

# SOIL STABILIZATION USING SILICON CARBIDE (SIC) NANOPARTICLES: CONFIRMATION USING XRD, SEM, AND FTIR

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**Abstract.** The current research focuses on nanoparticles' ground-improvement potential using clayey soil mixed with varying amounts of the nanoparticles "Silicon Carbide". With an increase in the amount of nanomaterial, a tendency of improvement has been recorded in liquid and plastic limits, as well as the plasticity index. The maximum reduction in liquid limit (15.8%), plastic limit (13.6%), and plastic index (18.7%) was recorded at 0.25 gm of Silicon Carbide as compared to control (0 gm of SiC). There was a 26.7% and 33.3% increase in the cohesion of soil at 0.25 gm and 0.3 gm of Silicon Carbide, respectively. Furthermore, when the Silicon Carbide content increased from 0.25 gm, the rate of increment of friction angle also increased. It was 87.5% and 137.5% at 0.25 gm and 0.3 gm of Silicon Carbide, respectively. Furthermore, 0.3 gm of Silicon Carbide, is found to be optimal within the scope of the experiment as at this amount of Silicon Carbide both cohesion and angle of friction attained maximum. XRD, SEM, and FTIR were used to confirm the findings. It concludes that by using even a small amount of nanomaterial, an appreciable change in the properties of clayey soil can be obtained in the field.

Keywords: nano-material, clayey soil, Silicon Carbide, index properties, cohesion, angle of friction.

# Introduction

The use of nanotechnology in civil engineering is opening up new avenues for soil stabilization research and development. Clay rich soil was termed as problematic soil for civil engineering usage, especially in foundations, as well as subgrade for road and rail beds. More specifically, when clayey soil comes in contact with water, it starts deteriorating chemically and mechanically, posing a lot of difficulty in saving the structures over or near them (Das & Sivakugan, 2018). Soil stabilization is a strategy that can be employed accordingly and as per the needs of a specific site (Hausmann, 1990). As we know, that "All Grounds are not Terra Firma" and "Unstable Grounds Do Exist", we need to have ready methods for slope stabilization as "Site Shifting" is not always possible.

In clayey soils, when moisture content reaches towards its liquid limit, it can develop thixotropy and may result in construction settlement, during construction, after completion or at any time during their life span (Firoozi et al., 2017). Swelling of Clayey Soil is another problem that

persists very commonly in alluvial plains due to clay-rich deposits along paludal zones with high organic content due to floral and faunal bioturbation. Hence, stabilization of soil aims to have higher strength so that deformability can be minimized. So, to tackle this problem, various solutions have been attempted and new methods have already been employed, like the traditional use of lime, cement, readymade admixtures, waste from industries, etc. (Chittoori, 2008; Pedarla et al., 2011). Clayey soils are typically firm when they are dry and lose their firmness as they become saturated. Soft clay is related to low compressive strength and extravagant settlement. This decrease in strength because of dampness prompts serious harm to the foundation and superstructures. Designing structures on sites with clay deposits poses challenges to designers and engineers. The damage caused by settlement of structures on expansive soil is estimated to be a billion dollars around the world. The damage related to expansive clayey soils is not only the result of the absence of deficient

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engineering remedies but also the inability to distinguish the presence of expansive soil in the subsurface due to inadequate exploration during the preliminary phase of the project investigation.

The use of Nano emulsions for the encapsulation of desirable chemicals in hair-related products enhances their distribution into the deeper hair shafts. The use of zinc and titanium micronized NPs in sunscreen lotions results in transparency, a less oily texture, and less odour, as well as making the lotions more absorbable into the skin's layers (Hameed et al., 2019). The food sector has expanded its demand for NP-based materials since most of them include critical components and are non-toxic and stable at high temperatures and pressures (Khajehei et al., 2019). NPs have significant benefits for treating water at considerable depths and in any region where traditional methods have failed (Samuel et al., 2018). Green nanomaterials provide a wide variety of capabilities for treating water polluted by harmful metal ions, organic and inorganic solutes, and pathogenic microbes (Needhidasan et al., 2014). Drugs with NPs have excellent immunogenicity, biorecognition, and pharmacodynamics, which increases pharmaceutical efficacy pharmacokinetics, non-specific toxicity (Janagam et al., 2017). Cancer is a fatal illness that still faces obstacles. NPs are the ultimate platform for biological purposes and therapeutic treatments. As a result, there is an urgent demand for anti-cancer medication. Failure to create a comprehensive anti-cancer response in malignant and normal cells [g]. Recently, researchers discovered that metal oxide NPs such as Zn and Ce oxide NPs offer great potential as anti-cancer medications (Rasmussen et al., 2010; Bisht & Rayamajhi, 2016). The goal of treating loose sub-grade soil is to reduce settlement and improve loadbearing ability to serve diverse foundation purposes. As a result, throughout the years, stabilization has been one of the most sought strategies for improving the efficiency of subgrade soil strength characteristics (Chen & Lin, 2009). It is the modification of soil to boost its physical attributes for a civil engineering works, and it might be utilized on pavements, roadways, airports, railways, and a variety of other areas where subsoils are inappropriate for building (Ingles & Metcalf, 1972).

