EXPERIMENTAL INVESTIGATION ON CEMENT MORTAR BLENDED WITH NANO SILICA SYNTHESIZED FROM RICE HUSK ASH

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Abstract—The residences of mortar in which substitute of cement cement is executed withNanosilica is proposed to be investigated. Cement is to be partly replaced via nanosilica of 1.5%, 3% and 4.5% respectively.. Rice husk ash, an agriculture waste is used to provide nanosilica. while rice husk ash is burnt in a muffle furnace at 650° C for four hours amorphous silica is received. Pure silica is extracted by titration technique the usage of 5N, H₂SO₄ solution with constant stirring temperature by refluxing method 98% of pure nanosilica may be extracted from pure silica. A literature survey on beyond studies become accomplished and the results display that the compressive and split tensile power at seventh day and 28th day of cement mortar blended with the nano particles (i.e., nanosilica), had been better than that of undeniable cement mortar and partial substitute the usage of nanosilica improves the mechanical and structural behaviour of mortar however lower the putting time. The water permeability check showed that the nano - SiO₂ concrete has higher water permeability resistant behaviour that the normal concrete. The electricity carried out by way of the usage of nanosilica is extra as compared to that of regular portland cement. There may be a top notch boom inside the early energy. SEM results show the debris are in agglomeration form and measurement is 80mm. XRD power pattern of nanosilica confirms the amorphous nature of the substance. An FTIR spectrum provides proof of indicating the presence of nanosilica.

I. INTRODUCTION

In recent years, the use of nano-particles has fabricate materials with new functionalities has gotten a lot of attention in a lot of fields. When ultra-fine particles are mixed into portland-cement paste, mortar or concrete, materials with characteristics that differ from traditional materials are produced. The performance of these cement-based materials is strongly influenced by nano-sized solid particles, such as calcium silicate hydrates(CSH) particles, or nano-sized porosity at the cement-aggregate interfacial transition region.

Weight , resilience, shrinkage, and steel-bond are some of the properties that nano sized particles or voids can affect. SiO_2 nanoparticles (nS) can serve as a nano-filler by filling the gaps between CCSH gel particles. Futhermore, the amount of CSH increase as a result of the pozzolanic reaction with calcium hydroxide , resulting in a higher densification of the matrix, which enhances the strength and durability of the material.

Previous researchhas shown that the addition of nanoparticles alters the properties of the fresh and hardened states, even as opposed to traditional mineral addictions. Amorphous silica colloidal particles tend to have a major effect of the hydration C_3S .

1.1 BENEFITS OF NANOSILICA

As compared to silica fume (SF), nano-silica reduced mortar setting time and reduced bleeding water and segregation while enhancing the cohesiveness of the mixture in the fresh state. When used in combination with ultra-fine ash, it ensures better efficiency than silica fume alone.

Furthermore , as opposed to formulations without silica fume, the compressive strength of mortar or concrete with silica fume improves. Some authors argue that required percentage of nS should be low (1-5 wt%) due to agglomeration caused by difficulties in disprsing the particles during mixing , while they argue that higher dosag can boost properties as well, of about 10%, if the formulation is adjusted properly to avoid excessive self- desiccation and microcracking , which could weaken the power.

The effects of nano-sized amorphous silica on the rheological properties, setting time air quality, and apparent density of cement-based mortars are discussed in this project.

Cement paste heat of hydration and phase determination were also calculated.

II. OBJECTIVE

The following goals were set in order to investigate the impact of partial cement replacement with nanosilica on the mechanical and physical properties of cement mortar.

- SEM and XRD were used to classify Nanosilica and ordinary portland cement in order to study their chemical composition and physical properties.
- > The aim of this research was to see how partial replacement of cement with nanosilica affected the consistency, initial and final setting times.
- ➤ To prepare , perform compressive strength tests on cement mortar cubes with nanosilica (1.5%, 3% and 4.5%) and compare the results to assess the effect of nanosilica.

III. METHODOLOGY

The aim of this project is to create nanosilica from rice husk ash and investigate the mechanical and physical properties of nanosilica-based cement mortar.

- Nanosilica is made from an extract of rice husk ash , which is an agriculture waste. Amorphous silica is generated by burning rice husk ash at 650° C for 4 hours in a muffle furnace.
- > The extraction of pure silica is achieved using a titration process with a $5N H_2SO_4$ solution and continuous stirring at a controlled temperature .
- > 98% pure nanosilica can be seperated from pure silica using the refluxing process.
- > Nanosilica of 1.5%, 3.0% and 4.5% is used to partly substitute cement in mortar cubes.
- > The properties of nanosilica mortar have been determined and compared to OPC mortar.

IV. EXPERIMENTAL PROGRAMS

4.1 Materials

Cement- Ordinary Portland cement (OPC) of grade 43 was used ,which met indian standard IS 8112-1989.

Sand- Screened river sand passed through 1.18mm sieve with fineness modulus of 2.85 and specific gravity of 2.55 (as per IS 383 -1970) was used.

Nanosilica- Using titration and refluxing processes, nano silica was synthesised from rice husk ash. SEM micrographs are taken with a SEM model, and XRD analysis is done with an X-rays diffractometer.

