

# Strengthening of cement blended soft clay with nano-silica particles

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**Abstract.** In recent years, Nano-technology significantly invaded the field of Geotechnical engineering, particularly in soil stabilisation techniques. Stabilisation of weak soil is envisioned to modify various soil characteristics by the addition of natural or synthetic materials into the virgin soil. In the present study, laboratory experiments were executed to investigate the influence of nano-silica particles in the consistency limits, compressive strength of the soft clay blended with cement. The results revealed that the high compressibility behaviour of soft clay modified to medium-stiff condition with fewer dosages of cement and nano-silica. The mechanism behind the strength development is verified with the previous researches as well as from Fourier Transform Infrared spectroscopy (FTIR), X-ray diffraction test (XRD) and Scanning Electron Microscopy (SEM) analysis. Based on the results, the presence of nano-silica in soft clay blended with cement has a positive effect on the behaviour of soil. This technique proves to be very economical and less detrimental to the environment.

**Keywords:** cement; nano-silica; soil stabilisation; soft clay; strength improvement

## 1. Introduction

Every soil is unique in the way in which it has evolved, its basic characteristics and the behavioural changes during which any of its conditions have changed. While selecting a site for any construction, the soil should have a minimum required engineering properties to come in the 'marginal' category. The soil fails to provide even the minimum value of engineering properties, and it is termed as 'poor' soil; some alternatives are suggested, which includes 1. Abandon the project, when it is possible to find another site or the construction in the particular site is impractical, especially in terms of stability and economy 2. Excavate and replace the 'poor' soil, if the cost of hauling, availability of new soil, environmental issues are under control 3. Redesign the project to accommodate the soil and site conditions; this is impractical when the bearing strata is at a deeper depth, and the cost of the deep foundation is not feasible in the case of smaller works and 4. Modify the soil to improve its properties, choose an effective ground improvement technique to make the soil stable which includes mechanical modifications, chemical stabilisation etc. Traditional methods of chemical stabilisation were focused on soil modified with some common materials like cement, lime, bitumen, fly ash etc. (Bahmani *et al.* 2014, Eujine *et al.* 2017, Arasan and Nasirpur 2015, Yilmaz *et al.* 2018). Their usefulness is successfully proved and implemented in various sites all over the world. Even though the addition of these traditional stabilisers into the soil, can upsurge the strength and stiffness considerably, the higher dosages do

result in the alterations in pH of the soil which thereby impacts on the environment as well as vegetable growth (Ateş 2016, Kutanaei and Choobbasti 2017).

Nanotechnology is a new emerging trend in the field of soil mechanics, and many experimental investigations are still going on to reveal its applicability in improving the soil properties. Unfortunately, the usefulness of Nanomaterials in the field of ground improvement is still not been recognized completely. The first hint about the concept of particles on a small scale was introduced by Dr. Richard Feynman while delivering a seminar at the yearly meeting of the American Physical Society at Caltech in 1959. The talk revealed the importance of decreasing the particle size to get more information about the object and explained it with different cases that were relevant at that time because that was the first time in history one could explain the concept of Technology in Nano-Scale (Feynman 1960). Due to the smaller size of particles, the specific surface area of the Nanoparticles is more, so a small percentage of Nanoparticle in the soil can give rise to in a sizeable enhancement in physical and chemical features of the soil in microstructural level (Shahin *et al.* 2015).

A variety of nano additives were introduced in the field of ground improvement, and many of them proved their efficacy in improving various properties of the soil. Correia and Rasteiro (2016) used Multiwall Carbon Nanotubes soft soil with higher silica content blended with Ordinary Portland cement to investigate the behavioural changes in the soil. The results revealed that the nanotubes were found to be very useful in pore space reduction and reinforce the soil-cement matrix in the nano-scale level. The compressive strength of soft soil was found to be dependent on the quality of the dispersion of Nanoparticle in the aqueous medium (water or surfactant). The proper dispersion of Nanoparticles in the solution was monitored with the aid of Dynamic Light Scattering (DLS). The Unconfined

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compressive strength was increased noticeably when the quality of particle dispersion in the suspension increased, which enhanced the void filling ability in the soil-cement matrix. Effect of carbon nano-fibers and carbon nanotubes were also compared in soils with various plasticity character, and Taha *et al.* (2018) summarized that nano-fibers showed better performance in soft soils.

Some researchers examined the effect of Nano Magnesium Oxide, Nano clay, and Nano copper in soil stabilisation and observed the influence in the Consistency limits, compaction characteristics and strength improvement (Taha *et al.* 2015, Majeed *et al.* 2014). Clay with high compressibility and the soil with organic matter were taken into consideration, and the positive effect of nano-materials was found to be profound in high compressible clay. A declining trend in the liquid limit, plastic limit, plasticity index, linear shrinkage and optimum moisture content of the soil and a rise in the maximum dry density was detected with the increase in nano stabilizer content in soil up to a specific limit then the excess dosages of additive caused agglomeration of particles affected the mechanical properties adversely.

Nano-silica can act as an accelerator in hydration of cement or fly ash, which imparts the strength development due to accelerated pozzolanic reactions in soil-cement or cement/ fly ash pastes (Qing 2007, Stefanidou and Papayianni 2012, Stephan 2012, Choobbasti *et al.* 2015). Irrespective of the cohesion parameter of the soil, nano-silica was proven to be effective in both sandy (Choobbasti *et al.* 2015, Kutanaei and Choobbasti 2017) and clayey soils (Pashabavandpouri and Jahangiri 2015, Moayed and Rahmani 2017) which disables the demerits of many other additives. Abrasion resistance of concrete pavement could be improved significantly when the concrete is mixed with nano-silica (Li *et al.* 2006). Moayed and Rahmani (2017) made a study on clay with kaolinitic nature Stabilized with Nano-silica solution in 5 different contents ranging from 0-5%. To get a clear idea about its effect on soil, they compared the values of unconfined compressive strength and Elastic modulus of the treated soil with the natural soil. Based on the outcomes, it can be noticed that the UCS (Unconfined Compressive Strength) value, as well as E value increases as the Nano-silica content increases in the soil and the strength, could be amplified up to 1.43 times in comparison with the values of untreated soil.

Bahmani *et al.* (2014) investigated the influence of silica nanoparticles (15 nm and 80 nm) in residual soil and concluded that the practice of using nanoparticles was very effective in stabilising cement-treated (4, 6, and 8% of cement) residual soil. The C-S-H (Calcium Silicate Hydrate) gel produced in hydration imparts a reduction in porosity and a rise in strength of the soil. The present study is aimed at the strength improvement of soft soil using nano-silica along with a minimum dosage of cement which can be beneficial both economically and environmentally.

