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VALORISATION OF PADDY HUSK: EXTRACTION OF SILICA FOR SUSTAINABLE DETERGENT FORMULATION

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ABSTRACT
The increasing emphasis on environmental sustainability and waste management has driven interest in the valorisation of agricultural residues. Rice husk, a by-product of rice milling, is often considered waste with limited commercial applications. This study explores the extraction of silica from rice husk ash (RHA) using thermal treatment and chemical extraction techniques, followed by its utilization as an abrasive agent in detergent formulations. FTIR analysis confirmed the presence of silica in the extracted material. Detergent samples formulated with sodium silicate derived from RHA were evaluated for foam stability, free alkali content, moisture content, and pH. The lab-formulated detergent exhibited a pH of approximately 9, a foam retention time of 13 minutes, foam height of 4.2 cm and a free alkali content of 0.0224 %. Comparative analyses with conventional detergents demonstrated that the RHA-based detergents exhibit comparable cleaning efficacy while being non-toxic and biodegradable. This study presents a sustainable approach to transforming agricultural waste into a valuable raw material for the detergent industry, contributing to circular economy practices and eco-friendly product development.

KEYWORDS: Rice husk ash, Sodium silicate, FTIR, foam stability, pH value, free alkali content.

1. INTRODUCTION

The detergent industry is undergoing a transformative shift towards sustainability by integrating innovative raw materials with traditional manufacturing techniques. One such revolutionary approach involves harnessing the untapped potential of rice husk ash (RHA) for detergent powder production. This strategy not only aligns with sustainable product development but also addresses pressing environmental concerns and resource efficiency challenges.[1,2]

Paddy husk, a major by-product of rice milling, is produced in vast quantities worldwide.[3-5] Traditionally regarded as agricultural waste, its disposal poses significant environmental challenges, contributing to pollution through open burning or improper waste management.

Rice husk contains about 15–20% silica by weight, making it a valuable source for high-purity silica extraction. The silica is primarily present in an amorphous form, embedded in the husk's lignocellulosic matrix. Extraction methods include thermal treatment**,** acid leaching**,** and alkaline extraction to obtain silica in various morphologies such as nanoparticles and mesoporous structures. Rice husk-derived silica is widely used in rubber reinforcement**,** ceramics**,** adsorbents, and catalysts due to its high surface area and thermal stability. It also finds applications in biomedical fields for drug delivery and in construction for producing high-performance concrete and insulation materials.[5] Paddy husk is rich in silica and cellulose, is a valuable raw material for various industrial applications, including sustainable detergent formulation.[6,7]

Telangana rice husk varieties possess distinct physical, chemical, and thermal properties that make them highly versatile. The husk is typically light brown to golden, with a low bulk density of 100–150 kg/m³, high porosity, and a tough, fibrous structure due to its rich silica content. Chemically, it comprises 15–25% silica, 30–40% cellulose, 20–25% lignin, and 15–20% ash after combustion, making it valuable for industrial and agricultural applications. Varieties such as Sona Masoori, RNR 15048 (Telangana Sona), BPT 5204 (Samba Masuri), and MTU 1010 exhibit different husk compositions, making them suitable for specialized applications, including biofuel, composting, and industrial processes.

Conventional detergent manufacturing heavily depends on petrochemicals and synthetic materials, which are not only expensive but also contribute to environmental degradation due to their non-biodegradable nature and potential to cause water pollution.[8-10] Most modern detergents contain complex formulations, including surfactants, builders, bleaching agents, fillers, and additives. While builders enhance washing efficiency, commonly used phosphates lead to eutrophication, severely impacting aquatic ecosystems.[11-13] The demand for eco-friendly alternatives has driven research into sustainable and biodegradable detergent formulations.