This project included the following experiments to assess physical and mechanical properties

4.2 Tests carried out

a) Physical Properties

- XRD (X-ray diffraction) -To determine the crystalline properties
- SEM (Scanning Electron Microscopy) -To determine particle size and surface morphology
- Check for accuracy, Initial and final setting times, and specific gravity test.

b) Mechanical Properties

• Compressive strength test

4.3 Synthesis of nanosilica

80~ml of 2.5 N sodium hydroxide solution prepared with triple distilled water is stirred with 10g of treated RHA sample. To dissolve the silica (RHA) and obtain a sodium silicate solution, the solution is heated at 100° C in a sealed 250 ml beaker for 4 hours with constant stirring. In a silica crucible , the solution is extracted , and the excess is washed with warm distilled water. The sodium silicate solution obtained is viscous , clean , and colourless , and it is allowed to cool to room temperature . The reaction is

$$(Ash)$$
 (Caustic soda) $(Sodium silicate)$ (Water) (Ash)

The titration process , which uses a 5N $\rm H_2SO_4$ solution and constant stirring under controlled conditions, can be used to extract pure silica. With normal air pressure, the temperature is in the range of 90°C-100^O C . The acidic environment pH2 means that the reaction has almost fully precipitated silica from sodium silicate.

$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O ---(2)$$

(Silica) (Sodium sulphate)

Sulphate impurities are removed by washing the gel - formed silica with warm distilled water. The amorphous silica is paved when the commodity is baked at 70° C for 15 hours. Nanosilica is derived from pure silica using the refluxing process. 6 hours of continuous refluxing at 80° C- 90° C with pure silica and 6NHCl solution after that , the sample is thoroughly cleaned with warm water to remove any alkali.

After that , 80 ml of 2.5N NaOH is applied to the silica powder and stirred continuously for 10 hours with a magnetic stirrer. The solution is then treated with conc. H_2SO_4 until a white precipitate forms. The precipitate is washed with warm distilled water several times until the filtrate is alkali-free. The precipitated nanosilica is dried for 30 hours in a hot air oven. A flow chart of the process is shown in fig 1.

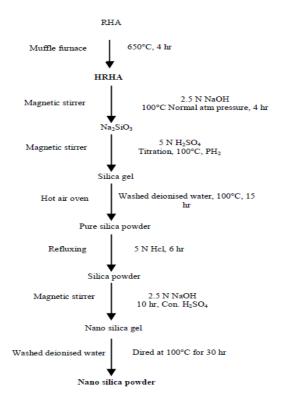


Figure 1: Flow diagram of Nanosilica synthesis.



Fig.2 EXPERIMENTAL SETUP FOR SYNTHESIZES PROCESS



Fig 4 NAOH SOLUTION Fig 5 MIXING OF RICE

Fig 6 RICE HUSK ASH (TOP) NANOSILICA (BOTTOM)

4.4 SEM (Scanning Electron Microscope)

The scanning electron microscope (SEM) is a type of <u>electron microscope</u> that uses a high-energy beam of electrons to scan the sample surface in a raster scan pattern to image it. The electrons communicate with the atoms in the sample to produce signals that include details about the sample's surface topography, composition ,and other properties including conductivity. The morphologies of three specimens of nanocement and ordinary cement were determined using SEM.

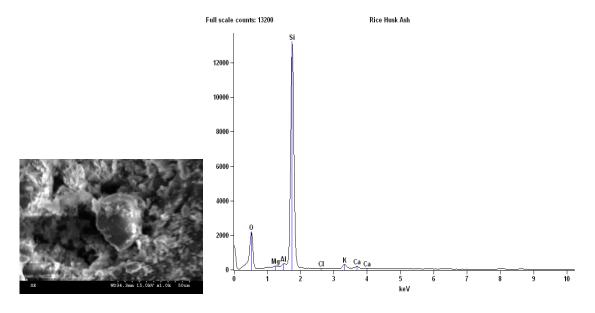


Fig7 SEM IMAGE OF RICE HUSK ASHSEM -EDAX TEST

Element	Net Counts	Weight %	Atom %
0	20716	49.54	63.57
Mg	693	0.27	0.23
Al	1087	0.38	0.29
Si	151493	47.39	34.64
Cl	271	0.14	0.08
K	2933	1.45	0.76
Ca	1567	0.83	0.43
		100.00	100.00

Table no.1 Quantitative Results of Rice Husk Ash

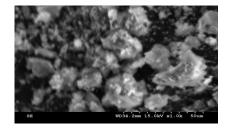


Fig 8 SEM IMAGE OF NANOSILICA

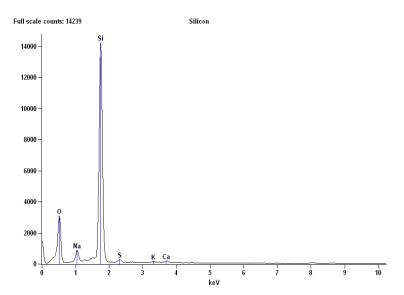


Table no.2 Quantitative Results of Nanosilica

Element	Net Counts	Weight %	Atom %
0	28829	50.37	63.69
Na	7630	5.17	4.55
Si	162928	42.56	30.66
S	2531	1.08	0.68
K	789	0.31	0.16
Ca	1186	0.50	0.25
Total		100.00	100.00

4.5 XRD (X-ray diffraction)

X-ray diffraction uses X-rays to determine the geometry or form of a molecule. The elastic scattering of X-rays from structures with long range order is the basis for X-ray diffraction techniques. Diffraction is the most detailed explanation of scattering from crystals. Using the X-ray diffraction method, all particle sizes of nanocement and ordinary cement are determined.