## 2. Materials used and methodology

A piling site at Kadavanthra, Kochi, in the state of Kerala (Coordinates of location 9.9564°N, 76.3015°E) was

Table 1 Features of Cement procured for the present study

Sl. No.	Physical Properties	Value
1	Specific Gravity	3.05
2	Consistency (%)	32
3	Initial Setting Time (min)	38
4	Final Setting Time (hrs.)	7.5

Table 2 Features of Nano-silica acquired for the current research

Sl. No.	Properties	Value
1	Specific Surface Area (m <sup>2</sup> /g)	202
2	Particle Size (Nano Meter)	17
3	pH Value	4.12
4	Specific Gravity	2.2-2.4
5	SiO <sub>2</sub> Content (%)	99.88



Fig. 1 Nano-Silica in powder form

Table 3 Engineering properties of test soils (Thomas and Rangaswamy 2019)

Sl. No.	Properties	Value
1	Specific gravity	2.50
2	Liquid Limit (%)	91
3	Plastic Limit (%)	33
4	Plasticity Index (%)	58
5	Clay (%)	46
6	Soil Classification (ISSCS)	CH
7	Maximum Dry Density (kN/m <sup>3</sup> )	14
8	Optimum Moisture Content (%)	28.5
9	Unconfined Compressive Strength (kPa)	28

\*ISSCS: Indian Soil Classification; CH: Clay with high compressibility

chosen for the sampling of soil used for the current research. Soil samples were procured from the same layer of soil to get a homogenous mixture of soil. Cement utilized in this study was manufactured by Ramco Cements Ltd, India. Table 1 and Table 2 show various features of the cement and Silica nanoparticles procured for the present work (Saranya *et al.* 2019, Thomas & Rangaswamy 2019), respectively. Nano-silica used in the current investigation is of 17 Nanometer-sized fine powder (white colour) which was purchased from Astrra chemicals, Chennai (See Fig. 1).

The collected soil was air-dried for one month for determining the index and geotechnical properties. After dehydrating the soil, the chunks of soil were ground well by ramming and sieved through 4.75 mm IS sieve. The

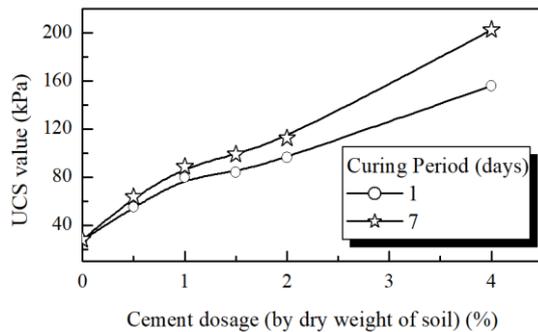


Fig. 2 Strength improvement of soil with cement dosage

pulverised soil after sieving is mixed thoroughly and kept in plastic buckets with lids to ensure the homogeneity since the soil collected from the same depth is taken from different boreholes. Soil samples were dried and prepared the dry powdered sample as per IS 2720 (Part 1)1983. Index and Strength Properties of tested soil are listed in Table 3 (Thomas & Rangaswamy 2019).

Reconstituted samples (3.8 cm diameter and 7.6 cm height) were prepared as per IS 2720 (Part 10) -1991 in a split mould at maximum dry density (MDD) and optimum moisture content (OMC) obtained from standard proctor test. Dry mixing of cement was adopted to prepare a uniform soil-cement mixture for avoiding the formation of cement lumps. Since Nano-Silica is a lightweight and bulky powder, the mixing of Nano-silica was cautiously monitored without losing minute particles from it. Dry mixing method is sufficient to prepare a uniform soil-additive mixtures which avoid the segregation of nanoparticles in the soil and is agreed by many researchers such as Pashabavandpouri and Jahangiri (2015), Choobbasti *et al.* (2015a), Changizi and Haddad (2015), Hanson *et al.* (2016), Kutanaei and Choobbasti (2017), Moayed and Rahmani (2017), Bahmani *et al.* (2014). Moulded cylindrical soil samples with and without additive mixing were cured in a desiccator. Prepared samples of raw and stabilised soil were carefully retained in a desiccator for a stabilising period ranging from 1- 4 weeks. Unconfined compressive strength (UCS) testing machine provided by Heico, Hydraulic & engineering instruments, was used at a strain rate of 1.5 mm/min. Unconfined compressive strength (UCS) of untreated soil sample (28 kPa) points toward the fact that the soil belongs to the soft soil category (Terzaghi *et al.* 1948). Samples were tested for Liquid limit and plastic limit tests as per IS 2720 (Part 5-1985) after 28 days of curing.

Cement content used in the present study is fixed as 1% by dry weight of the soil. Although the relationship between cement content and the strength improvement is linear, the objective of the current research is to minimise the use of the traditional additive to the maximum. Unconfined compressive strength tests on untreated soils with varying percentages of cement (0-4% by dry weight of soil) showed that the rate of increase is persistent even after 1% of cement (refer Fig. 2). Cement used in the study acts as an accelerator of the reaction between nanoparticles and clay flakes. Percentage of Nano-silica (abbreviated as NS throughout the paper) was varied from 0 to 20% by dry

weight of soil to identify the optimum dosage for the present work. Stabilised samples were tested on 1, 7, 14, 21, and 28 days of curing to evaluate the rate of increase in strength with time. Studies were done by Yilmaz and Goren (2018) also identified that the cement could accelerate the unconfined compressive strength of high plastic clays. To analyse the mechanism behind the changes in strength and consistency limits of treated soils Chemical and Microstructural analyses were conducted on dried powdered samples. Rigaku miniflex 600 X-ray diffractometer with Cu-k alpha radiation was used for XRD analysis in the present study. Agilent Cary 630 FTIR spectrometer and SEM JOEL model 2100 were used to conduct FTIR spectroscopy and SEM analysis.

### 3. Results and discussions

#### 3.1 Soil- nano-silica mixtures-strength benefits

Nanoparticles, particularly silica Nanoparticles, were mixed to the soft clay in various proportions to inspect the influence on unconfined compressive strength of the treated soil. The effect on undrained shear strength ( $q_u$ ) is summarised in Table 4. The stress-strain relationship obtained from various unconfined compressive strength tests with different Nano-silica content for 1 and 28 days of curing is plotted in Figs. 3 and 4, respectively. Some additional dosages of Nano-Silica (5, 10 and 20% by dry weight) were also taken for the trial testing to finalise the optimum dosage of Nano-silica in the present study. But they were limited to 1 and 7 days of curing because an idea about the strength improvement could be extracted from the initial days of curing itself.

