© 2025 IJRTI | Volume 10, Issue 10 October 2025 | ISSN: 2456-3315

Forms of Micro Silica and it's impacts on Concrete

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Abstract

This study evaluates the chemical characteristics of microsilica produced from rice husk ash (RHA) through controlled combustion. The investigation emphasizes the role of amorphous silica and crystalline silica forms in influencing concrete's pozzolanic activity, strength, and durability. Controlled RHA burning between 500°C and 600°C preserves the reactive amorphous silica while minimizing deleterious crystalline silica, optimizing microsilica's efficacy as a supplementary cementitious material.

The global construction industry is increasingly seeking sustainable alternatives to traditional materials to mitigate its environmental footprint. This study evaluates the chemical and physical characteristics of microsilica produced from rice husk ash (RHA), an agricultural byproduct, through precisely controlled combustion processes. The investigation focuses on the critical role of silica's polymorphic forms—specifically, reactive amorphous silica versus deleterious crystalline silica—in influencing the performance of concrete. The pozzolanic activity, which is fundamental to the strength and durability enhancement in concrete, is predominantly governed by the presence of amorphous silica. This non-crystalline phase reacts with calcium hydroxide (

$Ca(OH)2$), a byproduct of cement hydration, to form additional calcium silicate hydrate (C-S-H), the primary binding agent in concrete. Conversely, the formation of crystalline silica phases, such as cristobalite and tridymite, at elevated temperatures, contributes minimally to strength and can initiate harmful alkali-silica reactions (ASR), leading to long-term durability problems. This research demonstrates that by controlling the RHA combustion temperature within an optimal range of

500°C to 600°C, it is possible to maximize the yield of reactive amorphous silica while minimizing the crystallization process. This optimization is key to producing high-quality RHA-based microsilica that serves as an effective supplementary cementitious material (SCM). The findings underscore the importance of production parameters in tailoring the properties of RHA for high-performance concrete applications, promoting a circular economy by transforming agricultural waste into a valuable construction material.

Keywords: Microsilica, Rice Husk Ash, Amorphous Silica, Crystalline Silica, Pozzolanic Activity.

1. Introduction

Microsilica, derived as a supplementary cementitious material from agricultural waste like rice husk ash (RHA), significantly influences concrete properties. The proportion of highly reactive amorphous silica over crystalline silica phases critically affects this material's performance. Controlled burning temperature during RHA production is a key factor.

The production of Portland cement, the most common binder in concrete, is an energy-intensive process responsible for approximately 8% of global carbon dioxide (CO_2) emissions. As infrastructure demands continue to grow worldwide, the concrete industry faces immense pressure to adopt more sustainable practices. One of the most effective strategies for reducing the carbon footprint of concrete is the partial replacement of cement with **supplementary cementitious materials (SCMs)**. SCMs are materials that, when used in conjunction with Portland cement, contribute to the properties of hardened concrete through hydraulic or pozzolanic activity. Their use not only reduces the volume of cement required but can also significantly enhance the final properties of the concrete, such as its long-term strength, impermeability, and resistance to chemical attack.

Among the various SCMs available, those derived from agricultural waste streams are gaining significant attention.

Rice husk ash (RHA) is a promising candidate, as it is a byproduct of the rice milling industry, with global rice production generating millions of tons of husks annually. When incinerated, these husks produce an ash that is exceptionally rich in silicon dioxide (SiO_2). However, the value of RHA as an SCM is not merely dependent on its silica content but, more critically, on the **morphology of this silica**. The silicon dioxide in RHA can exist in either a highly reactive, disordered **amorphous** state or a stable, non-reactive **crystalline** state.

The amorphous form of silica is highly pozzolanic. A pozzolan is a siliceous or aluminosiliceous material that, in a finely divided form and in the presence of water, chemically reacts with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties. In concrete, this reaction consumes the soluble $Ca(OH)_2$ produced during the hydration of cement and converts it into additional C-S-H gel. This secondary C-S-H fills capillary pores within the cement matrix, leading to a denser, stronger, and more durable microstructure.

