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Investigation on Effectiveness of the Top Down Nanotechnology in Mechanical Activation of High Calcium Fly Ash in Mortar

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Abstract

Background/Objectives: Many synthetic additives have been using for improve cement mortar and concrete characteristics, but natural additive is a friendly environment option.In this work, the attempt is made to study, to reduce the pollution from cement production and other materials used in the construction by replacing of Secondary Cementitious Materials. **Methods**: In this study, the effects of cementitious materials like Ultra-Fine Fly Ash (UFFA) and Nano Silica (NS) on strength development, water permeability of mortar and the optimum use of UFFA and NS in mortars are investigated. Class C Fly Ash which is used as partial replacement of cement was subjected to ball milling for a total duration of 2 hours to obtain UFFA. The NS is a by product obtains from the silicon industries are also used as a replacement material of cement. Cement was partially replaced with four percentages (15%, 30%, 45% and 60%) of UFFA and NS by weight. The specimens used to determine the compressive strength and split tensile strength at the age of 3, 7, 14 and 28 days. Crystallite phase and grain size of UFFA and NS were determined by using X-ray diffraction test and the shape and texture was studied using SEM analysis. **Findings:** The test results show that strength increases with increase of secondary cementitious materials up to 30% of replacements of cement. Test results indicates that the physical, chemical, mechanical and durability properties of the cement are also enhanced with the influence of the secondary cementitious material. **Application/Improvements:** Increase in use of fly ash in cement mortar in the construction industry reduces the consumptions of cement as well as the cost of construction.

Keywords: Compressive Strength, Durability Properties, Fly Ash, Micro Structural Study

1. Introduction

The cement production in the world is around 4200 million metric tons per year. In China it is 2250 million metric tons, follows India which is having 250 million metric tons cement production per year. The cement industry is one of the primary producers of carbon dioxide. As an environmentally concerned world, the CO_2 emission should be controlled somehow. The use of cement cannot be controlled in any developing or developed nations[1,2]. But through the replacement of cement any other cementitious materials we can control the production of cement. The complete replacement of cement with any supplementary cementitious material is also not possible. But the partial replacement of the cementitious materials like Silica Fume, Fly Ash, Ground Granulated Blast Furnace Slag, Nano Silica and natural pozzolans etc. are successful possibilities[3–5]. In the secondary cementitious materials, most of them can be collected from the industrial wastes. For example the fly ash is a byproduct from coal used factories and thermal power plant.

Sustainability is the important strategy that we can adopt to reduce the industrial wastes. Coal fired power plants are the main electricity source in the world[6–8]. Especially in India the electricity produced by the coal fired power plants is 69%. So the amount of fly ash that

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produced while power plant running is huge. This waste fly ash can be used as a supplementary cementitious material. There can be reducing the industrial waste deposit by the coal fired power plant. So it is obvious that the fly ash can meet any high demand in the industrial market with cheap price tag. Concrete and mortar are the most used construction material, has high potentials recycling wastes in point of returning them into new and useful products or improving existing products. According to that fly ash is the best recycling waste. Through the partial replacement of the cement with fly ash we can reduce the production of the cement and there by the emission of CO_2due to the cement production[9,10]. Overall 600 million tons waste fly ash is produced. India produces about 80 million tons per year. This waste fly ash will remain in the earth for long years. The decomposition of this waste is not an easy process. But we can use this fly ash material as a supplementary cementitious material. Fly ash is considered as effective supplementary cementitious material in the construction industry. The waste fly ash that can be collected from the industries and the coal power plants is named as a raw Fly Ash[11–13].

In this study the different percentages of Ultra Fine Fly Ash (UFFA) and Nano Silica (NS) that can be used in the preparation of mortar by the replacement of cement is investigated. The rheological properties of the fly ash mortar are evaluated. The compressive strength, split tensile strength, water permeability and drying shrinkage tests are carried out on the mortar samples to find the mechanical strength properties. SEM and FTIR tests are conducted to analyze the micro structural properties.