There is continuous research with nanomaterials and technology to enhance soil strength. Nanotechnology is a nearly six-decade-old concept. It was brought to light when Feynman (1960) gave a speech titled "There is Plenty of Room at the Bottom", yet it did not achieve popularity or acceptance at the time. The use of nanotechnology in geotechnical engineering has brought a new dimension to the creation of "Engineered Soil", which has a variety of desired and enhanced features (Majeed & Taha, 2012). Nanoparticles have the ability to cation and anion exchange, fixation and cementation properties. It has been found that only an appreciable change in the properties of particles at the micro-level can be achieved by the addition of modifiers, but when the same is reduced to the nano level, the changes are more pronounced (Firoozi et al., 2017). The predominance of changes obtained in the mass properties of particles is in the form of changes in basic micro-geometry, textural arrangement, and the development of new chemical potentials. Nanoparticles are very quick in reactions with other particles, which effectively brings appreciable changes in the properties of material, whether it is physical or chemical. This is achieved by adding an economical amount of nanomaterial to the desired material (Ghasabkolaei et al., 2017). Mercier et al. (2003) suggested electromagnetic forces as the dominant process as compared to gravitational forces in soil stabilization. Alireza et al. (2013) investigated that using lime there was little improvement in soil, but as soon as nano silica was mixed into the lime-stabilized soil, remarkable results were shown, including CBR strength of the soil considerably increased. Studies on future research prospects of nanomaterials suggest that there are emerging possibilities and prospects in time to come using nanomaterials (Al-Rawas & Goosen, 2006; Arora et al., 2019; Huang & Wang, 2016; Majeed & Taha, 2012). Various experimental studies have shown that there are many advantages and potentials for the utilization of nanomaterials in various applications (Arabani et al., 2012; Bahmani et al., 2014; Ghazavi & Bolhasani, 2010; Ghazi et al., 2011; Khalid et al., 2015; Sani et al., 2010; Taha, 2009; Zhang, 2007). A brief tabular data has been presented in Table 1.

At present, the application of nanoparticles is encouragingly increasing, but there is a need to find out costbenefit analysis, easy invention of nanoparticles, amount optimization, easy mixing technology, placement and impact on the environment. The present study aims at stabilization of low plastic clay which is available in several areas of northern India. Nanoparticles of silicon carbide (SiC) have been added to the clay and its geotechnical behavior at micro and macro level have been observed.

The SiC nanoparticles has been adopted due to its unique properties such as wide and tunable band gap, resistive towards high temperatures, chemical inertness, high tensile strength and hardness.

#### 2. Methodology and experimental studies

Clay samples were collected from a dry pond nearby the Aligarh Muslim University (AMU) campus in Aligarh district (UP, India), from a pit excavated to a depth of 1 m. Aligarh is situated on the alluvial soil of the Indo Gangetic plane and is historically important for its educational institutions like AMU.

The specific gravity of 2.52, Soil classification CL, Liquid limit 38, Plasticity index 16%, Optimum water content 19%, Maximum dry unit weight ( $g/cm^3$ ) 1.64. The angle of friction of the soil 8 and cohesion of 45 KPa (Table 2).

Whereas, the SiC nanoparticles was bought from Sisco Research Laboratories Pvt. Ltd. (SRL) – India.

