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Experimental Investigation on Cement Mortar Blended With Nano Silica Synthesized From Rice Husk Ash

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Abstract: In this paper work the properties of blended cement mortar in which cement is replaced with synthesized nanosilica is investigated. Cement replacement levels of 3%, 5% were adopted. When rice husk ash is burnt in a muffle furnace at 650°C for 4 hrs Amorphous Silica is obtained. Pure Silica is extracted by titration method using 5N, H2SO4 solution with constant stirring at controlled temperature. By refluxing method 98% of pure Nanosilica is extracted from pure silica. SEM, XRD and FTIR tests were carried out to determine the chemical and physical properties of the rice husk ash and synthesized Nanosilica. SEM results show the particles are in agglomeration form. XRD powder pattern of nanosilica confirms the amorphous nature of the substance. An FTIR spectrum adds evidence of the presence of nanosilica. Properties of cement blended with synthesized Nanosilica of 3%, 5% were determined as per codal provisions. The compressive strength increases from 16% to 53% and 11% to 62% respectively for 7 and 28 days and density increase from 2% to 8%, for the increase in Nanosilica content from 3 to 5%.Partial replacement of Nanosilica improves the mechanical behavior of mortar but decrease the setting time. The strength achieved by using Nanosilica is greater compared to that of Ordinary Portland Cement.

I. INTRODUCTION

In recent years, the use of nano-particles has received particular attention in many fields of applications to fabricate materials with new functionalities. When ultra-fine particles are incorporated into Portland-cement paste, mortar or concrete, materials with different characteristics were obtained. The performance of these cement based materials is strongly dependent on nano-sized solid particles, such as particles of calcium–silicate–hydrates (C–S–H), or nano-sized porosity at the interfacial transition zone between cement and aggregate particles. Typical properties affected by nano-sized particles or voids are strength, durability, shrinkage and steel-bond. Nano-particles of SiO_2 (nS) can fill the spaces between particles of gel of C– S–H, acting as a nano-filler. Furthermore, by the pozzolanic reaction with calcium hydroxide, the amount of C–S–H increases, resulting a higher densification of the matrix, which improves the strength and durability of the material. Previous research indicates that the inclusion of nano-particles modifies fresh and hardened state properties of cement mortar. Colloidal particles of amorphous silica appear to have considerable impact on the process of C_3S hydration. In this paper work, nanosilica was introduced in cement paste. And the effects of nano-sized amorphous silica on setting time, compressive strength and apparent density of cement-based mortars are investigated.

The Mechanical behavior of concrete materials depends on structural elements and phenomena that occur on a micro and nano scale. As a result, nanotechnology can modify the molecular structure of concrete material to improve the material's bulk properties; improve significantly the mechanical performance, volume stability, durability and sustainability of concrete and have revolutionary effects, allowing the development of cost-effective, high – performance, and long-lasting products and processes for cement and concrete, within the ideals of sustainable development.

"We need to better know to control the timing of concrete setting. " The evolution of the hydrogen profiles shows the timing of the surface layer's breakdown. This information can be used to study the concrete setting process

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as function of time, temperature, cement chemistry, and other factors. For example, researcher used NRRA to determine that in cement hydrating at 30 0ºC, the breakdown occurs at 1.5 hours. The surface disintegration then released accumulated silicate in to the surrounding solution, where it reacts with calcium ions to form a calcium-silicate hydrate gel, which binds cement grains together and sets the concrete.

The aim of this paper work is to study the effect of partial replacement of cement with nanosilica on mechanical and physical properties of cement mortar.

II. NANOSILICA

The construction industry uses concrete to a large extent. About 14 bln ton were used in 2007 Concrete is used in infrastructure and in buildings. It is composed of granular materials of different sizes and the size range of the composed solid mix covers wide intervals. The overall grading of the mix, containing particles from 300 nm to 32 mm determines the mix properties of the concrete. The properties in fresh state (flow properties and workability) are for instance governed by the particle size distribution (PSD), but the properties of the concrete in hardened state, such as strength and durability, are affected by the mix grading and resulting particle packing One way to further improve the packing is to increase the solid size range, e.g. by including particles with sizes below 300 nm. Possible materials which are currently available are limestone and silica fines (Sf), silica fume (SF) and nano-silica (nS). However, these products are synthesized in a rather complex way, resulting in high purity and complex processes that make them non-feasible for the construction industry.

