

Sustainable Clay Tile Production Using Partial Replacement of Precipitated Silica

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Abstract - The usage of ceramic tiles as an efficient construction material for flooring and roofing has increased with the advancements in the manufacturing processes. Ceramic tiles are traditionally made of plastic and non-plastic clay. An attempt was made to study the performance of the clay tiles which the replacement of Precipitated Silica (PS) at 2.5, 5, 7.5 and 10 percentages. It was proposed to optimize the production of tiles with an objective of sustainable development. PS is derived from quartz sand in the form of amorphous silicon dioxide. It is also a product of rice husk ash. So Precipitated Silica was used in the unglazed tile manufacturing process and the partially replaced tile specimens were investigated for its water absorption, shrinkage and strength characteristics. The mixing of raw materials was done in a ball mill and the pressing of the tiles was performed in a hydraulic press. The specimens were fired at temperature ranging from 200°C to 1500°C in the furnace. Shrinkage test, water absorption test and flexural strength test were carried out and the performance of 2.5% Precipitated Silica replaced tiles was found to be similar to that of the conventional tiles. The studies also proved that the ceramic tiles performed better than PS replaced tiles at higher proportions due to the lack of densification and porous nature of PS.

Keywords: *Precipitated Silica; Tiles; Flexural strength; Water absorption; Shrinkage;*

INTRODUCTION

Tiles are thin flat slabs or blocks usually made up of burned clay, glazed or unglazed used either structural aspects or decorative aspects of buildings. It is a manufactured piece of hard-wearing material installed in roofs, floors and walls. The introduction of tunnel kiln around the 19th century increased the automation of tile manufacture. The manufacture of tiles does not require heavy and harmful chemicals and also there is no negative impact on the environment for obtaining the raw materials. They are made from naturally available raw materials like clay and can also adopt the usage of recycled materials. Clay has a very low permeability as its particles are very closely packed so that it doesn't allow water to seep through it, making it the right choice for tile making. But the over usage of clay observed in the tile making process all over the world calls for engineering solutions.

Silva, et al, (2017) studied the effect of Rice Husk Ash [RHA] on mixing with clay roof tiles to know its structural and thermal properties. The physical and chemical properties of RHA and clay were also determined. With a replacement of 10% RHA, the clay tile showed an increase in strength of about 45.97%. It is due to the high amount of silica present in the RHA that contributes to the strength. Higher amounts of RHA mixing are also not desirable. Water absorption of the tile increased with RHA content. **Gobinath, et al, (2016)** studied the engineering characteristics of black cotton soil stabilized with precipitated silica. There was an increase in the California Bearing Ratio [CBR] value along with the coefficient of permeability (in addition with 50% of PS). As the Black Cotton [BC] soil was found to be platy in its microstructure, which on addition and mixing with PS which was found to deposit in between the platy structure, thus reducing the friction in between them leading to limited increase in strength. **El Maghraby et al, (2011)**, studied the granite as flux in stoneware tile manufacturing and have confirmed that the viability of usage of natural granite in a ceramic preparation. The laboratory samples showed the bending strength similar to commercial ceramic tile. **Music, et al, (2011)** made experimental studies on the precipitation of silica particles and their properties. Field Emission Scanning Electron Microscopy [FE-SEM] inspection showed primary silica particles were of 15-30nm in size. **Subbukrishna, et al, (2007)** performed studies on the production of precipitated silica with rice husk using Indian Institute of Science Precipitated Silica Technology [IPSIT] process by varying from 20% to 25% of rice husk and the major constituent being silica. **Luo, et al, (2014)** made experimental studies on the use of Waste Foundry Sand [WFS] as a replacement for clay in tile manufacture. Up to a

replacement of 15%, the bending strength increased as the surface particles bonded with each other and the pore sizes were small. In the interior structure there was an improved adhesion interaction due to the formation of necks. **Jordán, et al, (2005)** studied the usage of sewage sludge in ceramic tile manufacture. It was evident that it would be preferable, if the sludge is used for ceramic tiles manufacture rather than in agriculture. **Sombatsompop et al, (2004)** studied the usage of fly ash particles and precipitated silica as fillers in natural rubber and Styrene-Butadiene rubber. It was analysed that the aggregation of silica substantially barred the rubber from being readily vulcanized.

From the literatures, it can be concluded that the modification of commercially available tile by using other sustainable materials has always been an area of interest. It was also found that using precipitated silica could lead to sustainable development and reutilization of wastes since precipitated silica is made from rice husk ash. Hence an attempt was made to produce eco-friendly tiles replaced with PS.