Soil used	Nano Particle utilized	Type of experiment	Increase	Decrease	Reference
Low plastic clay	Nano silica + Lime	CBR	CBR strength of the soil and soil-lime mixture up to 21 and 7.5 times, respectively	_	Alireza et al. (2013)
Kaolonite clay	Nano MgO and Nano Al <sub>2</sub> O <sub>3</sub>	Attenberg limits, MDD (maximum dry density), OMC (optimum moisture content)	Maximum dry density	Liquid limit, plastic limit, Swelling potential and plasticity index	Sanjeev et al. (2017)
Sand	Nano CuO, Nano MgO, and Nano Clay	Maximum dry density and the optimum moisture content, unconfined compressive strength	Maximum dry density and the optimum moisture content, unconfined compressive strength	_	Majeed and Taha (2012)
Sand with clay	Nano Clay	Direct shear test and Atterberg limits	Shear strength	PI	Sani et al. (2010)
Cement- treated residual soil	SiO <sub>2</sub>	Consistency, compaction, hydraulic conductivity, and compressive strength	Unconfined compressive strength	Hydraulic conductivity	Bahmani et al. (2014)
Sand	Modified Montmorillonite Nano Clay	Plasticity and strength characteristic	Liquid limit and plasticity index, unconfined compressive strength	_	Ghazi et al. (2011)
Subgrade soil	Biocompatible silica nanoparticles	Atterberg Limits, CBR	California Bearing Ratio (CBR)	Biocompatible silica nanoparticles	Buazar (2019)

Table 1. Use of nanomaterials for soil stabilization available in open literature

Table 2. Physical and chemical properties of the sample soil

Properties	Soil
Specific gravity	2.52
Soil classification	CL
Liquid limit	38%
Plasticity index	16%
Optimum water content	19%
Maximum dry unit weight (g/cm <sup>3</sup> )	1.64

# 2.1. Synthesis of nanocomposites of clayey Soil and SiC nanoparticles

The nanocomposites mixture was prepared mechanically using the "Coning and Quartering" method. Table 3 shows the different percentages of SiC nanoparticles by weight that was added.

The experimental program with varying SiC nanoparticles has been carried out under the standard codes of practice. Liquid Limit of the soil mix has been determined using Casagrande apparatus as guided in IS 2720-5 (Bureau of Indian Standards, 1985). Further, the Plastic Limit of soil silica mix has been obtained as per IS 2720-6 (Bureau of Indian Standards, 1972).

Shear strength parameters of a soil sample, determined in unconsolidated undrained triaxial compression without pore water measurement. The strain controlled triaxial apparatus were used following IS 2720-11 (Bureau of Indian

Table 3. Synthesis details of nanocomposites of Clay and SiC nanoparticles

Sample Identity	Clay (gm)	SiC (%)
SO	300	0
S1	300	0.033
S2	300	0.050
S3	300	0.066
S4	300	0.083
S5	300	0.10

Standards, 1993). Swelling Potential (SP) of soil, calculated from the values of Plasticity Index. By using the given relation:

SP =  $(2.16 \times 10^{-3})$  (PI) 2.44.

For working out the compaction behavior of modified soils, Proctors Compaction test was carried out at OMC as per IS 2720-7 (Bureau of Indian Standards, 1980) recommendations (reaffirmed in 2011).

SEM (Scanning Electron Microscopy) and XRD (X-Ray Diffraction), analysis of plain sample and with mixed nanomaterial was also obtained. For SEM analysis, a small quantity of the sample was fixed on the SEM holder and coated with gold with the help of a gold sputter coater unit. The sample was mounted on the aluminum stub and observed under a Scanning electron microscope (JSM 651 LV-JEOL, Japan) at 15 kV. Fourier Transform Infrared (FTIR), investigation on the samples was also conducted.

### 3. Results

A comprehensive set of experimental programs were carried out to determine the geotechnical properties of clay soil in natural state and after SiC nanoparticles mixing. Further behavior of the mix was also monitored as the percentage of mixed SiC nanoparticles has been varied. The SiC nanomaterial is available on online and offline market in most of Asian nations. The price of this material is subject to demand and supply and vary from place to place and season to season also.

The liquid limit, plastic limit, plasticity index, and other index properties decreased as SiC nanoparticles were added to it (Figure 1) up to 0.25 gm. The maximum percent reduction in liquid limit (15.8%), plastic limit (13.6%), and plastic index (18.7%) was recorded at 0.25 gm of SiC as compared to control (0 gm of SiC). However, further increment of the SiC leads to reverse the trend. Lessening the plasticity indices indicates that soil properties have improved. Hence, very small amount of SiC nanoparticles can significantly increase strength and improve the properties of soil. The results are in line with the behavior of nanoparticles reported earlier (Alireza et al., 2013; Taha, 2009).