The stress-strain behaviour of soil treated with Nano-silica particles showed that the increase in Nano-silica content as well curing period increases the strength of soft clay. From all the curves at various curing periods, 1% of Nano-silica showed a steeper curve than any other dosages. The percentages of Nano-silica brought the stress-strain curves up than the preceding dosage. The Unconfined compressive strength (UCS) value of each percentage of

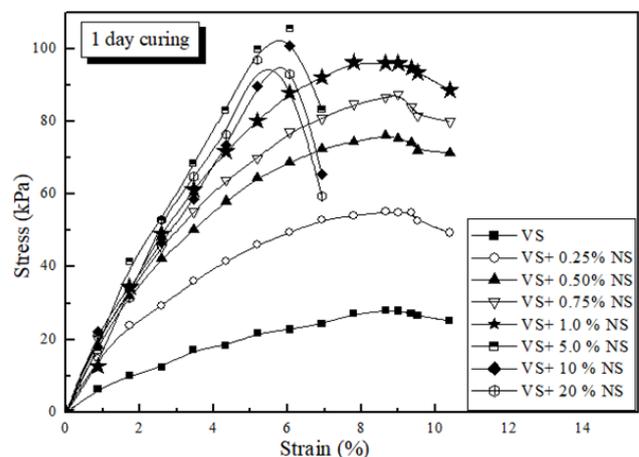


Fig. 3 Stress-strain behavior of clay-nano-SiO<sub>2</sub> blends at 1 day curing

Table 4 Effect of Nano-silica on unconfined compressive strength (UCS) of soil combinations at different cure timings

Sl. No.	Combinations	Nano SiO <sub>2</sub> (%)	Unconfined compressive strength (kPa) at curing time (days) of				
			1	7	14	21	28
1	VS+ NS 0.25	0.25	55.10	80.83	99.74	105.08	118.24
2	VS+ NS 0.50	0.50	76.09	87.99	110.47	122.86	129.33
3	VS+ NS 0.75	0.75	87.50	102.29	128.48	135.26	143.72
4	VS+ NS 1.0	1.00	96.24	132.29	161.13	166.51	170.48
5	VS+ NS 5.0	5.00	105.54	142.86	-	-	-
6	VS+ NS 10	10.00	100.74	131.19	-	-	-
7	VS+ NS 20	20.00	97.14	112.89	-	-	-

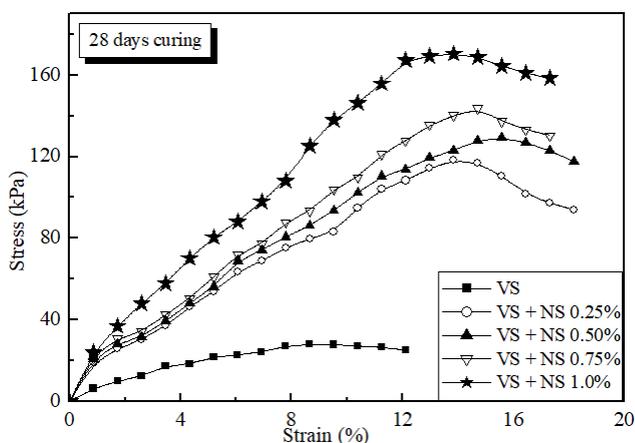


Fig. 4 Stress-strain behaviour of clay-nano-SiO<sub>2</sub> blends at 28 days curing

Table 5 Changes observed in Consistency limits of soft soils in the presence of cement and silica nanoparticles

Sl. No.	Description	Liquid Limit (LL)%	Plastic Limit (PL)%	Plasticity Index (PI)%	Soil Classification (ISSCS)
1	VS	91.0	33.0	58.0	Inorganic clay of high plasticity (CH)
2	VS+ C1	49.0	30.1	18.9	Inorganic or organic silt of Intermediate plasticity (MI / OI)
3	VS+ C1 + NS 0.25	48.0	32.0	16.0	
4	VS+ C1 + NS 0.50	47.0	32.0	15.0	
5	VS+ C1 + NS 0.75	46.7	32.0	14.7	
6	VS+ C1 + NS 1.0	45.7	32.0	13.7	
7	VS+ C1 + NS 2.0	47.0	33.0	14.0	

\*ISSCS: Indian Standard Soil Classification System; C: Clay; M: Silt

Nano-silica for various curing period point toward that a minimum of around 55 kPa can be achieved with a smaller quantity (0.25%) in just 24 hours of resting period which can be ascended to 118 kPa in 28 days.

Strength Development for different curing periods in clay treated with Nano-silica is summarised below. The strength of the tested soft clay was increased 3.22 times

within 28 days as that of virgin soil in the presence of 0.25% of Nano-silica. Though a maximum of around 5 times strength as that of natural soil can be attained within 28 days in the presence of 1% Nano-silica, even if 5% of Nano-silica could produce a higher strength than 1% Nano-silica, the rate of increase was found to be declined after 7 days of curing. Also, a decrease in strength was observed with an excess dosage of Nano-silica beyond 10%. Above mentioned results are pointing out the following conclusions that as the dosage of Nano-silica can increase in strength considerably, but beyond a specific limit, the strength increases keep a steady-state followed by a declination in strength gain. A similar trend was observed in the research work done by Hou *et al.* (2012).

The Nano-sized particles cover the micro-sized particles of clay, thereby increases the wettable surface area of the matrix, which in turn reduces the moisture content in the soil (Qing 2007). Once the pozzolanic reaction started, the increased bonding between the cementitious products formed and the clay particles results in a tougher soil matrix. But the excess dosage of Nano-silica causes accumulation of Nanoparticles inside the soil matrix separately, which are having weak bond strength. Comparatively a quicker pozzolanic reactions in the initial stages of curing owing to the higher specific surface area of nano-silica results in a coating all over the clay particles. Beyond a limit of the addition of Nano-silica hinders the cementitious products from further hydration. Thus a reduction in strength could be observed (Hou *et al.* 2012). The increase in the dosage of the Nano-silica made the soil into a brittle one as its demand for the moisture is higher for soil-nano-silica mixture. The dosage beyond 5% results in sudden failure of the sample which is not advisable since a better trend in the stress-strain relationship was observed when 1% of Nano-silica was used, more ductile than higher dosages but stiffer matrix than lower dosages. Results of Moayed and Rahmani (2017) shows a closer agreement with the trend, as mentioned above.

### 3.2 Soil- cement- nano-silica mixtures- changes in consistency limits

Treatment with cement and cement plus nanoparticles have changed the consistency limits of the soft clay. Liquid limit and plastic limit tests were piloted on untreated and treated soils after 28 days of curing period. Table 5 summarizes the values of Liquid limit (LL) and plastic limit (PI) of untreated and treated soils by varying dosages of additives after a curing period of 4 weeks. A radical reduction in LL is observed with the addition of 1% of cement to the soil. The rise in the dose of Nano-silica slightly reduced the LL, but the reduction in trend is not estimable. Concerning the modifications in the plastic limit, the values appeared to be stationary per rising in the prescribed amount of nano-silica even though the soil-cement showed the lowest PL value in the absence of nanoparticles. The plasticity index (PI) of untreated soil was belonging to soils with very high plasticity (PI greater than 40), which is improved to the category of soils with medium plasticity (PI= 10-20). Smaller the PI value, larger be the apparent friable zone. The decrease in LL and no