4.6 Laser scattering analyzer

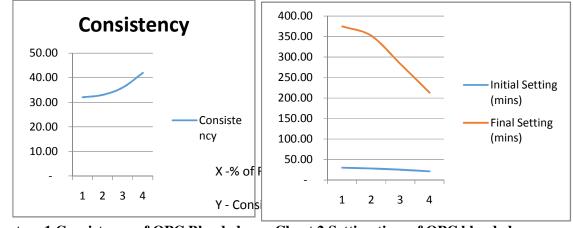
The most commonly used method for particle size analysis is laser diffraction. A representative cloud or ensemble of particles passes through a broadened beam of laser light in laser diffraction particle size analysis, scattering the incident light onto a Fourier lens. The scattered light is focused onto a detector array, and a particle size distribution is estimated from the collected diffracted light data using an inversion algorithm. The particle sizes of nanocement were calculated using a laser scattering analyzer.

4.7 CONSISTENCY AND SETTING TIME OF CEMENT (BLENDED WITH NANOSILICA)

OPC's norm quality value was discovered to be 32 percent . in the case of nanosilica, the standard consistency increased from 32% to 42%. the addition of nanosilica to cement resulted in a significant increase in surface area and natural consistency.

NAME OF TEST	OPC	OPC With 1.5% nanosilica	OPC With 3.0% nanosilica	OPC With 4.5% nanosilica
Normal consistency	32%	33%	36%	42%
Intial setting time	30 min	28 min	25 min	21 min
Final setting time	375 min	352 min	283 min	214 min

Table.3 Consistency and setting time of OPC Blended with nanosilica



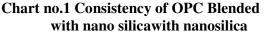


Chart 2 Setting time of OPC blended

4.8 DENSITY OF MARTOR CUBE

SL.NO	PARTICULARS	DENSITY
		(kg/ m3)
1	OPC	2188
2	NS 1.5%	2245
3	NS 3.0%	2260
4	NS 4.5%	2450



Fig.9 MIXING OF NANOSILICA

Fig.10 .MORTAR CUBE

A. 4.9CUBE COMPRESSIVE STRENGTH

After 7 days of curing , the results of cube compressive strength of OPC and cement partially substituted with nanosilica are tabulated . OPC had a cube compressive power of 14.88 MPa after 7 days of curing. The cube compressive strength gradually increased when the cement was replaced with nano silica (1.5% to 4.5%) . the compressive strength of all blended cement mortar specimens with OPC

During the hydration of OPC, nanosilica reacts with water, liberating free calcium hydroxide and forming calcium hydro silicate gel compound.

 $2(3CaO.SiO_2) + 6H_2O \longrightarrow 3 CaO.2SiO_2 \cdot 3 H_2O + 3Ca (OH)_2$

This can be again simplified as

 $3 C_3 S + 6 H C_3 S_2 H_3 + 3 Ca (OH)_2 - SiO_2$

С- S -Н

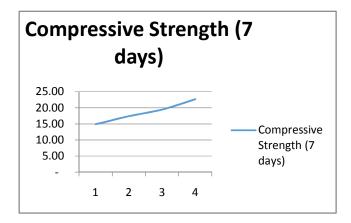
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The pore size of the blended cement mortar is significantly *leaucea, and* CSH gel forms on the aggregate surface . as compared to OPC concrete, the bulk hydrated cement paste is denser, which increase the hydration age.

Table.5 Influence of nanosilica on compressive strength

identification	Comp.strength (7 daysMpa)	% of incresese
OPC	14.88	-
NS 1.5%	17.38	16.8
NS 3.0%	19.43	13.77
NS 4.5%	22.64	21.57

Chart no3 Strength test on mortar cube



CONCLUSION

- ✓ When rice is burned in a muffle furnace at 650° C for 4 hours, an extract of nanosilica from rice husk ash, an agriculture waste, is used to generate nano silica.
- ✓ The extarction of pure silica is achieved using a titration process with a 5N H_2SO_4 solution and continuous stirring at a controlled temperature.
- ✓ The refluxing process removes 98% pure nanosilica from pure silica.
- ✓ Nanosilica replaces cement in 1.5%, 3% and 4.5% of cases.
- \checkmark As compared to ordinary portland cement , nanosilica has a higher consistency .
- ✓ As compared to ordinary portland cement, nanosilica has a faster initial and final setting time.
- ✓ As compared to portland cement paste, the inclusion of nanosilica in the cement paste increases the compressive strength.

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