Characterization of zeolite influence to improve the performance of concrete

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Abstract. The key characteristic of the research is to provide sustainable concrete by reducing cement quantity by using modified pozzolanic materials, such as fly ash and zeolite. Concrete is the primary material used for construction where as there are several drawbacks and adverse impacts on the environment. There are also efficient ways of minimizing carbon footprint / global warming by minimizing the cement contents of concrete preparation. The approach pursued here in this paper is greater cement replacement with fly ash and zeolite and its concrete efficiencies in terms of workability, strength and durability. In this paper concrete grade M40 is adopted and adjusted concrete with UFFA replacement was done for different levels, the optimum levels were carried, and further zeolite replacement was done in different levels with cement. For 30% replacement of UFFA and 10% of Zeolite, the compressive strength increased to 8.2%, flexural strength increased to 4.1% and split tensile strength to 7.7% on comparison with conventional concrete. The micro structural studies confirmed the increase in strength was due to the fineness of the SCM’s and its void filling ability.

1. Introduction
In the construction sector the concrete is commonly used material hence the utilization of cement is predominantly in civil industry, so the natural resources are being used in an alarming rate for the production on cement causing global warming. The most important greenhouse gas for global warming is the carbon dioxide. A greater understanding of the global warming crisis has called for a rapid reduction of CO$_2$ emissions. The CO$_2$ emissions vary, depending on the processing process for 1 ton of cement usually 0.73 to 0.99t of CO$_2$ is released. The use of additional cement material (SCMs) in concrete production is one successful way of meeting this demand and is the ideal way for excellence in structural efficiency, durability and mechanical properties. [1, 2].

Fly ash is used as an extra cement ingredient in the making of Portland concrete, which improves the property of concrete by hydraulic and pozzolanic activity, or both. A lot of research was performed using fly ash as an additional cementitious material. Fly ash, which until recently was regarded as waste and a source of air and water pollution, is indeed a resource and has also proven its worth over. The production of fly ash is extreme high as compared to the utilization of fly ash quantity which resultin large disposal problem [3]. Global demand for energy is expected to increase by approximately 50 per cent between 2016 and 2040. Many of this growth will remain concentrated in the developing countries, particularly China and India; while industrialisation, population growth and unexploited medium-sized expansion will push energy needs in general, especially coal.[4].
Fly Ash switched from the "Materials from waste" division to the "Materials" division by creating & applying technologies. Fly Ash is used in building components such as bricks, walls, door frames, etc. very efficiently and economically. Fly Ash is also used in road and embankment construction, with some modifications in nature [5]. In recent years fly ash has become a key interest for many researchers, fly ash can also be used in concrete with combination of recronfibers, coconut fibers and silica fume for the improving the tensile properties [6, 7]. In the construction industry, the use of fly ash-silica fume blended geopolymer has tremendous potential to boost sustainability. In fact, waste management is to be efficiently realized through the usage of consumer goods on concrete[8]. Coarse aggregate of paste-coated nanomaterials will increase the strength of high-volume fly ash concrete while enhancing the transport properties of high-volume fly ash concrete (e.g., decreasing its permeability. In addition, if the coated rough aggregate increases the resistance to freezing / thawing cycles to a sufficient degree when mixed into high volume fly ash concrete, even without air [9]. The application of high volume fly ash as part of the concrete cement substitute decreases contamination in the atmosphere and conserves natural resources [10]. The synergistic behavior of fly ash, nanoparticles and inhibitors has facilitated the rise in early age strength and toughness characteristics [11]. There are other early age fly ash output issues, time lag, poor early age intensity and its healing conditions. Nano fly ash modifications are introduced for overcoming this deficiency [12].

Zeolites is commonly used as beds for ion exchange in water purification, softening, and other domestic and commercial applications, due to various research works and advances zeolite nowadays is used in construction industry as a replacement of cement. The perfect way to boost simple physical performance, mechanical and functional features, durability and hygric and thermal properties in concrete is the 20 per cent zeolite content in the blended binder [13]. Zeolite will decrease the 28-day shrinkage when partially substituted with sand and increase the strength of Engineered CementitiousComposite (ECC) [14]. Using zeolite, the filter increases the ability to reduce emission parameters of wastewater forms and does not compromise the physical properties of porous concrete by using pumice materials[15,16]. 10% of cement substituted with natural zeolite positively affected strength production pattern of self-consolidated fiber reinforced concrete (FRSCC) together with the creation of environmentally friendly concrete [17]. Synthetic Zeolite resulting from Alumina fluoride synthesizing waste products, improves concrete porosity and durability when replaced by 10 percent of concrete cement [18]. The use of superplasticizers, air entraining agents in zeolite-including concrete demonstrates the positive outcome of water penetration, drying shrinkage and freezing-thaw resistance in concrete [19]. The use of zeolite increases concrete density, strength, durability and reduces chloride penetration freeze-thaw resistance and corrosion rate of concrete [20, 21, 22]. The addition of 20 percent natural zeolite has resulted in a significant increased mixture strength and electric resistivity, particularly in older ages [23]. Zeolite reduces the resistance to corrosion in carbon steel by improving the creation of a denser safety firm on the carbon steel surface [24].