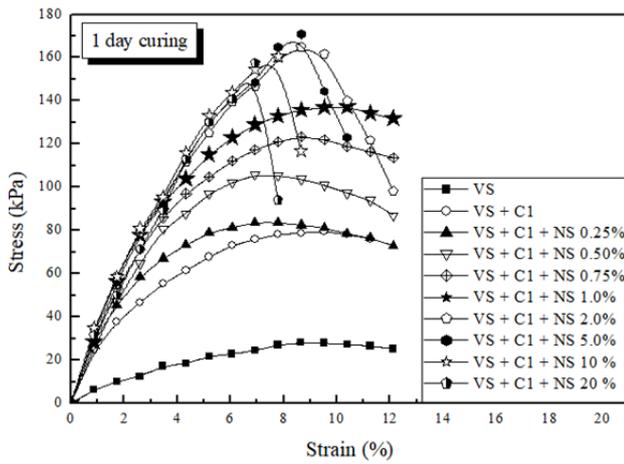


Fig. 5 Stress-strain behaviour of cemented clay- nano-SiO<sub>2</sub> blends at 1 day curing

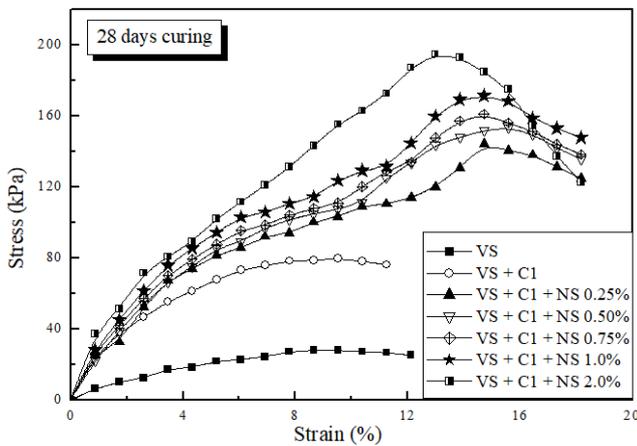


Fig. 6 Stress-strain behaviour of Soil-Cement-nano-SiO<sub>2</sub> mixtures at 28 days curing

significant change in PL showed that the dry strength and toughness near PL would be more, and the permeability and rate of volume change are lesser for treated soils compared to untreated soils.

### 3.3 Soil- cement- nano-silica mixtures- strength benefits

The addition of Nano-silica on cement-treated clay soil resulted in strength improvement from unconfined compressive strength tests. The stress-strain curves shown below prove the statement as mentioned earlier (see Fig. 5-6). The slope of the curves become steeper with the Nanoparticle dosage. The unconfined compressive strength value has raised a little with the mixing of 0.25% Nano-silica into the soil-cement compared to that obtained for 0% Nano-silica in cement-treated soil in 24 hours. But the curing period has played a significant role in improving the strength from the lower value to a maximum of around 144 kPa in 28 days of curing with the same 0.25% Nanoparticle dosage (see Table 5). Dosage 2 and 3 (0.5 and 0.75% Nano-silica in 1% cement-treated soil) showed a similar trend in stress-strain behaviour as well as unconfined compressive

Table 6 Influence of Silica nanoparticles on unconfined compressive strength (UCS) in soil-cement mixtures for various cure timings

Sl. No.	Combinations	Nano SiO <sub>2</sub> (%)	Unconfined compressive strength (kPa) at curing time (days) of				
			1	7	14	21	28
1	VS	0	28.00	-	-	-	-
2	VS + C 1	0	79.70	-	-	-	-
3	VS + C 1 + NS 0.25	0.25	83.77	99.22	116.13	125.21	144.27
4	VS + C 1 + NS 0.5	0.50	105.76	113.12	120.67	146.16	153.58
5	VS + C 1 + NS 0.75	0.75	123.32	127.46	134.73	154.66	161.14
6	VS + C 1 + NS 1.0	1.00	137.29	142.19	152.96	163.87	171.49
7	VS + C 1 + NS 2.0	2.00	165.01	169.75	173.23	183.67	194.90
8	VS + C 1 + NS 5.0	5.00	170.84	178.42	-	-	-
9	VS + C 1 + NS 10	10.00	160.69	165.40	-	-	-
10	VS + C 1 + NS 20	20.00	157.44	162.20	-	-	-

strength value obtained for various curing periods. A minimum value of unconfined compressive strength around 144 kPa can be assured with just 0.25% of Nano-silica in the tested cement treated soft clay although the improvement becomes more with the rise in the dosage of Nanoparticle into the soil. Nano-silica at 2% dosage showed that further increase beyond 1% doesn't seem like producing a remarkable improvement in the tested soil.

With 1% Nano-silica, the unconfined compressive strength value has become 2 fold in just one day compared to that of the soil-cement mix without Nanoparticles. For finalizing the optimum dosage of Nano-silica, dosages were increased to 5, 10, and 20% as well. A similar trend was observed when the soil was treated with Nano-silica alone. An increase in strength up to 5% of Nano-silica could be seen from the results, but the rate of increase was getting diminished with the higher dosages beyond 1%. Also, 10 and 20% of Nano-silica showed an adverse effect on strength development which agrees with the results obtained by Bahmani *et al.* 2014. But an increased cement consumption (4- 6%) is not advisable even though the optimum dosage of Nano-silica is 0.4%. This drawback of the investigation by Bahmani *et al.* (2014) can be covered by the use of 1% cement and 1% Nano-silica. The increase in unconfined compressive strength agrees with the results obtained for the compressive strength of geopolymer composites in research work done by Khater (2016) using nano-silica. Around 3% nano-silica was found to be appropriate for producing the maximum strength in the investigation by Khater (2016), which showed close relation to the optimum dosage obtained in the present work. Also, the observations recorded by Murthy and Ganesh (2019) agree with the ability of nano-silica in improving the compressive strength as well as fracture properties of the concrete.

In Table 6, the effect of the addition of silica Nanoparticles on the soil-cement mix with an increase in

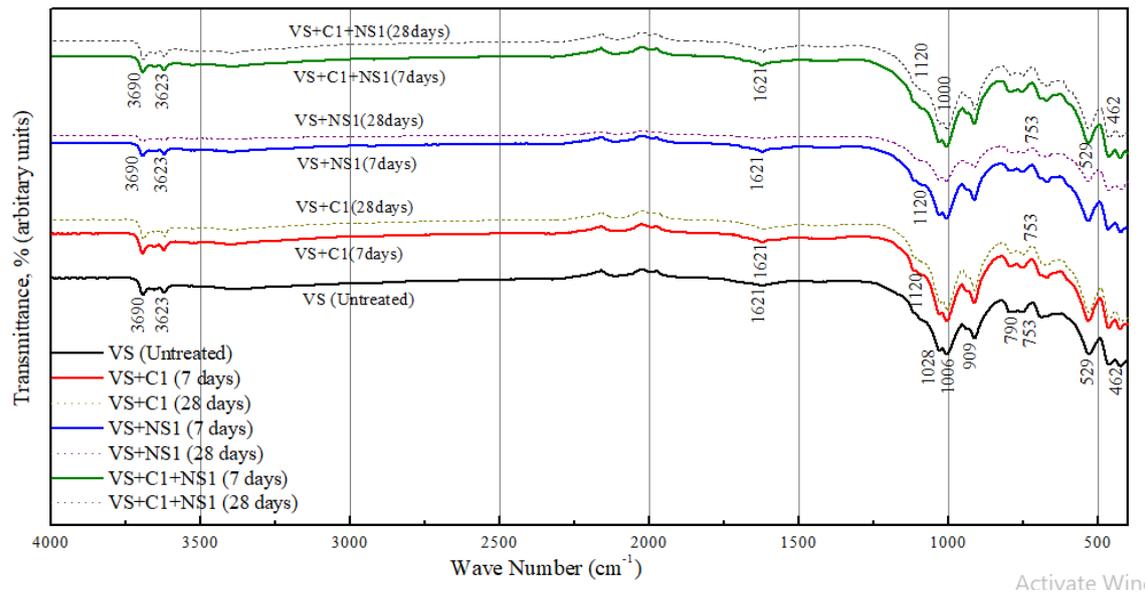


Fig. 7 Patterns of virgin clay and clay stabilized with cement and nanoparticles from FTIR

