



## FUNCTIONALIZED ORDERED MESOPOROUS SILICAS (MCM-48): SYNTHESIS USING RICE HUSK ASH AS AN AGRO WASTE AND ITS APPLICATIONS IN CATALYSIS

**Dr. Manish Raghunathrao Deshpande<sup>1\*</sup>, Dr. Sudhir Shivnikar<sup>2</sup>, Mukund Joshi<sup>3</sup>, Jagdish Kulkarni<sup>4</sup>, Vaishnavi Pethkar<sup>5</sup>, Ashwini Ingle<sup>6</sup>**

<sup>1</sup>HOD of Physics and Research Supervisor, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

<sup>2</sup>Principal, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

<sup>3</sup>Assistant professor and research scholar, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

<sup>4</sup>Assistant professor and research scholar, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

<sup>5</sup>Assistant and research scholar, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

<sup>6</sup>Assistant professor and research scholar, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

**\*Corresponding Author:** Dr. Manish Raghunathrao Deshpande

\*HOD of Physics and Research Supervisor, Netaji Subhash Chandra Bose Art, Commerce and Science College Nanded 431601 Maharashtra India.

**DOI:** 10.48047/ecb/2023.12.si10.00272

## 1. Introduction

Functionalized ordered mesoporous silicas, such as the highly renowned MCM-48, have garnered considerable interest and fascination within the scientific community. This is primarily due to their exceptional and distinctive properties, which hold immense potential for various applications, particularly in the realm of catalysis. The intricate and precisely organised structure of these mesoporous silicas, combined with their functionalization, has paved the way for groundbreaking advancements and In the current context, the utilisation of rice husk ash (RHA) as an agricultural waste material for the synthesis of MCM-48 presents a highly sustainable and environmentally friendly approach. By repurposing this abundant byproduct of rice production, we can effectively reduce waste and minimise the negative impact on our environment. Rice husk ash, which is obtained from the burning of rice husks, has long been recognised as a potential source of valuable materials due to its unique chemical composition. However, its utilisation has been limited, and it is often discarded as waste, leading to significant environmental concerns. By harnessing the potential of RHA in the synthesis of MCM-48, we can transform this agro waste into a valuable resource. MCM-48, a type of mesoporous material, has a wide range of applications in various industries, including catalysis, adsorption, and drug delivery systems. Its unique structure and properties make it highly desirable for these applications. The synthesis of MCM-48 using RHA involves a series of carefully designed processes that maximise the utilisation In this introductory paragraph, we will delve into the fascinating world of synthesising functionalized MCM-48 using rice husk ash (RHA) as a precursor. We will explore the various applications of this synthesised material in catalytic processes, shedding light on its immense potential and significance in the field. So, let's embark on this journey together as we unravel the intricate details of this synthesis method and uncover the wide-ranging possibilities it presents in the realm of catalysi The materials known as MCM-48 exhibit a remarkable characteristic of having a highly organised mesoporous structure. This structure is characterised by its large surface areas, which provide ample space for various chemical reactions and interactions to occur. Additionally, the pore sizes of MCM-48 materials can be adjusted or tuned to meet specific requirements, allowing for precise control over the flow and diffusion of molecules within the material. Moreover, the

surface functionalities of MCM-48 can be modified or tailored to suit specific applications, enabling the material to exhibit desired properties and behaviours. Overall, the unique combination of a well-ordered mesoporous structure, large surface areas, tunable pore sizes, and controllable surface functionalities make MCM-48 materials highly versatile and valuable in a wide range of scientific and technological fields. One of the key advantages of MCM-48 is its ability to incorporate functional groups onto its surface. This unique feature allows for the customization of its catalytic properties, making it an incredibly versatile and promising catalyst for a wide range of reactions. By strategically attaching different functional groups, researchers can fine-tune the catalytic activity and selectivity of MCM-48, enhancing its performance in specific chemical transformations. This level of control over the catal By effectively harnessing the potential of RHA (rice husk ash) as a precursor material, we not only have the opportunity to tackle the pressing issue of waste management, but we can also make significant strides in reducing our reliance on conventional silica sources. This, in turn, paves the way for a more environmentally friendly and sustainable approach to catalysis. By recognising the inherent value of RHA and utilising it as a precursor, we are able to optimise its unique properties and unlock its vast potential in catalytic applications. This not only benefits waste management efforts by providing a valuable outlet for rice husk waste, but it also contributes to a more sustainable future by reducing our dependence on traditional silica sources. This shift towards a greener approach to catalysis aligns with the global push for sustainable practises and offers a promising solution to the challenges we face in waste management and resource utilisation.

