#### **RESEARCH PAPER**



# An Experimental Analysis of Precipitated Silica in Petroleum-Contaminated Clay for the Strengthening of Soil Characteristics

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#### **Abstract**

In recent times, the environmental impacts of petroleum-contaminated clay (PC clay) have intensified, leading to more severe detrimental effects. To address this issue, the use of precipitated silica (P silica) has proven to be an effective solution. P silica offers improved treatment for the contaminated clay and enhances the engineering properties of the soil. A series of experiments such as pH test, electrical conductivity test, unconfined compressive strength (UCS) test, California bearing ratio (CBR) test, free swell index (FSI) test, compaction test and Atterberg limit tests were conducted to compare the soil characteristics of the PC clay with and without the treatment of P silica. The PC clay soil specimens were tested after the inclusion of P silica with varying dosages of 0%, 3%, 6%, 9% and 12% by dry weight of clay soil. The inclusion of P silica at a dosage of 6% resulted in the optimum strength of 275.12 kPa. This dosage also led to a reduction in free swell index (FSI) and maximum dry density (MDD), indicating a flocculation mechanism caused by the presence of P silica. Additionally, there was an improvement in stiffness and strength parameters, as evidenced by the enhancement in CBR (California bearing ratio). Therefore, the effective utilization of P silica in contaminated clay soil provides a variety of applications in the geotechnical field.

Keywords Petroleum-contaminated clay · Precipitated silica · Flocculation · Soil strength · Maximum dry density

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#### 1 Introduction

The ecological system has been significantly impacted by petroleum production in recent times. Soil, an essential resource for life on Earth, is particularly affected by hydrocarbon emissions into the environment. These emissions, which result from activities such as oil exploration, transportation, production, processing and oil spills, have detrimental effects on the safety of structures. They are also responsible for the contamination of soil with hydrocarbons. This contamination not only affects the quality, productivity and ecology of the soil but also reduces its bearing capacity. Additionally, hydrocarbons serve as a major source of heavy metals in the soil, further exacerbating these negative effects (Jabbar et al. 2022). The soil can be affected by oil spills, including petrol and diesel. The soil contaminated with petroleum oil altered the geomechanical properties of the soil. Annually, petroleum production was found to be 12 million metric tonnes (Failed 2013). The released petroleum products produce changes in the engineering behaviour of



soil, ultimately leading to the unsuitability for using the contaminated clay as bases and sub-bases in road construction, land cover materials and topping layers in parking garages. The uncontrolled emission of hydrocarbons also affects the soil and water resources (Koshlaf and Ball 2017; Alardhi et al. 2020). These are released into the soil using leakage in the storage tanks, disposal in ponds from refineries, drilling processes and also due to transportation (Ławniczak et al. 2020; Rathi and Yadav 2019). The presence of hydrocarbons in soil has a significant impact on its properties, including pH, texture, cation exchange capacity and mineral and organic matter content. To ensure the productive use of soil and mitigate the effects of spills, it is crucial to remove contaminants from the environment. Various techniques have been employed to remove hydrocarbons from soil and improve its geotechnical characteristics. These techniques include bioremediation and biodegradation of oil sludge through a two-phase composting system, Fenton treatment, surfactant-enhanced remediation, precipitation method using alkali derived from green wastes, extraction of heavy metals and column extraction of metals using ethylenediaminetetraacetic acid (EDTA) and nitrile-triacetic acid (NTA). However, it has been observed that these techniques, including other chemical methods, can have negative effects and may not be compatible with the soil. To enhance the characteristics of clay soil, the use of silica fume, lime and cement has been found effective. Additionally, clay can be stabilized with sodium silicate, sodium hydroxide and nano-silica in combination with randomly distributed fibres to further enhance its strengthening characteristics (Kulanthaivel et al. 2022; Selvakumar et al. 2021; Kulanthaivel et al. 2022). The nano-silica and sodium silicate in clay was utilized to improve the soil characteristics and Young's modulus of the treated soil (Selvakumar et al. 2021). Also, nano-silica combined with randomly distributed fibres was used in the treatment (Kulanthaivel et al. 2022).

The main focus of the current study was to stabilize clay contaminated with petroleum (referred to as PC Clay) using precipitated silica (referred to as P Silica). The purpose of this stabilization is to improve the design and performance of geotechnical structures. Several experiments were conducted to assess the characteristics of the clay specimens, including unconfined compressive strength, free swell index, pH, electrical conductivity, compaction characteristics and plasticity characteristics. The P silica was added in varying percentages of 3%, 6%, 9% and 12%. By comparing the properties of the PC clay specimens with those of the P silica-stabilized clay specimens, the geotechnical properties of the PC clay were evaluated.