So far from all the literature reviewed, that the uses of ultrafine fly ash and zeolite in concrete is a good secondary cementitious replacement material in terms of strength. The combined analysis of the use of ultrafine fly ash and natural zeolite in concrete has not been carried out to date and this paper therefore includes compressive research studies covering successful factors on the strength and workability including its micro structural studies of natural zeolite and ultrafine fly ash blended concrete.

2. Material and Methods
2.1 Materials
The main materials included in the analysis are OPC grade 53 cement meeting the standard IS 12269-2013, fly ash used is obtained from Ennore thermal plant and is pulverized for size reduction by using 120 minutes of ball milling apparatus for the preparation of UFFA, zeolite used was of the clinoptilolite kind collected in the form K. Mohan & Company which in this analysis is all in powder form, some of the basic test was conducted such as specific gravity, fineness and blaine fineness to
determine its physical properties having specific gravity of 3.13, 2.97 and 1.87, fineness of 5, 1.8 and 4.8 for cement, UFFA and Zeolite respectively. The coarse aggregate of maximum size used was 10mm, the physical properties of coarse aggregate were found out by conducting few basic tests as per IS-2386 (part III)-1963 with specific gravity of 2.95 and water absorption of 1.0. Well graded river sand moving through 4.75mm was used as fine aggregate throughout the study and some basic test shows that specific gravity was 2.61 and water absorption as 2.3. The chemical composition of OPC, UFFA and zeolite is shown in table 1. For raw and calcined natural pozzolan class N, the total pozzolanic operation equal to the sum of SiO$_2$, Al$_2$O$_3$ and ferric oxide (Fe$_2$O$_3$) should be 70 per cent. This property was 85.32% for the zeolite used, and 87.3% for ultrafine fly ash.

**Table 1. Chemical composition.**

|       | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | CaO  | MgO  | K$_2$O | Na$_2$O | SO$_3$ | LOI  | Blaine Fineness |
|-------|---------|-------------|-------------|------|------|-------|---------|-------|------|-----------------|
| OPC   | 21.41   | 5.54        | 4.47        | 62.32| 1.23 | 0      | 0       | 2.83  | 1.05 | 331             |
| UFFA  | 57.31   | 23.53       | 6.46        | 3.59 | 1.58 | 1.37  | 1.04    | 0.7   | 2.14 | 608.44          |
| Zeolite| 75.97   | 7.10        | 2.25        | 3.10 | 0.90 | 0.20  | 0       | 0     | 6.20 | 1130            |

2.2 Preparation of Ultrafine fly ash
The fly ash pulverization is carried out using fly ash ball mill for the gradual reduction in particle size. The ball mill apparatus consists of three cylinders made of stainless steel with inner diameter of 15cm and coupled with motor having a power of 2HP run at a speed of 120 rpm for 2 hours. After the pulverization of fly ash the particle size reduction is observed around 4 to 8 microns and named as ultrafine fly ash.

2.3 Mix Proportions
The mix ratio for concrete grade M40 is made for the cube, cylinder, and beam and also measures the material for certain specimens. In terms of ratio, the mix pattern can be shown as Cement: Sand: Aggregates. The concrete mix design requires several steps in finding the optimal material and calculations necessary to be performed in compliance with IS 456:2000(41). Ratio of blends is 1: 1.508: 1.85 whereas cement content is 520 kg / m$^3$, coarse aggregate is 960.284 kg / m$^3$, sand is 784.252 kg / m$^3$ and water is 208 1 / m$^3$.

2.4 Preparation of samples
Ultrafine fly ash and zeolite are incorporated into concrete to enhance strength and reduce environmental damage. The concrete was casted in 100 mm side-length iron molds and cured in a water tank after removal. At 7 days, 14 days and 28 days of age the mechanical property of the sample was checked. The first element UFFA has been replaced by four cement levels, namely 15%, 30%, 45% and 60% this optimum level has been replaced by additional zeolite, which is 5%, 10%, 15% and 20% with cement. For the entire analysis, the entire sample was prepared using cement with 0.4 water ration. Before casting the specimens, the workability of the concrete was tested.

