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Sustainable Green Approach of Silica Nanoparticle Synthesis Using an Agro-waste Rice Husk

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ABSTRACT

Agro-waste can provide a non-metallic, environmentally friendly bio-precursor for the production of green silica nanoparticles. To manufacture silica nanoparticles from rice husk, biogenic silica nanoparticles were generated using an alkaline precipitation approach. Rice husk as a source of silica nanoparticles is environmentally and economically valuable because it is a plentiful lower price agricultural derivative that can be used to help with waste management. During the synthesis process, the dose of rice husk ash was used at 5 g at pH 7, alkali dose concentration of 0.5 M, reaction period of 3.5 h, and temperature of 90°C that produced maximum silica nanoparticles with a yield of 88.5%. To optimize the silica nanoparticle production from rice husk ash Box Behnken Design (BBD) a subcategory of the response surface methodology (RSM) was accomplished. BBD model was successfully matched, as evidenced by the high correlation values of adjusted R^2 0.9989 and predicted R^2 0.9977. Silica nanoparticles' amorphous form generated from rice husk ash is indicated by XRD analysis 2Ɵ peak at 22.12° and UV-Vis Spectroscopy absorbance peak at 312 nm. The amorphous shape of silica is amorphous and crystalline defined through XRD. nanoparticles generated from rice husk ash is indicated by FESEM analysis and EDX analysis, confirming that the SiO_2 elemental configuration comprises the highest concentration of Si and O. The existence of a siloxane group in the produced compound was revealed by FTIR spectra stretching vibrations at 803.69 and 1089.05 cm^{-1}

INTRODUCTION

Rice is among the greatest crops cultivated around the world since it is one of the most important food varieties and a supplier of nourishment for individuals. In contrast to 2020, the estimated global rice production has increased from 513.2 million tons to 519.7 million tons in 2021 (FAO 2021). As a result of generation and refining in the industry of agriculture, rice husk accounts for the major residue. Due to its characteristics like lignin, cellulose, hemicellulose content, hard surface, a small quantity of proteins, and high silicon quantity, rice husk can't be bacterial decomposed easily and is also insoluble in water (Soltani et al. 2015). In many countries, mostly rice husk has pointlessly burned and it is responsible for the air pollution problem. Additionally, composting of rice husk generates a huge quantity of methane. Rice husk contains approximately 20% of rice in weight ratio and incineration of these rice husks under 500-700°C of controlled temperature can generate ash and also amorphous silica (Kang et al. 2019). As compared to other crops, silica act as a unique crop residue in rice (Setiawan & Chiang 2021). The silica-separated rice husk is eco-accommodating as a result of its procurement from natural items and affordable because of the low-cost raw substance value.

The development of silica dioxide nanoparticles has drawn gigantic consideration in the world of science and technology in light of their broad use in different fields like biomedical fields, drug delivery systems, pesticides degradation, thermal insulators, humidity sensors, and electronic devices (Bharti et al. 2015, Bapat et al. 2016, Nazeran & Moghaddas 2017, Chong et al. 2018, Kano et al. 2019). According to previous studies, SiO_2 nanoparticles have been synthesized using natural resources like rice husk, marine sponges and diatom, coal fly ash, sand, and sugarcane bagasse (Sankar et al. 2018, Falk et al. 2019, Aphane et al. 2020, Ismail et al. 2021). One of these natural resources that are easily accessible in huge amounts is a byproduct of rice. There are different methods of silica nanoparticles synthesis like biotransformation method, microwave synthesis, thermal decomposition technique, laser ablation, chemical precipitation, plasma-assisted aerosol precipitation, sol-gel method, vapor-phase reaction, sonochemical synthesis, mechano-chemical method, hydrothermal synthesis, precipitation method, and pyrolysis, etc. (Krishna et al. 2021). Due to some environmental and technical problems like harsh experimental conditions, costly chemicals, and long-time and complex technologies in all these above syntheses, they are not environment friendly and economical to produce silica nanoparticles, thus, it is intriguing to build up flexible and elective techniques for acquiring nano silica from such biomass. There is a solution approach to a vital field of nanotechnology's potential applications for the present innovation. Furthermore, because the reuse of biomass assets has the potential to be extremely beneficial for eco-companion nanotechnology and nanoscience, the production of SiO_2 nanoparticles from rice husk has been extensively researched utilizing several exploratory methodologies. Chemical reaction techniques such as hydrothermal synthesis, acid-alkali leaching, microwave, combustion synthesis, precipitation, sonochemical, pyrolysis, and sol-gel are popular methods for producing SiO_2 nanoparticles from rice husk (Dubey et al. 2015, Sankar et al. 2016, Gao et al. 2017, Peres et al. 2018, Sankar et al. 2018, Almeida et al. 2019, Bui et al. 2020). When involving the biomass asset, rice husk is used to create silica as a siliceous substance at an unimaginable pace of gig tons/year, since rice husk contains an amorphous form of silica 90% (Hossain et al. 2018). After reviewing the literature, we found a persuasive and straightforward procedure for producing bio-created silica from a variety of rice husk sources. We used a rapid sonochemical method to demonstrate the properties of amorphous silica nanoparticles obtained from brown rice husk, which is one of the simplest methods for obtaining excellent silica of high quality from siliceous biomass reserves. The surface-to-volume ratio and microstructural size of silica oxide nanoparticles can be easily regulated by adjusting the sonication period throughout the sonochemical process. The optical, textural, morphological, and structural aspects of rice husk-derived silica oxide nanoparticles were investigated, as well as the impacts of sonochemical procedure duration on the objective properties (Sankar et al.2018). Because of the foregoing, rice husk is regarded as the greatest economically significant silica source.