#### 2 Materials and Methodology

#### 2.1 Petroleum-Contaminated Clay

The PC clay was excavated from Chemancherry, Chennai, by auger-driven boreholes. Clay was mixed with 2% of commonly produced petroleum to make PC clay. The fuel content was restricted to 2% because of the lubricating qualities of oil, clay's propensity to become liquid and the challenges in testing. The engineering properties of the PC clay are tabulated in Table 1. Based on the index and engineering properties PC clay soil, it was classified as high-compressible clay (CH) as per the Indian Standard.

#### 2.2 Precipitated Silica

P silica, a type of silica product, holds significant commercial value and can be utilized in various ways. It is primarily composed of amorphous silica, which is obtained from sources such as rice husk and quartz. Silica accounts for approximately 89% of its composition. The process of creating P silica involves the formation of a slurry by agitating mineral acid with sodium silicate, which helps regulate the mechanical properties like particle size and porosity. The focus is to achieve a filter cake through the slurry of precipitated silica. The resulting product is then rinsed to remove any salt by-products formed during the precipitation process. Once the salts are eliminated, the silica is dried to achieve the desired water content. To ensure a less dusty form, the dried silica is ground and classified based on particle size distribution. Finally, it can be supplied to commercial markets.

**Table 1** Index and engineering properties of PC clay

Index and engineering properties	PC CLAY
Sand (4.75–0.075 mm), %	0.87
Silt (0.075–0.002 mm), %	10.03
Clay (<0.002 mm), %	89.1
Free swell index, %	73
California bearing ratio (CBR), %	2.5
Unconfined compressive strength (UCS), kPa	275.12
Liquid limit (%)	56
Plastic limit (%)	35
Plasticity index (%)	21
Optimum moisture content (%)	30
Maximum dry density (g/cc)	2.23
Electrical conductivity	0.159
pH	8.81
Soil classification (as Per IS)	СН



#### 2.3 pH and Electrical Conductivity

The pH tests were performed according to IS 2720-26 (1987). The pozzolanic reactions between PC clay and P silica were observed for the soil specimens using pH tests. The pozzolanic reaction is a chemical reaction that take place when the PC clay soil admixed with P silica in presence of water. The amorphous silica in the P silica reacts with PC clay to form calcium silicate hydrate (C–S–H) gel, which was one of the primary binding agents to improve the strength of the soil. The pH metre was calibrated to 4.0, 7.0 and 9.2 as standard reference values for each specimen. The pH values for slurries were noted after 1.5 h. The electrical conductivity tests were also performed similarly to the pH tests.

#### 2.4 Unconfined Compression Test

The unconfined compressive strength test was conducted as per IS 2720 (Part 10) (1973). A cylindrical soil specimen was obtained after the static compaction. The diameter and height of the cylindrical soil specimen were 38 mm and 76 mm, respectively. The prepared soil specimen was under the axial load with constant strain rate of about 1.2 mm/min. The PC clay included the P silica in 3%, 6%, 9% and 12%. The samples were cured and tested after one day. The procedure was performed three times in order to get the accurate results.

#### 2.5 Free Swell Index Test

The free swell index test of the soil specimen was carried out as per IS 2720 (Part 40)—1985. The soil samples were sieved with the help of a 425µ sieve. The measuring cylinders of 100 ml capacity were filled with distilled water and kerosene, respectively. Ten gram of PC clay soil were put it into the measuring cylinders, which has distilled water and kerosene. After 24 h, free swell index value of soil sample was correlated from initial and final volume of soil samples.

#### 2.6 California Bearing Ratio Test

The CBR test was performed according to IS: 2720 (Part 16): 1987. The soil specimens were prepared based on the optimum moisture content and maximum dry density, which was obtained from modified proctor compaction test. The soil specimen was applied with axial load with constant strain rate of about 1.25 mm/min.

#### 2.7 Atterberg's Limits Test

The Atterberg's limit test was conducted as per IS: 2720 (Part 5) 1985. The PC clay was admixed with 0%, 3%, 6%,

9% and 12% P silica, and the liquid limit test was performed with the help of Casagrande apparatus. The plasticity index of the soil was calculated from the numerical difference between the liquid and plastic limits.