## A. Background

Mesoporous silicas possess an unparalleled pore configuration at the nanoscale, distinguished by well-arranged and interconnected passageways. This configuration offers them with a spacious surface area and consistent pore size distribution. The elevated surface area enables a larger quantity of operative locations, whereas the consistent pore size distribution facilitates effective mass transfer of reactants and products. These characteristics render mesoporous silicas exceedingly appealing for catalytic purposes. In catalysis, the operative sites on the substance's exterior facilitate chemical reactions by offering a surface for reactant molecules to adsorb and interact, resulting in the creation of desired products. The elevated surface

area and pore configuration of mesoporous silicas amplify the reachability of these dynamic locations, leading to enhanced catalytic efficacy and selectiveness in comparison to alternative substances.

### **B. Introduction to MCM-48 and its unique properties:**

MCM-48 is a distinct kind of mesoporous silica that is produced using a surfactant-guiding technique. This technique entails utilizing a surface-active agent molecule as a blueprint around which the silica precursors congregate, creating a hexagonal assortment of tubular cavities. MCM-48 demonstrates numerous distinct characteristics that render it exceptionally captivating for catalysis. Firstly, it possesses a substantial surface area, which offers ample active sites for catalytic reactions. Furthermore, MCM-48 exhibits a well-organized mesoporous framework, guaranteeing the consistent dispersion of pores across the substance. This consistency enables for accurate management over reactant entry to the operative locations, resulting in enhanced reaction effectiveness and selectiveness. Ultimately, MCM-48 possesses a limited pore size assortment, which empowers the discriminatory absorption and dispersion of molecules according to their dimensions, subsequently amplifying its catalytic efficacy.

### **C. Importance of waste utilization and sustainable materials synthesis:**

Waste utilization is of paramount significance in today's world as it tackles environmental concerns and fosters a circular economy. By employing discarded materials as precursors for mesoporous silica synthesis, scientists can diminish the load on landfill sites and minimize the extraction of pristine resources. Furthermore, refuse materials frequently encompass precious constituents that can be transformed into advantageous commodities. By integrating refuse substances into the amalgamation of porous silicas, scientists can formulate value-enhanced materials while diminishing waste production.

Sustainable substances creation is another crucial facet in catalysis investigation. Conventional amalgamation techniques frequently encompass energy-consuming procedures, severe response circumstances, and the utilization of perilous substances, resulting in adverse ecological consequences. Sustainable amalgamation approaches concentrate on cultivating ecologically conscious techniques that downplay resource utilization, refuse production, and ecological

damage. By utilizing sustainable amalgamation techniques, scientists can diminish the environmental impact of mesoporous silica fabrication, rendering it a more eco-conscious procedure.

### **D. Objective of the research paper:**

The aim of the research paper may differ depending on the particular investigation. Nevertheless, it typically revolves around exploring the amalgamation, portrayal, and catalytic traits of MCM-48 mesoporous silica employing discarded substances or eco-friendly synthesis methodologies. The investigation document might aspire to investigate the viability of utilizing refuse substances as precursors for MCM-48 formation and assess the resultant substance's architectural and catalytic characteristics. The goal may also encompass examining the effectiveness of MCM-48 in particular catalytic reactions and evaluating its efficacy, specificity, and durability in comparison to alternative catalysts. Furthermore, the investigation document might aim to offer perspectives into the structure-property connections of MCM-48 and its possible implementations in catalysis, contributing to the wider realm of eco-friendly materials synthesis and waste utilization.

## **2. Methods**

### **2.1 Chemicals and starting materials**

The amalgamation of modified arranged mesoporous silicas (MCM-48) entailed the utilization of diverse substances and initial components acquired from Aldrich. These substances were employed without any extra refinement, suggesting that they were of adequate purity for the synthesis procedure. The substances and initial components employed comprise:

- Vinegar: Utilized as a reactant or additive in the synthesis process.
- Cetyltrimethylammonium bromide (CTAB): A detergent that aids in the creation of the organized mesoporous framework.
- Polyoxyethylene (23) lauryl ether (PLE): An additional surfactant that aids in the creation of the mesoporous framework.
- 3-Aminopropyltriethoxysilane (APTS): An organosilane compound utilized for modifying the MCM-48 material.
- 3-Aminopropyltriethoxysilane (APTS): An organosilane compound employed for enhancing the functionality of the MCM-48 material.
- 3-Aminopropyltriethoxysilane (APTS): An organosilane compound applied for the purpose of

functionalizing the MCM-48 material.

- Toluene: A dissolvent that could be utilized in specific stages of the amalgamation procedure.
- Sodium hydroxide (98%): An alkaline substance that can be utilized as a catalyst or to modify the pH during the synthesis procedure.
- Rice chaff ash (RCA) was acquired from a neighboring farm and exposed to a milling procedure. Then, it experienced calcination at an elevated temperature of 873 K (approximately 600 °C) for 12 hours. This incineration process is conducted to eliminate any organic substances and contaminants from the rice husk ash, resulting in the desired RHA substance.

