Journal of Chemistry and Chemical Sciences, *Vol.8(5), 850-855, May 2018* *ISSN 2229-760X (Print)*
(An International Research Journal), *www.chemistry-journal.org* *ISSN 2319-7625 (Online)*

Copper doped Mesoporous MCM-41 Synthesized Using Coconut Oil and Characterized by Spectroscopic Techniques

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(Received on: May 1, 2018)

ABSTRACT

Mesoporous Cu doped Sodium Silicate (MCM-41) where synthesized systematically. Powder MCM- 41 was crystallized from a solution containing sodium silicate, coconut oil, sulfuric acid and deionized water respectively. After 24 hours of crystallization at room temperature the Cu- MCM- 41 powder was filtered washed dried and calcined. Cu-MCM -41 characterized using spectroscopic techniques like low angle XRD, FT-IR, SEM spectra. Cu- MCM- 41 achieved higher performance at low concentrations in wastewater treatment. This was due to the successful pore opening using coconut oil. It was concluded that might be a good adsorbent for the removal of organic dyes from wastewater.

Keywords: Coconut oil; Sodium silicate; Cu-MCM-41; Adsorbent; Pore size.

1. INTRODUCTION

Removal of dyes from wastewater was one of the main problems occurs in most of the industries such as chemical, printing, leather, dyes, food, cosmetics, textile etc. Many of the methods were accessible and reported for the treatment of dyes and each of them having merits and demerits. Activated carbon was used to separate various types of coloring materials. But the main drawback of the activated carbon was the high manufacture cost and difficult to regenerate. Alternatively the natural clays can act as a collector in separate colour from the polluted water. Investigators worked on non-conventional low-cost adsorbents like natural

materials, biosorbents, and waste materials from industry, agriculture for the dye separate from aqueous solutions communicate in a few literatures.[1,2.] Various biosorbents was analysis in numerous literatures.[3,4,5,6] Mobil Oil Corporation were synthesized a series of mesopores alumino silicate in which MCM-41 was a novel mesoporous zeolite having cylindrical pore structure and large pore size, which made analysis different adsorption studies.[7] The large surface area and pore sizes ranging from 2 to 50 nm adsorbent useful for the removal of dyes from wastewater. Additionally it can be rejuvenate by using NaOH or HCL solution to recover the adsorbents and adsorbed dyes. Modified classical synthesis procedures for MCM-41 using novel silica precursors at room temperature were studied by few authors.[8,9,10] Some investigator examine the adsorption of methylene blue using MCM-41 as an adsorbent.[11,7,12] The objective of the current research work is to synthesize MCM-41 mesoporous material with high surface area[13] by a slight modification of classical synthesis method. The synthesized MCM-41, Cu doped MCM-41 and surface area, pore size, morphology are characterized based on the analyses of the XRD patterns SEM and FT-IR. To find adsorption capacity and removal of Organic dye from wastewater.

2. EXPERIMENTAL

2.1 Synthesis of Mesoporous (Cu-MCM-41) Material

Synthesis of Mesoporous Cu-MCM-41 was prepared by dissolving 0.1 mole sodium silicate in deionized water. A homogeneous and clear solution was made by continuously stirring. Posterior that 0.1 mole ratio of Copper sulfate, sulfuric acid was added to the homogeneous solution and the mixture was stirred for 5 min. Then the requested amount of coconut oil was added with stirring. 0.1 molar composition of (Na_2SiO_3:12.206 M, $CuSO_4$:15.9606 M, H_2S0_4:6.808 M, H_2O:1.8 M (2.24 ml), $C_4H_8NNaO_2$:12.5103 M) the gel was formed. The mixture was stirred at room temperatures and white precipitate was filtered, washed successional with deionized water then dried. Finally silica crucible use to calcined in air at 500 °C.

3. CHARACTERIZATION OF THE MCM-41

3.1 X-ray Diffraction

X-ray diffraction (XRD) patterns of MCM-41, Cu-MCM-41 samples were carried out under air atmosphere at room temperature on a Profile Data Ascii Dump (XRD) instrument using Co radiation operating at 30 kV and 30 mA between 2Theta range of 0.000-25.000 and 2° with a 0.02° 0.60 sec scan speed.

3.2 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) was performed using JSM-6360 Instrument. Analyses the morphology of synthesized MCM-41, Cu-MCM-41 Mesopores Nano composite were examined. SEM operated at an acceleration voltage of 10 kV.

3.3 FT-IR Spectroscopy

Infrared spectra of the sample were recorded on a FT-IR Equinox 55 Spectrophotometer. Mesoporous materials in the frame work region were obtained.