Materials

Precipitated Silica

Precipitated silica is derived from quartz sand which is in the form of amorphous silicon dioxide. Sodium silicate is formed by the reaction of silica and caustic soda. The solution is then sieved to excerpt the residual ash. The next step is precipitation of silica from the sodium silicate solution by passing carbon di-oxide at specific flow rate conditions along with continuous agitation throughout the process during which silica precipitates out. The remaining soluble salts will be washed out. Sodium carbonate and calcium reacts and forms calcium carbonate and in turn those are converted into sodium hydroxide when it reacts with calcium hydroxide. Filtration is followed to remove any solid calcium carbonate leftovers. Precipitated silica has a good binding nature, which is supposed to increase the adhesion between the particles in which it is used resulting in strength increase.

Terracotta clay

Terracotta is derived from the Latin word *terra cocta* which means baked earth. Terracotta is the term normally used to refer sculptures that are made as earthenware. It is also used for manufacture of vessels, waste water pipes, roofing tiles, bricks, building components etc.

Methodology

The methodology of the making of tiles is compiled in Figure 1.

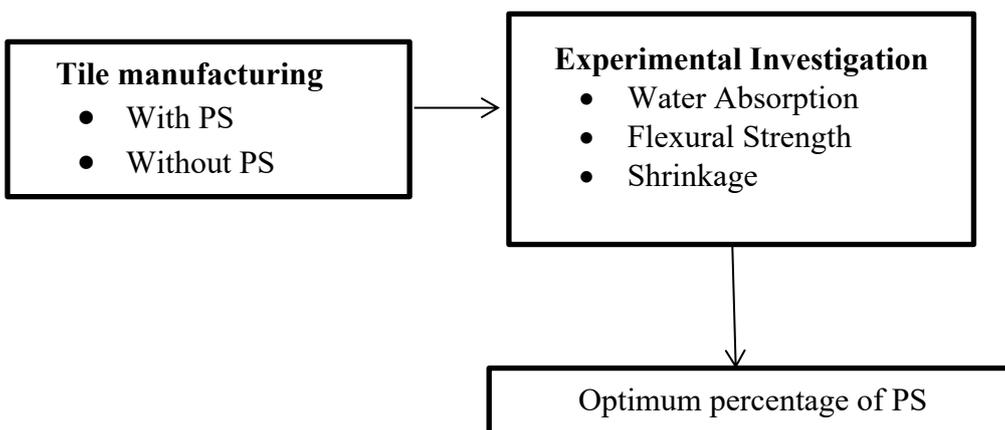


Fig 1 Methodology of the Study

Tile Manufacturing

The manufacturing process of tiles involved the following steps.

- Step 1: Raw material collection
- Step 2: Quality monitoring
- Step 3: Raw material Grinding
- Step 4: Storage of raw material

- Step 5: Moulding and Pressing
- Step 6: Drying of green tiles
- Step 7: Burning of green tiles
- Step 8: Packing and Dispatch

The raw material used usually for the manufacturing of tiles was argillaceous terracotta clay. As the study involved partial replacement of clay with PS, powder of precipitated silica was added at the proportions of 2.5, 5, 7.5 and 10 percentages. The limits of the proportion were based on the optimum percentage obtained from the previous studies on clay with PS by Gobinath, et al, (2016). His studies revealed that 50% replacement of tile with PS provide better results. Hence setting the upper limit as 50% replacement of tile with PS, the proportions were incremented in 2.5 percentages. As the tile showed steady decline of strength with PS increments beyond 2.5%, the study has been limited to 10%. The material stored under sunlight was taken for quality testing in the R&D lab and were tested for its plasticity. After the quality check, the clay was transported to the ball mills for grinding. The coarse materials from the ball mills were segregated using sieves of 90 μ . The fine materials after jolting were stored in various silos, with addition of moisture. Then the clay from conveyor belts were poured in moulds of size 8.75 x 5cm. For pressing, a pressure of 100-150 kg/cm² was applied. In order to remove the water content, were dried for 40 minutes at a temperature of 168°C to produce the green tiles as shown in Figure 2. Finally, the green tiles were burned in kilns to maximum temperature of 1500°C. Then the manufacture tiles were perfectly sculpted, marked, packaged and despatched.