The chemical constitution of the RHA, as presented in Table 1, enumerates the proportions of diverse components existing in the residue. This comprises significant constituents such as silica (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), ferric oxide (Fe<sub>2</sub>O<sub>3</sub>), lime (CaO), magnesia (MgO), potash (K<sub>2</sub>O), soda (Na<sub>2</sub>O), titania (TiO<sub>2</sub>), manganous oxide (MnO), phosphorus pentoxide (P<sub>2</sub>O<sub>5</sub>), and an element for combustion loss. This composition examination provides understanding into the fundamental constitution of the RHA employed in the formation of MCM-48.

## 2.2 Syntheses of MCM-48 (RHA) and APTS-MCM-48 (RHA)

The amalgamation of permeable MCM-48 silica employing a blend of affirmative-neutral surfactants and rice husk ash (RHA) as the blueprint and silica origin, correspondingly. The amalgamation process encompasses the subsequent stages:

- Preparation of Sodium Silicate Solution: RHA, encompassing 93% SiO<sub>2</sub>, is stirred and warmed with sodium hydroxide in water at 350 K. This response leads to the creation of a sodium silicate solution.
- Preparation of Surfactant Blend: A blend of cetyltrimethylammonium bromide (CTAB) and polyoxyethylene(23) lauryl ether (PLE) is dissolved in water and heated to 350 K.
- Combining of Solutions: The sodium silicate

solution and the surfactant solution are blended and energetically stirred for 30 minutes. This stride results in the creation of a surfactant-silica gel with the preferred MCM-48 mesophase.

- Gel Maturing: The gel concoction is maintained at 373 K for 2 days, enabling the surfactant-silica gel to additionally cultivate the MCM-48 mesophase.
- pH Modification: The pH of the gel mixture is modified to 10 by incorporating vinegar (30% weight proportion). The concoction is subsequently maintained at 373 K for an extra 2 days.
- Filtration and Cleansing: The resulting gel is strained to segregate the solid product, which is then rinsed with demineralized water to eliminate any lingering impurities.
- Dehydration: The cleansed product is dehydrated overnight at 373 K to eliminate any remaining moisture and attain a desiccated substance.
- Calcination: To eliminate any organic remnants from the desiccated substance, it is exposed to calcination in atmosphere at 823 K for 4 hours. The warming speed is 1 K/minute, and middle pauses at 373 K, 473 K, and 623 K are incorporated in the procedure.

The final product acquired from RHA is known as MCM-48 (RHA), indicating that it is MCM-48 silica generated using rice husk ash as the silica source.

To prepare amine-altered MCM-48, specifically APTS-MCM-48 (RHA), the method proposed by Kim et al. (2005) is followed. A concoction comprising 0.146 g of 3-aminopropyltriethoxysilane (APTS) in 50 mL of dehydrated benzene is merged with 1 g of MCM-48 (RHA) obtained from the previous production phase. This procedure involves embedding APTS onto the surface of MCM-48 (RHA) to integrate amine functional groups, resulting in the formation of APTS-MCM-48 (RHA).

In general, the depicted procedure delineates the amalgamation of MCM-48 (RHA) and subsequent modification to acquire APTS-MCM-48 (RHA) with amine clusters on the exterior.

**Table 1 Chemical composition of the rice husk ash.**

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	Loss on ignition
93.2	0.13	0.07	1.23	0.25	0.78	0.08	0.006	0.33	0.15	3.66

The passage describes the utilization of rice chaff ash (RCA) as the silicon source in the synthesis of MCM-48, a type of material with a unique pore structure. The chemical composition of the RCA used in the formation of MCM-48 is provided, indicating the percentages of various oxides present in the ash. It contains primarily silicon

dioxide (SiO<sub>2</sub>), accounting for 93.2% of its composition. Additionally, it contains small amounts of aluminum oxide (Al<sub>2</sub>O<sub>3</sub>), iron oxide (Fe<sub>2</sub>O<sub>3</sub>), calcium oxide (CaO), magnesium oxide (MgO), potassium oxide (K<sub>2</sub>O), sodium oxide (Na<sub>2</sub>O), titanium dioxide (TiO<sub>2</sub>), manganese oxide (MnO), phosphorus pentoxide (P<sub>2</sub>O<sub>5</sub>), and a

component for combustion loss.

To prepare APTS-MCM-48 (RHA), the MCM-48 (RHA) sample was subjected to a boiling process in a mixture. The distillation took place at a temperature of 313 Kelvin for a duration of 24 hours. After reflux, the particles were filtered and washed sequentially with toluene, ethyl alcohol, and diethyl ether. Subsequently, the rinsed particles underwent dehydration under vacuum conditions at a temperature of 343 K for 8 hours. The resulting material was then designated as APTS-MCM-48 (RHA).