Conversely, crystalline silica, which forms when RHA is incinerated at excessively high temperatures (typically above 700°C), is largely inert and does not contribute to the pozzolanic reaction. Worse, certain crystalline polymorphs like cristobalite and tridymite are known to be reactive in the highly alkaline environment of concrete, triggering a deleterious expansion known as the **alkali-silica reaction (ASR)**. ASR occurs when these reactive silica forms react with alkali hydroxides (from the cement) to form a hygroscopic gel that absorbs water, swells, and exerts internal pressure, causing extensive cracking and compromising the structural integrity of the concrete. Therefore, the efficacy of RHA as a high-quality SCM is directly tied to the manufacturing process.

Controlled burning temperature is the most critical factor in producing RHA with a high amorphous silica content and minimal crystalline phases. By maintaining combustion temperatures within a carefully controlled window, typically between 500°C and 600°C, the organic components of the rice husk can be burned off while preserving the silica in its reactive amorphous state. This study aims to elucidate the profound impact of silica forms in RHA on concrete properties, emphasizing the necessity of controlled combustion to engineer a sustainable and high-performance construction material. It highlights the chemical transformations that govern the material's performance, providing a scientific basis for the efficient utilization of this abundant agricultural waste in modern concrete technology.

2. Role of Silica Forms

Silicon dioxide (SiO_2) in microsilica exists mainly as:

- **Amorphous silica**, a non-crystalline phase enabling pozzolanic reactions with calcium hydroxide to form calcium silicate hydrate (C-S-H), enhancing strength and durability.
- **Crystalline silica** including cristobalite and tridymite, less reactive and prone to producing alkali-silica reaction (ASR) gels that damage concrete.

Amorphous silica shows high pozzolanic activity, reacting with calcium hydroxide to form additional strength-enhancing C-S-H gel.

SiO_2(amorphous)+$Ca(OH)_2$+H_2O→ C-S-H(calcium silicate hydrate)

Crystalline silica is usually inert, contributing little to strength, and may increase impurities. The balance of amorphous to crystalline silica affects overall reactivity and durability of cementitious materials. materials. **Cristobalite** and **Tridymite** are highly reactive forms of crystalline silica.

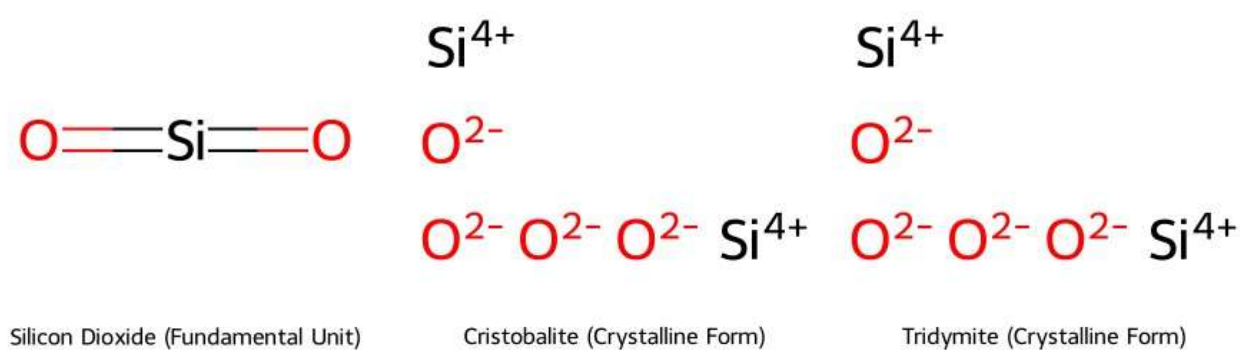

FIGURE 1 : crystal structures

The core chemical difference is highlighting is the structure of silicon dioxide (SiO_2). In its amorphous state, the SiO_4 tetrahedra are arranged randomly, which allows for high reactivity. In crystalline forms like cristobalite, these tetrahedra are arranged in a highly ordered, repeating lattice, making them less reactive.