curing periods is displayed. The increase in unconfined compressive strength (UCS) value has become steady after 21 days of curing with the mixing of Nano-silica in cement treated soil. Well-organized structure of a calcium silicate hydrate gel evolved due to the hydration process cannot remain in a dispersed manner due to the excess dosage of Nanoparticles as well as the aggregation of cementitious products, this results in a reduction in strength after 5%. A similar trend was observed beyond 20% (Li *et al.* 2006). Progress in the strength of clay soil is contributing a considerable reduction in the amount of Calcium Hydroxide in the treated soil which has no role in improving the strength of soil unlike C-S-H gel, and the silica nanoparticles act as a nucleus surrounded by the gel which coats all the clay particles (Choobbasti *et al.* 2015).

Even a small dosage (0.25%) of Nano-silica along with cement could result in a considerable increase in strength in 3 - 7 days of curing, and it could reduce the fluidity of mortar (Sobolev *et al.* 2006). Many authors used the particles of Nano-silica in a range of 0 to 5% as in the present study that blended with cement, lime or fibers, etc. (Changizi and Haddad 2015, Pashabavandpouri and Jahangiri 2015, Choobbasti *et al.* 2015, Hanson *et al.* 2016, Stefanidou and Papayianni 2012, Sobolev *et al.* 2006, 2009). With the higher doses of Nano-silica in the soil-cement mix, the relative strength development has arrived at a peak in 21 days after that period, the rate of increase in strength was found to be slower. Specific vital observations can be pointed that the unconfined compressive strength (UCS) value, as well as stress-strain behavior, becomes better in the presence of Nanoparticles. 1% of Nano-silica can provide a commendable improvement in 28 days around 5 fold that of the virgin soil. 2% of Nano-silica could provide 6 fold relative strength development, which indicates that beyond 1%, Nano-silica could not be responsible for a proportionate development in unconfined compressive strength (UCS) value.

Moreover, beyond 1% of Nano-silica shifts, the nature

of the failure to a highly brittle one and the adverse effect is that once the peak has reached, in no time, the soil shatters down without giving a warning. Hence, the optimum dosage of Nano-silica is decided as 1% by dry weight of the soil when it is used with and without the presence of a traditional additive like cement since both the results show a similar trend in the strength improvement. Even though the results of 2 and 5% of Nano-silica showed some improvement compared to that of 1%, by keeping justification to the aim of the present work that is “to develop an ideal combination of Traditional and Non Traditional additive which neither hinders the environment nor compromise on the strength of the treated soil”. The difficulty in using higher dosages like 5, 10 etc. is that (1) the Nanoparticles demand more water to form a uniform soil mixture, (2) the silica Nanoparticles occupy double or triple the volume of the soil, hence the mixing is difficult. The good quality of the mixture could not be assured, (3) soil become highly brittle which fails suddenly without prompting with the spreading of cracks on the sample, (4) since the cost/kg of nano-silica is high (around 935 Indian Rs./kg), the higher dosages for stabilization is not economical.

#### 4. Microstructural and chemical characterization of treated clay soils

Chemical and Microstructural Analysis was conducted for the detailed investigation on the reason behind the strength enhancement and the changes in soil structure. Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction tests (XRD), and Scanning Electron Microscopy (SEM) were adopted for the current research and the fine points are summarized in the following sessions.