### 2.3 Characterization

The passage elucidates the experimental methodologies and apparatus employed to scrutinise and delineate the attributes of the materials in query.

- Powder X-ray Diffractometry (XRD): The XRD diagrams were gathered utilising a Rigaku Miniflex diffractometer with Cu Ka radiation, where Cu alludes to copper as the X-ray origin, and Ka indicates the particular wavelength ( $\lambda = 1.54 \text{ \AA}$ ). The diffraction information were documented in the  $2\theta$  scope from  $1.5^\circ$  to  $108^\circ$  with an  $18^\circ$  augmentation and 1-second augmentation duration. X-ray diffraction (XRD) examination offers details regarding the crystalline arrangement and stage constitution of the substances.
- Nitrogen Adsorption-Desorption: Nitrogen adsorption-desorption isotherms were determined at 77 K (cryogenic nitrogen temperature) employing a Micromeritics ASAP 2010 volumetric sorption analyzer. The specimens were preprocessed by depletion at 473 K (approximately  $200^\circ\text{C}$ ) prior to each adsorption assessment. This method aids in ascertaining the precise surface area (SBET) of the substances. The examination is grounded on the adsorption and desorption of nitrogen gas on the substance's exterior, offering details about its permeable composition and surface expanse.
- Elemental Assessment: The elemental constitution of rice husk ash was ascertained utilising an inductively coupled plasma (ICP) spectrometer. The particular apparatus utilised was the Jobin Yuon JY-38 VHR prototype. ICP scrutiny encompasses ionising the specimen in a plasma condition and subsequently gauging the emitted radiation to recognise and assess the constituents existing in the specimen.
- Fourier Metamorphose Infrared Spectroscopy (FT-IR): FT-IR spectra of the samples were acquired at room temperature using a Nicolet 6700 spectrometer equipped with an attenuated

total reflection (ATR) enclosure. The spectrums were gathered across a span of  $4000\text{--}400 \text{ cm}^{-1}$ . Fourier-transform infrared (FT-IR) examination offers insights regarding the functional clusters and chemical linkages existing in the substance.

- Scoping Electron Microscopy (SEM): The formations of the specimens were inspected using scanning electron microscopy. The particular device utilised was an FEI Quanta 200 machine functioning at 30 kiloelectron volts and equipped with an energy-dispersive X-ray (EDX) detector. SEM examination permits the scrutiny of the substance's exterior structure and the elemental constitution of diverse areas employing the EDX sensor.

These methodologies together offer valuable perspectives into the crystalline arrangement, surface characteristics, elemental makeup, chemical linkages, and form of the materials under examination.

### 2.4 CO<sub>2</sub> adsorption

The passage elucidates the experimental arrangement and methodologies for performing carbon dioxide (CO<sub>2</sub>) absorption measurements on MCM-48 (RHA) and APTS-MCM-48 (RHA) utilising a self-made stationary-bed reactor.

- Setup: A stationary-bed reactor, as illustrated in Scheme 1, was employed for the adsorption measurements. Roughly 300 milligrammes of the adsorbent (MCM-48 or APTS-MCM-48) was placed into a pottery sample pan in the fixed-bed setup. The reactor enables the regulated passage of gases through the absorbent layer.
- Activation: Before the adsorption trials, the MCM-48 sample underwent an initial activation procedure. It was warmed at 383 K (approximately  $110^\circ\text{C}$ ) for 2 hours under a helium (He) atmosphere. This initiation phase aids in readying the sorbent for adsorption by eliminating any lingering gases or contaminants.
- Adsorption Trial: Throughout the adsorption trials, a gas blend comprising 1.04% carbon dioxide (CO<sub>2</sub>) diluted in helium gas was introduced into the immobile-bed reactor. The flux rate of the gas mixture was regulated at 10 mL/min using a mass flow controller. This stream rate guarantees a steady and regulated provision of the gas amalgamation to the adsorbent layer.
- Gas Examination: To oversee the advancement of CO<sub>2</sub> absorption, the outflow gases from the reactor were scrutinised utilising a gas chromatograph (GC). The gas chromatograph utilised for this examination was an Agilent

model 6890 N. The vapours were transferred through a Hayesep Q column (2 m) and a thermal conductivity sensor (TCS) for identification and measurement of carbon dioxide. The gas chromatography examination offers details regarding the quantity of carbon dioxide absorbed by the absorbent throughout the duration.