When they react with the alkali hydroxides in the concrete pore solution, they form a hydrophilic gel.

$SiO_2 + 2NaOH \rightarrow Na_2SiO_3$ (sodium silicate) + H_2O

$SiO_2 + 2KOH \rightarrow K_2SiO_3$ (potassium silicate) + H_2O

This gel absorbs water from the surrounding concrete, swells, and creates internal pressure. This pressure leads to cracking and spalling, which weakens the concrete and significantly reduces its durability and service life.

FIGURE 2 : Concrete Spalling and cracking

3. Controlled Burning Temperature Effects

RHA burned at 500-600°C mostly yields amorphous silica, maintaining pozzolanic properties. Above 700°C, crystalline silica forms, reducing reactivity and increasing ASR risk. Hence, controlled temperature combustion is essential.

This study was designed to investigate the influence of RHA combustion temperature on the resulting silica structure and its subsequent impact on key concrete properties. The methodology involved the production of RHA under controlled laboratory conditions, comprehensive material characterization, and systematic testing of concrete specimens.

To study the effect of temperature, three batches of RHA were produced in a programmable muffle furnace:

- **RHA-600:** Produced by burning the rice husks at a controlled temperature of 600°C for 4 hours. This temperature falls within the optimal range for producing amorphous silica.
- **RHA-900:** Produced by burning the rice husks at 900°C for 4 hours. This temperature is expected to induce the formation of crystalline silica phases.
- **RHA-UC (Uncontrolled):** Produced by open-air burning in a steel drum to simulate a common, less-controlled field practice, which often results in high levels of unburnt carbon.

After incineration, all three ashes were allowed to cool and then ground in a roller mill for 90 minutes to achieve a fine, uniform particle size comparable to that of cement.

Table 1: Physical and Mineralogical Properties of RHA Samples

Property	RHA-600	RHA-900	RHA-UC
Silica Content (SiO_2) (%)	91.2	94.1	88.7
Loss on Ignition (LOI) (%)	1.8	1.1	4.6
Predominant Silica Form	Amorphous	Crystalline (Cristobalite)	Amorphous with impurities

4.Concrete Mix Design and Preparation

- **Cement:** Ordinary Portland Cement (OPC) conforming to standard specifications was used as the primary binder for all mixtures.

- **Aggregates:** Locally sourced natural sand was used as fine aggregate, and crushed granite was used as coarse aggregate. Both were washed, dried, and graded according to standard protocols.

- **Micro silica:** Rice husks ash were obtained from a local mill in Panagarh, West Bengal. They were grind in a roller mill to achieve a fine powder form (fineness 94% on 45µ IS Sieve).

- **Water:** Potable tap water was used for mixing concrete.

Four concrete mixtures were designed with a constant water-to-binder ratio of 0.45. The mix proportions for the four concrete mixtures are detailed in Table 1.

Table 2: Concrete Mix Proportions (per m³)

Material	Control Mix (CM)	RHA600 + CM	RHA900 + CM	RHAUC +CM
Cement (kg)	400	360	360	360
RHA based MS(kg)	0	40	40	40
Water (kg)	180	180	180	180
Fine Aggregate (kg)	750	750	750	750
Coarse Aggregate (kg)	1100	1100	1100	1100
Water/Binder Ratio	0.45	0.45	0.45	0.45

5. Testing Procedures

- **Fresh Concrete Properties:** The workability of each concrete mix was evaluated using the **Slump Test**.
- **Hardened Concrete Properties:**
    - **Compressive Strength:** Cubic specimens (150mm x 150mm x 150mm) were cast for each mix. The cubes were demolded after 24 hours and cured in water. Compressive strength was tested at 7, 28, and 90 days.
    - **Alkali-Silica Reaction (ASR) Potential:** An **Accelerated Mortar Bar Test** was performed. Mortar bars were cast using crushed borosilicate glass as a reactive aggregate for the control mix and the two RHA mixes (RHA-600 and RHA-900). The bars were stored in a 1N NaOH solution at 80°C, and their length change (expansion) was measured periodically over 16 days. Significant expansion indicates a potential for deleterious ASR.