The reduction in the plasticity index results in a reduction in the soil's swelling potential. When SiC nanoparticles were added to soil up to 0.25 gm, the swelling potential of the soil decreased to 40.1% as compared to control (Figure 2). This decrease in swelling potential could be attributed to the fact that it allows less water to enter the pores, which in turn reduces the swelling of the montmorillonite because SiC nanoparticles fill the pores of the soil mineral.

Soil shear strength is controlled by both cohesion (C) and angle of friction ( $\Phi$ ). The chemical bonding force is represented by cohesion, whereas the angle of friction is an important factor influencing inter-grain slippage and friction strength. In clay, the cohesion value is expected to be greater than the angle of friction, and vice versa in sand. To observe the relationship between the SiC nanoparticles content and the shear strength parameters, the parameters (C and  $\Phi$ ) were plotted against the SiC content.

A graph of the SiC nanoparticles content and the cohesion, angle of friction is shown in Figure 3. The angle of friction and cohesion increased as the SiC nanoparticles content increased. The angle of friction for control (without SiC) was 8, while the angle of friction for 0.3 gm of SiC was 19. In contrast, soil cohesion varies from 45 to 60 depending on the percentage of SiC present. When SiC nanoparticles are introduced into the soil, the SiC nanoparticles fill the spaces between the soil and begin to induce interlocking behavior in the soil. This helps to explain why clayey soils have high cohesion and angle of friction values as SiC nanoparticles content increases. There was a 26.7% and 33.3% increase in the cohesion of soil at 0.25 gm and 0.3 gm of SiC, respectively, as compared to control. Further, when the SiC content increases from 0.25 gm, the rate of increment of friction angel also



Figure 1. Effect of SiC nanoparticles and water content on engineering properties of soil



Figure 2. Effect of SiC nanoparticles on swelling potential of the soil



Figure 3. Effect of SiC nanoparticles on Cohesion of the soil and Angle of friction of the soil (degree)

sharpens (87.5%). A 137.5% increase in angle of friction was observed at 0.3 gm of SiC as compared to control.

The improvement in clay strength after incorporating SiC nanoparticles into it can be attributed to the strong "Coulombic Interaction" between silicon carbide nanoparticles and clay particles as illustrated in Figure 4. The silicon carbide nanoparticles have a large surface area on which clay particles are uniformly blended, which maintains the strength of the clay particles by intercalating between them (Ahmad et al., 2016).

The SEM image of pure SiC nanoparticles, which appear to be very fine granulated, is shown in Figure 5. Figure 6 shows SEM images of sample S0, S1, S3, and S5 nanocomposites at 5  $\mu$ m. Figure 6a depicts a micrograph of pure clay with a flakes-like structure. Clay/SiC nanocomposites (Figure 6b, 6c, and 6d) almost have the same morphology as pure clay but appear to be more agglomerated due to strong columbic interaction between silicon



Figure 4. Schematic representation of interaction between clay and silicon carbide nanoparticles



Figure 5. SEM image of SiC nanoparticles



Figure 6. SEM images of: a - S0; b - S1; c - S3; d - S5 nanocomposites

carbide nanoparticles and clay particles. Furthermore, no pure silicon carbide nanoparticles appear, indicating that the soil has been completely mixed with SiC nanoparticles.

Figure 7 depicts the FTIR spectra of S0, S1, S3, and S5 nanocomposite. In the spectra of pure clay (Figure 7a), the peak at 3620 cm<sup>-1</sup> is due to O-H bond stretching vibration, while the peak at 3436 cm<sup>-1</sup> is due to O-H (hydration) bond stretching vibration. O-H bending vibrations cause the peak at 1638 cm<sup>-1</sup>. Si-O in plane stretching vibration has a peak at 1020 cm<sup>-1</sup>. In case of Clayey soil/SiC (S1) (Figure 7b), the peak due to O-H bond stretching is slightly shifted to lower wavenumber and appeared at 3610 cm<sup>-1</sup> but the hydration (O-H) bond stretching vibra-

tion found at 3435 cm<sup>-1</sup> which is almost similar to pure clay. The peak due to stretching vibration of Si-O plane is appeared to broaden and slightly shifted to higher wavenumber at around 1030 cm<sup>-1</sup>. FTIR spectra of S3 and S5 nanocomposites showed the same pattern as that of S1 nanocomposite as shown (Figure 7c and Figure 7d). The shifting of peaks in S1, S2 and S3 nanocomposites indicating that the columbic interaction has taken place between clay and silicon carbide nanoparticles. It is in line with the previous finding (Bel Hadjltaief et al., 2018).