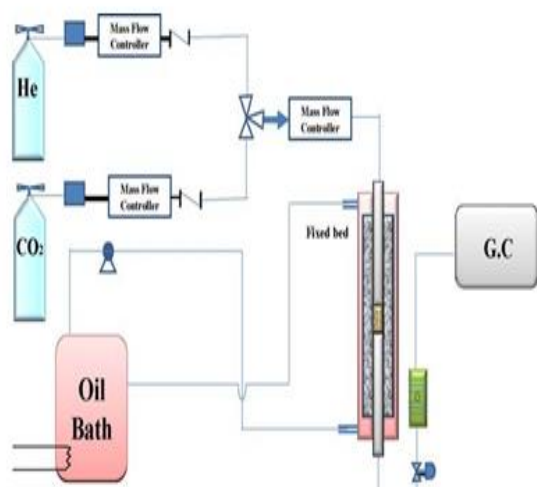
- The investigations were carried out at three separate temperatures: 298 K (roughly 25 °C), 323 K (roughly 50 °C), and 348 K (roughly 75 °C). By altering the temperature, the adsorption conduct of CO<sub>2</sub> on the sorbent can be examined under diverse thermodynamic circumstances.

In general, this experimental arrangement and examination technique enable the exploration of carbon dioxide absorption characteristics of the MCM-48 (RHA) and APTS-MCM-48 (RHA) substances. The outcomes acquired from the gas chromatograph examination furnish details regarding the adsorption capability and dynamics of carbon dioxide on the sorbents at varying temperatures.

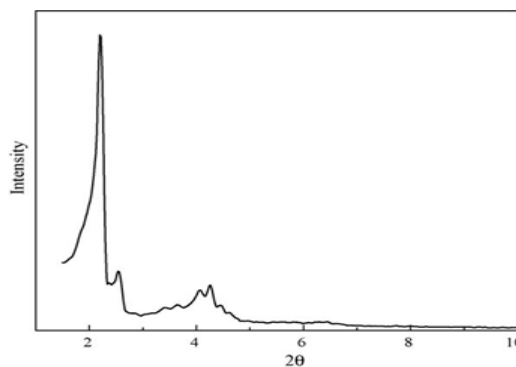
### 3. Results and discussion

#### X-ray diffraction (XRD)

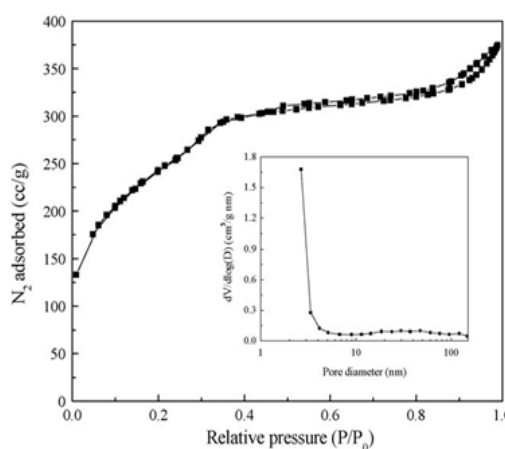
The pulverised X-ray diffraction blueprint of calcined MCM-48 (RHA) is displayed in Fig. 1. The XRD diagram of the MCM-48 (RHA) displays a distinct d211 Bragg reflection, a faint d220 Bragg reflection shoulder, and multiple indeterminate peaks between 38 and 58 2 $\theta$ , suggesting the presence of the Ia3d bicontinuous cubic phase, which resembles the conventional siliceous MCM-48 made from tetraethylortho-silicate (TEOS).



**Scheme 1.** Diagram of fixed bed reactor for CO<sub>2</sub> adsorption.



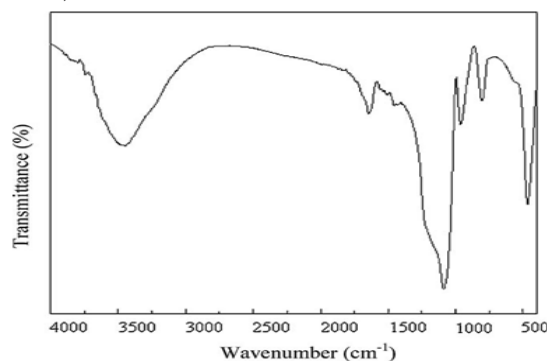
**Fig. 1.** X-ray powder diffraction pattern for MCM-48 (RHA).



**Fig. 2.** The N<sub>2</sub> adsorption–desorption isotherm of MCM-48 (RHA) powder at 77 K.

#### Nitrogen adsorption–desorption isotherms

Fig. 2 displays nitrogen adsorption–desorption of the calcined MCM-48 (RHA) demonstrated a characteristic type IV profiles, suggesting that the pore dimensions of the resulting cubic formation were within the mesopore range. The isotherm of the MCM-48 (RHA) additionally displays the kind H1 hysteresis loop linked with the unsealed cylindrical passage with consistent dimensions and form. Table 2 exhibits the precise BET surface regions, pore capacities, pore sizes for the MCM-48 (RHA) along with those for the MCM-48 (TEOS).