Subsequently, we present a simple technique for synthesizing biogenic silica nanoparticles of high purity from rice husk and evaluated their biocompatible qualities. The silica nanoparticle was well characterized by different methods like X-Ray Diffraction, UV-VIS spectroscopy, Fourier Transform Infrared, Field Emission Scanning Electron Microscopy, and Energy Dispersive X-Ray. Silica nanoparticles have been made synthetically by various methods involving substance reductants.

MATERIALS AND METHODS

Material

For this study, rice husk (RH) was taken as a raw material from a rice mill. An analytical-grade chemical was used to make the nanoparticles.

Process of Silica Nanoparticles Synthesis

To remove dust and soil, rice husk was cleaned thoroughly with tap water adhering to it until the water was clear. The pH was then neutralized by washing it with distilled water. The rice husk was then rinsed and dried out in the sunlight for two days before being dried for 3 h at 90°C. The dried rice husk was ground into flour. The dried rice husk was then ignited at 600°C for 4 h to generate a grey powder of rice husk ash (RHA). A 500 mL NaOH (0.5M) solution was used to disperse the rice husk ash. For dissolving silica, stirring was done with a magnetic stirrer at 200 rpm for 3.5 h at a particular temperature of 90°C to form a solution of sodium silicate. Whatman no. 41 filter paper was used to filter the resultant solution. The filtrate from the sodium silicate solution was permitted to cool to room temperature. To generate silica precipitation, with regular stirring the sodium silicate solution was titrated with H_2SO_4 acid solution to pH 7. The solution was then agitated for one day before being aged for two days to let the silica gel to develop. Finally, using distilled water, the gel-containing solution was filtered, fragmented, and washed, yielding a fresh silica gel that was lyophilized overnight to get rid of water. For further characterization, the produced SiO_2 nanoparticles are stored in vacuum desiccators. Fig. 1 represents an alkali-based silica extraction process.

$$SiO_2 + 2NaOH \rightarrow Na_2SO_3 + H_2O$$
$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2.H_2O + Na_2SO_4$$

Optimization and Characterization of Silica Nanoparticles

For value addition, silica production from rice husk using alkali digestion was precisely carried out at optimum pH, alkali dose concentration, digestion time, temperature, and adsorbent concentration.

Optimization has a lengthy history of research, notably in the subject of operational analysis, which has resulted in

