the low-heat conductivity and unit weight. Today, lightweight property of concrete has been still used in constructional elements such as brick, plaster, pipe, wall and floor block. The high rise structures have been influenced by earthquakes because of high unit weight of concrete. The substitution of Perlite Powder in concrete might be the solution for diminishing damages of earthquakes attributable to lightweight property [1]. However, mixed concretes with perlite may cause strength losses at early ages contrasted with Portland cement, then the strength is enhanced by pozzolanic reactions in further [3].

The concrete material is highly susceptible to elevated temperatures evolved during fire accidents and proximity to reactors or furnaces. Their characteristic properties such as modulus of elasticity, volume deformation, structural integrity, strength of the structural elements, etc., are degraded during these exposures. This leads to adverse structural changes and failures. So, a study on behaviour of concrete material is crucial. As the temperature is increased, surface water and capillary water is lost due to the reduction of cohesive forces. Around 110°C dehydration will cause to escape chemically bonded water from CSH. From 800C to 150°C dehydration of ettringite takes place and at the same time decomposition of gypsum between 150°C and 170°C. After reaching 300°C dehydration leads to the expansion of aggregate, which increases internal stresses and therefore micro cracks develops in concrete material [5]. As the temperature is raised, further decomposition of portlandite occurs between 400°C and 540°C. At around 530°C the calcium hydroxide which stops its interference within the material leads to shrinkage of concrete [6]. When the temperature increases beyond 4000C the concrete strength decreases rapidly due to the degradation of calcium-silica-hydrate (C-S-H) [7]. Concrete reaches disintegration stage at 800°C and at 1150°C where melting of feldspar takes place. Then above that all the minerals in the compounds changes to glass phase from cement phase [5].

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This makes the concrete material lose their strength and leads to collapse of structure. Hence, it is necessary to study the behavior of concrete material with PP under elevated temperatures.

Thermal conductivity of a material is the quantity of heat transmitted through a unit thickness in a direction perpendicular to a surface of unit area, due to a unit temperature gradient under given conditions. Thermal expansion and contraction behaviour of concrete varies primarily with aggregate type, cementitious material content, w/c ratio, temperature range, concrete age, and ambient relative humidity. Of these factors, aggregate type was found to have the greatest influence on the expansion and contraction of concrete [7]. Moisture content, w/c ratio, and type of cement were found to only affect the thermal expansion at relatively low temperatures, i.e., $T < 200\text{ }^{\circ}\text{C}$ [7]. For a constant amount of water, the coefficient of thermal expansion of concrete reduces with decreasing cement content [7]. Thermal conductivity of concrete increases with increasing cement content and aggregate [8].

The concrete having fire resistivity is appreciable, whether it is in material form or in structural form. The issues related to the elevated temperature risk can be reduced by the application of concrete as a structural material. Use of perlite powder replacement with cement makes the concrete inert for fire and safety structural design. The formation of bubbles, after vaporisation, reduces the heat transfer mechanism thereby increasing fire resistance to the concrete. Economic and environmental imperatives are bound to increment in the upcoming years and one of its development industries is to acquire more energy efficient buildings and construction materials. An essential way to achieve such buildings is to improve the thermal insulation properties. Reduction of the heat loss in buildings decreases the consumption of energy, thus, reduces the cost of heating and cooling [9]. Due to its higher porosity, lightweight concrete is a suitable material for thermal insulation of structures [9]. Reduction in portland cement utilization helps to diminish the CO_2 discharge and the cost because of the lower strength utilization amid calcining and granulating. Since more than half of the electrical energy utilization accompanied by concrete for granulating crude materials and clinker [10].

Even though such pozzolanic parameters were explored by few researches, no studies have been conducted on the usage of perlite in manufacturing of cements. Currently, perlite powder has been used as a replacement of aggregate in concrete all over the world. Further studies can be conducted to investigate in this area. Therefore, the present study is a part of experimental program carried out to identify the suitable and optimum PP proportion to replace cement in concrete when subjected to elevated temperatures.