#### 2.8 Standard Proctor Compaction Test

The standard proctor compaction test was performed based on IS:2720 (Part VII). The standard proctor compaction test was conducted to obtain the relationship between maximum dry density (MDD) and optimum moisture content (OMC) of PC clay soil admixed with different percentages of P silica.

#### 3 Results and Discussion

# 3.1 Effect of P Silica on pH and Electrical Conductivity of PC Clay Soil

Figure 1 illustrates the changes in pH and electrical conductivity resulting from the addition of P silica into PC clay. As the concentration of P silica increased in the PC clay, the pH of the soil specimen also increased. The pH values recorded for different levels of P silica inclusion 0%, 3%, 6%, 9% and 12% were 8.81, 9.23, 9.54, 9.37 and 9.19, respectively. This tendency of increment was observed due to the silicate component generation in the soil after the treatment with the additive. The contaminated clay was slightly acidic, whereas after adding P silica, soil acidity was removed. The electrical conductivity of the PC clay soil treated with P silica was decreased. The electrical conductivity of PC clay soil admixed with 0%, 3%, 6%, 9% and 12% P silica was observed as 0.159 ms/cm, 0.153 ms/cm, 0.147 ms/cm, 0.141 ms/cm and 0.136 ms/cm, respectively. The electrical

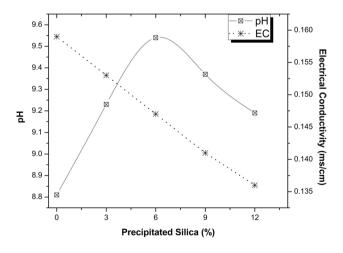


Fig. 1 Effect of P silica on pH and electrical conductivity of PC clay soil



conductivity of the PC clay soil mixed with P silica was decreased. The reason behind that was P silica hindered the ionic movement in the PC clay soil. It improved efficiently when the P silica addition effectively enhanced contaminants' withdrawal in PC clay. The optimum pH of the crude oil-contaminated clay with silica encapsulation was approximately 5, which is lower than the optimum pH attained from this study (Akpoveta 2020). The pH and electrical conductivity of the petroleum incinerated bottom ash treated with nano-silica and silica fume obtained considerably the same results (Nikravan and RamezanianpnourAA 2018). The electrical conductivity reduction significantly agreed with the results from the crude oil-polluted soil (Vincent et al. 2011).

#### 3.2 Effect of P Silica on Unconfined Compressive Strength of PC Clay Soil

The influence of varying dosages of P silica on unconfined compressive strength of PC clay soil is presented in Fig. 2. The percentages of P silica inclusion in PC clay were 0%, 3%, 6%, 9% and 12%. The specimens were treated and tested after the curing period of one day. The UCS of the PC clay soil was increased by inclusion of P silica up to 6%. On further addition of the additive, the UCS of the specimens were decreased. The strength improvement was attained due to the potential of P silica in void filling and the cation exchange mechanism between PC clay and P Silica at an earlier stage, followed by a pozzolanic reaction later. The further addition of P silica beyond 6% was due to the formation of lump-like fragments that were unstable and weak, resulting in the UCS reduction in soil specimens. Hence, the influence of P silica on PC clay exhibited better performance at an optimum percentage of

300 0% PS 270 Unconfined Compressive Strength (kPa) 3% PS 6% PS 240 9% PS 210 180 150 120 90 60 30 Axial Strain (%)

Fig. 2 Effect of P silica on unconfined compressive strength of PC clay soil

6% inclusion. The UCS from the dispersive clay treated with nano-silica exhibited higher results due to the variation like clay and treatment with a different type of admixture (Masrour et al. 2021). Soil stabilized with sodium silicate represented consistent results compared to this study (Gobinath et al. 2020). The UCS obtained for low-plasticity clay treated with nano-calcium carbonate was found to have higher strength since the plasticity of the soil differs (Kannan et al. 2022). The soil stabilized with nano-silica—cement mixes provided the same results as the current study (Kulkarni and Mandal 2022).