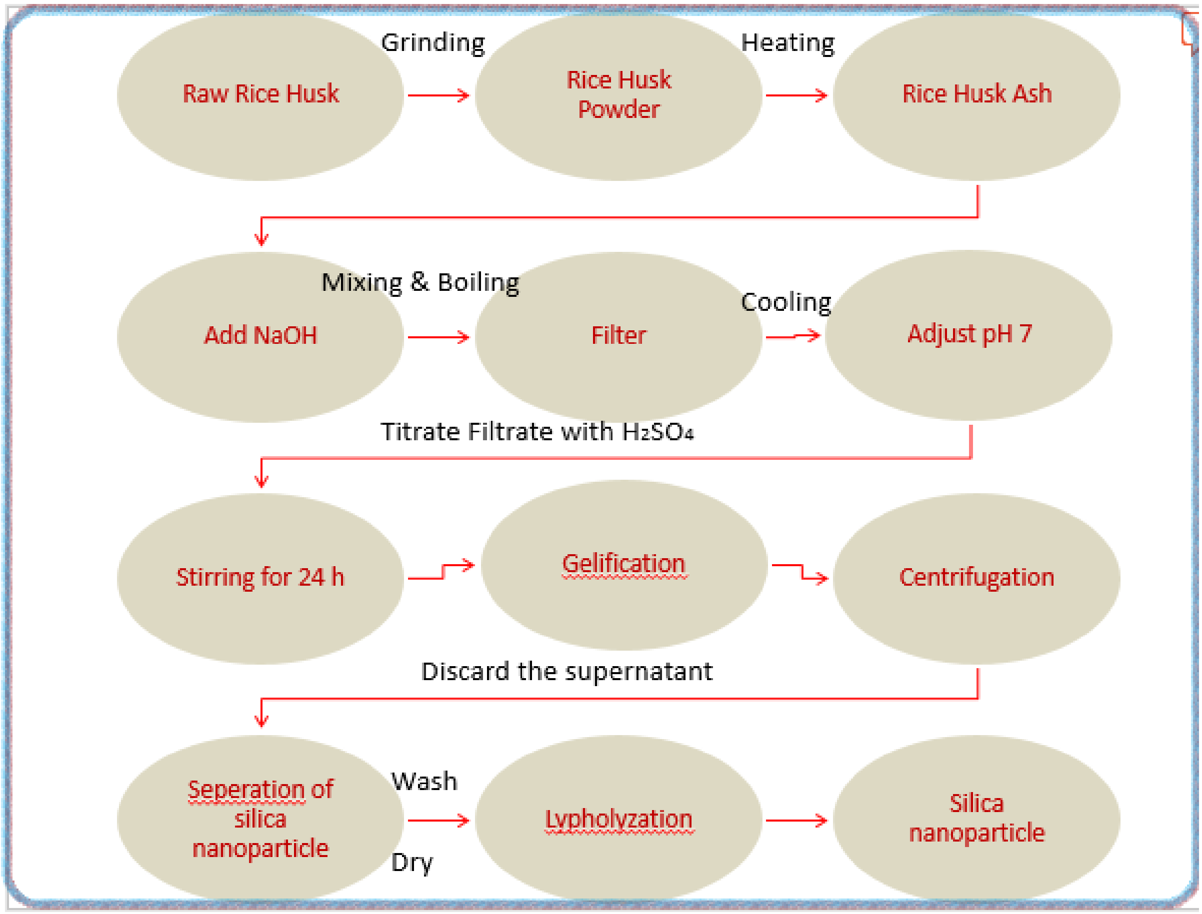


Fig. 1: Extraction process of silica nanoparticles from rice husk.

a plethora of methodologies. The influence of interaction among the elements is overlooked in traditional single-factor time testing because the experimenter modifies a single factor while keeping the other factors constant. Response surface methodology is a systematic analytical strategy for investigating the correlations between design features and responses to gain better overall knowledge with the fewest possible experiments (Cheng et al.2015). The Box Behnken generates designs that have favorable statistical features, allowing the quadratic model to be used. Response surface methodology optimizes processes and products by using quantitative data in an experimental design to discover and simultaneously solve multivariate equations. (Li et al. 2019).

The standard RSM, the Box–Behnken design model, for optimization, was built using Design Expert software 13. The optimum pH, NaOH concentration, temperature, time, and raw material dose were determined using a five-variable Box Behnken design. The design was chosen because it meets the majority of the requirements for silica nanoparticle production optimization. The fundamental goal of response surface methodology is to find the process's optimum practical conditions that meet the operating criteria. There were 46 experiments in the quadratic model's Box Behnken design (BBD). The design variables of BBD for silica nanoparticle production include pH (2-12), NaOH concentration (0.3-0.8 M), Temperature (40-130°C), time (2-5 h), and raw material dose (3-8 gm). To characterize the connection between independent variables and their observed responses, the equation employed was a quadratic polynomial (Cheng et al. 2015). The following is the model equation:

$$Y_{Pred} = \beta_0 + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n-1} \sum_{j=1}^{n} \beta_{ij} x_i x_j + \sum_{i=1}^{n} \beta_{ii} x_i^2 + e \quad \ldots(1)$$

Where, Y_{pred} is the approximation value of the reactive variable, β_0 is constant, β_i, β_{ii}, β_{ij} are the linear, quadratic, and interaction constants of the regression coefficients, and x_i and x_j are the independent variables in the form of coded values and e is the residual error. The consequences of the silica nanoparticle manufacturing were analyzed using model graphs and analysis of variance (ANOVA) like three-dimensional graphs, expected vs actual value plots, and contour plots.

The properties of the Silica nanoparticles like functional groups, phase identification, surface topography, size analysis, determining elemental composition and absorbance were determined using Scanning Electron Microscopy (JSM-7610F Plus, JEOL Japan), X-Ray Diffraction (Rigaku Miniflex-II Diffractometer, Japan), Energy Dispersive X-Ray Analysis (EDX), UV-Vis Spectroscopy (Shimadzu) and Fourier Transform Infrared (Spectrum Two, Perkin Elmer).

Optimization of Silica Nanoparticles Synthesis

Effect of pH: The influence of pH on silica extraction was studied from pH 2 to pH 10 using 5 gm rice husk ash dissolving in 0.5 M NaOH solution at 100°C for 4 hours. Silica extraction productivity improved when the pH was raised from 2 to 7, which was 29.56% to 88.5% (Fig. 2(a)). Further increases in pH did not lead to a significant increase in silica nanoparticle yield. The results show that the optimum pH of 7 results in a dose for getting the maximum output of silica nanoparticles from rice husk ash. This optimum pH for silica synthesis was 7. Similar findings were made by Yang et al. (2019) of China in their ing vestigation on mesoporous silica aerogels.

Effect of alkali dose: To study the NaOH concentration effect on the digestion of 5 g rice husk ash to manufacture silica nanoparticles varying dose of alkali was studied at pH 7 for 4 hours at 100°C. When the NaOH concentration was elevated from 0.3 M to 0.5 M, the silica elimination rose from 40.6 % to 88.5 %. (Fig. 2(b)). Further rise in the alkali dose did not result in a substantial increase in the production of silica nanoparticles. As a result, in the given digestion conditions, the optimum dose of NaOH concentration for silica extraction was found as 0.5 M. In their research, Ghorbani et al. (2015), of Iran study on silica nanoparticles, employed a similar NaOH concentration and observed similar results.