2. Experimental Works

2.1 Materials Used

Materials used in this investigation consist of Ordinary Portland Cement (OPC) 43 grade confirming to ASTM C 150. The supplementary cementitious materials like fly ash is used in this study collected from Neyveli Lignite Corporation, Neyveli, Tamil Nadu. The Fly ash is ball milled into fine particles to obtain an Ultra Fine Fly Ash. The reduction in particles size of fly ash from micron level to ultrafine particles was carried out using planetary impact ball mill. The duration of grinding of particles is 2 hours. The ball to powder ratio is 10:1 was maintained. The 16mm diameter stainless steel ball are used to grind the particles with the rotation speed of 160 rev/min[14]. The distilled water which is having pH value of 7 is used for the preparation of cube and cylinder mortar specimens. The Nano Silica (NS) is used as a powder form obtained from Elkem product services Mumbai. The standard Ennore sand is used as a fine aggregates consist of equal proportions of three grade of sand, Grade I (1mm to 2mm), Grade II (0.5mm to 1mm) and Grade III (0.09 to 0.5mm). The Conplast SP430 (SP) phenolphthalein based super plasticizer is used in the investigation along with lesser water binder ratio of 0.4 used to improve the workability[15,16].

2.2 Mix Proportions

The mortar mix proportions of 1:2.75 (Binder:Sand) mix ratio is adopted according to the IS 10262-2009. The binder consists of OPC, UFFA and NS. The cement was partially replaced by weight of 15, 30, 45 and 60% of NS and UFFA separately. Two water binder ratios of 0.485 and 0.4 + 1% SPL was used in the preparation[17,18] of all mortar specimens with and without partial replacement of NS and also the UFFA.

2.3 Specimen Preparation and Curing

To determine the compressive strength and water absorption test on mortar cube, the specimens were prepared using 50mm cube mould by placing the mortar in two layers and well compacted as per IS:1727. For the measurement of drying shrinkage also the mortar specimen was placed in two layers and compacted well in the mould of 250x25x25mm size. To calculate the split tensile strength of the mortar, the specimens were prepared in 50 mm diameter and 100 mm height of steel mould. All the cubes, cylinder and bar specimens of different mix proportions prepared are cured in saturated lime water for various curing periods as per IS:1727.

2.4 Method of Testing

The mortar cubes prepared without and with partial replacement of NS and UFFA were tested to determine the compressive strength at various curing periods of 3, 7, 14, 28 days in accordance with ASTM C109. Tests were conducted as per ASTMC 496 to determine the split tensile strength on the cylinder specimens prepared without and with the replacements of NS and UFFA at the various curing periods adopted in the study. The drying shrinkage of mortar mix is also determined by testing the shrinkage bars of various mix used in the study at the specified curing periods as per IS4031:1988 and ASTMC 598. The water permeability tests on mortar cube were also conducted on all the mix as per IS:3085-1965 at the age of 28days.

3. Results and Discussions

3.1 Physical Properties

The basic physical properties like specific gravity, Blaine`s fineness and particle size of OPC, UFFA, NS and Sand was determined and presented in the Table 1 and the particle size distribution of OPC, UFFA, NS samples shown in Figure 1. From the results, it was observed that the NS is finer than UFFA and cement. But UFFA is slightly finer than cement. It is also evident from the higher and lower Blaine's fineness values of NS and Cement respectively.

3.2 Chemical Composition of Materials

The Chemical composition of OPC, UFFA and NS are given in Table 2. From the observation of carbon content, the Ultra Fine Fly Ash has very low values while compare to cement, it may lead to the good and green environment[19,20]. In nanosilica it was observed that the presence of silica content was much higher than the UFFA and cement which leads to early setting.