The specimens are kept under elevated temperatures namely 27°C , 200°C , 400°C , 600°C , 800°C by replacing the cement with perlite powder for 0%, 1%, 3%, 5% and 7% mix proportions. For elevated temperatures muffle furnace is used at constant temperature up to 1 hour after reaching the target temperature. Then the specimens were tested according to the code of provisions. Samples of various mix proportions were given for micro structural analysis to identify the element/mineral composition and chemical compounds in it.

II. MATERIALS AND PROPERTIES

A. Perlite powder

Perlite powder has high water substance and contains the property of uncommonly expansion when exposed to high temperature. The PP has a significant pozzolanic effect and is an active mineral admixture, and also has high freeze-thaw resistance and fire protection capability [1]. PP was used as a pozzolanic mineral by replacing 1%, 3%, 5%, 7% of cement by weight. PP was purchased from Astra Chemicals, Chennai, India. The physical and chemical properties of PP are given in Table 1 and 2 respectively.

Table 1 Physical Properties of PP

PRODUCT NAME	PERLITE (– SF POWDER)
Colour	White
Bulk Density	40 – 50 kgs. per cu. m
Ph	Neutral
Thermal Conductivity	0.040 W/M ² K At 0 ⁰ C
Moisture Content	0.5% Max.
Organic Content	0.1% Max.
Specific Gravity	2.36

Table 2 Chemical Composition of PP

Chemical Compounds	% of Composition
Silicon dioxide (SiO_2)	72-76 %
Aluminum oxide (Al_2O_3)	11-16 %
Sodium oxide (Na_2O)	2-5 %
Magnesium oxide (MgO)	0.1-1 %
Potassium oxide (K_2O)	1-5 %
Calcium oxide (CaO)	0.5-2.5%

B. Cement

A 53-grade Ordinary Portland Cement has been used and at most care was taken to keep it from getting influenced by dampness. The physical properties of cement are shown in the Table 3

Table 3 Physical Properties of Cement

Tests Conducted	Value	
Fineness of Cement	7.63%	
Specific Gravity of Cement	3.15	
Setting Time of Cement	Initial	36 min
	Final	369 min

C. Coarse Aggregate

Coarse Aggregate obtained from locally available pulverized rock, sieved through 20 mm sieve is used. It was washed to remove dirt, dust and dried under dry conditions.



The aggregates tested according to the Indian Standard Specifications IS 383-1970. Table 4 shows the properties of the coarse aggregate used for this study.

D. Fine Aggregate

Sand confining to Zone-II of IS 383-1970 was used as a fine aggregate. It should be dirt-free, inactive, free from natural clay and silt. Table 5 shows the properties of the fine aggregate used for this study

Table 4 Properties of the coarse aggregate

Tests Conducted	Value
Specific Gravity	3.06
Water absorption	0.201%
Fineness modulus	7.30

Table 5 Properties of the Fine aggregate

Tests Conducted	Value
Specific Gravity	2.44
Water absorption	0.416%
Fineness modulus	2.69

III. EXPERIMENTAL PROCEDURE

A. Mix Proportioning of Cement

The experimental work carried out by replacing cement with Perlite Powder (PP). The Table 6 shows the quantity of the ingredients per cubic meter as per the mix design made according to IS 10262-2009.

Table 6 Mix Proportions

Materials	Quantity
Cement	438.35 (kg/m ³)
Fine Aggregate	690.29 (kg/m ³)
Coarse Aggregate	1123.76 (kg/m ³)
Water Cement Ratio	0.43

B. Casting of Specimens

The ingredients such as cement, Perlite Powder (PP) are mixed homogenously to make the mix in uniformly. The quantities used for the preparation of specimens have been given in Table 7. The Slump value of 50 mm to 70 mm was maintained uniformly for all the mixes. The Specimens were demoulded after 24 hours of casting and kept for curing in curing tank. The various mix proportions of the concrete specimens replaced with PP to the cement were named as NC (Normal Concrete), 1PP, 3PP, 5PP, 7PP for 0% PP, 1% PP, 3% PP, 5% PP, 7% PP mix proportions respectively.