##### 4.1 Fourier transform infrared spectroscopy (FTIR)

Spectral response of IR spectroscopy is typically

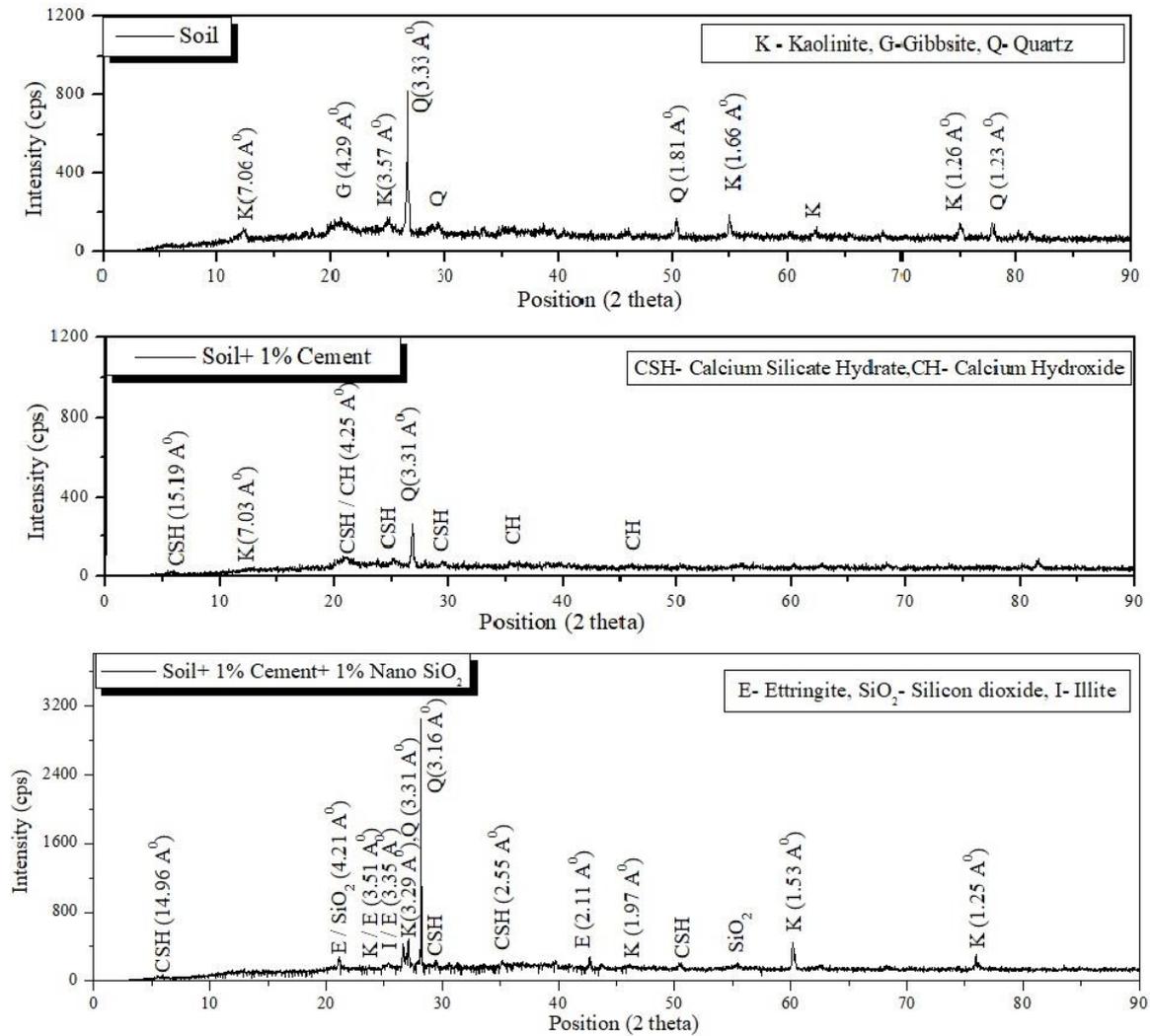


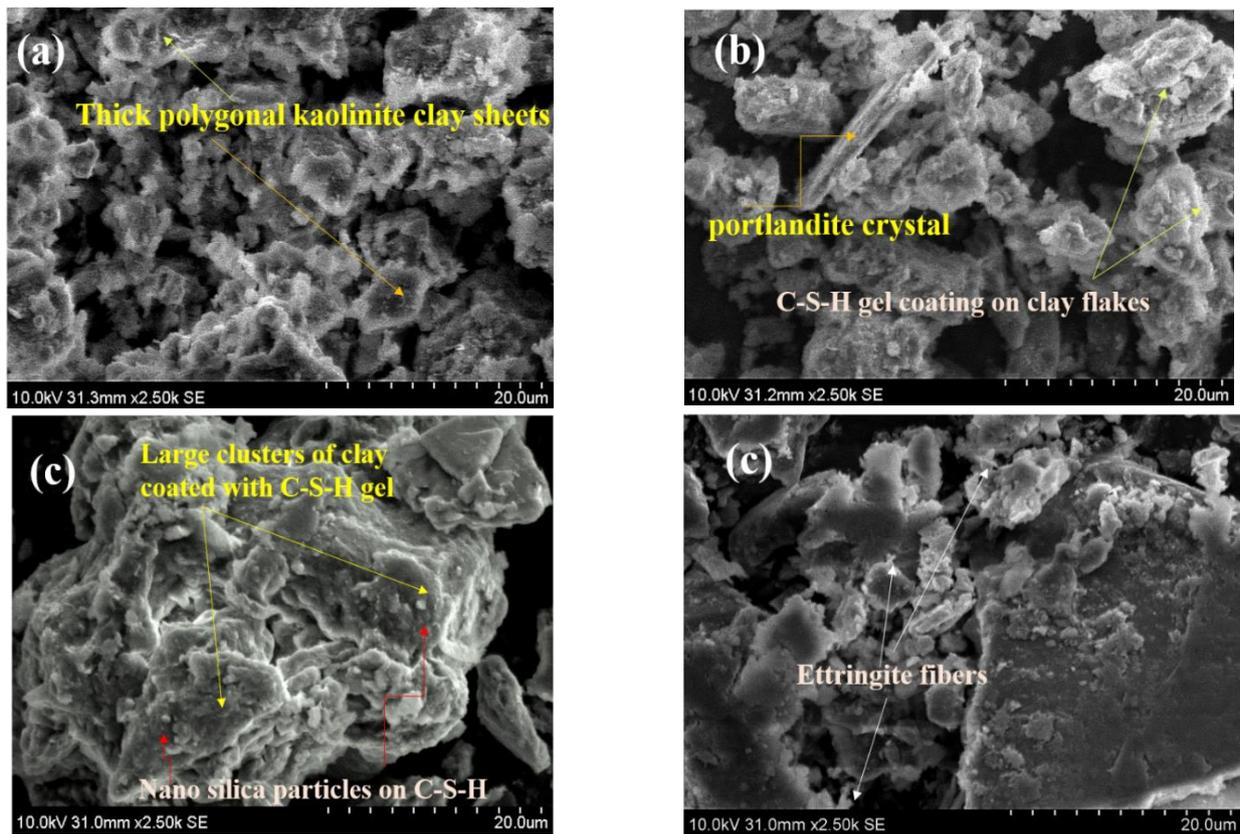
Fig. 8 XRD pattern of untreated soil and soil treated with cement and nano-SiO<sub>2</sub> after a curing period of 28 days

displayed in the range of 4000 and 400  $\text{cm}^{-1}$  in which stretching and bending vibrations of OH and Si-O groups. Soft clay stabilized with cement and silica nanoparticles were inspected with the aid of the Fourier Transform Infrared Spectroscopy technique for analyzing the effect of the curing period. Fig. 7 demonstrates the FTIR spectra of soil without any additives and soil stabilized with cement and nano-silica after a keeping in a desiccator for 7 days and 28 days. A wide-ranging group of Si-O-Si band in the region of 600–1500  $\text{cm}^{-1}$  was observed in the spectra of stabilized soil. That region possibly is associated with the complicated spectra of C-S-H (Hessam *et al.* 2014). The differences in the transmittance measurements and locations of the peaks in the raw soil, the cement-treated soil and the soil-cement blend with nano-silica possibly divulge that the nature and volume of the C-S-H segment has reformed. Along with this influence of C-S-H on the stiffness of the soil, the surplus C-S-H could lessen the soil perviousness by clogging the capillary apertures and consequently refining the structure of the soil in micro-level, which may well contribute to the improved compressive strength (Hessam *et al.* 2014).

The wide-ranging groups appearing at 3653  $\text{cm}^{-1}$  in the

spectra of all soil samples linked to the overlapping stretching vibrations of the structural -OH groups of Calcium hydroxide evolved in the course of the hydration of tri-calcium silicates and di-calcium silicates, and the free -OH groups of water particles existing in the blend. This group decreased for specimens wherein soil was cured with cement and cement plus nano-silica as the hydration process progressed, indicating a diminution in free water owing to C-S-H bond development. The reduction in the strength of this band for VS+ C1 and VS+C1+NS1 was the most prominent after 28 days of hydration. This evidence shows well agreement with the trend obtained for Biricik and Sarier, 2014.