6. Results

6.1. Fresh Concrete Properties The slump test results, shown in Table 3, reflected the impact of RHA purity. The high-carbon RHA-UC mix experienced a significant loss in workability due to the adsorption of mix water by the carbon particles.

Table 3: Fresh Concrete Properties (Slump Test)

Mix ID	Slump (mm)
Control Mix (CM)	110
CM-RHA600	95
CM-RHA900	98
CM-RHAUC	60

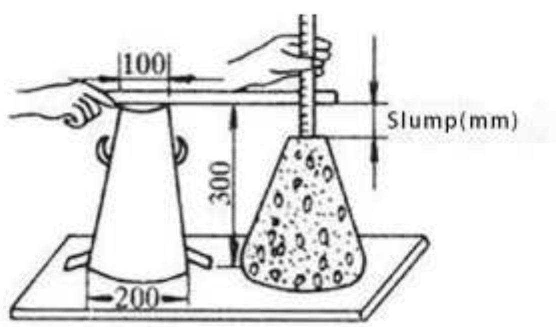

FIGURE 3 : Concrete Slum Test

6.2. Hardened Concrete Properties The compressive strength development over 90 days is presented in Table 4. The CM-RHA600 mix showed superior long-term strength due to its pozzolanic reactivity, while the CM-RHAUC mix consistently underperformed.

Table 4: Compressive Strength of Concrete Mixes (MPa)

Mix ID	**7-Day Strength**	**28-Day Strength**	**90-Day Strength**
Control Mix (CM)	28.5	39.2	42.5
CM-RHA600	25.1	43.8	51.0
CM-RHA900	24.5	39.5	43.1
CM-RHAUC	19.8	31.4	34.0

The potential for ASR was evaluated using the accelerated mortar bar test, with the final expansion results shown in Table 5. The RHA-600 mix effectively mitigated ASR, while the RHA-900 mix, containing crystalline silica, exacerbated it.

FIGURE 4 : Concrete Compressive Strength Test

Table 5: Accelerated Mortar Bar Test Results (ASR Potential)

Mix ID	Final Expansion after 16 days (%)
Control Mortar	0.10
Mortar with RHA-600	0.04
Mortar with RHA-900	0.25

Note: Expansion > 0.10% is generally considered deleterious.

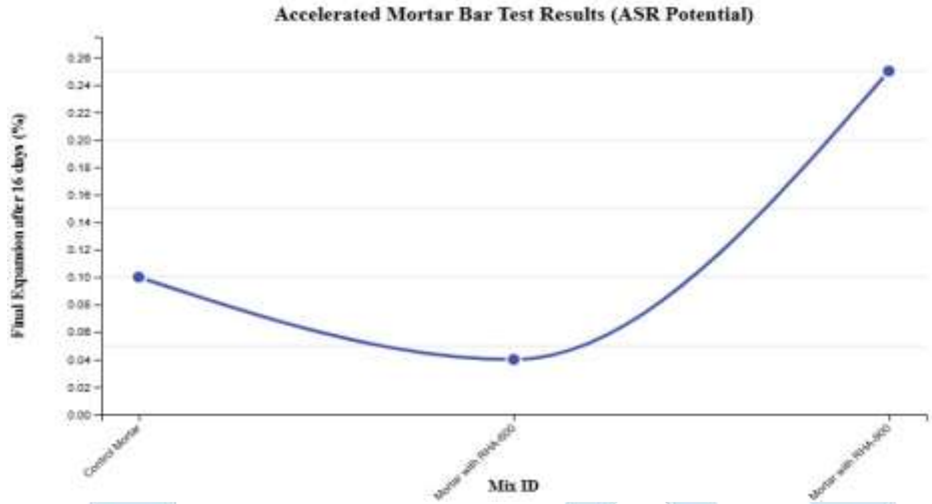

FIGURE 5 : Accelerated Mortar Bar Test

7. Purity Considerations

Loss on Ignition (LOI) informs about impurities (e.g., unburnt carbon). Low LOI is ideal for predictable hydration and concrete quality.