3.3 Compressive Strength of Mortar

The compressive strength of mortar cubes are tested at the ages of 3, 7, 14 and 28 days and are compared with the cubes prepared with the partial replacement of NS and UFFA of 15, 30, 45 and 60 % for the water binder ratio of 0.485 and 0.4 with 1% SPL and it is shown in Figure 2(a), 2(b), 2(c) and 2(d) respectively. From the results, it

Table 1. Physical property of materials

Type of Sample	Specific Gravity	% finer (45µ)	Blaine`s fineness (cm²/g)	Median particle size (µm)
OPC	3.15	86.32	2630	23.28
UFFA	2.56	99.12	7277	8.68
NS	2.1	100	16000	0.019
Sand	2.64	-	-	-

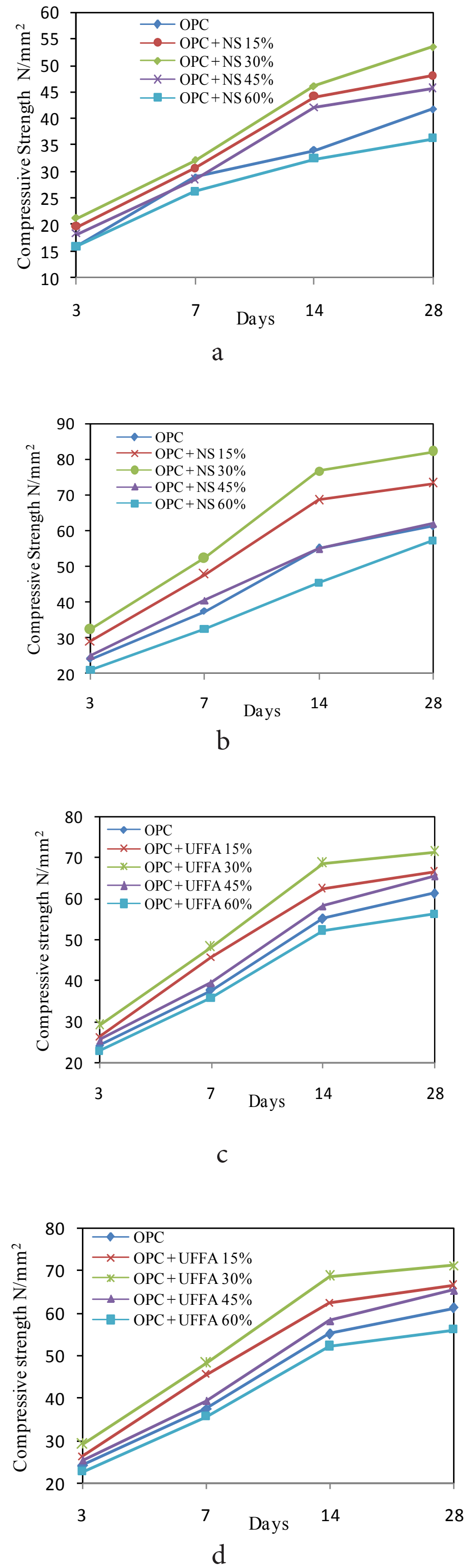


Figure 1. The particle size distribution of OPC, UFFA, NS samples.