### 3.3 Effect of P Silica on Free Swell Index of PC Clay Soil

The variation in FSI concerning the P silica inclusion of 0%, 3%, 6%, 9% and 12% is represented in Fig. 3. The FSI of the soil specimens tended to decrease as P silica concentration in PC clay increased. The FSI values for 0%, 3%, 6%, 9% and 12% addition of P silica were found to be 73%, 56%, 42%, 31% and 23%, respectively. The P silica governed the swelling characteristics of the soil by accumulating in pore spaces, thereby enhancing the infusion of voids. This infusion effectively stiffens the soil matrix, which resists the swelling characteristics of the soil specimen. The FSI of the treated specimens reduced significantly with the inclusion of P silica compared to the untreated soil specimens. The FSI was efficiently reduced with P silica in PC clay compared to the reduction of about 56% by lime sludge and cement (Kulkarni and Mandal 2022).

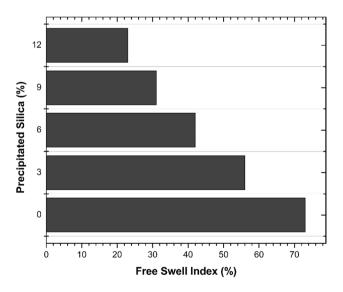
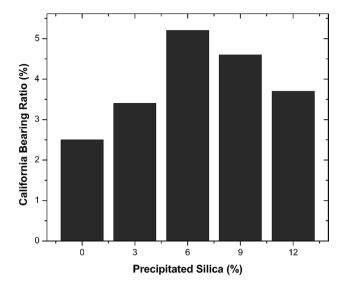


Fig. 3 Effect of P silica on free swell index of PC clay soil







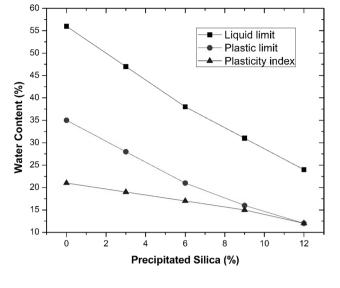


Fig. 5 Effect of P silica on plasticity characteristics of PC clay soil

# 3.4 Effect of P Silica on California Bearing Ratio of PC Clay Soil

The variation in CBR values concerning the inclusion of P silica is shown in Fig. 4. The CBR values of PC clay increased with an increase in P silica content in the PC clay. The percentage inclusion of P silica was found to be 3%, 6%, 9% and 12%. The CBR values were found to be increased till the addition of 6% inclusion of P silica. However, further addition of P silica decreased the CBR values. The CBR values increased from 2.5 to 5.2 when 6% P silica was included in PC clay.

Further inclusion of P silica decreased the CBR values to 3.7%, representing an optimum value of 6%. The increment in CBR represents an improvement and the stiffness of the soil matrix. This is due to the pozzolanic reaction after the P silica was included in PC clay. The P silica exhibited gapping of pores, and hence the strength increased significantly. A better improvement of CBR was observed at 6% inclusion of P silica in PC clay. Adding silicate soil provided slightly higher results than P silica due to the variance in the type of soil and its application (Marik et al., 2022). This study's reduction percentage in CBR values was 32.4%, significantly higher than the 27.28% obtained from contaminated soil treated with sodium silicate (Adlin Rose et al. 2021).

# 3.5 Effect of P Silica on Plasticity Characteristics of PC Clay Soil

The variation of plasticity characteristics, such as liquid and plastic limits concerning the inclusion of P silica, is elucidated in Fig. 5. The P silica was added in 0%, 3%, 6%, 9% and 12%. The liquid and plastic limits of the PC clay

decreased with the addition of P silica, thereby decreasing the plasticity index of the clay. Therefore, it can be observed that the increase in the P silica concentration in PC clay provided a greater influence on the expansive characteristics of clay. This phenomenon was due to the chemical reactions between P silica and PC clay resulting in the specimens being more brittle than the untreated soil. The plasticity characteristics of the soil altered because of the flocculation, cation exchange and agglomeration mechanisms involved after additive inclusion. The values were observed after a contamination period of 14 days. The 6% inclusion of P silica significantly altered the cohesive behaviour and nature of the clay soil. The plasticity characteristics of the clay were reduced after the 6% inclusion of clay soil. This tendency was observed as the concentration of cations with higher valence increased.

Further addition of P silica beyond 6% increased as the mixtures turned non-homogeneous. Hence, the P silica inclusion in soil decreases the swelling characteristics of PC clay. This tendency made the 6% inclusion of P silica be optimum value and highly efficient. The soil stabilization with sodium silicate was found to be varied slightly with the results obtained from the present study due to the type of admixture utilized (Gobinath et al. 2020).