**Fig. 3.** FT-IR spectrum of MCM-48 (RHA).

The BET surface region was 1290 m<sup>2</sup>/g for the MCM-48 from tetraethyl orthosilicate (TEOS) and 1024 m<sup>2</sup>/g for the MCM-48 from rice husk ash. The corresponding pore dimensions were 2.58 and 4.02 nanometers and pore capacities were 1.15 and 2.58 cubic centimetres per gramme for the MCM-48 (TEOS) and MCM-48 (RHA), respectively. The particular surface region, cavity capacity, cavity size are tinier for amine modified mesoporous substance, APTS-MCM-48 (RHA), than those for original equivalent, MCM-48 (RHA). Such substantial decrease of textural characteristics for small-pore materials upon amine grafting was in excellent accordance with the literature.

The nitrogen proportion acquired from the elemental examination was employed to ascertain the surface occupancy of 3-aminopropyl clusters. As can be observed in Table 2, 2.4 millimoles per gramme of amine functional entities were affixed to the MCM-48 (RHA) pore exterior. When TEOS

was utilised as a source to formulate the MCM-48, comparable outcomes were achieved.

#### FT-IR spectrum

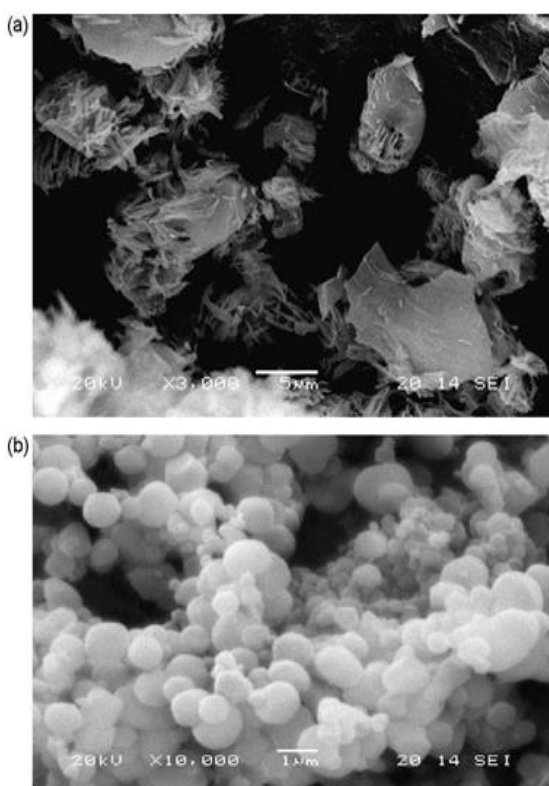
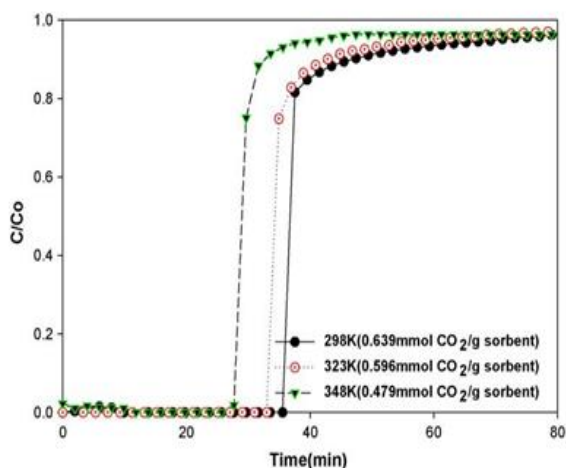
Figure 3 displays the FT-IR diagram of an MCM-48 (RHA) specimen across the span of 4000–400 cm<sup>-1</sup>. The infrared spectrum for MCM-48 (RHA) closely corresponds with MCM-48 (TEOS) that was documented in the literature. The Si–OH pinnacle emerges at approximately 3460 cm<sup>-1</sup>, whereas pinnacles for the feeble solitary Si–OH units derived from the germinal Si–OH units were detected at 3742 cm<sup>-1</sup>. The specimen additionally displayed peaks at 805 cm<sup>-1</sup>, ascribed to the Si–O–Si elongation oscillation, and 957 cm<sup>-1</sup>, which was allocated to the harmonious elongation oscillation of the Si–OH clusters. These findings validate that the exterior of MCM-48 (RHA) indeed possesses silanol clusters, as anticipated.