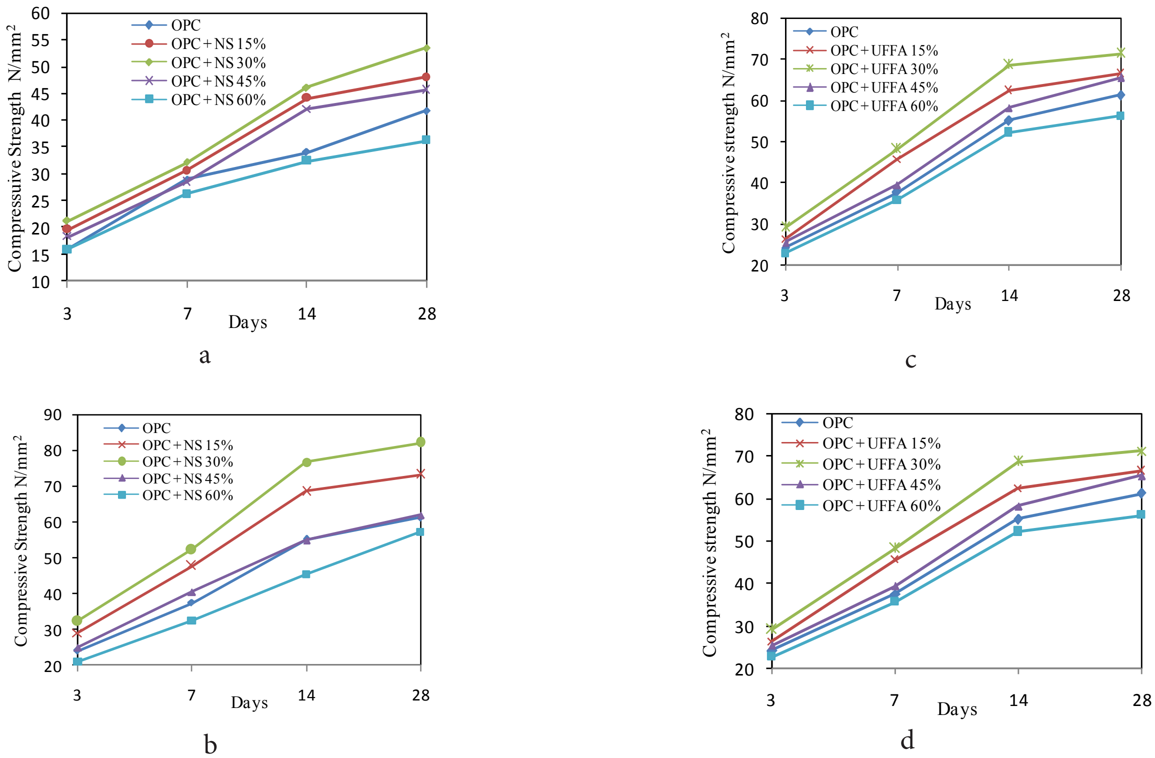


Figure 2. (a) Compressive strength of OPC mortar specimens with different replacement of NS (w/b = 0.485). (b) Compressive strength OPC mortar specimens with different replacement of NS (0.4+1%SPL). (c) Compressive strength of OPC with different replacement UFFA with 0.485 w/b ratio mortar specimens. (d) Compressive strength of OPC with different replacement UFFA with 0.4+SPL w/b ratio mortar specimens.

Table 2. Chemical composition of materials

Composition	Cement	UFFA	NS
SiO_2	21.41	35.2	96
Al_2O_3	5.54	27.4	-
Fe_2O_3	4.47	6.83	-
CaO	62.32	19.2	-
MgO	1.23	1.73	-
K_2O	0	0	-
Na_2O	0	0	-
SO_3	2.83	4.24	-
LOI	1.05	3.08	-

is observed that, compressive strength of mortar cubes prepared with partial replacement of NS with 0.485 w/b ratio and 0.4 w/b + 1% SPL shows increase in trend upto 30%, beyond which the addition of NS reduce the compressive strength. The maximum increase in compressive strength with the addition of NS for the water binder ratio 0.485 and 0.4+1% SP is 12 and 21 N/mm^2 respectively[21].

Similar trend was observed with the addition of UFFA of 30% also shows the strength at the maximum of 7 and 10 N/mm^2. The addition of SP with the water binder shows the higher strength on both the partial replacement of NS and UFFA with the cement.

3.4 Split Tensile Strength of Mortars

The split tensile strength of mortar were also tested at the ages of 3, 7, 14 and 28 days on cylinder specimens prepared with partial replacement of NS and UFFA similar to compressive strength and it is shown in Figure 3(a), 3(b), 3(c) and 3(d) respectively. From the results, it is observed that, split tensile strength of mortar cylinder with NS at 0.485 and 0.4 + 1% SP w/b ratio for replacement of 30% gives 18 and 22% of higher strength while compare to the control mortar at the age of 28 days[22]. Similar trend was observed with the addition of UFFA of 30% also shows the strength at the maximum of 11 and 10% respectively. Increase in UFFA and NS beyond 30% shows the reduction in tensile strength.