Fig 2 Green Tiles with and without PS

Experimental Investigation

Water absorption characteristics

The water absorption test was used to determine the sorptivity of water by the terracotta powder by measuring the increase in mass of a specimen after immersion in water. Initially, the dry weight of the tiles was noted. Two tiles were reserved in each proportion of precipitated silica such as 0, 2.5%, 5%, 7.5% and 10% for conducting water absorption test as per Indian Standards. The tiles were immersed in hot water for two hours and then immersed in cold water for about 30 minutes. The immersed tiles were immediately wiped with a dry cloth. The final weight of the tiles was noted. Water absorption can be computed by subtracting the final weight with initial weight and then dividing by initial weight. Water absorption is expressed in percentage. The results are compiled in Table 1.

Table 1 Change in water absorption

| Replacement % - Tile Number | Initial weight (g) | Final weight (g) | Water absorption (%) | Average (%) |
|--------------------------------|--------------------------|------------------------|----------------------------|----------------|
| 0% -1 | 71.99 | 74.6 | 3.62 | 3.745 |
| 0% -2 | 71.76 | 74.54 | 3.87 | |
| 2.5% -1 | 71.84 | 74.82 | 4.14 | 4.475 |
| 2.5% -2 | 71.58 | 75.02 | 4.81 | |
| 5% -1 | 71.86 | 77.21 | 7.43 | 7.525 |
| 5% -2 | 71.58 | 77.03 | 7.62 | |
| 7.5% -1 | 72.02 | 78.24 | 8.60 | 8.55 |

| | | | | |
|---------|-------|-------|-------|--------|
| 7.5% -2 | 71.99 | 78.12 | 8.51 | 10.735 |
| 10% -1 | 71.67 | 79.32 | 10.67 | |
| 10% -2 | 71.56 | 79.29 | 10.80 | |

Shrinkage Characteristics

In order to study the dilatometric behaviour of the tiles, shrinkage test was conducted. Lines of suitable lengths were drawn on tiles and were measured before and after to find the shrinkage. Two tiles were taken in each proportion of precipitated silica such as 0, 2.5%, 5%, 7.5% and 10% and are heated in the kiln at 350-1200°C for 48 minutes to study its shrinkage characteristics. Tile shrinkage has no units as it is defined as the ratio of the lengths of the specimen before and after firing. All the tiles with and without PS initially measured a length of 9.5 cm before firing. From Table 2 the variations of shrinkage ratios with respect to PS increment can be inferred.

Table 2 Shrinkage Characteristics

| Replacement % | Size of the specimen (cm) | Shrinkage |
|---------------|---------------------------|-----------|
| 0% | 9.1 | 1.04 |
| 2.5% | 8.9 | 1.06 |
| 5% | 8.75 | 1.08 |
| 7.5% | 8.6 | 1.10 |
| 10% | 8.5 | 1.11 |

Strength characteristics

The flexural strength of fired samples was measured by 3-point loading method. For clay roofing tiles, the flexure strength of the tiles was tested using a double lever loading machine. The tiles were straddled between rollers of 12 mm diameter as per the IS requirement. After mounting the tiles, it was brought in contact to the loading frame. The terminal bucket of lever arm assembly was opened and the nickel balls weight acting as the load on the tiles was poured into the bucket. The load was applied using the lead balls which automatically stopped when the tiles broke. The accuracy of load indication is within $\pm 2\%$. The apparatus is designed in such a way that, when the tiles break, the lead balls that pours automatically into the buckets gets stopped. After failure of tiles, the total weight of the bucket with lead balls were calculated and it was converted into load. The flexural strength was calculated by using standard formula as per IS.

From Table 3 the variations of flexure strength characteristics can be observed.

Table 3 Strength Characteristics

| Replacement % | Length (cm) | Breadth (cm) | Thickness (cm) | Force acting on the tile (N) | Flexural Strength (N/cm ²) |
|---------------|-------------|--------------|----------------|------------------------------|--|
| 0% | 9.1 | 4.5 | 0.7 | 92.4 | 572.1 |
| 2.5% | 8.9 | 4.5 | 0.8 | 74.2 | 343.9 |
| 5% | 8.75 | 4.4 | 0.9 | 62.2 | 240.1 |
| 7.5% | 8.6 | 4.3 | 1 | 49 | 147.2 |
| 10% | 8.5 | 4.2 | 1 | 37.5 | 113.8 |

RESULTS AND DISCUSSION

Water absorption characteristics

It can be observed from Table 1, that as the proportion of precipitated silica increases, the water absorption increases gradually. Initially, upon 2.5% addition of precipitated silica, there is a 0.73 percent rise in water absorption. With 5% addition of precipitated silica, there is a steep

increment of water absorption by 3.05%. At 7.5% and 10% replacement of precipitated silica, the water absorption increment patterns are flat and steeper similar to that of 2.5% and 5% respectively as shown in Figure 3. However, there is a tremendous increment of 6.99% of water absorption upon 10% addition of precipitated silica. This may be due to the rice husk ash which has higher water content. So, increase in proportion of precipitated silica gradually increases the water content in the tiles.

