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Extraction and characterization of silica from rice husk for use in food industries

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Abstract

Rice husk, in large quantities, is an easily available agricultural residue in India. It is a silica rich raw material comprising of about 90-98% silica. Owing to its large ash content and the presence of sodium silicate in its ash, rice husk becomes an economical source to extract silica from the rice husk ash, which has an extensive market and also helps in ash removal. Rice husk is a widely popular boiler fuel and the ash produced usually creates disposal issues. The silica extraction process, not only offers a solution for waste clearance, but also recovers a valuable product, together with certain useful associated applications. Silica gel's high specific surface area allows it to adsorb water readily, making it suitable as a desiccant (drying agent) in various purposes. Moreover, silica's inherent antifungal property makes it a potential antifungal agent.

In this study, an attempt is made to extract and characterize amorphous silica, from rice husk ash, for its potential use in the food industry. At the same time, resolving the disposal issue of rice husk ash and safeguarding the environment from pollution.

Keywords: agricultural byproducts, extraction, rice husk ash (RHA), silica, antifungal property

1. Introduction

In today's times, when we are facing an ever-increasing crunch in every aspect of our lives and running out of resources to find sustainable solutions, the answer lies in waste, as our primary resource. We have waste in abundance in every nook and corner, but without its judicious utilization, there cannot be sustainable development. Food waste along with its by-products that aren't edible, contain many valuable products which may not only give us green alternatives, but also benefit the economy, especially of the developing nations.

Rice husk is an agricultural byproduct, i.e., an unavoidable food waste, which is abundantly available in rice producing countries. India produces nearly 12 million tons of rice husk annually. Rice husk has a high ash varying from 18-20%. Silica is the key component of rice husk ash varying from 85-95% [1].

A major problem for rice growers is the disposal of rice hulls. Until now they are discarded either by open incineration or burying. Uncontrolled burning of rice husk causes the ash, which is principally silica, to be converted into crystalline form and also renders it less reactive [2, 3]. Carbon monoxide (CO) is discharged into the atmosphere, by burning of rice hulls that is harmful to the environment as well as humans. Space for combustion is not easily accessible in densely inhabited regions and combustion generates adverse atmospheric effluence. However, there is a large quantity of biogenetic silica in rice hulls which has great potential economic gains for developing countries that have high amounts of rice hulls readily available and essentially, free of cost [3].

Several approaches have been developed to obtain silica from rice husk ash at low cost. It has been reported that the influence of chemical treatment on the purity of silica is higher [4] than thermal treatment [5]. Rice husk contains silica (SiO_2) in its hydrated amorphous form i.e. silica gel. By combusting rice husk at moderate temperature, the ash obtained has approximately 92-97% of amorphous silica and some metallic impurities that can be further removed by acid leaching treatment [6, 3]. Using rice husk as the raw material two million tons of pure high grade silica can be produced to meet the high demand of various industries utilizing it [7].

Silica gel has adsorption properties which have been credited to its surface hydroxyl groups. Usually, some are free silanol groups, i.e. free standing hydroxyl groups, whereas some are hydrogen bonded to adjoining silanol groups. It has been observed that for effective adsorption of water molecules, a high silanol number and balanced concentration proportionality between these two different types of hydroxyl groups is essential [8]. It is economical to extract silica from the silica- rich rice husk ash, which has wide market and also reduces the issue of ash clearance [9].

Silica is a bioactive element implicated as having fungicidal properties [10]. Numerous studies have revealed increased resistance to plant, fungal diseases in response to silicon applications. It has been proposed as a viable alternative to conventional control techniques. Silica can improve environmental stress tolerance and increase crop productivity [11, 12]. It has been shown to have increased resistance to powdery mildew in cucumbers [13, 14], in grapes [15], in muskmelons [16], as well as *Pythium spp.* and *Cladosporium spp*. in cucumbers [17]. In the recent past, in vitro studies have also been performed to investigate the antifungal effect of silica and in vitro inhibition of mycelial growth of several phytopathogenic fungi grown on potassium silicate amended media has been reported [10].

2. Material and Methods

2.1 Materials

Rice husk was collected from a rice mill in Uttar Pradesh. The rice husk was converted into rice husk ash (RHA) by incinerating the husk at 650ºC in a muffle furnace. All chemicals were of analytical grade and used without any purification. The chemicals used in the process were obtained from Sisco Research Laboratories (SRL) Pvt. Ltd and Himedia, Mumbai, India. Distilled water was used during the entire procedure.