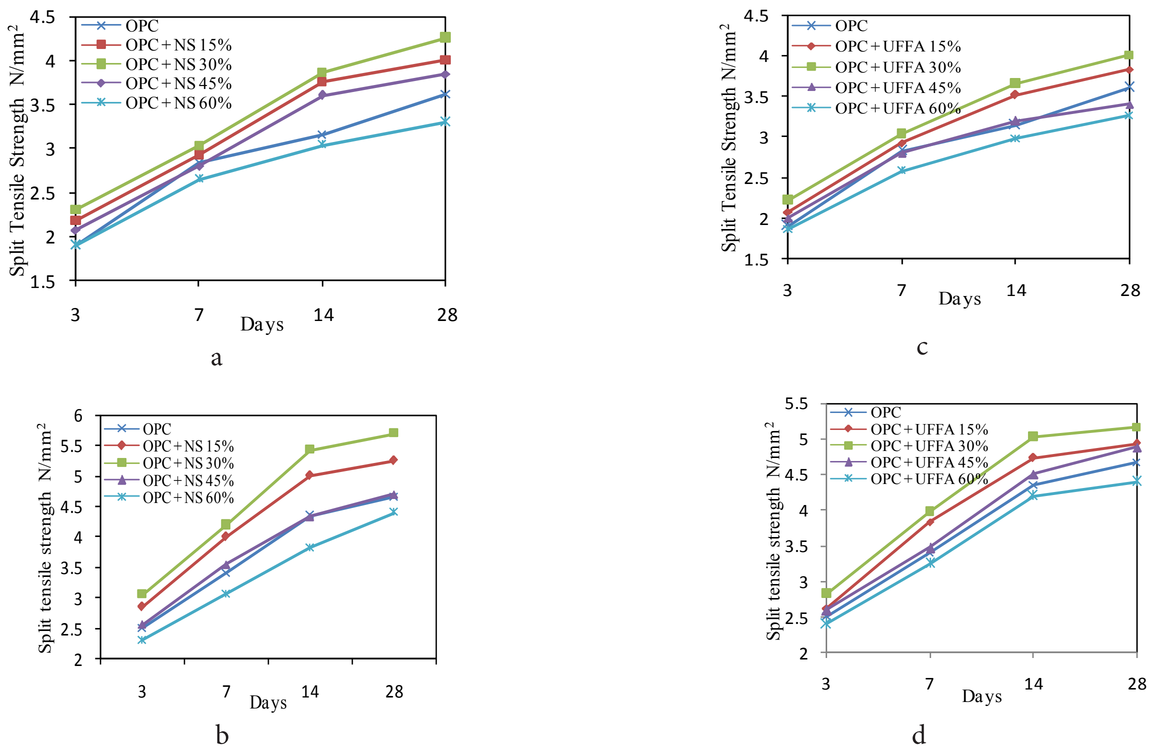


Figure 3. (a) Split tensile strength of OPC with different replacement NS with 0.485 w/b ratio mortar specimens. (b) Split tensile strength of OPC with different replacement NS with 0.4+SPL w/b ratio mortar specimens. (c) Split tensile strength of OPC with different replacement UFFA with 0.485 w/b ratio mortar specimens. (d) Split tensile strength of OPC with different replacement UFFA with 0.4+SPL w/b ratio mortar specimens.

3.5 Water Permeability

Permeability test were conducted to find the water absorption capacity of the specimen. The water permeability of the OPC, UFFA and NS samples for the different replacement level with 0.485 and 0.4+1% SPL w/b ratio is shows in Figure 4. It is observed that the permeability of UFFA samples has the less water permeability than the OPC samples but little higher than the NS mortar[23]. Due to the reduction in cement content the water was absorbed in lesser amounts in UFFA and NS mortar samples.