C. Exposure of Specimens to Elevated Temperatures

After 28 days of curing, the specimens such as cubes, beams and cylinders were taken out and dried for 24 hours before they kept in the furnace. The specimens after reaching the target temperature they were kept in furnace for one hour respectively. For 200⁰C and 400⁰C the time taken to reach the target temperature is 30 minutes whereas for 600⁰C and 800⁰C it took 60 minutes and 80 minutes respectively. After sometime the specimens are taken out from the furnace and were left outside for cooling. Then the specimens were tested for strength tests to determine the load carrying capacity. The Figure 1 shows the rate of rise in temperature of the specimens kept in the furnace.

D. Testing of Specimens

The concrete cube specimens are tested in compressive testing machine, Split tensile test on cylinder specimens and flexural test on beam specimens. To study the micro-structural analysis, XRD and SEM analysis were conducted.

For this experimental work a total number of 225 specimens were prepared. In that 75 cube specimens of size 100 mm x 100 mm x 100 mm and cylinders of 75 numbers with a size of 100 mm x 200 mm and 75 numbers of beams of size 500 mm x 100 mm x 100 mm were cast. Then 75 specimens of every percentage replacement of PP mix proportion were tested and the values were averaged to get optimum results.

Table 7 Mix Proportions of Binary concrete

Mix ID	Cement (kg/m ³)	Perlite Powder (kg/m ³)	Fine Aggregate (kg/m ³)	Coarse Aggregate (kg/m ³)	Water (litres)
NC	438.35	0.00	690.29	1123.76	188.49
1PP	433.97	4.38	690.29	1123.76	188.49
3PP	425.20	13.15	690.29	1123.76	188.49
5PP	416.43	21.92	690.29	1123.76	188.49
7PP	407.66	30.68	690.29	1123.76	188.49

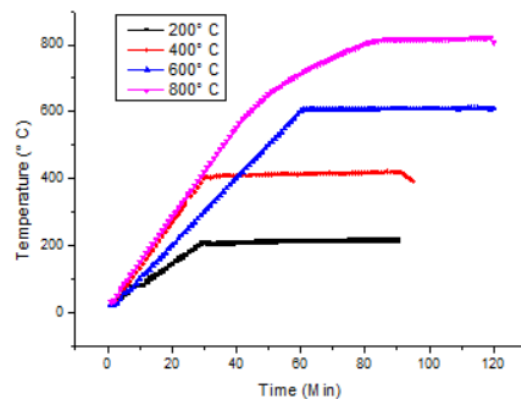


Figure 1 Time vs Temperature Graph

IV. RESULTS AND DISCUSSIONS

A. Compressive Strength Test:

The recommendations from IS: 516 is considered for compressive strength test [11]. From Figure 2, it infers that the 5PP concrete mix has attained more strength when compared with other percentages of PP replacement. For 5PP concrete mix, the strength has increases by 3% from 27⁰C to 200⁰C and from 200⁰C to 400⁰C strength increase by 5%. This is due to the increase in strength after exposing to 300⁰C, as further hydration of non-hydrated cement will produce more CSH which in turn makes the concrete strengthened [12]. Further increase of temperature results in reduction in strength by 8% from 400⁰C



to 600°C and further reduction of strength by 21% from 600°C to 800°C because of degradation of C-S-H (Calcium Silicate Hydrate) compound and chemically bond water escapes from C-S-H which rapidly decreasing strength beyond 400°C.