2.2 Extraction Process
RHA was pre-treated with acid to remove metallic impurities and enhance silica purity. pH, of RHA samples dispersed in water, was adjusted to 7 using an acid (HCl, HNO_3, H_2SO_4). The samples were stirred for 2 hours, filtered and the residues were washed with water. After the pre- treatment, the residues were dispersed in NaOH and boiled for 1 hour. The solutions were filtered and the carbon residues were rinsed with boiling water. The washings were collected and cooled to room temperature, before titrating against HCl until the pH was 7. Silica precipitation started when pH was below 10. The precipitated silica gels were aged for at least 12 hours. The gels were crushed to form a slurry with deionized water, which was then centrifuged at 2500 rpm. The supernatant was discarded and the washing was repeated, if required. The gels were then dried in a beaker at 80ºC to form Xerogel.

2.3 Characterization of Silica
The functional groups in the extracted silica sample were examined by FTIR equipped with attenuated total reflectance (ATR) accessory in the range of 400-4000cm^{-1}.
The particle size of silica was determined by ZetaPlus Zeta Potential Analyzer. The metallic impurities in silica were analyzed with the help of Inductively Coupled Plasma- Mass Spectroscopy (ICP-MS).

2.4 Antifungal Assay
Antifungal activity of silica was tested on Malt Extract Agar (MEA) plates against molds (*Penicillium spp.*) isolated from bread using well diffusion test [18]. The bread was kept in damp conditions sealed in a plastic bag until fuzzy (blue-green mold) growth was observed. Fungal culture plates were prepared by isolating the area of bread, having blue green fuzzy growth. The isolated part was cut and placed onto MEA plates and incubated at 30ºC for 3-4 days.
The MEA plates were prepared. They were labelled and partitioned into two equal halves, to create wells. Mold sections, from previously cultured plates, were placed onto the plates. About 10g of silica gel was added to each plate and incubated for 3-4 days. The plates were observed regularly for mold growth and zone of inhibition.

3. Results and Discussion
3.1 Bio-Silica extraction from Rice Husk Ash (RHA)
The extracted Bio-Silica was in the form of sodium silicate after the addition of sodium hydroxide. As the pH was shifted from basic to neutral with the addition of hydrochloric acid, the sodium silicate started to precipitate. Addition of hydrochloric acid also removes the traces of metal impurities in RHA.

Fig 1: Silica obtained after Extraction process

The precipitated sodium silicate was aged in distilled water so that the silica particles are dispersed uniformly throughout the gel and to remove impurities and strengthen the gel. Thereafter, the gel was repeatedly washed and centrifuged to remove residual impurities and finally heated at 80ºC to form amorphous silica. The rice husk ash (RHA) produced after controlled thermal treatment of rice husk was white in color that suggested complete removal of carbonaceous compounds present in rice husk which would have interfered with the extraction of silica in later stages. The temperature was kept below 700ºC to enhance the possibility of extracting amorphous silica with high purity levels.

RHA produced was treated with alkali to solubilize silica present in rice husk to form a silicate compound that precipitated by the addition of acid, which also eliminated metal impurities, giving pure amorphous silica (Bio-Silica) after drying. The pH was closely monitored while precipitating silica gel and aged as long as possible to get rid of impurities that might lower the purity of extracted silica. 2.5 g of white silica powder was extracted from 10 g rice husk ash.

3.2 Fourier Transform- Infrared (FTIR) analysis of extracted Silica
The FTIR spectra of silica (extracted at 650°C) is shown in Fig. 2.

