



**SYNTHESIS OF ORDERED MESOPOROUS SILICA MCM-41
USING RICE HUSK ASH AS AN AGRO WASTE WITH
CONTROLLED MORPHOLOGY FOR POTENTIAL
APPLICATION IN CONTROLLED DRUG DELIVERY SYSTEMS**

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1. Introduction

In recent years, there has been a growing interest and focus on the development of advanced materials that possess controlled morphologies and exhibit desirable properties. This trend has been observed across a wide range of fields, including the field of pharmaceutical science. The ability to engineer materials with specific morphologies and properties has opened up new avenues for research and innovation. Scientists and researchers are now able to tailor the characteristics of materials to meet specific requirements. When considering the wide range of materials available for various applications, it is worth noting that mesoporous silica materials have emerged as particularly promising candidates, especially in the field of controlled drug delivery systems. These materials have demonstrated remarkable potential and have garnered significant attention from researchers and scientists alike. With their unique structural properties and characteristics, mesoporous silica materials offer a multitude of advantages: One type of mesoporous silica that has garnered considerable attention is MCM-41. This particular material has captured the interest of researchers and scientists alike due to its remarkable characteristics, namely its meticulously organised pore structure and its expansive surface area. These features make MCM-41 an exceptionally promising candidate for various applications, particularly in the realm of drug loading and release. The well-ordered pore structure of MCM-41 allows for efficient encapsulation of drugs within its porous framework, ensuring optimal drug loading capacity. Additionally, the high surface area of MCM-41 provides ample space for drug molecules to interact with the surrounding environment, facilitating effective drug release. These combined attributes make MCM-41 an ideal choice for enhancing the efficiency and efficacy of drug delivery systems. The process of synthesising mesoporous silica materials has traditionally relied on the utilisation of expensive and specialised chemicals, which can significantly impact the overall cost and complexity of the procedure. Over the past few years, there has been a growing interest among researchers in the exploration of alternative and sustainable sources for silica precursors. This shift in focus is driven by the need to find more environmentally friendly and economically viable options for obtaining silica, a vital component in various industries. Traditionally, silica precursors have been obtained from non-renewable sources such as quartz and sand. However, the extraction and processing of these materials often involve energy-intensive

methods and can have detrimental effects on the environment. As a result, scientists and engineers have been actively seeking out alternative sources that are both renewable and sustainable. One promising avenue of research involves the utilisation of agricultural waste. One example of a readily available and environmentally friendly source is rice husk ash (RHA), which is derived from the husks of rice grains. Rice husk ash is a byproduct of the rice milling process and is considered an abundant agricultural waste product. With the increasing demand for sustainable and renewable resources, RHA has gained attention as a potential raw material for various applications. Its abundance and easy accessibility make it an attractive option for industries looking to reduce their environmental impact. By utilising RHA, we can not only minimise waste but also tap into its potential benefits in different ways. Rice husk, which is a byproduct of the rice milling process, is often disposed of, resulting in significant environmental concerns. This waste material, consisting of the outer protective layer of the rice grain, is commonly overlooked and disregarded, despite its potential for various beneficial applications. Given the increasing global focus on sustainability and the urgent need to address environmental issues, it is crucial to explore innovative ways to utilise rice husk effectively and minimise its negative impact on the environment. On the other hand, it is worth noting that RHA, or rice husk ash, possesses a considerable quantity of silica, which renders it a highly appealing and environmentally sustainable substitute for the production of mesoporous silica materials. This characteristic of RHA holds great potential in various applications and industries, as it offers a viable solution that is both efficient and eco-friendly. By harnessing the silica content present in RHA, researchers and manufacturers can explore new avenues for the synthesis of mesoporous silica materials, thereby contributing to the advancement of sustainable technologies and reducing the reliance on traditional methods that may have adverse environmental impacts. Consequently, the utilisation of RHA as an alternative source for silica synthesis not only presents a promising solution but also aligns with the growing global emphasis on sustainability and eco-conscious practises.