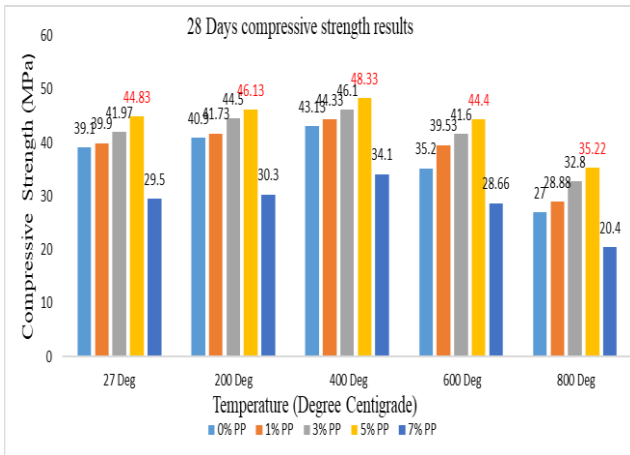


Figure 2 28 Days Compressive Strength Test Results



Figure 3 Compressive Strength Test

B. Flexural Strength Test

Flexural Strength Test was performed as per code of provisions IS 516- 1959 recommendations [11]. The flexural strength test of the concrete specimens was tested after 28 days of curing.

From the Figure 4 it infers that the 5% PP concrete mix, flexural strength increases by 6% from room temperature to 200 °C and from 200 °C to 400 °C it increases by 7%. The maximum flexural strength has attained at 400 °C. Further increase of temperature leads a reduction in strength by 15% and decreases by 47% from 600°C to 800°C. It clearly shows that the flexural strength test followed the similar trend as observed in Compressive Strength test.

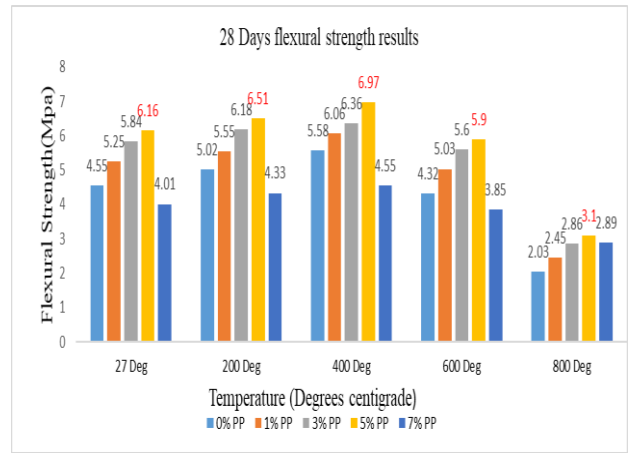


Figure 4 28 Days Flexural Strength Test Results



Figure 5 Flexural Strength Testing Machine

C. Split- Tensile Strength Test

Split Tensile Strength Test was done as per code of provisions IS 5816 recommendations [13] and the results were taken after 28 days of curing.

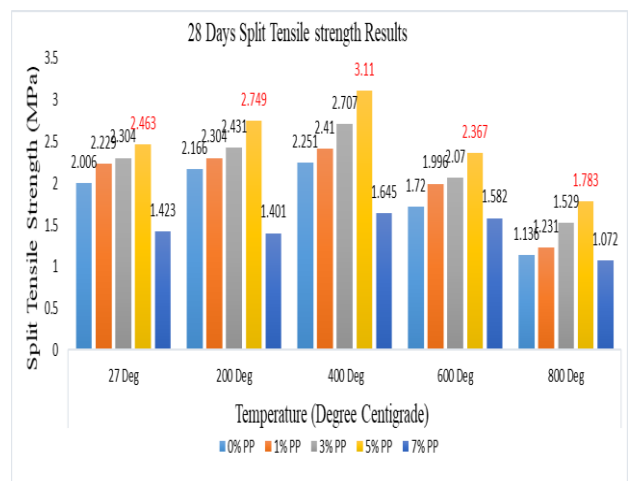


Figure 6 28 Days Split Tensile Strength Results



From Figure 6, it is seen that for 5PP mix, there is an increase in tensile strength by 12% from room temperature to 200°C and 13% from 200°C to 400°C. Further increase of temperature leads reduction of tensile strength by 24% from 400°C to 600°C and decreases by 25% from 600°C to 800°C. It clearly shows that the tensile strength test followed the similar trend as observed in compressive strength and flexural strength tests.