FIG.1 NANO SILIC

The main mechanism of this working principle is related to the high surface area of nS, because it works as nucleation site for the precipitation of CSH¬gel. However, according to Bjornstrom et al. it has not yet been determined whether the more rapid hydration of cement in the presence of nS is due to its chemical reactivity upon dissolution (pozzolanic activity) or to their considerable surface activity. Also the accelerating effect of nS addition was established indirectly by measuring the viscosity change (rheology) of cement paste and mortars. The viscosity test results shown that cement paste and mortar with nS addition needs more water in order to keep the workability of the mixtures constant, also concluded that nS exhibits stronger tendency for adsorption of ionic species in the aqueous medium and the formation of agglomerates is expected. In the latter case, it is necessary to use a dispersing additive or plasticizer to minimize this effect.

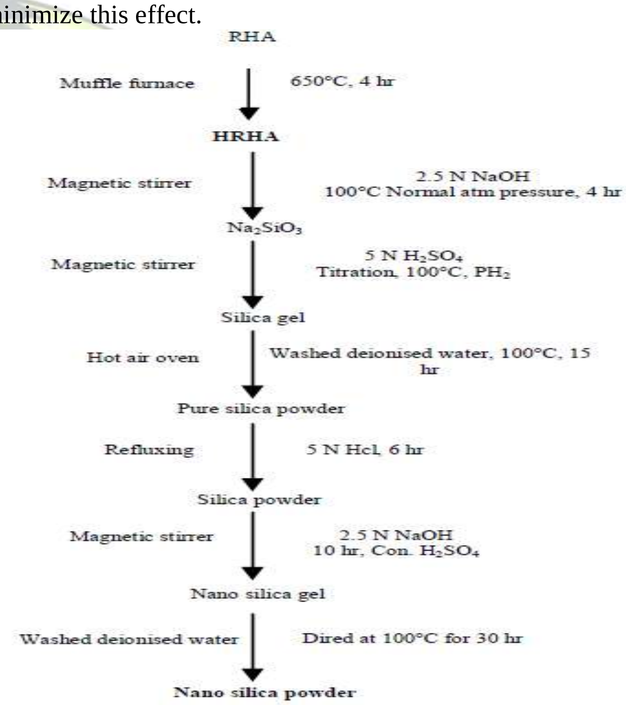

FIG.2 FLOW DIAGRAM OF NANOSILICA SYNTHESIS

About 10 g of this treated RHA sample is stirred with 80 ml of 2.5 N sodium hydroxide solution prepared using triple distilled water. The solution is heated at 100ºC in a covered 250 ml beaker for 4 hrs with constant stirring to dissolve the silica (RHA) and to produce a sodium silicate solution. The solution is filtered in a silica crucible and then the residue is washed with warm distilled water. The obtained viscous, transparent, colorless sodium silicate solution is allowed to cool to room temperature. Pure silica is extracted by titration method using 5N H_2SO_4 solution under constant stirring at controlled condition. The temperature is in the range of 90°-100°C with normal

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atmospheric pressure. The acidic condition pH2 indicates approximately the complete precipitation of silica from sodium silicate by the reaction. Washing the gel formed silica using warm distilled water removes the sulphate impurities. When the product is in an oven at 70ºC for 15 hrs paves the amorphous silica. By refluxing method nanosilica is extracted from pure silica. Pure silica with 6NHCI solution is subjected to 6 hrs of continuous refluxing at 80°-90°C. The sample is then thoroughly washed with warm water to become alkali free. Afterwards 80 ml of 2.5N NaOH is added to the silica powder under continuous stirring using a magnetic stirrer for 10 hrs. Then con.H_2SO_4 is added to the solution until a white precipitate is formed. The precipitate is washed repeatedly with warm distilled water until the filtrate become alkali free. The obtained precipitated nanosilica is dried in a hot air oven for 20 hrs.

Fig.3 RICE HUSK ASH (TOP) NANOSILICA (BOTTOM).

III. EXPERIMENTAL PROGRAMME

This section describes in detail the materials used, mix proportions, test specimens preparation and the various tests conducted as part of the experimental work.

MATERIALS

Cement - Ordinary Portland cement (OPC) of 43 grade, conforming to Indian standard IS 8112- 1989 was used.

Sand - screened River sand passed through 1.18mm sieve with fineness modulus of 2.85 and specific gravity of 2.55.

Rice husk ash - Rice husk ash were taken from industry in Madurai.

Nanosilica - Nanosilica from rice husk ash was obtained by titration and refluxing methods.

IV. TESTS CARRIED OUT

The following tests to determine Chemical, Physical and Mechanical properties were carried out in this paper.

Cement

Test for Initial setting time and final setting time.
Consistency test.
Specific gravity test.