As per the Indian standards, the water absorption values for a normal roof tile should be of the range 3-6%. Beyond 6% water absorption, the tiles are categorised as unfit for construction purposes. From the above results, it was evident that the tiles without precipitated silica and 2.5% replaced silica tiles can be used for construction purposes. The increment in water absorption upon addition of precipitated silica may be attributed to the porosity and high-water absorbing nature of precipitated silica. However, this can be further studied by observing its strength characteristics. The water absorption characteristics firmly depends on the micro structure and porosity. As the porosity increases with PS increment due to the attenuation of densifying process. Lack of densification led to the porosity increment. This performance was observed to be contradictory to the performance of tiles with granite as reported by El Maghraby et.al, (2011). Also, the platy structure of the clay was disturbed due to PS addition which contribute to the increment of inter-connected spaces. This may be the reason behind the water absorption increment with PS as stated by Gobinath et.al,(2016). The suitable percentage of silica replacement was found to be 2.5% which showed suitable water absorption percentage within the range of 3 – 6%. This makes the tiles with 2.5% suitable for construction purposes as per IS 2690-1.

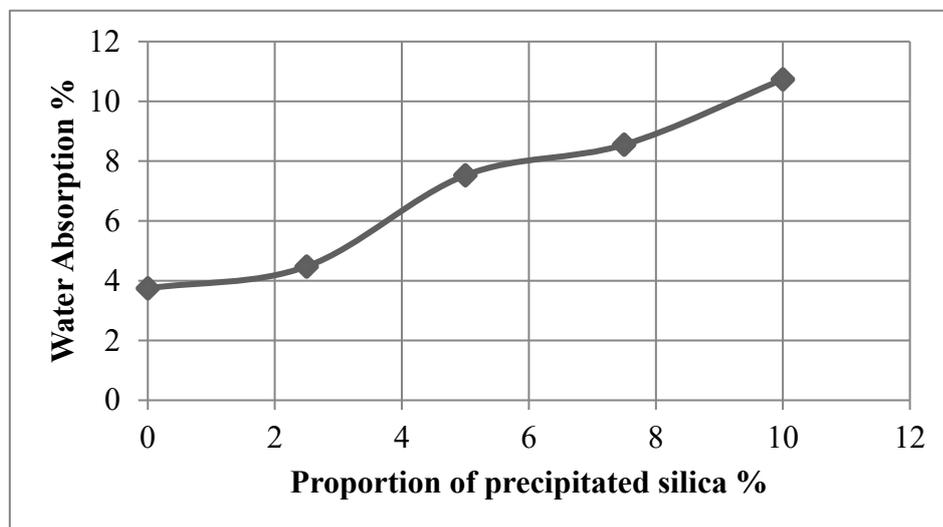


Figure 3 Variation of water absorption with proportions of PS

Shrinkage Characteristics

From Table 2, it can be observed that the tiles shrink with PS increment. The shrinkage in the tiles may be due to the destruction of diffused double layer from the clay particles upon heating. More the shrinkage of tiles more is its susceptibility to volumetric changes. This also shows that as shrinkage in the tiles increases with proportion of precipitated silica, the swell property in the tiles decreases as shrinkage is inversely proportional with the swell parameters.