Effect of Digestion Time

The influence of processing time on silica extraction was studied for 5 grams of rice husk ash at pH 7, 0.5 M NaOH, and 100°C digesting temperature over time intervals ranging from 0.5 to 6 hours. It has been detected that the silica production was elevated from 16.91 % to 88.45 % when the digestion period was increased from 0.5 to 3.5 hours [Fig. 2(c)]. However, increasing the duration beyond 3.5 hours there was no considerable increase in silica nanoparticle extraction, hence 3.5 hours was considered to be the ideal reaction time for rice husk ash working dose aforementioned specified digestion states for silica extraction. According to Yang et al. (2019), in a China study on mesoporous silica aerogels, the optimal time was 4 hours since there was no significant extraction after that.

Effect of temperature: To study the influence of digestion temperature ranging from 30°C to 150°C on silica extraction from rice husk ash (5 gm) in 0.5 M NaOH solution at pH 7, with a digestion time of 3.5 hours. The silica extraction efficiency escalated from 14.8 % to 88.45% when the digestion temperature was elevated from 30°C to 90°C (Fig. 2(d)). However, an increase in the temperature did not result in a substantial increase in nanoparticle extraction. The optimum temperature for extraction of silica nanoparticles was found 90°C. Manaa (2015) from Egypt's study on silica products also noted comparable outcomes.

Raw material dose: A variable dose of 2 gm to 10 gm of rice husk ash was used to extract silica nanoparticles while keeping the alkali dose constant at 0.5 M NaOH and a digestion period of 3.5 hours at 90°C. As shown in Fig. 2(e), the raw dose concentration increasing from 2 to 5 gm increased the silica extraction from 54.8% to 88.5 %. Though, raising it after 5 gm did not yield a remarkable rise in silica nanoparticle extraction. As a consequence, a 5 g dose of rice husk ash was discovered to be ideal for the largest yield of silica nanoparticles. Ghorbani et al. (2015) revealed a 5.0 g optimum dose of rice husk ash in their research.

RSM-BBD Analysis

Design Expert Software 13 was used to analyze the data performance for regression analysis. The encoded versions of a second-order polynomial equation, Eq. (2), that reveals silica nanoparticle production is shown below:

Silica Production (%)

$$Y_{Pred} = +89.17 + 26\,A + 6.38\,B + 10.63\,C + 2.56\,D + 2.56\,E + 2.00\,AB + 2.00\,AC + 2.50\,AD + 0.0000\,AE - 4.25\,BC - 1.75\,BD - 1.50\,B - +1.25\,CD - 1.0000\,CE + 2.75\,DE - 31.29\,A^2 - 4.29\,B^2 - 6.79C^2 - 2.37\,D^2 - 1.04E^2 \quad \text{...(2)}$$

Fisher's F-test was accustomed to determining the arithmetical significance of the polynomial equation. Table 1 shows the study of data variability for the response surface quadratic model. Table 1 also includes the regression coefficients for the quadratic, linear, intercept, and interaction factors of the model. The p-values of the model terms were used to determine their significance. An F-test revealed that the model was extremely effective, with a Fvalue of 2122.78 and a p-value < 0.0001. The "lack-of-fit" F-value of 3.81

and p-value of 0.0716 suggested that the “lack-of-fit” was insignificant in comparison to the pure error.

In Predicted vs. Actual (Fig. 3), Contour (Fig. 4), and 3D response-surface (Fig. 5) plots show the kind of interactions between the five studied variables, as well as the relationship between experimental levels and responses of each variable.

Characterization of Silica Nanoparticles

Fourier transform infra-red (FTIR) spectroscopy: Fig. 6 shows the FTIR spectrum of silica dioxide nanoparticles. Specifically, the Si-O-Si vibration peak could be seen. The bending vibration, asymmetric stretching vibration, and symmetric stretching vibration are assigned to the transmittance peaks of Si-O-Si at 469.49 cm^{-1}, 1089.05 cm^{-1}, and 803.69 cm^{-1}, correspondingly (Mohd et al. 2017, Nandiyanto et al. 2016, Wibowo et al. 2017). The silica surfaces produced a broad peak at 3149.79 cm^{-1} due to the hydroxyl stretching vibration produced by the remaining adsorbed water and the silanol group vibration. (Chen et al. 2014). The H-O-H bond in molecular water is liable for the bending vibration, the band saw at roughly 1626.26 cm^{-1} (Chen et al. 2014). The carboxyl side groups show a symmetric stretching peak at 1406.01 cm^{-1} (Sarkar et al.2014). As in charged amines ($C=NH^+$), the 2359.78 cm^{-1} band exhibits NH^+ stretching (Lade et al. 2015). The findings are consistent with previous research on silica nanoparticles (Ghorbani et al. 2015, Manna 2015).

X-Ray powder diffraction: The SiO_2 nanoparticles XRD patterns made from rice husk ash shown in Fig. 7. The broad peak in the XRD pattern of rice husk ash at 22.12° established the amorphous nature of silica (Wibowo et al. 2017), which is appropriate for the formation of sodium silicate solution, however sharp peaks at 31.37°, 45.16°, 55.99°, and 75.02° specify the silica nanoparticles crystalline nature (Wahab et al. 2019). As a result of these XRD peaks, it was concluded