2.5 Mechanical properties.
Compressive strength is the ability of concrete structures to withstand loads and tends to break down when compressed. The compressive strength test of the cube specimens of 100x100x100 mm with a capacity of 2.5 KN/s until the specimen fails to withstand the load based on IS: 516-1959 has been performed. The specimens were brushed and washed to remove the loosely bound materials before processing. Tensile strength is very important in determining the pressure at which the concrete splits due to loss of stress. The typical size of 100 X300 mm cylindrical concrete specimen was mounted horizontally between the machine's loading surfaces. Compression measuring machine of 2000KN...
was used for split tensile strength calculation. Flexural strength was conducted using concrete rectangular prisms of standard size 100X100X500 mm. The load added to the specimen was 180 kg / min, and for all tests concrete were cured for 7, 14 and 28 days in fresh water.

2.6 Micro structural studies

The SEM imaging of the concrete specimens are analysed by placing the sample in high vacuum. The SEM image of the concrete strata is obtained by testing the sample in high-resolution field electron microscope [FEI QUANTA 200], which provides the crystal structure and shows us how dense the concrete structure is. And the EDS data are obtained from EDAX 102mm Octane Prime EDS Detector. The EDS analysis helps us to know the possible elements available in the sample which helps to understand the elements necessary to improving structural efficiency. The X-ray Powder Diffraction test is done for the concrete sample to identify the crystalline compounds present in the sample also to find the quantity of the hydrated compounds in the concrete specimen. The sample were tested using X’ Pert Powder XRD system with a 2θ range of 0-150°, Cu-K α radiation was used for obtaining the diffraction pattern of samples. The XRD spectrum is analysed through Xpert High score plus, peaks are identified. This provides variation in the peaks of the sample with different mix combination. The principle behind the Fourier Transform infrared spectroscopy is that all the molecules have a capability of absorbing the light in infrared region, the instrument analyse the difference between the absorption and transmittance of molecules. The instrument used for this test is Bruker Alpha T with a spectral range of 500-4000 cm⁻¹. This FTIR spectrum is very much useful in identifying the bond nature and amount on which the bond is present in the sample with the strength attributes.

3. Results and Discussion

3.1 Consistency Test

Table 2 compares the values for the consistency. As with the rise in concrete zeolite content, the amount of water required to achieve consistency increases, while taking UFFA as a substitute material due to ultrafine particle in UFFA, water demand is lower than mixed concrete demand for Zeolite. Water demand in zeolite can be due to a wide surface area which needs more water to achieve consistency.

| Mix Combination     | Normal consistency | Penetration (mm) |
|---------------------|--------------------|------------------|
| OPC                 | 27%                | 45               |
| OPC+UFFA15%         | 30%                | 43.5             |
| OPC+UFFA30%         | 29%                | 44               |
| OPC+UFFA45%         | 30%                | 44               |
| OPC+UFFA60%         | 29%                | 45               |
| OPC+UFFA30%+ZL5%    | 32%                | 43               |
| OPC+UFFA30%+ZL10%   | 30%                | 43.5             |
| OPC+UFFA30%+ZL15%   | 35%                | 45               |
| OPC+UFFA30%+ZL20%   | 35%                | 45               |

3.2 Setting Time Test

Table 3 measures the setting time of cement paste with various percentages of UFFA and Zeolite. The setting time also increases proportionally, as with the increase in UFFA and Zeolite content. The initial setting time is the needle’s penetration depth exceeding 45 mm in the vicat apparatus. This means the depth of the penetration is slowly through. OPC’s setting time for separate UFFA and zeolite ratios is 24 hours higher than all other blended pastes from the study. This is because a significant amount of UFFA and Zeolite is available along with cement which is itself a retarder.
Table 3. Setting time of the mixes.

| Mix Combination                      | Initial Setting Time (mins) | Final Setting Time (mins) |
|--------------------------------------|----------------------------|---------------------------|
| OPC                                  | 45                         | 320                       |
| OPC+UFFA15%                          | 155                        | 350                       |
| OPC+UFFA30%                          | 230                        | 510                       |
| OPC+UFFA45%                          | 295                        | 660                       |
| OPC+UFFA60%                          | 350                        | 780                       |
| OPC+UFFA30%+ZL5%                     | 85                         | 300                       |
| OPC+UFFA30%+ZL10%                    | 125                        | 330                       |
| OPC+UFFA30%+ZL15%                    | 190                        | 480                       |
| OPC+UFFA30%+ZL20%                    | 240                        | 570                       |

3.3 Workability results
The values substituted with different percentage of UFFA and Zeolite mixes for OPC's workability was tested and in comparison, as the growth in UFFA content increases, its small size increases workability but as the rise in the Zeolite content increases in concrete the workability reduces, the amount of water needed to achieve the workability increases, the water demand may be due to a large area of the surface, which needs more water to achieve workability.