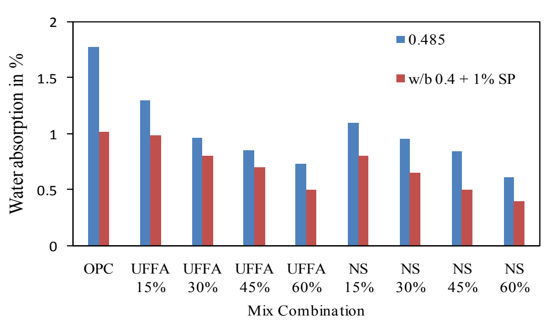
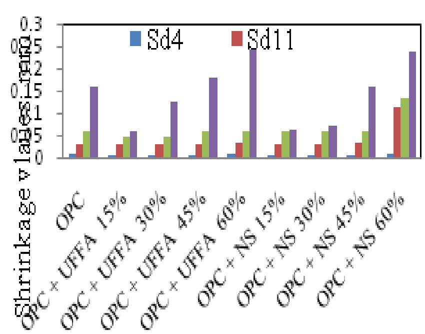


Figure 4. Water permeability of OPC, UFFA and NS samples.

3.6 Drying Shrinkage

Drying shrinkage test has been carried out to find the change in length of the bar specimens at the age of 4, 11, 18, 25 days are compared with partial replacement of NS and UFFA of 15, 30, 45 and 60 % for the water binder ratio of 0.485 and 0.4 with 1% SPL is shown in Figure



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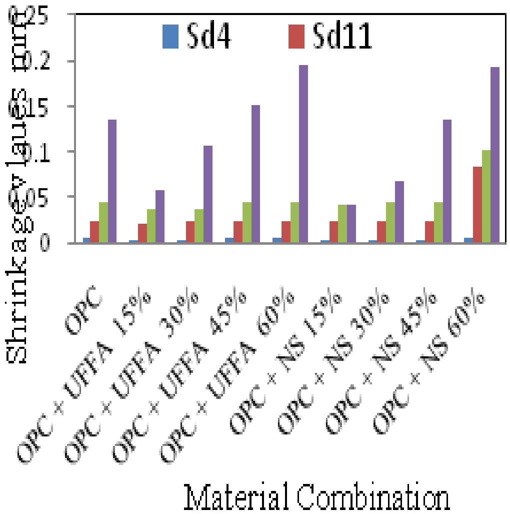

b

Figure 5. (a) Dry shrinkage of OPC, UFFA and NS samples with w/b ratio of .485 mortar specimen. (b) Dry shrinkage of OPC, UFFA and NS samples with w/b ratio of .4 + 1% SPL mortar specimens.

5(a) and 5(b). Results show that the cement without super plasticizer was higher shrinkage than the sample with super plasticizer[24]. Hence the addition of super plasticizer reduces the drying shrinkage along with the increase in strength.

4. Morphological Studies

4.1 Scanning Electron Microscopy (SEM)

The shape, texture and morphology of OPC, UFFA and NS were studied using Scanning Electron Microscopy (SEM). The Figure 6 (a) and (b) shows SEM images of OPC, Figure 6 (c) and (d) show SEM images of UFFA, Figure 6 (e) and (f) shows the SEM images of NS samples with the 30 µm and 5 µm magnitude. From the test results, the particle size of OPC, UFFA and NS samples were 3.82 µm, 185 nm, 96 nm respectively observed from the SEM images. The NS sample particle size is less compare to the OPC, which is the particle shape in spherical structure. In OPC samples, the solid spheres and irregular shaped particles of un-burnt carbon can be seen which is large in size compare to UFFA and NS samples. Also agglomerated spheres and irregularly shaped amorphous particles can be detected which may be due to inter particle fusion during sudden changes[25].