In this study, we aim to synthesize ordered mesoporous silica MCM-41 using rice husk ash as an agro waste material. The utilization of RHA not only offers a sustainable solution for waste management but also provides a cost-effective alternative for the synthesis of mesoporous silica materials. Additionally, the controlled morphology

of the synthesized MCM-41 allows for potential applications in controlled drug delivery systems. The synthesis process involves the extraction of silica from rice husk ash and the use of cetyltrimethyl ammonium bromide (CTAB) as a templating agent to create the well-ordered pore structure of MCM-41. The resulting material will be characterized using various analytical techniques to assess its structural and morphological properties, including X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and BET surface area analysis. The controlled drug delivery system offers numerous advantages, including targeted and sustained drug release, improved therapeutic efficacy, and reduced side effects. By incorporating drugs into the mesoporous structure of MCM-41, we can achieve precise control over the release kinetics, allowing for personalized and optimized therapeutic regimens.

2. Experimental

2.1 Reagents

- ✦ Rice husk ash from Blitar: Rice husk ash pertains to the leftover ash acquired from incinerating rice husks, which is the shielding outer covering of rice kernels. In this scenario, the cinder from Blitar was utilised as an agricultural waste material.
- ✦ Organised permeable silica MCM-41: MCM-41 is a kind of mesoporous silica substance recognised for its systematic pore configuration. It is frequently employed in diverse applications, such as catalysis and adsorption. In this trial, the rice husk ash was utilised in the creation of organised permeable silica MCM-41, suggesting that it was probably employed as a precursor or supplement in the manufacturing procedure.
- ✦ Cetyltrimethyl Ammonium Bromide (CTAB): CTAB is a quaternary ammonium compound and a detergent. It is frequently employed as an emulsifying agent or a blueprint in the fabrication of mesoporous substances such as MCM-41. In this instance, CTAB was probably employed as an emulsifying agent during the creation of organised permeable silica MCM-41.
- ✦ Hydrochloric acid 37% (HCl): Hydrochloric acid is a potent acid frequently utilised in scientific facilities. The intensity of 37% suggests the potency of the acidic solution. It is probable that hydrochloric acid was employed in this trial for diverse intentions, such as modifying the acidity level or corroding the

silica substance.

- ✦ Acetic Acid (CH_3COOH): Acetic acid, alternatively referred to as ethanoic acid, is a feeble acid frequently present in vinegar. The expression frosty could allude to a compact or concentrated form of acetic acid. Its particular objective in the trial is not specified.
- ✦ Sodium hydroxide (NaOH): Sodium hydroxide is a potent alkali frequently employed in diverse chemical procedures. It can be utilised to modify the pH, counterbalance acids, or stimulate specific chemical reactions. Its particular function in this trial is not specified.
- ✦ Methyl alcohol 100% (CH_3OH) and 96% Ethyl alcohol ($\text{CH}_3\text{CH}_2\text{OH}$): Methyl alcohol and ethyl alcohol are both varieties of alcoholic beverages. They are frequently employed as solvents or reagents in chemical reactions. The reference to their distinct concentrations (100% for methanol and 96% for ethanol) suggests the immaculateness of the solvents employed.
- ✦ Aquadest: Aquadest is probably an abbreviated version of aqua purificata, which pertains to purified water. Purified water is utilised in laboratory environments to guarantee the lack of contaminants that might disrupt experiments.