Table 1: Analysis of variance for response surface quadratic model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	22996.83	20	1149.84	2122.78	< 0.0001	significant
A-pH	10816.00	1	10816.00	19968.00	< 0.0001	
B-NaOH Concentration	650.25	1	650.25	1200.46	< 0.0001	
C-Temperature	1806.25	1	1806.25	3334.62	< 0.0001	
D-Time	105.06	1	105.06	193.96	< 0.0001	
E-Raw Material Dose	105.06	1	105.06	193.96	< 0.0001	
AB	16.00	1	16.00	29.54	< 0.0001	
AC	16.00	1	16.00	29.54	< 0.0001	
AD	25.00	1	25.00	46.15	< 0.0001	
AE	0.0000	1	0.0000	0.0000	1.0000	
BC	72.25	1	72.25	133.38	< 0.0001	
BD	12.25	1	12.25	22.62	< 0.0001	
BE	9.00	1	9.00	16.62	0.0004	
CD	6.25	1	6.25	11.54	0.0023	
CE	4.00	1	4.00	7.38	0.0118	
DE	30.25	1	30.25	55.85	< 0.0001	
A^2	8545.47	1	8545.47	15776.25	< 0.0001	
B^2	160.74	1	160.74	296.76	< 0.0001	
C^2	402.56	1	402.56	743.19	< 0.0001	
D^2	49.23	1	49.23	90.88	< 0.0001	
E^2	9.47	1	9.47	17.48	0.0003	
Residual	13.54	25	0.5417			
Lack of Fit	12.71	20	0.6354	3.81	0.0716	not significant
Pure Error	0.8333	5	0.1667			
Cor Total	23010.37	45				

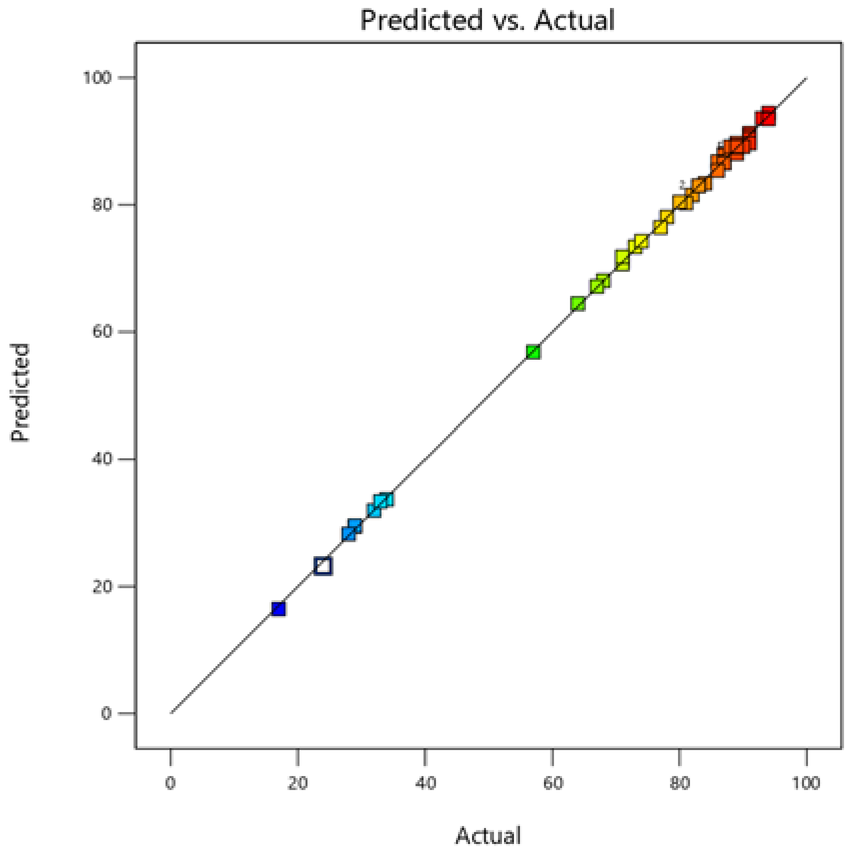


Fig. 3: Predicted vs. Actual plots.

that the rice husk contains a mixture of crystalline and amorphous silica phases. It was obvious that no other material impurities were present based on the peak positions of the observed spectra (Wahab et al. 2019). The creation of an amorphous form of silica nanoparticles has varied applications in our daily lives and adds to the beneficial effect (Raut & Panthi 2019). Peaks in the XRD pattern of silica nanoparticles generated by alkaline precipitation from rice husk ash are amorphous and contain a small proportion of crystalline silica (Wahab et al. 2019).

Scanning electron microscope (SEM) and EDX analysis: Scanning electron microscope images reveal the spherical shape structure of silica nanoparticles (Fig. 8). The silica nanoparticles produced were approximately 61.87 nm in size. A small portion of the generated SiO_2 particles formed an aggregation of SiO_2 nanoparticles, according to the findings. As a result, amorphous silica nanoparticles were discovered in nature, as seen in Fig. 8. Ahmad et al. (2017) also made similar observations.

EDX confirmed the chemical configuration of silica nanoparticles. The strongest peaks are displayed by oxygen and silica existent in silica nanoparticles, indicating that SiO_2 nanoparticles are generated. The non-appearance of other elements indicated that extensive washing with water had eliminated most of the soluble ions. During the thermal decomposition of rice husk, metal contaminants have also been from the rice husk transported along with the volatiles. Akhayere et al. (2019), reported similar results for synthetic silica in their elemental analysis.