Sand

Specific gravity test.
Fineness modulus.

Nanosilica

Initial setting time final setting time of Nanosilica.
Consistency test of Nanosilica.

Mortar cubes

Compressive strength test

V.TEST ON BLENDED CEMENT

Consistency and Setting Time of Blended Cement was determined as per IS guidelines (IS 4031-1995 part 5) for the cement blended with Nanosilica of 3%,5%. The paste with standard consistency was used to determine the initial setting time and final setting time.

VI. CASTING OF MORTOR CUBES

To determine the strength of blended cement three mortar cubes for each replacement levels were cast. The details are given below.

Cement mortar – 1:3
Replacement of cement by Nanosilica – 3%,5%.
Water cement ratio – 32% to 42%
Size of mortar cubes – 150*150*150mm
No of cubes cast – 4 x 6 (24 nos)

During moulding, the cubes were well compacted manually, after 24 hours, the specimens were demoulded and immediately submerged in clean fresh water. After 7 and 28 days of curing, the cubes were taken out of water and allowed to become dry for some hours and were tested in compression testing machine.

VII. STRENGTH TEST

28 days Compressive strength of OPC and OPC replaced with nanosilica at different percentages were determined as per IS guidelines. The load was applied at the rate of 140 kN/minute. In the ultimate load stage, the cube gets cracked and that load was taken for calculating the compressive strength.

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Fig 4. MIXING OF NANOSILICA

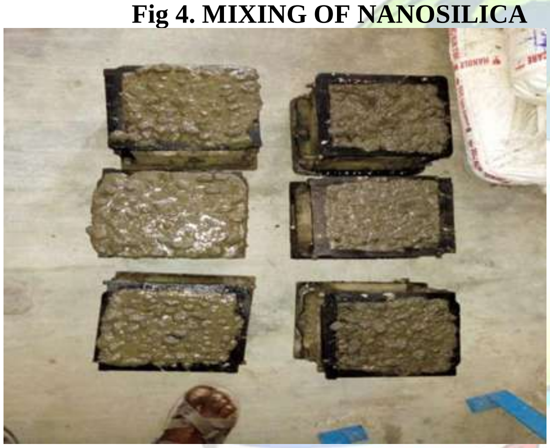

Fig 5. CASTING OF NANOSILICA

Fig 6. CURING MORTAR CUBE

The Standard Consistency value of OPC was found to be 32%. The Standard consistency increased from 32% to 42% for Nanosilica. Nanosilica addition resulted in large increase in surface area of cement, and the increase in normal consistency.

The table I shows that partial replacement of cement by nanosilica of 3%,5% modifies the properties of cement mortar as given below. Normal Consistency increases from 3% to 32%.Initial setting time decreases from 6% to 30%.Final setting time decreases from 6% to 42%.

The fig. 7 shows that normal consistency of OPC blended with nanosilica is gradually increased due to replacement of cement. The fig 8 shows that setting time of OPC blended with nanosilica is gradually decreased due to replacement of cement.

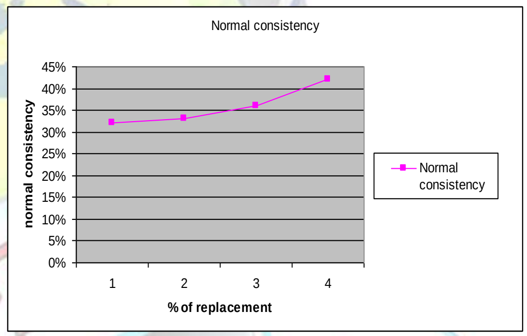

FIG.7 CONSISTENCY OF OPC BLENDED WITH NANO SILICA

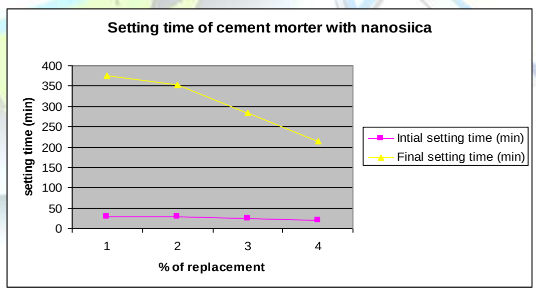

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FIG.7 CONSISTENCY OF OPC BLENDED WITH NANO SILICA

FIG.8 SETTING TIME OF OPC BLENDED WITH NANOSILICA

CUBE COMPRESSIVE STRENGTH

The results of Cube compressive strength of blended cement after 7 and 28 days of curing are shown in table 5.4.1 and 5.4.2 respectively. After 7 and 28 days of curing OPC showed cube compressive Strength of 14.88 Mpa and 22.67Mpa respectively. When the cement was replaced by nano silica (3% to 5%) the cube compressive strength showed a gradual increase. All blended Cement mortar specimens with nanosilica showed higher compressive strength than that of mortar specimen with OPC.