2.2 Synthesized mesoporous silica MCM 41 from rice husk

The given details elucidate the amalgamation procedure for acquiring mesoporous silica MCM-41 from rice husk ash (RHA). Here is a sequential breakdown of the process:

1. Rice chaff (husk) was washed with hydrochloric acid (HCl) at 80°C for 2 hours to eliminate impurities. This stride aids in purging the rice husk prior to subsequent handling.
2. Following the rinsing process, the rice husk was purified and cremated at a temperature of 600°C for a duration of 6 hours. This combustion process led to the creation of rice chaff ash or rice husk ash (RHA). Combustion at elevated temperatures eliminates biological constituents and leaves behind the inorganic residue.
3. 6 grammes of RHA was dissolved in 100 ml of 2M sodium hydroxide (NaOH) solution. The concoction was subsequently filtered using permeable parchment to segregate any indissoluble contaminants.
4. The ensuing filtrate (containing RHA) was titrated with 2M hydrochloric acid (HCl) until a coagulate formed. The titration procedure persisted until the pH reached 7, signifying neutralisation.
5. The gel formed in the preceding step was

strained using permeable paper to eliminate surplus liquid. The strained gel was subsequently desiccated at 100°C for 6 hours, yielding the retrieval of silica from the rice husk.

6. 6 grammes of silica (SiO₂) was combined with 100 ml of 2M caustic soda (NaOH) solution. The concoction was warmed at 80°C in an oil bath for 24 hours. This measure aids in the disintegration of SiO₂ and the creation of sodium silicate (Na₂SiO₃).
7. Cetyltrimethyl Ammonium Bromide (CTAB) was dissolved in various concentrations (1%, 1.25%, and 1.5%) in 100 ml of purified water (aquadest). CTAB functions as a surface-active agent and aids in the creation of the intended mesoporous configuration.
8. Vinegar acid (acetic acid) was titrated into the sodium silicate solution until the pH reached 10. The concoction was stirred for 6 hours. This measure aids in regulating the acidity level and encouraging the hydrolysis and condensation reactions of silicate compounds.
9. The thermohydro process was conducted at 100°C for 24 hours. Hydrothermal circumstances aid in the creation and organisation of the mesoporous configuration.
10. Following hydrothermal processing, the material was aged for an extra 24 hours, probably at room temperature. This stride permits for additional architectural growth and consolidation.
11. The fabricated substance was subsequently purified using a blend of distilled water and ethyl alcohol. This measure aids in eliminating contaminants and leftover reagents from the substance.
12. Refluxing was conducted utilising a mixture comprising 9 ml of hydrochloric acid (37%) and 160 ml of methanol (CH₃OH) for a duration of 12 hours. Refluxing aids in additional refining and elimination of any lingering contaminants.
13. The remainder acquired from boiling was isolated using a centrifuge. Centrifugation enables the segregation of solid substances from the aqueous phase.
14. The isolated remainder was desiccated at 80°C for 2 hours, probably utilising an oven or comparable apparatus. Desiccation eliminates all residual dampness and guarantees the durability of the synthesised mesoporous silica substance.

In general, this amalgamation procedure led to the production of organised porous silica MCM-41 from rice husk ash. The acquired substance

possesses a regulated formation, rendering it appropriate for potential uses in managed medication distribution systems and other domains necessitating managed permeability.

2.3 Characterization

The portrayal methods utilized in the investigation included Fourier transform infrared spectroscopy (FTIR) using Thermo Scientific Nicolet IS10 apparatus to ascertain the chemical bonding, x-ray diffraction (XRD) analysis conducted on PanAnalytical X'Pert Pro system to ascertain the crystalline structure. Microscopic examination was carried out employing scanning electron microscopy (SEM) with FEI Inspect S50 and transmission electron microscopy (TEM). For the resolution of distinct surface region, cavity magnitude, and cavity arrangement, the Brunauer-Emmett-Teller (BET) nitrogen adsorption-desorption technique was employed. This examination was conducted using a Quantochrome surface evaluator. These portrayal techniques provided valuable data on the chemical bonding, crystalline arrangement, microstructure, and surface characteristics of the synthesized ordered mesoporous silica MCM-41 material derived from rice husk ash.