In response to the growing emphasis on waste valorisation and circular economy principles, researchers have explored novel applications for agricultural residues.[14-16] Paddy husk, once an underutilized by-product, is now recognized for its potential in various industries, including the production of green detergents. The use of RHA-derived silica in detergent formulations offers a sustainable, cost-effective, and environmentally friendly alternative to conventional ingredients. Recent advancements in material science and green chemistry have further optimized the extraction and utilization of silica from rice husk, enhancing its applicability in cleaning products while reducing reliance on synthetic chemicals.[17-20]

This study investigates the feasibility of utilizing sodium silicate derived from RHA as an abrasive and functional ingredient in detergent formulations.[21] By integrating agricultural waste into detergent production, this approach promotes sustainability, resource efficiency, and eco-friendly product development, contributing to a cleaner environment and a circular economy.[22]

1.1 Detergent

Detergents are chemical compounds designed to effectively clean and remove dirt, grime, and stains from surfaces, fabrics, and materials. They achieve this through three primary mechanisms: reducing surface tension to allow water to penetrate and lift away dirt, breaking down oils into smaller particles for easy removal, and suspending and removing dirt and particles.[23] The key components of detergents include surface active agents, or surfactants, which penetrate fabric fibers to remove dirt and oil, enzymes that enhance the washing process by reducing energy and water consumption, and phosphates that remove stains, improve detergent performance, and soften water. Additionally, sodium laurel sulfate, a common ingredient in laundry detergents, provides strong cleansing power and high foam production, making it a crucial component in many cleaning products.[24] Figure 1 shows the surfactant molecule.

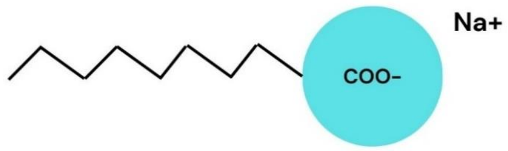

Figure 1: Molecular structure of a surfactant, illustrating the hydrophilic head and hydrophobic tail regions.

1.2 Classification of detergents

Detergents can be broadly classified into two main types: anionic and cationic detergents. Anionic detergents are the most commonly used in household laundry applications, known for their excellent cleansing and foaming properties. A widely used example is Sodium Dodecyl Sulfate (SDS), which effectively removes dirt and grease by interacting with water and oil molecules.[25] On the other hand, cationic detergents possess antimicrobial and fabric-softening properties, making them valuable in specific cleaning and conditioning applications. A well-known example of a cationic detergent is Cetyl Tri-methyl-Ammonium Bromide (CTAB), which is frequently used in disinfectants, fabric softeners, and certain hair care products.[26]

1.3 Applications

Detergents serve a wide range of applications across various industries. In household and industrial cleaning, they are commonly used for washing dishes, clothes, and surfaces, as well as for pre-washing, soaking, rinsing, and bleaching. Beyond cleaning, detergents play a crucial role in scientific research, particularly in cell biology, molecular biology, and biochemistry, where they are used for cell lysis, protein crystallization, and the purification of membrane proteins and lipids. In the automotive industry, detergents are added to fuels to prevent fouling in fuel injector components and carburettors of internal combustion engines, ensuring efficient performance. Similarly, they are incorporated into dry-cleaning solvents to aid in soil removal from garments. Additionally, detergents are blended into lubricating oils to prevent varnish-like deposits from accumulating on cylinder walls, thereby enhancing engine longevity and efficiency.

2. MATERIALS

Rice husk was generously provided by a milling facility at Narsapur, Telangana, India. Chemicals sodium hydroxide, orthophosphoric acid, caustic soda solution, soda ash, ammonia, hydrogen peroxide, sodium tri-polyphosphate, sodium silicate, sulphonic acid, additives are procured from Akshaya Chemicals Hyderabad. Distilled water is used in all experiments.

Equipment used: Hot air oven, Magnetic stirrer, Heater, muffle furnace, sieve set, screen.

3. EXPERIMENTAL PROCEDURE

3.1. Pretreatment of Rice Husk

In this study, rice husk was collected from the Narsapur region in Telangana, India. It was sun-dried for 48 hours to reduce moisture content, then sieved to remove any leftover rice grains, allowing it to burn more efficiently. A 150 g sample of rice husk was placed in crucible and heated in a furnace at 700 °C.[27] The rice husk ash as shown in figure 2 was then sieved to eliminate any remaining rice grains, ensuring optimal combustion efficiency.

Figure 2: Rice Husk ash after combustion.

3.2. Extraction of Sodium Silicate

Rice husk ash is converted into sodium silicate through a meticulous pre-treatment procedure. 10 g sample of RHA was reacted with a 1M sodium hydroxide (NaOH) solution. Initially, the mixture was stirred with a glass rod to ensure thorough blending. It was then placed in a hot air oven at 100 °C for one hour. After the heating process, the solution was poured onto filter paper, and the residues were dried in an oven at 60 °C for 30 minutes.[28] Finally, the synthesized sodium silicate was analysed using FTIR spectroscopy to verify the presence of silica functional groups.