Two stable groups at 3690 and 3623  $\text{cm}^{-1}$  recognized are allied with octahedral stretch vibrations from OH groups; typically, those 2 bands are conditioned to separate kaolinite from the other minerals (Lescinskis *et al.* 2018). The bands were also seen at 1028  $\text{cm}^{-1}$ , and 1006  $\text{cm}^{-1}$  and were attributed to Si-O stretching among clay units, this may be attributable to the reordering of clay lattice due to stabilization. The band at 909  $\text{cm}^{-1}$  matches with OH distortion of hydroxyl groups. Most other bands, such as Si-O vibrations viewed at 790  $\text{cm}^{-1}$ , 529  $\text{cm}^{-1}$ , and 462  $\text{cm}^{-1}$



(a) Untreated soil (b) Soil+ 1% Cement and (c) Soil +1 % Cement + 1% Nano SiO<sub>2</sub>

Fig. 9 SEM results of soil samples after a curing period of 28days

also confirmed the existence of kaolinite, though bands at  $1621\text{ cm}^{-1}$  marked bending vibrations of water molecules (H-O-H). The absorption peak in the  $1621\text{ cm}^{-1}$  region was attributed to the OH deformation genre of water which is found predominant in soil sample mixed with cement and nano-silica. Other bands observed, such as the Si-O vibrations at  $790\text{ cm}^{-1}$  and  $753\text{ cm}^{-1}$  also confirmed the existence of kaolinite (Madejova and Komadel, 2001, Latifi *et al.*, 2016). The bands were also observed at  $462\text{ cm}^{-1}$  were validated to Si-O-Si bending. The doublet at  $790\text{--}793\text{ cm}^{-1}$  is due to Si-O-Si inter tetrahedral bridging bonds in SiO<sub>2</sub>, and OH deformation band of gibbsite at  $1000\text{ cm}^{-1}$  are finger-prints of the characteristic vibrational modes which are recognized effortlessly (Saikia and Parthasarathy, 2010). The formation of Ettringite and stretching vibrations of the S-O bond of gypsum is validated by the presence of a band at  $1120\text{ cm}^{-1}$ . A change in the intensity of the corresponding band is observed with changes in the curing time and the additive which shows well agreement with the observations by Birick and Sarier (2014).

#### 4.2 X-ray diffraction tests (XRD)

XRD tests were piloted for investigating the crystal-like clay behaviour before and after stabilizing with cement and Nano-silica. Fig. 8 illustrates the XRD patterns of raw soil and soil treated with cement and Nano-silica. According to the pattern, virgin soil contains quartz and minerals of Kaolinite. The peaks of Kaolinite decreases with the

addition of cement and Nanoparticles. With the addition of cement, the soil-cement matrix exhibits peaks of both calcium hydroxide and C-S-H (Calcium Silicate Hydrate) gel made during the pozzolanic reaction of cement in the presence of water. The obtained strength improvement in the soil-cement mix is because of the formation of C-S-H gel produced. Consequently, it reduces the pore volume and increases the bond strength between the flaky structures coated with the gel.

The free Ca (OH)<sub>2</sub>, which has no role in strength improvement of soil, is consumed by the Nanoparticles added in the next phase of stabilization for generating surplus C-S-H gel within the soil matrix. The reductions in peaks corresponding to CH (Calcium Hydroxide) and an increase in peak intensity of C-S-H gel in the XRD pattern confirms the statement mentioned above. The peak corresponding to Ettringite (E) also declares that the treated soil matrix is firm and intact against drying shrinkage.

#### 4.3 Scanning electron microscopy (SEM)

Stavridakis and Hatzigogos (1999), Horpibulsuk (2006), Chew *et al.*, (2004), Bell (1976) made an effort to analyze the chemical properties of cement and minerals of clay and then their information about bonding due to cementation during the reaction between them by scanning electron microscopy (SEM). Based on the investigation, it is confirmed that the chemical interaction amongst calcium hydroxide and the flaky surface of clay results in the

evolution of novel compounds in the soil-additive matrix. SEM photographs in Fig. 9 (a)-9(c) helps to visualize the noticeable variations after 28 days occurred within the soil fabric before and after the introduction of stabilizers used in the present study. Since the new composites formed during the hydration process are missing in the raw soil, it exhibits an uneven structure with lots of voids (Solanki and Zaman, 2012). Unlike the structure of untreated soil (Fig. 9(a)), the SEM image of treated soil with the optimum dosage of cement (Fig. 9(b)) unveils needle-like growths in the soil-cement matrix formed by the accumulation of calcium aggregates. This finding can be taken as the cause of a definite increase in the unconfined compressive strength value of the soil-cement specimens. Due to the addition of cement into the soil, flocculation was formed which converts the cement-soil matrix into a granular arrangement. Comparable interpretations were made by Onitsuka *et al.* (2001). Unlike the growth of needle hydrates observed in almost all nano-modified soil samples, soil treated with silica nanoparticles was found to be denser in structure and a noticeable change in the size of hydrated crystals was also detected, this is found to be similar in the study of Stefanidou and Papayianni (2012). The existence of some particle packs observed in the raw soil was due to the micro-pores formed around large particle packs because of the adherence of clay particles in the presence of moisture. The volume of those pores was diminished, thereby increasing the compactness because few of the pores were clogged by the cementitious gel (Fig. 9(b)).

Fig. 9(c) explains the micrograph of clay stabilized using cement and nano-silica. This image reflects the formation of C-S-H gel, filling of soil pores with the gel, densification of the soil matrix, strength gain, and the wrapping of clay flakes with the secondary C-S-H gel developed by the reaction between cement and nano-silica. Increasing the packing density of the treated soil and uniform scattering of C-S-H in the matrix can be explained by the terms the nano-filling and nucleation effect of particles, respectively. The observations from SEM analysis shows well agreement with the research done by Hessam *et al.*, 2014. Stability of the secondary C-S-H gel formed (in the presence of nano-silica and cement) is more creditable and thereby densifying the soil-cement-nano-silica mixture.

## 5. The mechanism behind the strength gain of the soft clay soils after stabilization

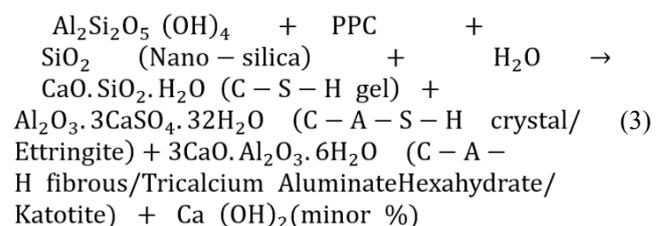
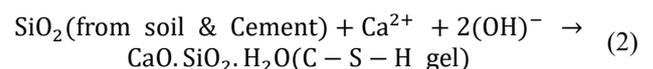
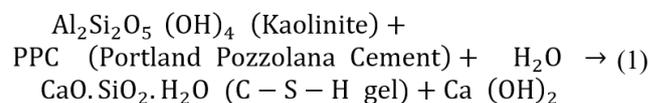
The significant findings from the chemical and microstructural analysis were combined in the following session that clear the whys and wherefores of the mechanism behind the strength improvement of soft soils treated with cement and nano-silica.