3. Result and Discussion

Illustration 1 exhibits the FTIR spectra of unadulterated CTAB and CTAB-induced silica mesoporous substances. The FTIR spectrum of unadulterated CTAB displayed distinctive peaks at frequencies of 2915.75 cm⁻¹, 2848.39 cm⁻¹, 1472.87 cm⁻¹, 960.31 cm⁻¹, 911.15 cm⁻¹, and 718.79 cm⁻¹. These summits corresponded to oscillatory bands of CH, CH₂, N(CH₃)₂, and CH functional clusters. Prior investigation conducted by Becerra (2012) documented analogous infrared spectra of the surfactant CTAB. The balanced (νs-(CH₂), d+) and dissimilar (νas(CH₂), d-) stretching oscillations of unadulterated CTAB were detected at 2849 and 2918 cm⁻¹, suggesting the existence of identical gauche imperfections. The crests at 3017 and 1487 cm⁻¹, and at 1473 and 1462 cm⁻¹, were ascribed to the uneven and even C-H oscillation movements of CH₃-N⁺ fragments and the CH₂ oscillation mode, respectively. The FTIR spectrum of CTAB-templated silica with different CTAB proportions (1%, 1.25%, and 1.5%) displayed noticeable wave frequencies at 3388.31 cm⁻¹, 1636.71 cm⁻¹, 1070.46 cm⁻¹, 956.95 cm⁻¹, 795.09 cm⁻¹, and 456.15 cm⁻¹. As per the Aldrich compendium repository, these wavenumbers corresponded to functional groups such as -OH, Si-O-Si, Si-OH, and Si-O. In contrast

to the unadulterated CTAB spectrum, the FTIR spectrum of CTAB-induced silica did not exhibit C-H oscillatory bands or other peaks detected in the unadulterated CTAB spectrum.

The handling of CTAB-structured silica with methanol reflux efficiently eliminated the surfactant template, as apparent from the lack of unadulterated CTAB functional groups in the silica reflux findings. The inclusion of CTAB to the extracted silica did not exhibit wavenumbers corresponding to the -CH₂-, N(CH₃)₂, and -CH₂-moieties. Furthermore, the existence of additional surface hydroxyl groups amplified the hydrophilic characteristic and altered the responsive capability of the pore channels, resulting in the creation of altered CTAB-templated silica mesoporous substances.

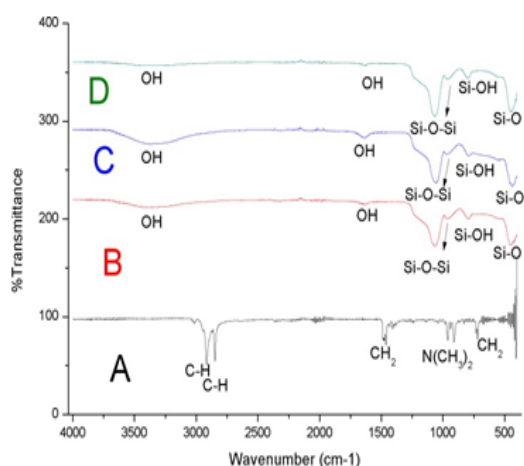


Figure 1. FTIR result of (A) pure CTAB, silica reflux results in concentration (B) 1% CTAB, (C) 1.25% CTAB and (D) 1.5% CTAB.

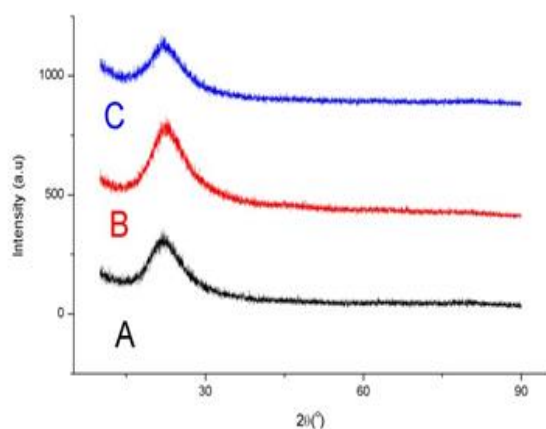


Figure 2. X-Ray diffraction curve of MCM 41 silicamesoporus with surfactant concentration (A) 1% CTAB, (B) 1.25% CTAB and (C) 1.5% CTAB.