3.3. Characterization

FTIR spectroscopy is a highly effective method for determining the chemical composition of different substances. By utilizing infrared radiation to examine samples, this technique provides crucial information about their structure and characteristics, including the detection of organic, polymeric, and certain inorganic compounds.[29] Rice husk ash was assessed using FTIR to identify the functional groups associated with silica.

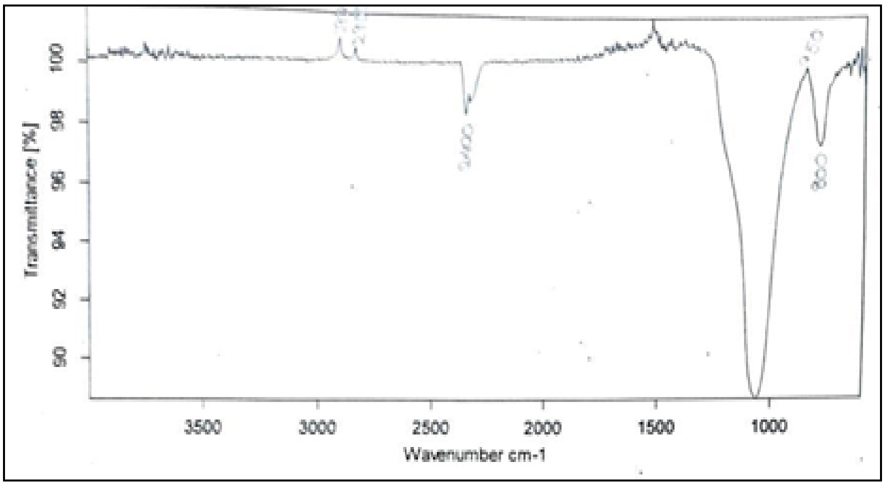

Figure 3: FTIR analysis of sodium silicate extracted form RHA.

From figure (3) it can be confirmed that.

- High Silica Content: Prominent peaks near 1100 cm⁻¹ and 800 cm⁻¹ signify a high concentration of silica, characteristic of thoroughly combusted rice husk ash.
- Organic Residue: Peaks observed around 2800-3000 cm⁻¹, 1700 cm⁻¹, and 1600 cm⁻¹ suggest the possibility of incomplete combustion, indicating the presence of organic residues.
- Moisture Content: A broad O-H stretching peak between 3400-3600 cm⁻¹ and a bending vibration around 1630-1650 cm⁻¹ point to the presence of moisture or hydroxyl groups.[29]

3.4. Preparation of Detergent

To 200 mL of sodium hydroxide solution 10 mL of sodium silicate solution (Sample A) and 250 mL of palm kernel oil, was added with continuous stirring to ensure thorough blending. While mixing, 400 g of soda ash and 50 g of sodium tri-polyphosphate (STPP) was gradually incorporated into the solution. To facilitate the detergent's formation, ammonia was added as a drying agent, while hydrogen peroxide was introduced to enhance the reaction, increasing the stirring speed for better homogenization. After 24 hours, the detergent had solidified, after which it was ground and sieved to obtain the final product as shown in figure 4. To assess its quality, foam stability, alkalinity level, and moisture content were evaluated.[27]