D. XRD Analysis

X-ray diffraction pattern of crystalline structures consists of many sharp peaks while those of the non-crystalline solids show diffuse humps. A hump indicates a short-range structure due to the irregular and non-repetitive arrangement of the atoms. Therefore, a hump in the XRD pattern indicates amorphous nature of the material [14].

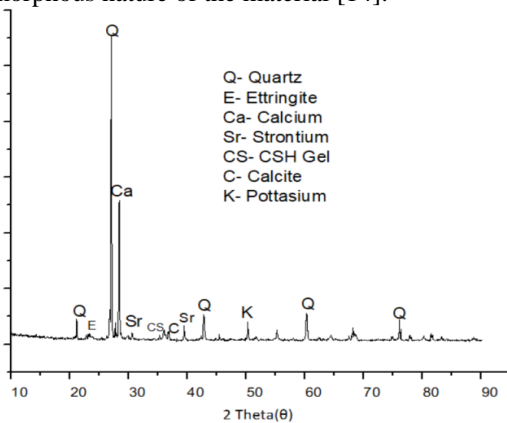


Figure 7(a) NC

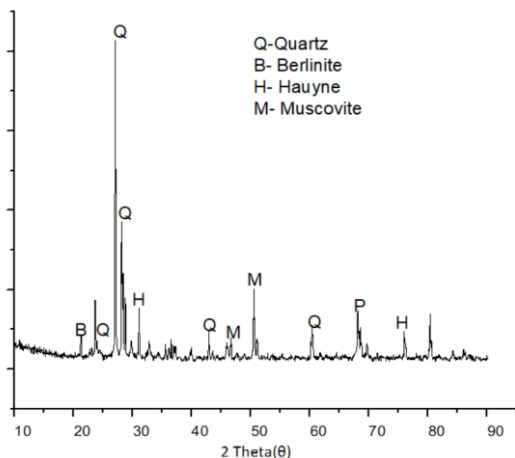


Figure 7(b) 1PP Mix

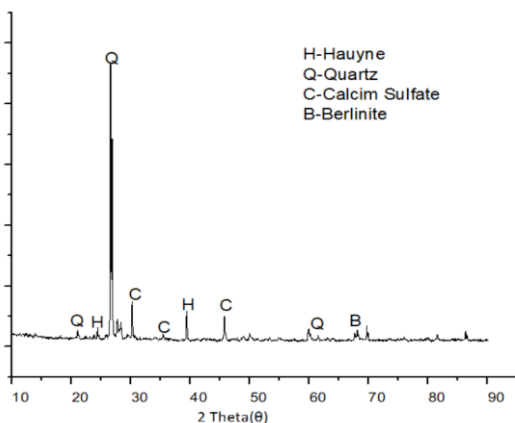


Figure 7(c) 3PP Mix

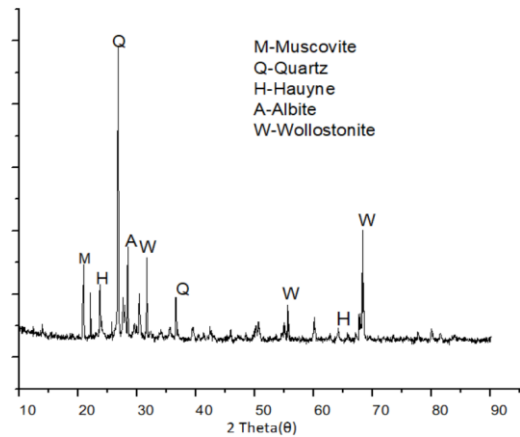


Figure 7(d) 5PP Mix

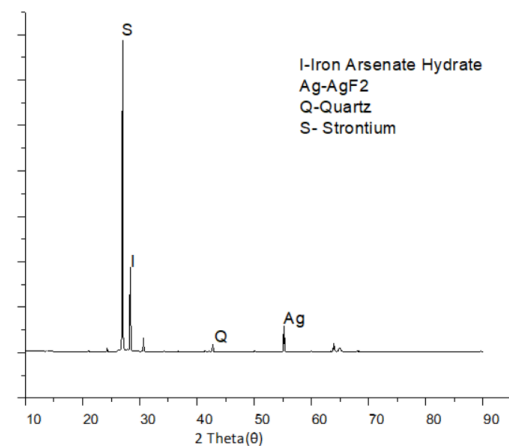


Figure 7(e) 7PP Mix

Figure 7 XRD Graphs of various mix proportions

From the Figure 7 it is evident that highest peaks in all mix proportions were observed for Quartz which is formed by the reaction of cement and water. Quartz helps in attaining more strength as it reacts with excess of Ca(OH)₂. Later on, Ettringite was formed which in turn got converted into Calcium Aluminium Sulphate. Wollastonite, contains more silica, was also formed and it reacts with water to form Calcium- Silicate- Hydrate (C-S-H). The C-S-H gel formed in 5PP was more compared to NC which is observed clearly from Figure 7(d) and the C-S-H gel plays a key role in increase of strength.

E. SEM Analysis

A scanning electronic microscope is a sort of electron magnifying lens that produces images of sample by filtering the surface with a focused beam of electrons. The electrons interact with atoms in the sample which produces the different signals that contain data about the surface topography and composition. An Energy Dispersive X Ray Analyzer (EDAX) is also used to determine the elemental composition and quantitative information.