### 5.1 Effect of nano-silica in soil-cement mix

Nano-silica acts as a catalyst which accelerates the pozzolanic reaction when it is added to the moist soil-cement mix. The elevated specific surface area of Nanoparticles can result in a higher rate of pozzolanic reaction, thereby more effective in reducing pore spaces in

the soil structure and compressibility. Even a small quantity of Nanoparticle could be responsible for strength improvement, but when used in excess amount, it can adversely affect the soil structure. But it was not proved in the present work. The pozzolanic reaction results in forming a gel called C-S-H (Calcium-Silicate-Hydrate), which further influence the bonding strength, clogging of pores, and reduction in calcium hydroxide crystals. Higher the amount of calcium hydroxide crystals, lower be the compressive strength (Sobolev *et al.* 2006, Sobolev *et al.* 2009, Jo *et al.* 2007). The reduction in Calcium hydroxide crystals not only helps in improving compressive strength but it can control the leaching of calcium ions which in turns helps positively in durability, compressibility since the calcium ion leaching accelerates the migration of calcium ions from cement-based materials making it a more porous structure (Farzadnia *et al.* 2012). Higher rate of strength development at early stages of curing and the lower rate at later stages can be expected as the effect of Nano-silica in Cement-soil mix cured at a higher temperature. The coating formed owing to C-S-H gel around the cement particles is thicker and harder at the early stages of curing, which provides higher initial strength. But the thicker coating formed in samples cured at higher temperatures hinders the further process of hydration of cement which results in a lower rate of strength increment at later stages. The cement-soil cured at lower or normal temperatures also possess high early strength and homogenous as well as less permeable C-S-H coating (Hou *et al.* 2012). When Nano-silica is used in Cement stabilization, it could replace a commendable amount of cement required for strength development. Lesser sized Nanoparticle (below 20 nm) can provide more effect in strength development and reduction in pore spaces within 28days (Stefanidou and Papayianni 2012) of curing than higher ones (more than 50 nm) (Givi *et al.* 2013).

The reason behind the strength gain and bonding in materials homogenous to cement, is the development of C-S-H gel in double phases: 1) During the cement hydration process in the presence of ample water content in soil-cement mixture 2) when nano-silica particles react with Calcium hydroxide. Because of this reason, the primary additive of the present work, i.e., Nano-silica with a slight dose of cement, is substantial in refining various Geotechnical properties of soft soils.



The above-listed equations can thoroughly explain the chemical changes that happened in the soil additive blend depending on the type of additive used. Eq. (1) shows that the Portland Pozzolana cement in the presence of ample moisture content into the kaolinite clay forms Calcium silicate hydrate (C-S-H) in gel form, which increases the bond strength as well as reduces the pore space considerably. Calcium hydroxide formed in this process is not having a significant role in the strength gain which is dissociated and combines with silicon dioxide in the soil and the cement to create secondary C-S-H gel (Eq. (2)). Addition of nano-silica in the kaolinitic clay with a small percentage of cement and sufficient water to form various products like Calcium silicate hydrate (C-S-H) in gel form, Ettringite (C-A-S-H crystals), mono-sulpho-aluminates (hexagonal /rose petal-shaped in SEM), C-A-H fibres (Tricalcium Aluminate Hexahydrate called as Katotite) etc. and a reduced amount of Calcium hydroxide (Eq. (3)). These combinations of gel, fibrous structures increases the stiffness of the soil and clogs the pores to the maximum when the additives are in optimum dose. Excess or reduced dosages might not be able to produce the same effect in the soil matrix.

In addition to this, the pH value of the untreated clay obtained was around 4.84, and that of the soil-cement mix was 6.47, which is slightly reduced to 6.18 with the addition of silica nanoparticles. The change indicates that the addition of Cement-nano-silica to soil in optimum dose is safer in the environmental aspects since the groundwater lies in 6 - 8.5 range and that of raw water lies in 6.5 - 8.5 range as per specifications are given by WHO Guidelines for Drinking Water Quality (3<sup>rd</sup> Edition, Vol. 1 Recommendations, 2008) and IS 10500: 2012 (Indian Standard Drinking Water — Specification (Second Revision)).

## 6. Comparison with traditional cement stabilization

Even though cement stabilization is effective in improving the properties of weak soils, it has some demerits in numerous ways. The average rate for the purchase of cement might be low, depend on the manufacturers, but it ranges around 125.6 US Dollars per metric ton in 2018 (T. Wang 2019). However, the average rate of Nano-silica is about 13-15 US Dollars per kg, which is expensive compared to the cement rate but lesser than that of many other nanoparticles such as Nano Titanium Dioxide, Nano-silver, etc. The optimum dosage of cement for cement stabilization, from many kinds of literature, is stated in between 6 and 10 % by dry weight of the soil. Andrew R.M (2018) in recent research, pointed out many distressing facts about global cement carbonation. The carbon dioxide emission from cement production alone and various other phases, including disposal methods, is about 900 Mt CO<sub>2</sub> (Xi *et al.* 2016). The rate of carbon dioxide removal from the atmosphere is around 94 - 232 \$USD /t-CO<sub>2</sub> (Keith *et al.* 2018). While considering all these factors on the harmful effect on the environment and the cost of saving the environment caused by the use of high dosages of cement

for the soil stabilisation, it is advisable to minimize the usage of cement in soil stabilization. The effect on the environment as well the cost for pollution control while considering the future factors, the introduction of nano-silica (1%) together with 1% cement is wiser compared to the traditional-economical cement stabilization.

## 7. Conclusions

A detailed, comprehensive study on the performance of various additives such as Nano-silica and cement was conducted on an Unconfined Compressive strength apparatus. Some significant findings can be listed as follows taken out from the results:

- The stress-strain behaviour of the cement-treated clay showed a brittle nature for all the curing periods.
- The combination of cement and Nanoparticle provides a plausible performance in the compressive strength of the soft clay.
- With the addition of the optimum dosage of Nanoparticle, the required cement dosage can be reduced to get ample improvement in the strength.
- The addition of the Nanoparticle in the presence of cement could reduce the peak strain in the treated soil samples compared to untreated and samples without cement. The overall rigidity of the treated soil is increased due to this stabilization.
- Based on the FTIR spectra, the presence of kaolinitic clay mineral and the rearrangement of clay structure with the inclusion of additives were confirmed. The C-S-H gel formed in the treated soil is identified, and the variations of the corresponding spectral peaks reveal the nature and quantity of the C-S-H gel evolved during the reaction process.
- The increased peaks of C-S-H and decreased peaks of Ca (OH)<sub>2</sub> exposes from the XRD patterns of soil treated assures the stiffness improvement of soil with the additive dosages.
- Reformation of flaky clay particles to large clusters coated with C-S-H gel, pore volume reduction, and increased pack density is perceptible while comparing the SEM images of untreated and treated soil.
- Formation of C-S-H gel during the pozzolanic reaction, reduction in Ca (OH)<sub>2</sub>, and clay lattice reformation are responsible for the elevated stiffness, reduced pore volume and better particle bonding etc. in nano-silica treated soil-cement mixture. The size of individual Nano-silica (17 nm) and the high reactivity of the Nano-silica particles also play a significant role in improving the soil strength in 28 days.
- The optimum of Nanoparticles with 1% cement is found to be an effective formula for the treatment of soft clay for converting it to stiff to very stiff clay.

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