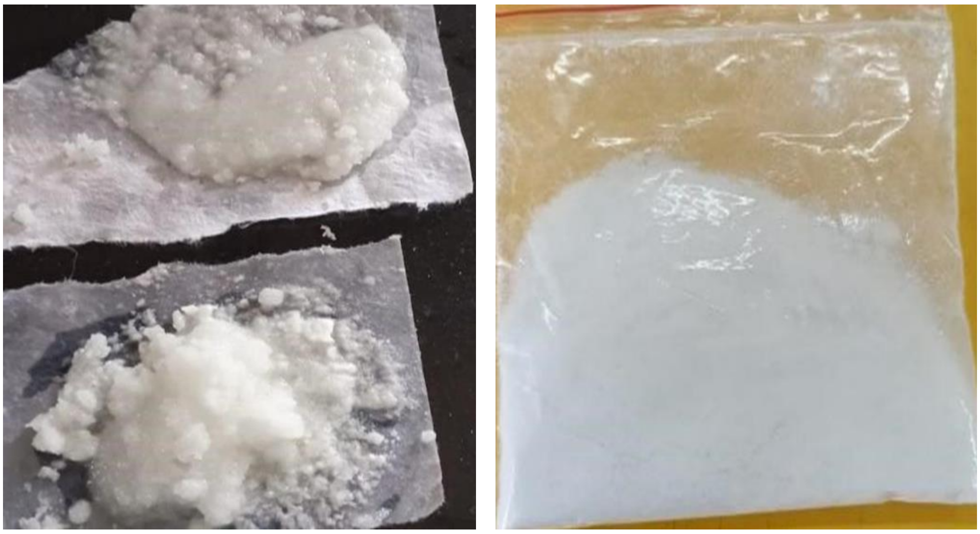

Figure 4: Detergent sample after filtration and drying.

4. QUALITY ASSURANCE OF THE LAB FORMULATED DETERGENT

4.1 Evaluation of Foam stability

The detergent's performance was evaluated by testing its ability to generate lather in hard water.[30] Using a stopwatch, the time taken for the foam to dissipate was measured. The durability of the foam is a key factor that distinguishes a high-quality soap or detergent. A dense, long-lasting foam helps lower surface tension, enhancing the cleaning process. Essentially, the more effectively the ingredients interact, the greater the foam production. The results are indicated in the table 1.

4.2 Analysis of pH value

The pH level is a key factor in ensuring a detergent's effectiveness and skin-friendliness. Ideally, an optimal detergent should have a pH between 9 and 11, with neutrality being the desired benchmark. To evaluate the detergent's pH, the prepared detergent solution was tested for its alkalinity or acidity using a pH meter. The pH value was found to around 9. It was confirmed that the detergent met quality standards and was safe for use.[27]

4.3 Test for Free Alkali

Determining the free alkali content in a detergent is essential, as an excess amount can be damaging to the skin. To assess a detergent's quality in relation to its alkali levels, a structured procedure can be carried out to precisely quantify and analyze the free alkali present.[30]

To the detergent sample 100 mL of neutralized alcohol is added and heated until the detergent is completely dissolved in the alcohol. Once fully dissolved, 10 cm^3 of Barium Chloride solution is introduced and two drops of phenolphthalein indicator is added to the flask.

Barium Chloride facilitates the precipitation of any sulfate ions present, while the phenolphthalein indicator produces a colour change to signal the titration endpoint. After adding these reagents, titration is done using a 0.1 M sulfuric acid solution. The sulfuric acid is gradually introduced until the solution turns colourless, indicating that the alkali has been completely neutralized by the acid.

The free alkali was calculated as per equation 1 and the results are indicated in table 1.

A= 3.1 x mv/w*100. (Eq 1)

where

m = Molarity of sulfuric acid solution, mol.L-1.

v = Volume of sulfuric acid solution used in the titration, mL

w= Weight of the detergent sample, g

A = Free alkali in %.

Table 1: Estimation of Detergent testing parameters.

COMPONENTS	LAB MADE DETERGENT	COMMERCIAL DETERGENT
pH	9-10	10-10.5
Foam Height	4.2 cm	0.06 m
Foam Retention Time	13 min	23 min
Percentage Free Alkali	0.0224 %	0.0155 %

5. CONCLUSION

The detergent formulated from Rice Husk Sodium Silicate demonstrates remarkable properties, including foam stability, balanced pH, and controlled free alkali content, making it competitive with commercial detergents. Additionally, it offers appealing qualities such as a pleasant texture, fragrance, and excellent water solubility. This eco-friendly detergent provides several advantages. It repurposes waste rice husk ash, reducing waste and supporting sustainable practices, making it an environmentally conscious product. Its production costs are lower than conventional detergents, ensuring affordability without compromising quality. The detergent delivers superior cleaning performance, matching the effectiveness of commercial products while appealing to eco-conscious consumers with its sustainable attributes. The lab-formulated detergent exhibited a pH of approximately 9, a foam retention time of 13 minutes, foam height of 4.2 cm and a free alkali content of 0.0224 %. Future development strategies include scaling up production to industrial levels for commercial success, assessing the quality of rice husk ash from different sources, and implementing targeted marketing strategies to attract environmentally aware consumers. Additionally, exploring its potential in industrial cleaning applications could further broaden its market reach and profitability.

Conflict of interest

The authors declare no conflict of interest.

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