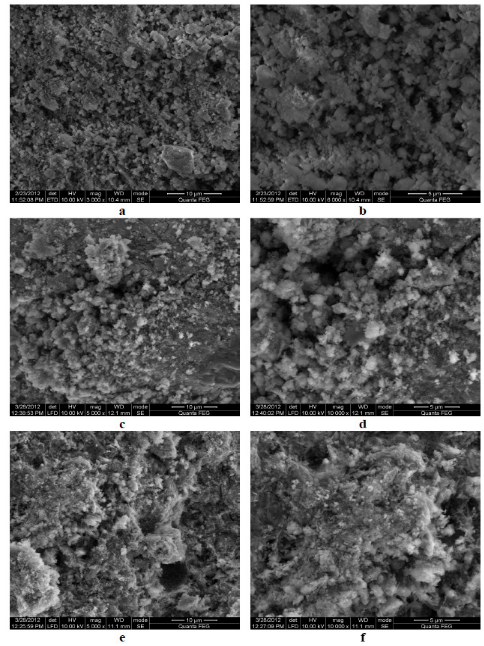

Figure 6. Particles shapes of OPC and UFFA, NS samples by SEM.

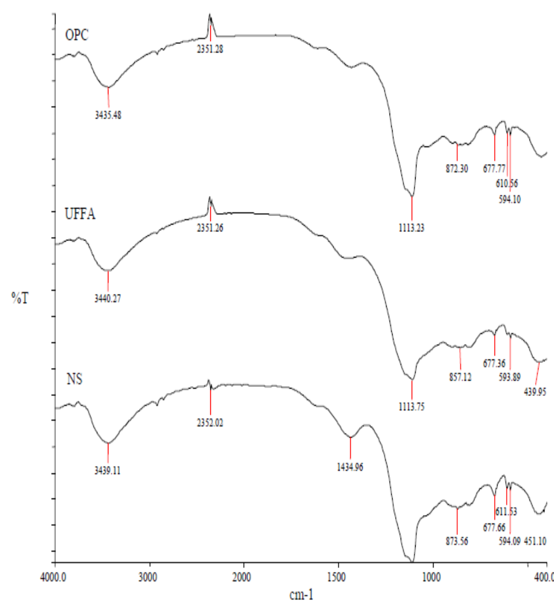

Figure 7. FT-IR spectra of OPC, UFFA and NS samples.

4.2 Fourier Transform Infrared Spectroscopy Analysis (FT-IR)

The surface structure and bond characteristics of molecules are determined from the FT-IR results, the analyses result of OPC, UFFA and NS samples are shown in Figure 7. The peak at 3439 cm^{-1} has identified in NS samples as compared to the OPC, which is O-H stretching. That the peak intensity at this wave number is found to increase due to decrease in particle size of NS samples. The peak wave number for UFFA is similarly to the OPC, due to particle size and characteristics, which is evident from the particles size analyses test results[26].

5. Conclusions

Based on the results of this study the following conclusions can be drawn.

UFFA and NS mortar provides satisfactory or higher strength as compared with OPC mortar. UFFA and NS mortar mix having various cement replacement level up to 30% exhibited satisfactory results for both compressive and tensile strength.

The optimum UFFA content is observed to be 30% of cement. The optimum NS content is 45% of cement, Mortars with 30% cement replacement shows around 14% higher compressive strength than OPC mortar after 28 days curing. The corresponding increase in tensile strength is reported to be around 8%.

The drying shrinkage of UFFA and NS mortar samples without super plasticizer was higher shrinkage than the sample with super plasticizer. The spherical shape of the NS leads to higher surface reaction and arrest the minor cracks.

The water permeability of UFFA and NS samples are less permeable with comparing of OPC mortar. UFFA and NS samples particles size and fineness is the major reason for voids reduction in mortar cube. The less voids mix gives the better durability to the structure.

The UFFA and NS mortar samples of FT-IR results shows that The peak at 3439cm^{-1} has identified as compared to the OPC, which is O-H stretching. That the peak intensity isincreases at this wave number due to decrease in particle size. In this process of cement hydration, CH is able to react with SiO_2 in further cementing system and form additional C-S-H to improve the compressive strength of mortars. From the SEM images, the OPC samples images shows irregular and non spherical shape of the particles. Comparing to OPC samples, UFFA have more spherical shape particles.

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