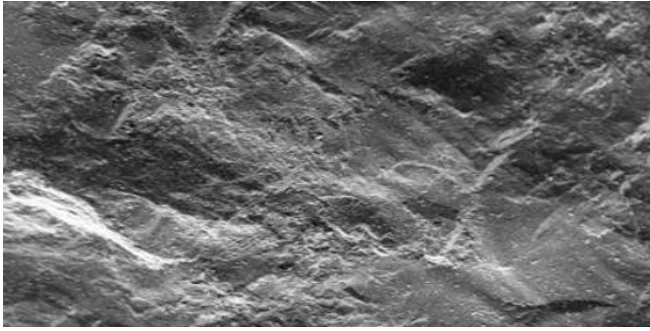


Figure (a) SEM Image of NC



Figure (e): SEM Image of 3PP

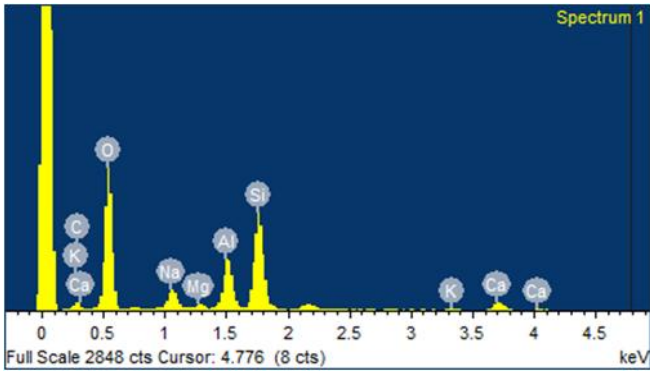


Figure (b) EDAX Graph of NC

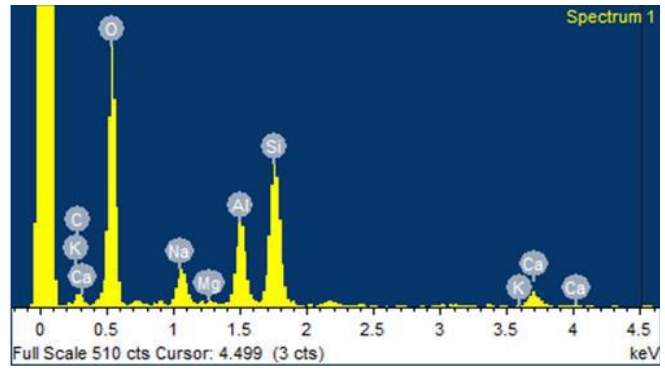


Figure (f): EDAX Graph of 3PP

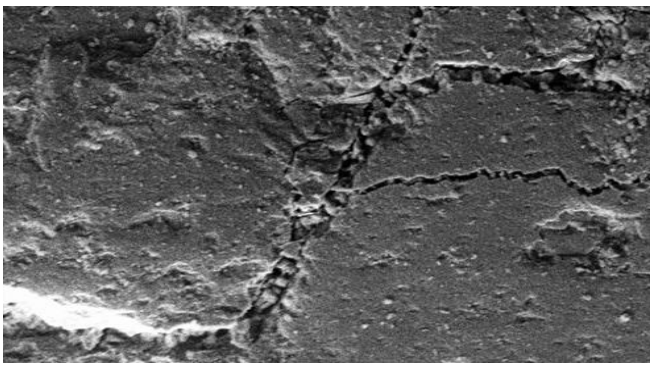


Figure (c): SEM Image of 1PP

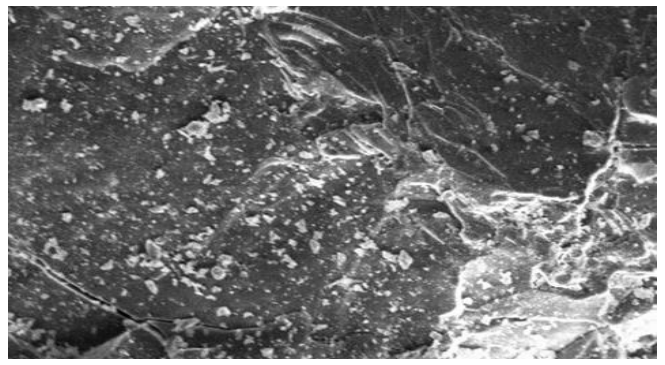


Figure (g): SEM Image of 5PP

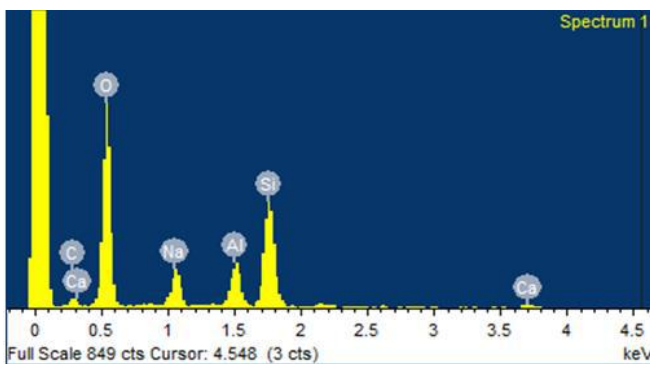


Figure (d): EDAX Graph of 1PP

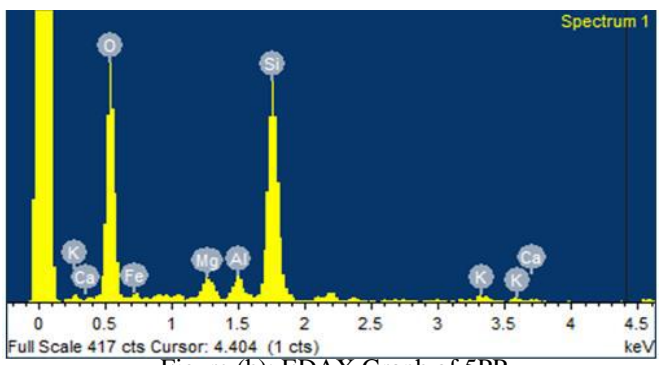


Figure (h): EDAX Graph of 5PP

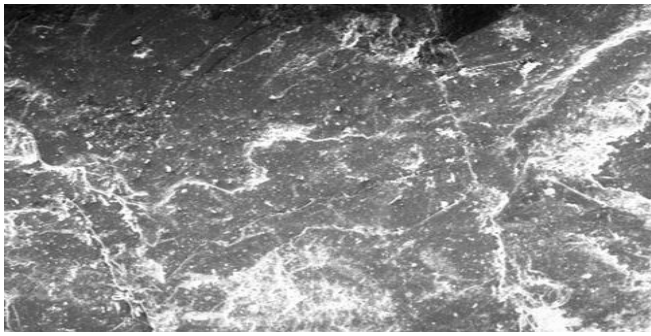


Figure (i): SEM Image of 7 PP

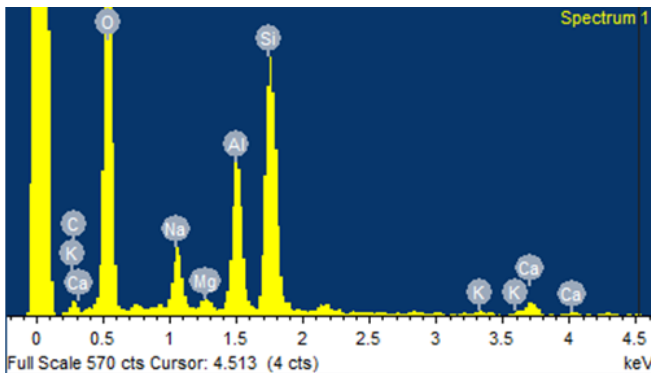


Figure (j): EDAX Graph of 7 PP

Figure 8: SEM and EDAX Analysis of Various Mix.

The results of SEM Analysis for all mix proportions (NC, 1PP, 3PP, 5PP, 7PP) are depicted in Figure 8. In NC the formation of pores and fractured plates are clearly observed. In 1PP mix, it is clearly shows that there is a presence of minerals such as Albite, Wollastonite whereas 3PP mix it shows the presence of feldspar. In addition to the above minerals 5PP mix shows the minerals such as Ferrite, Albite, Ettringite.