UV-visible spectroscopy: The absorption band edge of silica nanoparticles by UV-visible spectroscopy was analyzed between 200 and 700 nm and a major adsorption band has been discovered at 312 nm with an absorbance of 1.91 (Fig. 9). The absorbance of nanoparticles is strongly influenced by wavelength and sample amount. The presence of silica nanoparticles is indicated by these observations, which lead to a Si-O-Si link. Patil et al. (2018) reported comparable findings.

CONCLUSIONS

The percentage yield of nano silica produced from rice husk ash at 600°C was 88.5%. The surface response approach using

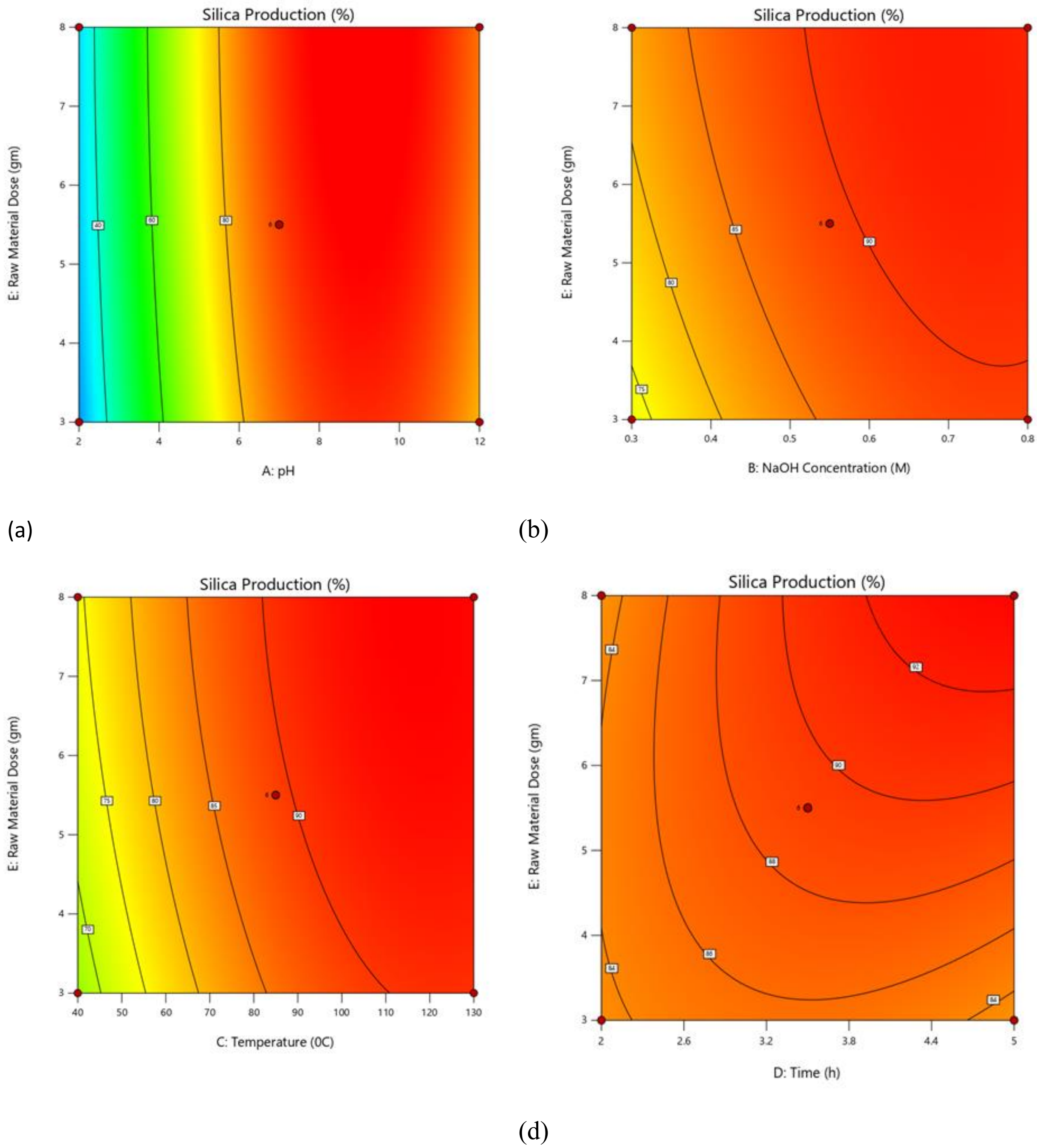


Fig. 4: Contour plots showing the interaction between (a) pH vs. raw material dose (b) NaOH concentration vs. raw material dose (c) Temperature vs raw material dose (d) Time vs. raw material dose.

the BBD model was successfully matched, as evidenced by the high correlation values of Adjusted R^2 0.9989 and Predicted R^2 0.9977. FESEM analysis of nano-silica particles from rice husk ash has shown its agglomeration form, with a particle diameter of 61.87 nm. The form of the particles was observed to be consistent. The presence of a significant broad peak at 22.12 on the XRD spectrum suggested that the nano-silica made from rice husk ash was mainly amorphous. The existence of hydrogen-linked groups siloxane and silanol in silica was established by FTIR data. The occurrence of O and Si in the ultimate formation, SiO_2 nanoparticles, was confirmed by EDX analysis. Biosynthesis