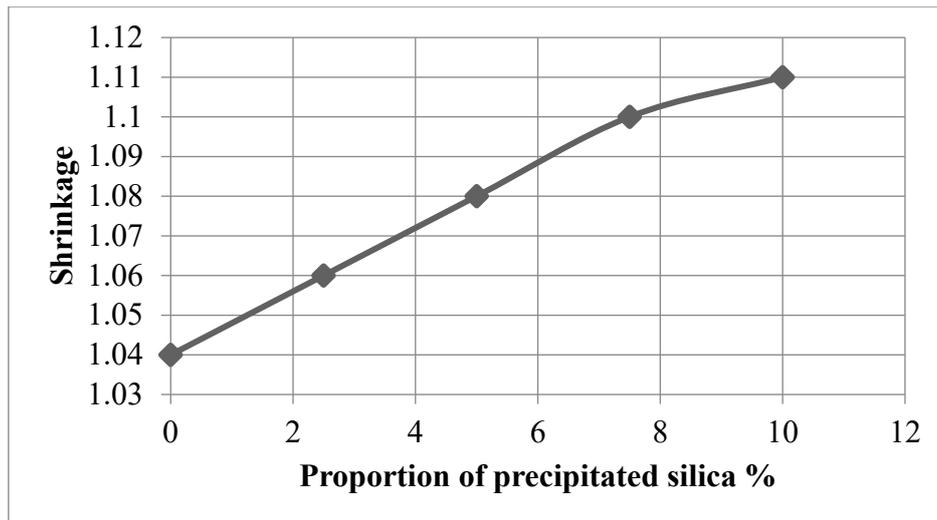


Figure 4 Variation of shrinkage with proportions of PS

While comparing the variation of shrinkage with PS as shown in Figure 4, with the work of El Maghrabyet.al., on tiles with granite which suggests that the fluxing nature of granite induces the shrinkage in tiles. Though the shrinkage of tiles increases with precipitated silica in this study, the lack of adhesion of precipitated silica were observed in water absorption and strength tests.

Strength characteristics

The clay tiles proved to be good in its flexural strength than the PS replaced tiles. The flexural strength of the tiles was gradually decreasing with increase in its addition of precipitated silica as shown in Figure 5. The conventional clay tiles had strength of 572.1 N/cm², while the 10% replaced tiles had a flexural strength of 113.8 N/cm².

Gobinath et al, observed that the Unconfined Compressive Strength [UCS] of the clay decreased with precipitated silica with a negligible increment initially. The negligible increment of strength was attributed to the frictional resistance between the plates of the clay and PS. But on overall observation, the strength decreased with PS. Similarly, the terracotta clay tiles exhibited decrement of flexural strength with increment of PS. It can be concluded that the frictional resistance between the clay plates and precipitated silica was very negligible.

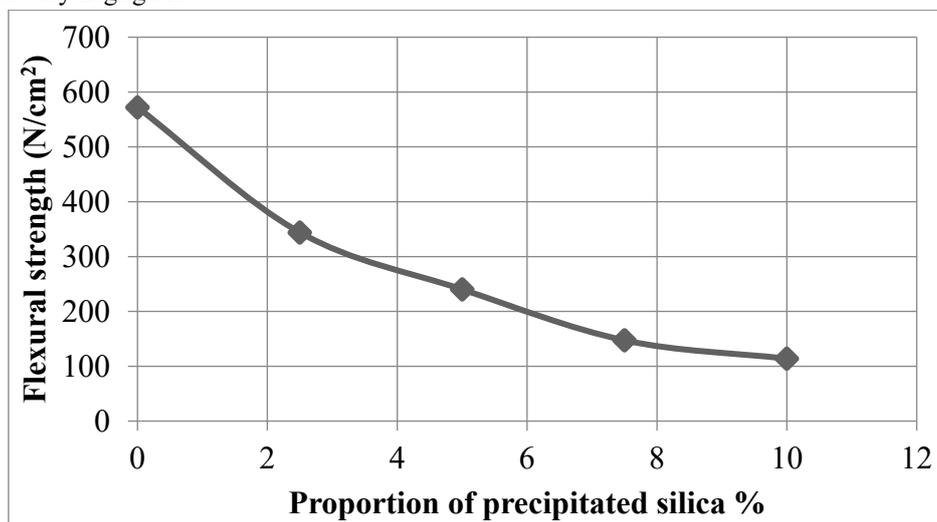


Figure 5 Variation of flexural strength with proportions of PS

CONCLUSION

Observations were made on the performance of precipitated silica replaced ceramic tiles and the following conclusions was made.

1. The water absorption of the tiles increased with the increment of precipitated silica content. This was due to the lack of densification of micro structure, increased porosity and distortion of platy structure by PS.

2. The shrinkage in the tiles increased with proportion of precipitated silica. The shrinkage in the tiles may be due to the destruction of diffused double layer, minimal flux nature and lack of adhesion.
3. The flexural strength of the tiles gradually decreased with increase in its addition of precipitated silica. The conventional clay tiles had strength of 572.1 N/cm², while the 10% replaced tiles had a flexural strength of 113.8 N/cm². The reason for the decrement in the strength may be due to less frictional resistance between the clay plates and precipitated silica.
4. The ceramic tiles performed better than PS replaced tiles at higher proportions due to the lack of densification and porous nature of PS. However, 2.5% of PS addition can perform similar to that of ceramic tiles.
5. Three important properties of tile validation for construction purposes such as Water Absorption, Flexural Strength and Shrinkage was evaluated through experimental studies. Furthermore, the studies can be expanded further for microscopic evaluation.
6. The usage of PS as a sustainable stabilizer in tile was analysed and found that at 2.5% of PS addition the objective of the study can be achieved.

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