4. RESULTS AND DISCUSSIONS

4.1 X-Ray Diffraction (XRD):

XRD patterns of the (MCM-41, Cu-MCM41) samples are given below figure 1. Only one low-angle peak for d100 plane perceive respective mesopores samples. Because the materials are not crystalline at the atomic level, reflections not produce at higher angles. The peak positions stay almost unchanged which specify that the structural arrangement is not affected during calcination. The intensity of the main peak of Cu-MCM-41 samples decreased and compare to MCM-41 sample. Width of the peak increased which indicate increase in copper content of the sample and simultaneously pore size increase, surface area decrease. These changes due to the partial substitution of Si^{4+} instead of Cu^{2+} ion resulting in the retain hexagonal structure of Cu-MCM-41 and decrease long range order. The X-ray diffraction pattern of Cu-MCM-411 also showed the presence of these peaks which denote that the structure of hexagonal arrangement was retain after introducing Copper into its structure. There was also a decrease in the relative intensity of the diffraction patterns of Cu-MCM-41 in comparison with MCM-41.

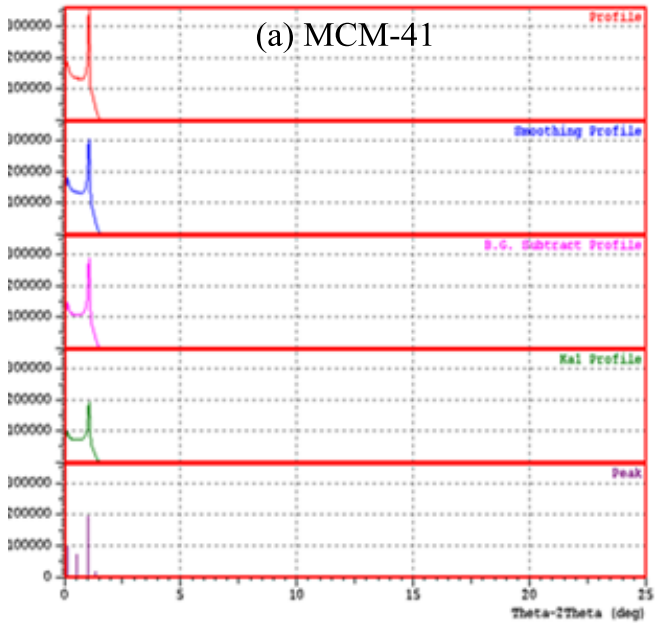

Figure 1. XRD patterns of a) MCM-41 Sample

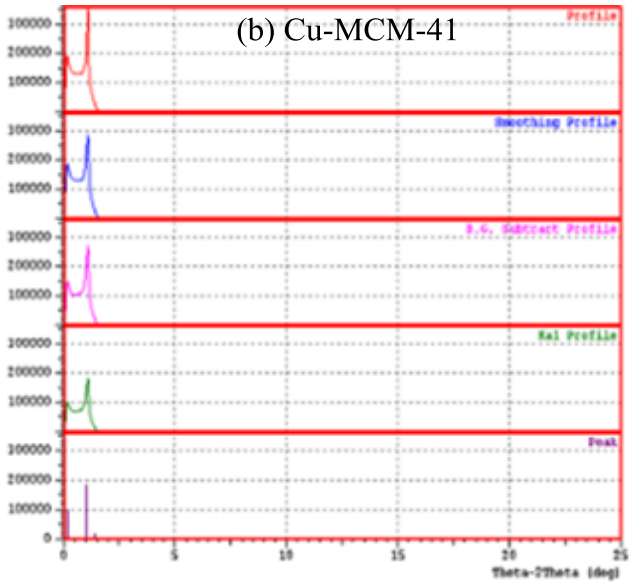

Figure 1. XRD patterns of b) Cu-MCM-41 Sample

4.2 Scanning electron microscopy

The Scanning Electron Microscope (SEM) Figures 2. (a) MCM-41, (b) Cu-MCM-41 clarify general morphology of mesopors sample respectively. The SEM micrograph of MCM-41 Fig.2 exhibit sample confirmed the mesopores particles sizes, morphology and showed hexagonal packing. Fig.2. (b) SEM image explain that the Cu metals are uniformly leap to the MCM-41 surface.