Figure 2 displays the X-ray diffraction (XRD) charts of various CTAB-templated silica samples, including 1% CTAB (Representation 2.A), 1.25% CTAB (Representation 2.B), and 1.5% CTAB (Representation 2.C). The X-ray diffractometer (XRD) profile of 1% hexadecyltrimethylammonium bromide (CTAB)-templated silica exhibited a peak diffraction at $2\theta = 22.0155^\circ$. Similarly, the XRD patterns of 1.25% CTAB and 1.5% CTAB-templated silica displayed analogous curves to that of 1% CTAB. All XRD patterns corresponded to the PDF card standard for the silica-cristobalite phase (ICDD # 00-001-0424). The diffraction patterns displayed similarities in shape (Figure 2), with diffraction peaks broadening around $2\theta = 22-24^\circ$, as observed in the diffraction pattern of the amorphous phase of silica.

Amorphous silicon dioxide is commonly found in organisms such as diatoms, radiolarians, silicoflagellates, and particular sponges. Amorphous or formless silica displays an arrangement of particles and compounds in a random and chaotic framework. Another inquiry has revealed that pinnacle broadening also indicates the formation of a porous structure. This hypothesis can be further confirmed through additional characterization techniques like TEM and BET surface analyzer.

Based on Figure 2, the diffraction peaks broaden further as the CTAB concentration increases. The 1.5% CTAB sample exhibits the widest diffractive peak when compared to the other samples. The widening of the diffraction peaks suggests the formation of shapeless structures triggered by pore creation assisted by the CTAB plan. Wanyika et al. (2011) have clarified that wide-angle X-ray diffraction (WAXRD) patterns reveal a chaotic-like profile corresponding to the presence of substantial voids in the material. Similar patterns are observed in Figure 2, indicating the porous structure of the silica. In essence, the peak broadening in the diffraction peaks of CTAB surfactant-templated silica is attributed to the presence of voids within the silica structure. Figure 3 exhibits scanning electron microscope (SEM) images of various weight ratios (wt. %) of CTAB-templated silica, including 1% CTAB (Figure 3.A), 1.25% CTAB (Figure 3.B), and 1.5% CTAB (Figure 3.C). The SEM images reveal a coral-like structure for the CTAB-directed silica. In Figure 3.A, the silica particles exhibit dimensions ranging from 476 nm to 627 nm, with an average particle size of 482 nm. Figure 3.B exhibits silica particles ranging from 343 nanometers to 542 nanometers, with

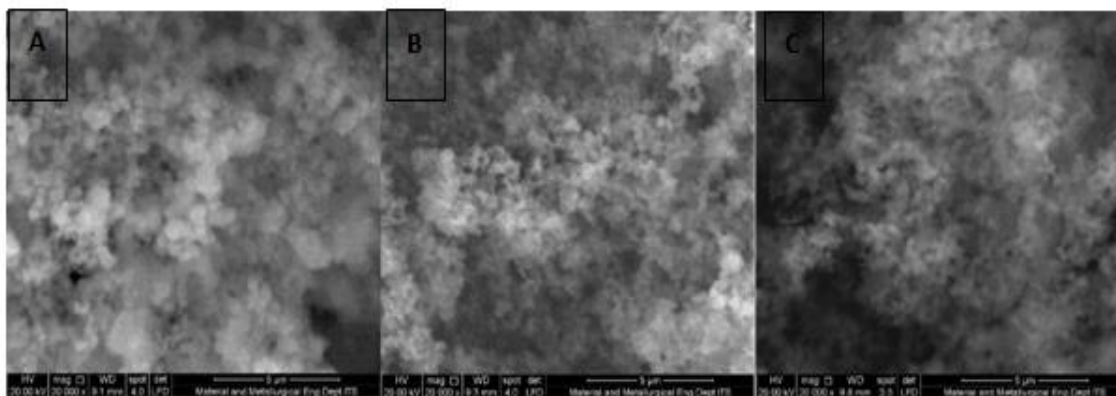


Figure 3. SEM images of silica with (A) 1% CTAB, (B) 1.25% CTAB, and (C) 1.5% CTAB.