# 3.6 Effect of P Silica on Compaction Characteristics of PC Clay Soil

The variation of compaction characteristics, such as optimum moisture content (OMC) and maximum dry density (MDD) concerning the inclusion of P silica, is elucidated in Fig. 6. The OMC and MDD values of the clay soil decreased initially and were found to increase after the



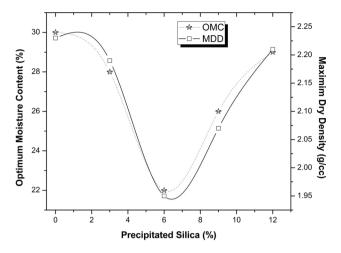
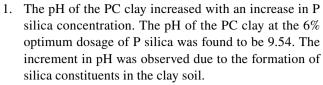


Fig. 6 Effect of P silica on compaction characteristics of PC clay soil

6% addition of the P silica. The OMC values obtained for 0%, 3%, 6%, 9% and 12% inclusion of P silica were observed to be 30%, 28%, 22%, 26% and 29%, respectively, whereas the MDD values were 2.23 g/cc, 2.19 g/ cc, 1.95 g/cc, 2.07 g/cc and 2.21 g/cc, respectively. The geometric arrangement of particles in PC clay was highly scattered at low percentage inclusion of P silica. However, MDD was found to be decreased after the addition of P silica in PC clay as the scattering characteristics increased. The reduction in MDD was due to the cation exchange process, eventually leading to the flocculation mechanism. At 12% inclusion of P silica, the characteristics of the PC clay were found to be unaltered. This occurred due to strength decrement in inter-particle interaction or bonding. The PC clay exhibited significant dry density and moisture content results at 6% inclusion of P silica. The results were found to be effective in comparison with the uncontaminated clay since the index properties of the PC clay were improved. The MDD reduction obtained from this study was considerably higher than the reduction obtained from dispersive clays treated with nano-silica (Masrour et al. 2021). The results were consistent with the values obtained from soil stabilized with sodium silicate (Gobinath et al. 2020).

#### 4 Conclusion

The research focuses on utilizing precipitated silica on petrol-contaminated clay and the stabilization phenomenon. The effective usage of P silica can significantly enhance the geotechnical properties of the PC clay. The experimental study on PC clay treated with P silica reveals the following conclusions.



- The electrical conductivity of the PC clay considerably decreased as the inclusion of P silica increased. The reduction of electrical conductivity was due to removing contaminants from the PC clay by adding an additive.
- 3. The UCS of the PC clay increased significantly with the inclusion of P silica. The optimum strength was achieved at a 6% addition of P silica and was observed to be 275.12 kPa. The strength increased concerning the cation exchange mechanism and soil's void-filling ability after the P silica treatment.
- 4. The FSI of the clay soil decreased when P silica was added. The FSI at 6% inclusion of P silica was 42%. The reduction was observed because of P silica's effective accumulation of pore spaces in PC clay.
- 5. The CBR of the PC clay improved with an increase in the P silica inclusion. The optimum value obtained at 6% inclusion of P silica was 5.2%. The increment in values was due to the pozzolanic reaction between the PC clay and P silica particles.
- 6. The liquid limit and plastic limit of the PC clay decreased with the increase in P silica concentration, eventually decreasing the soil's plasticity index. The reduction of plasticity characteristics was found owing to the mechanisms such as flocculation, cation exchange and aggregation.
- 7. The OMC and MDD decreased after including P silica in PC clay. The OMC and MDD attained the optimum of 6%, 22% and 1.95 g/cc, respectively. This tendency of change in compaction characteristics was observed because of the flocculation process in PC clay with P silica.

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**Data availability** The data used to support the findings of this study are available from the corresponding author upon request.

#### **Declarations**

**Conflict of interest** The authors declare no potential conflicts of interest concerning this article's research, authorship and publication.

**Ethical Approval** This material is the author's original work, which has not been previously published elsewhere. The paper is not cur-



rently being considered for publication elsewhere. The paper reflects the author's research and analysis truthfully and completely.

Research Involving Human Participants and Animals The authors at this moment assure that this research does not involve any human participants or animals.

**Informed Consent** All the authors read and understood the provided information and had the opportunity to ask questions. We understand that our participation is voluntary and free to withdraw at any time without giving a reason or cost. All the authors understand the study involves research, purposes of the research, their subject's participation, procedures to be followed and identification of any experimental procedures.

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