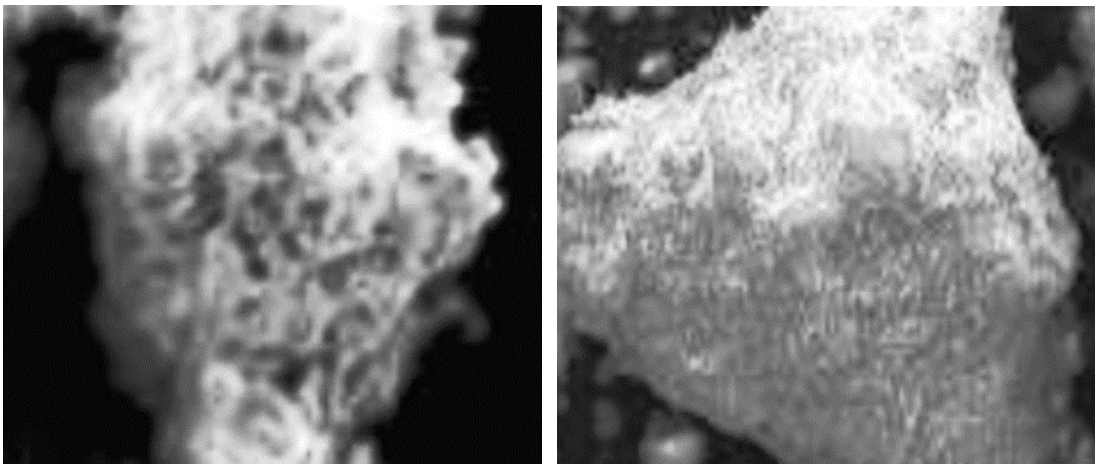

Figure 2. SEM image of (a) MCM-41 sample (b) Cu-MCM-41 sample

4.3 Spectroscopy in the FT-IR region

The infrared spectra of Figures 3. MCM-41 and Cu-MCM-41 samples showed bands in the region of 500-3500 cm^{-1}, which are characteristic of the fundamental vibration of MCM-41 structure with reference to the literature.[8] The band frequency at 3450 cm^{-1} is demonstrate to the stretching mode of Si-H_2O. The broad strong peak at 1064 cm^{-1} can be ascribed to the asymmetrical stretching of Si–O–Si groups. The band frequency at 803 cm^{-1} can be attributed to the symmetrical stretching of Si–O–Si. Above band frequency showed that Copper doped MCM-41 the original structure does not affected.

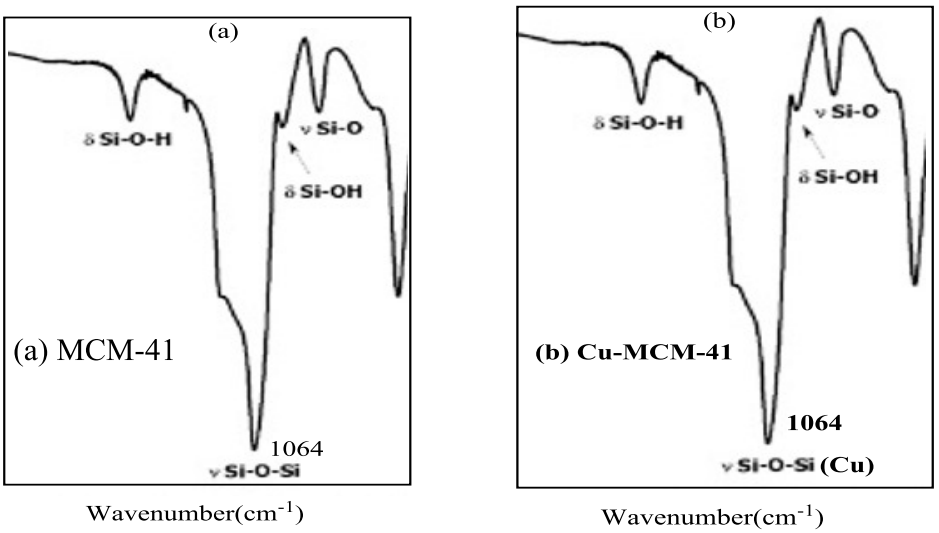

Figure 3. FT-IR spectral band of (a) MCM-41, (b) Cu-MCM-41

5. CONCLUSION

Synthesis procedure has been successfully adopted for the preparation of MCM-41, Cu-MCM-41 and characterized using XRD, SEM and FT-IR. Incorporation of Copper metal did not affect the structure ofCu-MCM-41, which was verified by the results obtained in the characterization of the material. XRD pattern observed that there is no peak shift, which confirms that the structure stability is not affected after calcination. SEM images explain atom in hexagonal arrangement, expected morphology and pore sizes. FT-IR band frequency showed that Copper doped MCM-41 the original structure does not affected. The above results show that the synthesized Cu-MCM-41 is having a maximum surface area, adsorption capacity with organic dyes in wastewater treatment. It is found that Coconut oil maintenance a considerable portion of the pore size with increase in surface area and adsorption capacity. This characterization results state that using this method can be used as an alternative technique in the synthesis of MCM-41 and suitable for adsorption studies.

6. ACKNOWLEDGEMENT

I would like to thank the JCT College of Engineering and Technology for helping the research work.

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