**Table 2 Structural properties of MCM-48 (RHA) and APTS-MCM-48 (RHA).**

Sample	BET surface	area	(m <sup>2</sup> /g)	BJH ads.	pore volume	(cm <sup>3</sup> /g)	BJH ads. pore	Diameter	(nm)	N (wt.%)	Amine group	content	(mmol/g)
MCM-48 (RHA)	1024	2.58			4.02								
APTS-MCM-48 (RHA)	102	0.098			10.24			3.34				2.4	
MCM-48 (TEOS) <sup>a</sup>	1290	1.15			2.58								
APTS-MCM-48 (TEOS) <sup>a</sup>	505	0.13			Microporous			3.42				2.45	

The BET expanse of MCM-48 (RHA) was calculated to be 1024 m<sup>2</sup>/g, signifying a substantial surface region accessible for adsorption. The BJH adsorption cavity capacity for MCM-48 (RHA) was established to be 2.58 cm<sup>3</sup>/g, indicating the existence of substantial mesoporous formation. The BJH adsorption pore radius for MCM-48 (RHA) was discovered to be 4.02 nanometers. The nitrogen concentration (N) of MCM-48 (RHA) was not furnished in the provided information. However, APTS-MCM-48 (RHA) displayed a notably diminished BET surface area of 102 m<sup>2</sup>/g, signifying a decreased surface area in contrast to MCM-48 (RHA). The BJH adsorption cavity capacity for APTS-MCM-48 (RHA) was assessed as 0.098 cm<sup>3</sup>/g, indicating a reduction in the cavity capacity following the amine-grafting procedure. The BJH adsorption pore size for APTS-MCM-48 (RHA) was established to be 10.24 nm, suggesting the existence of bigger mesopores in contrast to MCM-48 (RHA). The amine cluster content for APTS-MCM-48 (RHA) was determined to be 3.34 millimoles per gramme. In contrast to MCM-48 (RHA), MCM-48 (TEOS) demonstrated a greater

BET surface area of 1290 m<sup>2</sup>/g, suggesting a superior surface area accessible for adsorption. The BJH adsorption pore capacity for MCM-48 (TEOS) was assessed as 1.15 cm<sup>3</sup>/g, indicating the existence of a mesoporous configuration akin to MCM-48 (RHA). The BJH adsorption pore radius for MCM-48 (TEOS) was discovered to be 2.58 nanometers. Likewise, APTS-MCM-48 (tetraethyl orthosilicate) exhibited a reduced BET surface area of 505 m<sup>2</sup>/g in contrast to MCM-48 (tetraethyl orthosilicate). The BJH adsorption cavity capacity for APTS-MCM-48 (TEOS) was evaluated as 0.13 cm<sup>3</sup>/g, suggesting a decrease in the cavity capacity following the amine-grafting procedure. The BJH adsorption pore size for APTS-MCM-48 (TEOS) was ascertained to be 3.42 nm, indicating the existence of mesopores. The amine cluster content for APTS-MCM-48 (TEOS) was assessed as 2.45 millimoles per gramme.



**Fig. 4.** SEM images of (a) RHA and (b) MCM-48 (RHA).

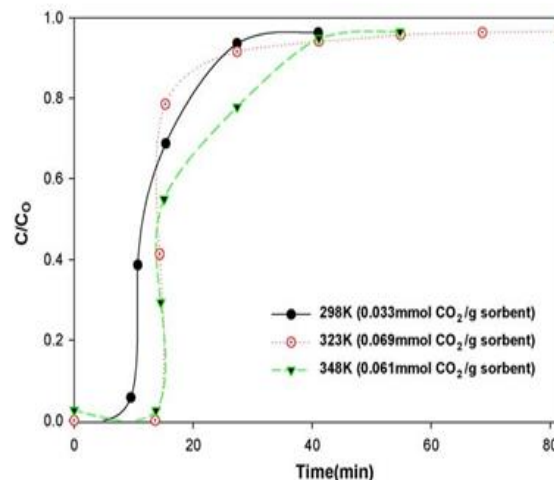
### Morphology

The arrangement of the RHA and MCM-48 (RHA) was investigated using SEM. Fig. 4a exhibits SEM images of RHA after heating at 873 K, where the ash maintains the rugged configuration of rice husk and mainly consists of fragments of separated scales with a framework resembling internal composition. Fig. 4b exhibits the SEM image of MCM-48 (RHA) comprised of splendid spherical particles of 1–1.5 μm in magnitude, resembling the formerly recorded.