This is because higher silicon content in synthesized Nanosilica reacts more readily with water and liberates free calcium hydroxide during the hydration of OPC and forms calcium hydro silicate gel compound.

Also in the blended cement mortar the pore size is much reduced and there is a development of CHS gel over the aggregate surface. The bulk hydrated cement paste is relatively denser compared to OPC concrete and this improves the hydration.

NAME	NORMAL CONSISTENCY	INITIAL SETTING TIME	FINAL SETTING TIME
	in %	in Min	in Min
OPC	32	30	375
3% NS	36	25	283
5%NS	42	21	214

S.NO	Area of specimen(mm)	Load (KN)	Compression strength(KN/m)
1	150x150	580	25.77
2	150x150	550	24.44
3	150x150	600	26.66

S.NO	Area of specimen(mm)	Load(KN)		Compression Strength(KN/m)	
		3%	5%	3%	5%
1	150x150	560	610	24.00	27.11
2	150x150	590	680	26.22	30.22
3	150x150	600	620	26.66	27.55

S.NO	Area Of specimen(mm)	Load(KN)	Compression Strength(KN/m)
1	150X150	650	28.88
2	150X150	680	30.22
3	150X150	630	28.00

S.NO	Area of specimen(mm)	Load(KN)		Compression Strength(KN/m)	
		3%	5%	3%	5%
1	150x150	630	800	28.00	35.55
2	150x150	690	750	30.66	33.33
3	150x150	700	760	31.11	33.77

The fig. 9 shows that the density of cement mortar cubes gradually increased due to replacement of cement by Nanosilica.

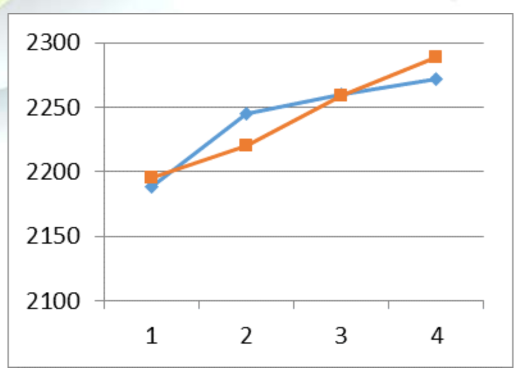

FIG 9 DENSITY OF MORTARCUBE

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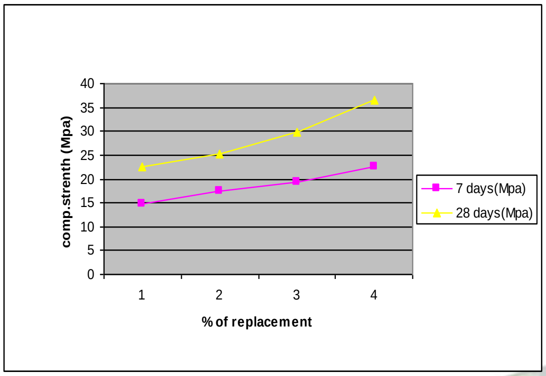

FIG.10 STRENGTH TEST ON MORTAR CUBE

Fig 11 COMPRESSION TEST FOR CUBE

Fig 12 COMPRESSION TEST

IX. CONCLUSION

An extract of Nanosilica from rice husk ash, an agriculture waste was used to produce nano silica by synthesized process. When rice husk ash is burnt in a muffle furnace at 650^0C for 4 hrs amorphous silica was obtained. Pure silica was extracted by titration method using 5N H_2SO_4 solution with constant stirring at controlled temperature.

From the results of the experimental investigation on Blended Cement with Nanosilica, it is concluded that Partial replacement of cement by nanosilica of 1.5%, 3% and 4.5% modifies the properties of cement mortar as given below.

- ✓ Normal Consistency increases from 3% to 32%.
- ✓ Initial setting time decreases from 6% to 30%.
- ✓ Final setting time decreases from 6% to 43%.
- ✓ 7 days and 28 days Compressive strength increases from 16 % to 52% & 11% to 62% respectively.
- ✓ And density increase from 2% - 8%.

Hence the incorporation of the nanosilica in the cement paste increases the compressive strength while reducing the setting time.

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