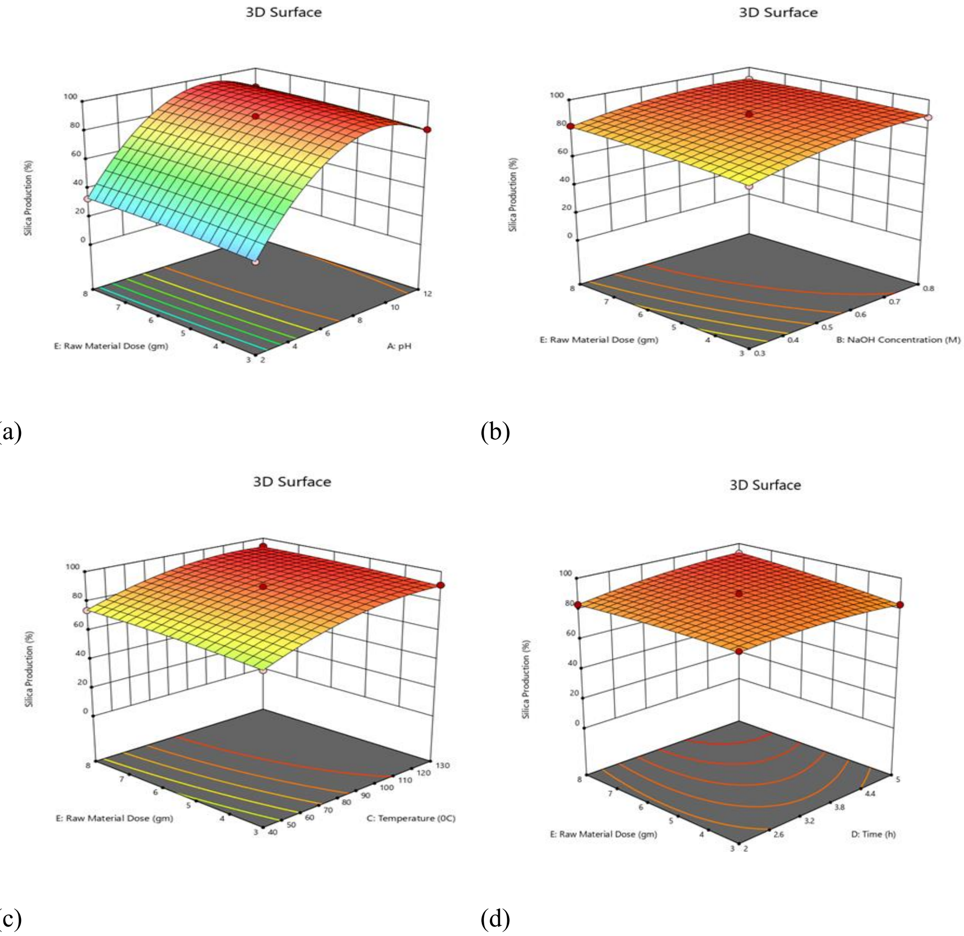


Fig. 5: 3D Response surface plots showing the interaction between (a) pH vs. raw material dose (b) NaOH concentration vs. raw material dose (c) Temperature vs. raw material dose (d) Time vs. raw material dose.

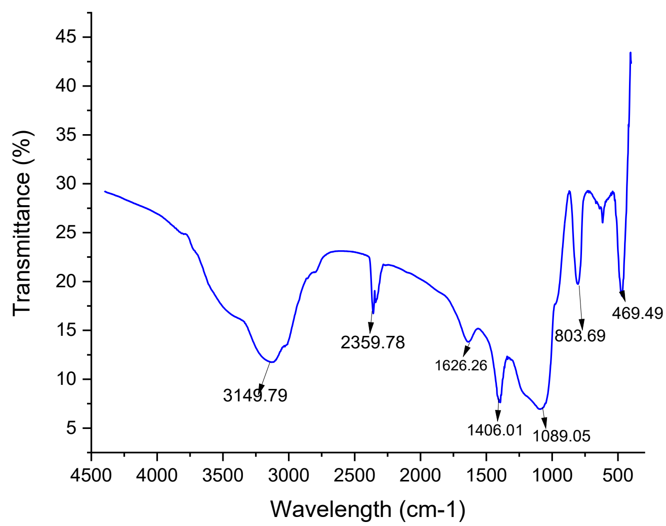


Fig. 6: FT-IR spectra SiO_2 nanoparticles prepared from rice husk.