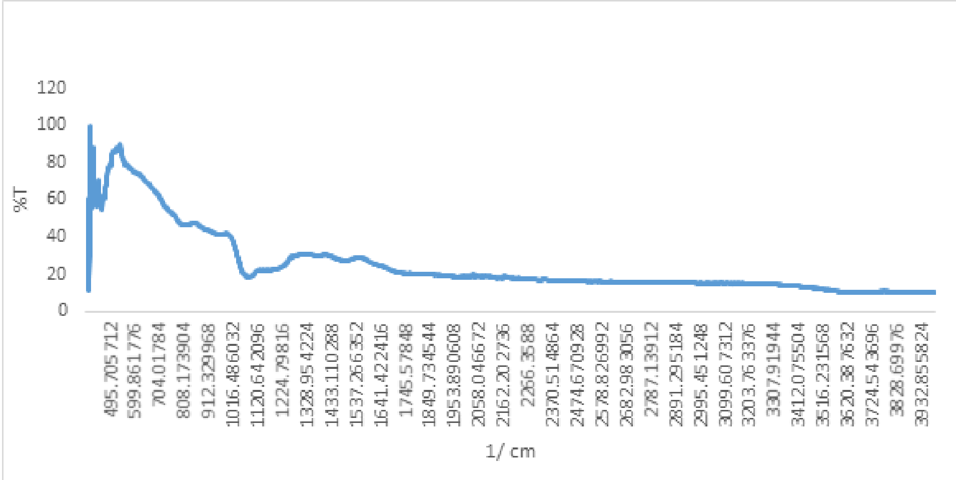

Fig 2: FTIR spectra of silica extracted from RHA

Table 1: Group frequency of silicone-oxy compounds (Interpretation of Infrared spectra [19])

Group Frequency (cm^{-1})	Functional group
1095–1075/1055–1020	Organic siloxane or silicone (Si-O-Si)
1110-1080	Organic siloxane or silicone (Si-O-C)

The range between 1076.27- 1095.56 cm^{-1} corresponds to Si-O-Si bonds, whereas 1080.13- 1109.69 cm^{-1} attributes to Si-O-C bonds in Fig. 3. Similar observations have been reported by Battegazzore *et al.* (2014), i.e. the Si–O–Si stretching and bending vibrations were detectable at 1047 and 800 cm^{-1} respectively [20].

3.3 Particle Size Analysis of extracted silica

The particle size of silica varied from 0.498 µm to 0.589 µm. This indicates that extracted silica particles are microparticles.

3.4 Inductively Coupled Plasma- Mass Spectrometry (ICP-MS) analysis of extracted silica

The ICP analysis detected the following trace metals in the extracted silica sample as shown in Table 2.

Table 2: ICP analysis of extracted silica

Element	Result (in ppm)
Chromium (Cr)	Not detected
Cobalt (Co)	Not detected
Nickel (Ni)	0.93
Arsenic (As)	Not detected
Cadmium (Cd)	Not detected
Antimony (Sb)	Not detected
Lead (Pb)	Not detected
Mercury (Hg)	Not detected

From the ICP analysis of the Bio-Silica, it is evident that there are no toxic heavy metals present in the sample except for nickel, which too is present within permissible limits. Though, this suggests that the rice husk was obtained from an area with high nickel content in either water/ soil. Thus to obtain pure Bio-Silica from RHA, it should be treated with activated carbon or other absorbents [21] to remove the residual nickel metal.

3.5 Antifungal activity of Bio-Silica

Fig 3: Isolation of *Penicillium spp.* from bread

Fig 4: Antifungal activity of Silica gel

Figure 3 shows the growth of isolated blue, green mold (*Penicillium spp.)* on malt extract agar plate. The inhibition zone was observed, though not uniformly distributed, in Figure 4 i.e. plate with silica gel in the wells. This result can be attributed to the addition of silica retarding fungal growth either by biochemical or physical mechanisms when host- pathogen interaction takes place like Silicon application induces the production of antifungal compounds after pathogen penetration of the epidermal cells [22]. The complete functioning of silicon towards retarding growth of fungi is still unknown both in plant systems as well as in vitro. A similar investigation was conducted wherein inhibition of fungal growth was reported with the use of potassium silicate [10].

4. Conclusion

This study illustrates that rice husk is not only an agricultural waste product, but a valuable by-product that can be used to extract amorphous silica, that has moisture adsorbing as well as antifungal properties. Amorphous silica is not easily available and is found in low concentrations in sand unlike crystalline silica that is naturally existing and plentiful. Though crystalline silica is naturally available, but due to its carcinogenic nature, its applications are restricted to certain fields including food industry. Meanwhile, amorphous silica that has been proven to be benign, can be used in food applications and various other fields where the particle size of amorphous silica is required like ink and rubber industries.

Due to improper disposal and burning approaches in developing countries where rice husk is burnt and used as fuel, rice husk ash, is a major environmental pollutant, though also very rich in silica (amorphous) content. Therefore, using RHA for extracting silica not only provides a valuable product but also helps in reducing the disposal problem, eventually having a positive impact on the nature by curbing pollution. Moreover, since the source of extracting (rice husk) amorphous silica is organic and a waste product it is non-hazardous and economical as the raw material has an almost negligible cost.

5. Acknowledgment

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6. References

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