The EDAX Graph in all the mix proportions shows the highest amount of SiO₂ which plays a major role in the hydration process and strength development. The peaks of SiO₂ were much higher compared with NC. An increase in the amount of SiO₂ is responsible for attaining maximum strength in 5PP concrete mix. In the case of NC, the EDAX graph shows chemical compounds like CaCO₃, SiO₂, Albite, Feldspar and Wollastonite; the same compounds existed in 5PP concrete mix too, except for CaCO₃, in addition a new material namely Fe also existed in the 5PP concrete mix. The chemical compound, CaCO₃ in 5PP mix acts as an inert filler and helps to act as a nucleation position for cement hydrates. It further helps to speed up the setting time and strength development. Due to the presence of finer grained phases such as ferrite, the reaction between cement materials have given better performance for 5PP mix. At the early age of hydration ferrite is formed in more quantities, later it slows down as it acts as a barrier and prevents further happening of the hydration process.

V. CONCLUSIONS

The following conclusions has been derived in this experimental study:

- (1) The specimens achieved high strength at temperature 4000C compared with the other temperature. This is due to the increase in strength after exposing to 3000C, as further hydration of non-hydrated cement will lead to produce more C-S-H gel which in turn makes the concrete strengthened.
- (2) The concrete with 5PP replacement of cement has achieved more strength compared with other percentages of replacement. This is due to the presence of ferrite (Fe) mineral, determined from the Micro-structural analysis, which is produced in huge amounts at the early age of hydration.
- (3) As the Perlite Powder replacement increases the cracks formed were reduced on the surface of the specimens.
- (4) Specimens has got approximately same strength at 270C and at 6000C for all the tests conducted.
- (5) The results of compressive strength, flexural strength, tensile strength had followed the same behaviour i.e., from 270C to 4000C strengths increased then further increase of temperature shows reduction in strength rapidly. Hence the replacement of PP with cement replacement of 5% by weight achieved better results at elevated temperatures when compared to other mix proportions.

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