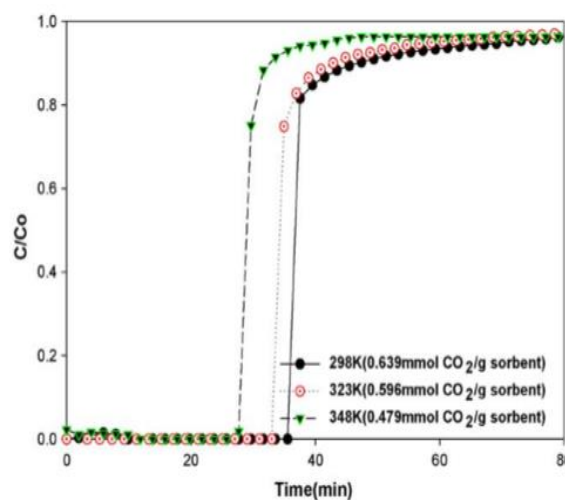
### Adsorption behavior study

To assess the carbon dioxide absorption capacity *Eur. Chem. Bull.* **2023**, *12*(Special Issue 10), 2259 – 2268

of MCM-48 (RHA), breakthrough experiments were carried out in a stationary bed reactor and the results are presented in Figure 5. Based on these groundbreaking contours, it is evident that the innovation span for CO<sub>2</sub> decreased as the temperature rose from 298 K to 348 K.



**Fig. 5.** Breakthrough curves for CO<sub>2</sub> on MCM-48 (RHA).



**Fig. 6.** Breakthrough curves for CO<sub>2</sub> on APTS-MCM-48 (RHA).

The steep slope observed for all the temperatures tested indicates that the CO<sub>2</sub> adsorption occurred very quickly, with minimal mass transfer and extremely fast kinetics. The carbon dioxide assimilation capacity can be calculated to be roughly 0.069 millimoles of carbon dioxide per gram of absorbent at 323 Kelvin on a molar basis in accordance with these innovative charts. Despite the fact that mesoporous silica exhibits uniform and extensive pores along with increased surface area, a considerable amount of active sites or adsorption sites, its deficient basic sites might conceivably be the cause of reduced CO<sub>2</sub> adsorption. Fortunately, it holds a plethora of



defective Si–OH clusters as showcased by the FT-IR plot and can be altered with an amine compound. Such altered mesoporous silica has been documented to possess beneficial CO<sub>2</sub> adsorption feature and the same exploration is to be carried out with MCM-48 (RHA) in the forthcoming. After the alteration of porous MCM-48 (RHA) surfaces with amine functional groups, we carried out the CO<sub>2</sub> adsorption capacity experiments in the presence of APTS-MCM-48 (RHA) to observe the influence of amine modification on the surface. In connection with breakthrough curve shape, the experimental results with APTS-MCM-48 (RHA) were similar to those of MCM-48 (RHA), as can be seen in Fig. 6. However, the carbon dioxide absorption capacity in the presence of APTS-MCM-48 (RHA) is approximately ten times higher than that of MCM-48 (RHA). This could be attributed to the fact that the amine functionality encompassing mesoporous substance possesses a unique interaction with CO<sub>2</sub>, which results in enhanced CO<sub>2</sub> adsorption capacity.

#### 4. Conclusions

In conclusion, the synthesis of functionalized ordered mesoporous silicas, particularly MCM-48, using rice husk ash (RHA) as a precursor material offers a sustainable and environmentally friendly approach. By repurposing RHA, an abundant agricultural waste, we can transform it into a valuable resource and reduce waste disposal issues. MCM-48 materials possess a well-organized mesoporous structure with large surface areas, tunable pore sizes, and controllable surface functionalities. These unique properties make them highly versatile and desirable for various applications, particularly in catalysis. The synthesis process involves several steps, including the preparation of a sodium silicate solution from RHA and sodium hydroxide, the mixing of surfactant solutions, gel aging, pH adjustment, filtration, washing, drying, and calcination. The resulting material, MCM-48 (RHA), is derived from RHA and exhibits the desired mesoporous structure. To further enhance the functionality of MCM-48 (RHA), amine-functionalized MCM-48, namely APTS-MCM-48 (RHA), can be prepared by grafting 3-aminopropyltriethoxysilane (APTS) onto the surface of MCM-48 (RHA). This introduces amine functional groups, which enhance the catalytic properties and adsorption capabilities of the material. Characterization techniques such as X-ray diffraction (XRD), nitrogen adsorption-desorption, Fourier-transform infrared spectroscopy (FT-IR), and scanning

electron microscopy (SEM) are employed to analyze the structure, surface properties, and elemental composition of the synthesized materials. CO<sub>2</sub> adsorption studies demonstrate the potential of MCM-48 (RHA) and APTS-MCM-48 (RHA) as adsorbents for carbon dioxide. The breakthrough curves indicate rapid adsorption kinetics, with APTS-MCM-48 (RHA) exhibiting a significantly higher CO<sub>2</sub> adsorption capacity compared to MCM-48 (RHA). Overall, the synthesis of functionalized MCM-48 using RHA as a precursor offers a sustainable and environmentally friendly approach to obtain mesoporous materials with desirable properties for catalytic applications. These materials have the potential to contribute to waste management efforts and promote a greener approach to catalysis, aligning with the global push for sustainability.

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