The significance of LOI in cement and concrete materials relates to purity and quality:

- A low LOI indicates fewer impurities (like unburnt carbon or excess moisture) in the cement or supplementary materials.
- Low impurities lead to more predictable hydration reactions during concrete curing, improving concrete strength and durability.

Unburnt carbon increases the Loss on Ignition (LOI) because when the sample is heated during the LOI test, the carbon burns off, contributing to the weight loss measured as LOI. This is why higher unburnt carbon content results in higher LOI values.

Regarding cement hydration and concrete properties, unburnt carbon affects these negatively in several ways:

- It adsorbs water and admixtures like air-entraining agents, which leads to higher water demand and poor air entrainment. This reduces workability and consistency of the fresh concrete.
- It can reduce the compressive strength of concrete because the carbon particles interfere with the bond between cement paste and aggregates.
- Unburnt carbon can cause discoloration of concrete and also increase electrical conductivity, which may accelerate corrosion of steel reinforcement.
- The pozzolanic reaction is less effective or less predictable, leading to reduced durability and hydration efficiency.
- High carbon content reduces the cement hydration predictability and results in inconsistent concrete quality.

5. Conclusion

Balancing amorphous and crystalline silica through controlled RHA combustion is crucial to producing microsilica that improves concrete durability and strength while mitigating ASR. This chemical understanding guides efficient use of sustainable microsilica in construction.

unburnt carbon raises LOI by burning off during ignition and adversely impacts cement hydration by increasing water demand, lowering strength, causing durability issues, and interfering with admixture effectiveness. Keeping low unburnt carbon content is necessary for better concrete performance and hydration control.

This investigation successfully demonstrated the critical impact of silica's polymorphic forms in rice husk ash (RHA) on the properties of concrete. The study confirms that the effectiveness of RHA as a supplementary cementitious material is not inherent but is engineered through precise control of the combustion process.

The primary conclusion is that

RHA produced at a controlled temperature of 600°C, which contains highly reactive **amorphous silica**, significantly enhances the long-term performance of concrete. This enhancement is driven by the pozzolanic reaction, where amorphous silica consumes calcium hydroxide to form additional strength-giving calcium silicate hydrate (C-S-H). This reaction leads to a denser, refined microstructure, resulting in superior 90-day compressive strength and a remarkable ability to mitigate the deleterious alkali-silica reaction (ASR) by consuming the necessary alkali hydroxides.

Conversely,

RHA produced at a high temperature of 900°C yields **crystalline silica** (cristobalite), which is largely non-pozzolanic and contributes minimally to strength. More critically, this crystalline form was found to be reactive in the ASR test, exacerbating expansion and posing a significant durability risk. This finding highlights the danger of using improperly calcined RHA in concrete.

Furthermore, the study reinforced the importance of purity, showing that RHA with a high content of

unburnt carbon (high LOI) negatively affects both fresh and hardened concrete properties. It impairs workability by adsorbing water and leads to a substantial reduction in compressive strength.

In summary, for RHA to be a viable and reliable SCM, its production must be carefully managed to **maximize amorphous silica content** and **minimize both crystalline phases and carbon impurities**. Controlled combustion within the 500-600°C range is essential for achieving these desired characteristics. By transforming an agricultural waste product into a high-performance material under scientifically controlled conditions, RHA can play a significant role in the development of sustainable, durable, and cost-effective concrete for the future, advancing the goals of a circular economy in the construction industry.

ACKNOWLEDGMENT

I would like to express my sincere gratitude to Ar. Nasreen parvez for the invaluable guidance, encouragement, and insightful feedback provided throughout this research. I also extend my appreciation to the Ultrafine Mineral and Admixtures pvt ltd for providing the necessary facilities and academic environment to complete this work.

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