3.4 Compressive strength
3.4.1 Impact of ultrafine fly ash on compressive strength. Various experiments were undertaken to enhance the concrete consistency of the area by replacing cement with secondary cemented materials that have adequate physical properties. Figure 1 shows the average compressive strength values of the specimen that comprises ultrafine fly ash and standard concrete after the 7, 14 and 28-day healing cycles. Since in all concrete formulations equal aggregate volume is used, the compressive strength of the ultrafine mixed concrete with a replacement of 30% is higher than all other concrete mixes, which is 7.2% higher than that of conventional concrete due to the efficient filling of pores in concrete and the creation of more hydrated product. However, compressive strength is decreased by removing ultrafine fly ash by 60 per cent due to the increase of light weight content the concrete density decreases and the concrete density is directly proportional to the concrete compressive strength.

![Figure 1. Compressive strength of concrete containing UFFA.](image-url)
3.4.2. Impact of Zeolite and ultrafine fly ash on compressive strength. In inclusion, the addition of zeolite in ultrafine fly ash-mixed concrete had a small impact on strength compared to ultrafine ash-containing concrete. Figure 2 shows the average values for the compressive strength. For 30 per cent of ultrafine fly ash and 10 per cent of zeolite integrated in concrete, which is 8.2% higher than conventional concrete and 1% higher than 30% UFFA due to the higher C-S-H bond and better pore filling, the maximum strength was achieved according to the tests. In the chemical properties of concrete, the further results in the compressive strength are studied.

![Figure 2. Compressive strength of concrete containing UFFA and Zeolite.](image)

3.5 Split Tensile strength

Split tensile strength values are shown in Figure 3. It shows that 30% of ultrafine fly ash and 10% zeolite replacement show higher values which are 3.62 N/mm², when compared conventional concrete it is higher by 7.7% and optimal UFFA replacement by 4.6% based on 28-day strength due to its pozzolanic reactions and the admixture filling capability which improves bonding strength between the paste and aggregate thus increases the load split resistance.
3.6 Flexural strength
The values of the flexural strength values are shown in Figure 4, indicating that 30 percent ultrafine fly ash in combination with 10 percent zeolite demonstrates higher strength than traditional and ideal substituted concrete UFFA. The value of the 30 percent UFFA showed higher values as measured in 7 days between the blends, but in 28-day intensity the higher values were obtained in 30% UFFA in cooperation with 10% zeolite concrete which is due to the improved bonding strength between the paste and the aggregate because of its pozzolanic reaction and its filling ability of the admixtures.

3.7 Microstructural studies
3.7.1. SEM analysis. SEM images of the binding material are shown in figure 5. The SEM images of 10-micron magnification are compared here.
Zeolite's SEM images show that zeolite tends to be small laminar crystals in the shape of rosettes attached, and the cement has been observed as an irregular particle size, while ultrafine fly ash appears in spherical form which in combination promotes higher bonding.

Figure 6 shows SEM images of concrete samples, the SEM analysis clearly indicates the hydrated and un-hydrated particles where the black spots indicate the un-hydrated particles and white regions indicate the hydrated particles. The black spots represent the pores present in the concrete. Ethtringite phase formation is high in traditional mix which significantly reduces hydration. Whereas the addition of UFFA and zeolite enhances C-S-H gel formation, since all these admixtures are in the silicate group. As well as the formation of crack is present in the conventional concrete but in optimum it shows higher.

3.7.2 EDS Analysis. The EDS analysis of the binding materials is shown in figure 7.
The analysis reveals that due to the presence of Si, Ca and Al in all the binding materials the strength of concrete will be increased. Compared to SEM research, to analyze the elemental composition, the energy dispersive X-ray spectroscopy analysis was performed with optimized admixtures to the different blends as shown in figure 8.

From the EDS analysis it shows that calcium, silicon, carbon, aluminum and oxygen are the major element found in that concrete sample, when comparing conventional concrete and optimum replaced concrete the C-S-H formation is higher in optimum due the compound present in optimum is more than conventional concrete.