Increasing the concentration of CTAB promotes the generation of micelle structures, resulting in stronger linkages between silica and surfactants. At diminished CTAB concentrations, silica particles that do not stick to surfactants have a propensity to adhere to Si-O-Si clusters, leading to amplified silica particle proportions. Zhao et al. (2013) clarified the process of mesoporous material creation, in which the $Rn-N+(R)3$ cluster attaches to silica on the entire outer surface. Silica generates elongated Si-O-Si filaments that metamorphose into particles.

Figure 4 displays TEM images of CTAB-templated silica at varying concentrations, revealing the presence of pore formations initiated by CTAB within the silica particles. Figure 4.A (1% CTAB) exhibits silica with a pore size of 3.58 nm, while silica-1.25% (Figure 4.B) and silica-1.5% (Figure 4.C) showcase pore sizes of 3.13 nm and 3.12 nm, respectively. The TEM images furthermore exhibit a honeycomb or hexagonal pore configuration, consistent with the characteristics of mesoporous silica MCM-41 observed in other inquiries.

Table 1 showcases the N₂ adsorption-desorption investigation (BET analysis) of the various CTAB-templated silica samples. The data illustrate that the

magnitude tends to increase with heightened CTAB concentrations. The entire pore volume also demonstrates an increasing trend with heightened CTAB concentrations, consistent with previous studies. Increasing the CTAB concentration allows for more templates to adhere to Si-O-H functional groups. Consequently, when eliminating the blueprint, a greater amount of pores are generated as the degree of the surfactant escalates. Yun-yu et al. (2012) mentioned that increasing the CTAB concentration leads to an increase in the surface area of the produced mesoporous silica.

Table 1 demonstrates that the superficial area data in this study align with the findings of Yun-yu et al. (2012), as the incorporation of CTAB concentration tends to augment the superficial area of the mesoporous silica, except for the 1.5% CTAB-templated silica. Further comprehensive examination is necessary to delve into the effects of integrating CTAB concentrations exceeding 1.5%. The TEM picture and BET surface analyzer results for 1.5% CTAB confirm that it falls under the category of mesoporous materials, with a pore size of approximately 3.12 nm as per TEM inspection and 5.814 nm as per BET surface analyzer.

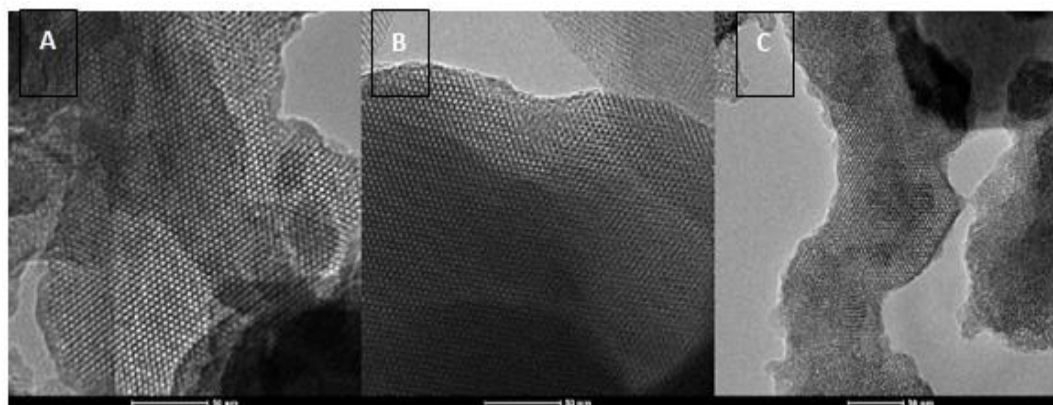


Figure 4. TEM results from silica with concentration (A) 1% CTAB, (B) 1.25% CTAB an (C) 1.5% CTAB.