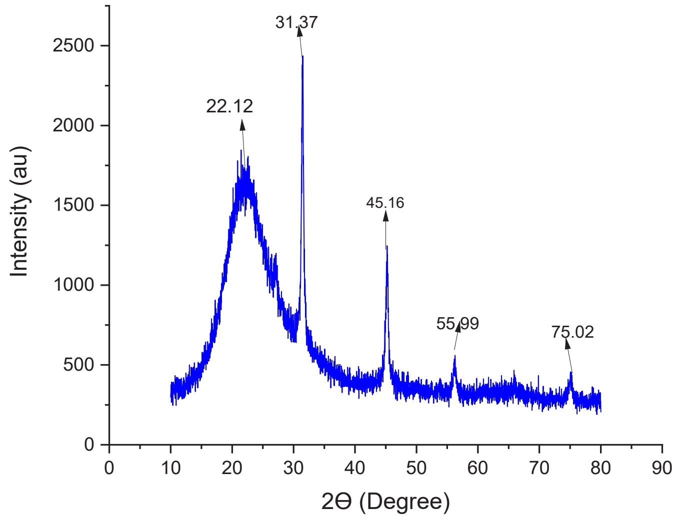
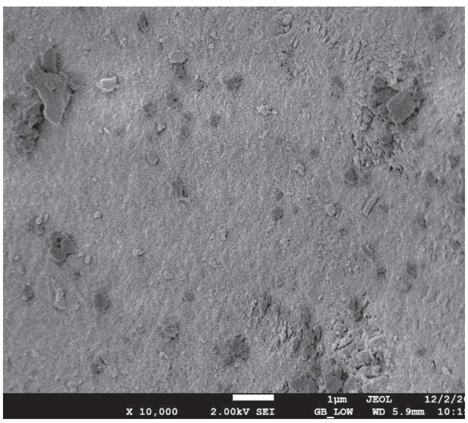
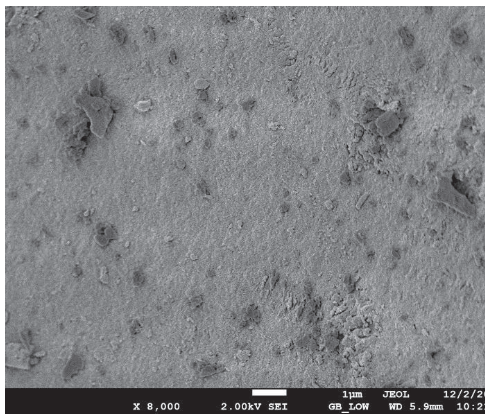


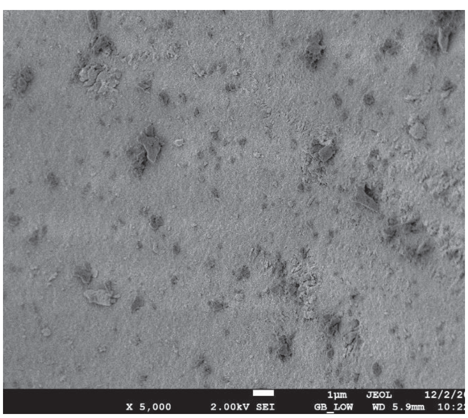
Fig. 7: XRD of SiO_2 nanoparticles prepared from rice husk.



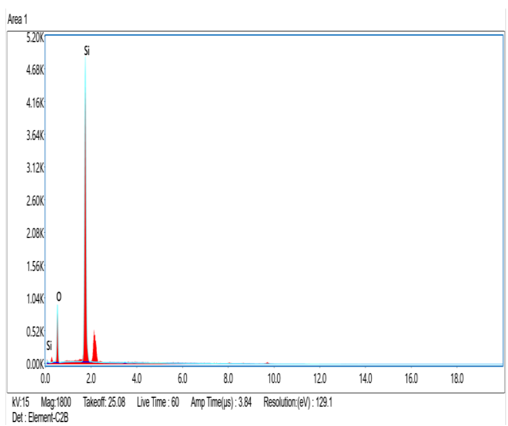
(a)



(b)



(c)



(d)

Fig. 8: (a) SEM images taken with 1 **µm** (10,000) index (b) SEM images taken with 1 **µm** (8,000) index (c) SEM images taken with 1 **µ**m (5,000) index (d) EDX of SiO_2 nanoparticles.

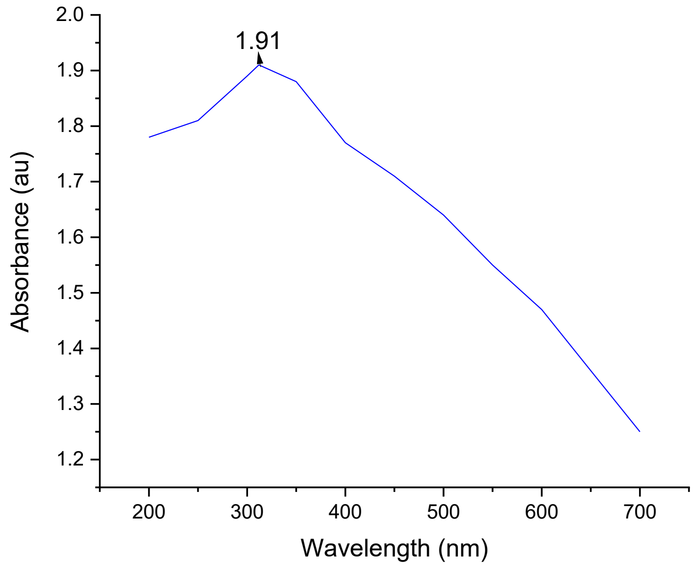


Fig. 9: UV-Visible Spectroscopy of SiO_2 nanoparticles procured from rice husk.

of silica nanoparticles using rice husk is an environmentally favorable, cost-effective green synthesis method. Rice husk as an origin of silica nanoparticles has a beneficial economic and environmental influence because it is a plentiful lower valuable agronomic by-product that can help with agro-waste clearance. In industry and agriculture, biosynthesized silica nanoparticles can be employed for a variety of applications.

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