3.7.3. XRD diffraction analysis: The composition of crystalline materials was studied by X-ray diffraction. Figure 9 shows the XRD pattern of zeolite, ultrafine fly ash and cement.
The pick indicated in the above figure represents the poly crystalline in the raw materials. The majority of the peaks in UFFA is due to the availability of quartz and mullite and in zeolite is due to the availability of quartz and clinoptilolite rest of the peak in zeolite is by other phases where as in cement the peaks are due to calcia, coesite, aluminium oxide and ironoxide. The XRD spectrum of concrete samples is shown in Figure 10. Of large compounds such as quartz, calcium silicate hydrate (C-S-H), calcite are the main peak and the spectrum has been allocated. The main peak indicates the difference in counts and according to the peaks the optimal samples at 28days show higher peaks than the conventional at 28days, suggesting the more available of C-S-H compound due to the more Si and Ca present in the test.

3.7.4. **FTIR.** The FTIR was conducted to study the bonding nature of the materials used in this study. Figure 11 shows the FTIR pattern of zeolite, cement and UFFA at the wave number ranging from 500 cm\(^{-1}\) to 4000 cm\(^{-1}\).
The mineralogical structure of the binding material according to FTIR results the peak of all the materials lies in between 500 to 1500 which represents single bonding in concrete. For cement the peaks are present at 513cm\(^{-1}\), 875.68cm\(^{-1}\), 1111cm\(^{-1}\) and 1423.47cm\(^{-1}\). The peak at 513cm\(^{-1}\) represents the presence of gypsum, the peak at 875.68cm\(^{-1}\) and 1111cm\(^{-1}\) corresponds to asymmetrical and symmetric vibrations of Si-O in quartz. The peak at 1423.47cm\(^{-1}\) represents the carbonated group. For UFFA the peaks are at 786.96cm\(^{-1}\) and 1056.06cm\(^{-1}\) which indicate mullite and quartz respectively. For zeolite the peaks at 422.4cm\(^{-1}\) represents bending vibration at O-Si-O occurring at antiphase, 530.42cm\(^{-1}\)represents symmetrically extending vibrations of Si-O-Si bridge bonds and vibrations bending O-Si-O, 914.26cm\(^{-1}\)represents symmetric stretching vibrations of bridge bonds Si-O-Si and 1031.92cm\(^{-1}\)represents bridge bonding asymmetrical vibrations Si-O(Si) and Si-O(Al).

By comparing of the spectrum of samples the availability of more hydrated product can be visible in optimum concrete blended with 30% UFFA and 10% Zeolite. The major peak for conventional concrete is shown in 534cm\(^{-1}\), 682.50cm\(^{-1}\) and 933cm\(^{-1}\) etc. The peak at 534cm\(^{-1}\) represents the presence of gypsum, the peak at 682.50cm\(^{-1}\) corresponds to the asymmetric and symmetric vibrations of Si-O-Si in quartz and peak at 933 cm\(^{-1}\) represents carbonated group. The major peak for optimum
concrete is shown in $952.84 \text{cm}^{-1}$, $1415.75 \text{cm}^{-1}$ and $1639.49 \text{cm}^{-1}$ where peak at $952.84 \text{cm}^{-1}$ represents symmetric stretching vibrations of bridge bonds Si-O-Si, peak at $1415.75 \text{cm}^{-1}$ represents the carbonated group including CaCO$_3$ and the peak at $1639.49 \text{cm}^{-1}$ indicates the water bond due to formation of hydrated products in concrete.

4. Conclusion

From the experimental analyses the following conclusion can be drawn,

- The optimum replacement percentage of UFFA and zeolite is found to be 30% and 10% respectively.
- The compressive strength using UFFA as a cement replacement with 30% shows the higher value which is 7.2% higher than that of the conventional concrete and with the optimum UFFA in combination with Zeolite10% shows 8.2% higher values than that of conventional concrete and 1% higher than that of optimum UFFA replaced concrete.
- The split tensile strength results shows that 30% UFFA in combination with 10% Zeolite gives 7.7% higher values as compared to conventional concrete and 4.6% higher values than optimum UFFA replaced concrete.
- The flexural strength results show that higher values were obtained in 30% ultrafine fly in co-operation with 10% zeolite concrete by 4.1% as compared to conventional concrete and 3.3% when compared to Optimum UFFA replaced concrete.
- The micro structural analysis based on 28days specimens including SEM, EDS, FTIR and XRD show that the Optimum replaced materials i.e., 30% UFFA and 10% Zeolite gives better results than the conventional concrete.

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