Table 1. Surface area, average diameter pore size, and total pore volume of silica samples with the addition of 1%, 1.25%, and 1.5% CTAB

No	Sample	Surface (m ² /g)	Area	Average diameter pore size(nm)	Total pore volume (cc/g)
1	1%	552.429		7.58248	1.047
2	1.25%	768.947		6.07107	1.167
3	1.50%	705.501		5.8142	1.025

The table provides the results of BET surface analysis for the CTAB-templated silica samples synthesized at different CTAB concentrations. BET analysis is a technique used to measure the specific surface area, average pore size, and total pore volume of porous materials. The specific surface area is an important parameter as it indicates the total area available for adsorption. In this study, as the CTAB concentration increases, the surface area also tends to increase. The sample with 1.25% CTAB concentration shows the highest surface area of 768.947 m²/g, indicating a larger surface available for interactions. The average pore size is a measure of the dimensions of the pores within the silica samples. It reflects the size of the empty spaces or voids present in the material. In this study, the average pore size tends to decrease as the CTAB concentration increases. The sample with 1% CTAB concentration has the largest average pore size of 7.58248 nm, suggesting the presence of relatively larger pores compared to the other samples. The total pore volume is a measure of the overall capacity of the material to hold fluids or molecules within its pore structure. The results in the table show some variation in the total pore volume with CTAB concentration, but there is no clear trend observed. The sample with 1.25% CTAB concentration exhibits the highest total pore volume of 1.167 cc/g, indicating a greater volume available for adsorption or storage of substances.

4. Conclusion

An extraordinary accomplishment has been achieved in effectively recovering the highly desired MCM-41 silica permeable material from the plentiful and frequently disregarded origin of rice husk ash. The synthesised MCM-41 silica material displays a range of functional entities, such as hydroxyl clusters (-OH), silicon-oxygen-silicon linkages (Si-O-Si), silanol clusters (Si-OH), and silicon-oxygen linkages (Si-O). These operative clusters perform vital functions in determining the characteristics and possible uses of the MCM-41 silica substance. Whilst scrutinising the particle dimensions, it has been noted that the tiniest magnitude of particles is detected at a concentration of 1.5% of CTAB. This measurement is taken using a contraption that

measures at a wavelength of 311 nm. Furthermore, the pore dimension of these particles is established to be 3.12 nanometers. The material, upon careful scrutiny, exhibits a captivating and elaborate honeycomb or hexagonal pore arrangement. This distinct configuration of pores, resembling the form of a beehive or a hexagon, incorporates a visually enchanting aspect to the material. The existence of this particular pore arrangement not just improves the substance's visual allure but also implies a possibility for proficient and successful movement or dispersion within its composition. The honeycomb or hexagonal When contemplating the diverse concentrations that were scrutinised, it was discovered that the utmost surface area was acquired with a concentration of 1.25% CTAB. This specific concentration produced an extraordinary surface area calculation of 768.947 m²/g. After scrutinising the specimen comprehensively, it has been ascertained that the average pore dimension of this specific sample measures exactly 6.07107 nanometers. Furthermore, the pore volume of the sample has been computed to be roughly 1.167 cubic centimetres per gramme. These measurements offer valuable perspectives into the intricate traits and qualities of the specimen, illuminating its distinct composition and potential uses in diverse scientific and industrial domains. This implies that the CTAB surfactant efficiently acts as a template for creating voids in the silica structure, resulting in the synthesis of mesoporous silica nanoparticles. In general, the victorious fusion of MCM-41 silica porous material employing rice husk ash as a precursor, alongside the control of particle size, pore diameter, and surface area through the modification of CTAB concentration, highlights the potential of these materials for various uses, especially in the domain of controlled drug delivery systems.

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