X-rays Diffraction Studies X-ray diffraction (XRD) has been developed as a rapid analytical technique. In this analysis the analyzed material must be grounded very



Figure 7. FT-IR spectra of: a – S0; b – S1; c – S3; d – S5 nanocomposites



Figure 8. XRD patterns of: a – S0; b – S1; c – S3; d – S5 nanocomposites (M – montmorillonite, Q – quartz, C – calcite)

finely, homogenized and average bulk composition is determined. XRD had been used primarily for phase identification of a material crystalline in nature. Unit cell dimensions can also be determined through this technique. Figure 8 depicts the XRD (X-ray diffraction) spectra of samples S0, S1, S3, and S5 nanocomposites. The clay's Xray diffraction pattern reveals that it is composed mainly of quartz, with minor amounts of montmorillonite and calcite (Figure 8a). As shown in Figures 8b, 8c, and 8d, the XRD spectrum of S1, S3, and S5 nanocomposites has peaks similar to those of clay (S0). The peak intensities of quartz are significantly reduced after incorporation of silicon carbide into the clay, but no additional peaks of silicon carbide are discernible due to its low amount.

#### 4. Discussion

The addition of a limited amount of SiC nanoparticles causes physiochemical interaction, which leads to an improvement in the geotechnical properties of the studied soil. This increased soil strength after incorporation of silicon carbide nanoparticles may be attributed to strong columbic interaction between silicon carbide nanoparticles and soil particles. The addition of SiC nanoparticles above the optimum amount resulted in a state of mass cluster bonded together, which influenced the mechanical properties of soils.

As no significant work on soil improvement using SiC has been available in the open literature. Its performance has been compared with other Nano materials. Sani et al. (2010) had performed the analysis of soil improvement using Nano-clay. Plastic and liquid limit was calculated, and it was found out that when 0.5 wt.% of Nano-clay was used then significant improvement was not seen but when 1 wt.% of Nano-clay was added then increment of 13% and 38% was observed in liquid limit and plastic limit, respectively, which in turn justifies that plasticity index was decreased by 40%. Moreover, Arabani et al. (2012) mixed clayey sand with 0.05-3% CNTs (Carbon Nano Tubes) by weight of the soil in the soil treatment sector. When compared to the original clayey soil, the compressive strength of soil containing 3% CNTs increased by about 120%. While shear strength increased, the Atterberg limit decreased.

Safety and reactivity of nanomaterials with environment and earth is under study. Risks and uncertainties of Zinc and Copper based nanomaterials had been recently studied (Keller et al., 2017; Rajput et al., 2020a, 2020b). However, the studies on SiC safety and reactivity of this nanomaterial is lacking behind.

#### Conclusions

All the index properties of soil, including liquid limit, plastic limit, plasticity index, swelling potential, and parameters C and  $\Phi$  were all assessed. These findings can help specialists promote better soil quality and other soil properties. The parameter cohesion appears to improve with further addition, and the angle of friction increases with the addition of SiC nanoparticles. The mechanical mixing method was used to successfully synthesize clayey soil/SiC nanocomposites, which were then characterized using SEM, XRD, and FTIR analysis. These studies have confirmed that clavey soil and SiC nanoparticle mix show different levels of chemical interactions. Results of this study reveal that an amount of 0.25 gm of SiC nanoparticles can cause a reduction in L.L., P.L., and P.I. by 15.8%, 13.6%, and 18.7%, respectively. It is evident from the current study that SiC affects the strength, indexes, and strength properties of soil and can be used for geotechnical engineering for soil improvement. Furthermore, the effects of nanoparticle safety and reactivity will need to be investigated.

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## Author contributions

Author Contributions: A. H. A. and M. R. S. were responsible for conceptualization, methodology and project administration. A. H. A. and K. P. are responsible for funding acquisition. J. Q. and M. R. S. are responsible for data acquisition, analysis, draft preparation and validation. M. R. S. & K. P. responsible for software, reviewing and editing. M. R. S and A. H. A. responsible for review and editing. S. A & A. S. B are responsible for editing of revised manuscript and validation.

#### **Disclosure statement**

Authors declare they have no competing financial, professional, or personal interests from other parties.

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