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30th October 2021, Day-Saturday

Jointly Organized by

One Day Multidisciplinary International e-Conference

On

Agriculture and Rural Development: Strategic Issues and Reform Options

CERTIFICATE

This is to certify that Prof./Dr./Mr./Ms. S. B. Shete has participated in One Day Multidisciplinary International e-Conference on 'Agriculture and Rural Development: Strategic Issues and Reform Options' Jointly Organized by Sant Ramdas Arts, Commerce and Science College, Ghansawangi Dist. Jalna and Sahakarbhushan S. K. Patil College, Kurundwad on 30th October 2021, Day-Saturday

He/She has participated and presented a research paper entitled conversion of Coal fly-Ash (C.F.A.) into MCM-4, mesoporous molecular sieves.

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PRINCIPAL

Conversion of Coal Fly Ash (C.F.A.) Into Mcm-41 Mesoporous Molecular Sieves

S. B. Shete

S.G.B.Mahavidyalaya, Purna

Email: s.bshete@rediffmail.com

Abstract :

Mesoporous molecular sieves in the hexagonal phase (MCM-41) is synthesized from fused coal fly ash (C.F.A.) solutions and CTAB surfactants. It is found that C.F.A. could be effectively transformed into mesoporous materials depending upon the hydrothermal conditions. It is also observed that a high concentration of Na⁺ ions in the supernatant of the fused coal fly ash is not critical in the formation of MCM-41 when prepared under controlled pH of gel, calcination temperature and calcinations duration conditions. We provide direct evidence of MCM-41 aluminosilicates with a homogeneous chemical composition of Si/Al=13.78 can be prepared with cationic surfactants. Our results resemble that coal combustion byproducts can be utilized for producing mesoporous molecular sieves even if containing significant amounts of impurities. The highest crystalline and well defined phase purity MCM-41 is obtained without hydrothermal treatment in short interval of time.

Keywords: Mesoporous material, surfactants, flies ash, hydrothermal synthesis

Introduction:

In India power sector has been receiving adequate priority ever since the process of planned development began in 1950. Hydro and coal based thermal power have been the main sources of generating electricity. In our country more than ninety million tones of coal fly ash (C.F.A.) is being generated annually with more than 6500 acres of land being occupied by ash-ponds and create environmental and health hazards. The recycling of C.F.A. has become an increasing concern in recent years due to increasing landfill costs and current interests in sustainable development of human society from view points of Energy economy and Environmental strategy, still about only 15% of total C.F.A. produced from coal combustion is reused in various applications i.e. in road construction, cement production, land reclamation and restoration of eroded soil.

Conversion of C.F.A. into useful Si-MCM-41 materials is one of the approaches to recycle C.F.A. which has extensive applications in basic sciences, petrochemical sciences, energy conservation, medicine, chemical sensor, air purification and waste remediation. This paper has reported A green, cost effective and fast method for production of Si-MCM 41 from Waste C.F.A. as an inorganic silica source & ethyl acetate as a mild acid hydrolyser. This method gives a high degree of crystallinity of Si-MCM 41 even in the comparatively short duration of time. This approach took 18 hrs at 298 K (R.T.) to produce Si-MCM 41 from C.F.A. which is so far as the knowledge, is the least time and lowest reaction temperature required to produce pure and ordered Si-MCM-41. However we came to conclusion that the change of pH of gel solution, change of calcination duration, change of calcinations temperature results into the varying yields and crystallinity of synthesized material. FTIR, XRD, N₂ Adsorption-desorption resembles crystal structure and porosity.

Porous solids are an important class of materials due to their wide applications in various separation, purification and catalytic processes. Although there are many kinds of amorphous porous materials which contain micro porous, mesoporous and macroporous, but there were no materials possessing uniform mesoporous until the early 1990s. Mobil corporation scientists first reported M41S family of materials¹⁻³ with pore size from 2nm to 50 nm. Because of high specific areas and large uniform pore sizes, mesoporous Si-MCM-41 materials found a lot of applications in selective catalysis, adsorption of gases and liquids⁴⁻⁷ etc. Perhaps the large scale production of MCM-41 became very expensive due to costly silica sources and harmful due to toxic chemicals.⁸

Resource recovery of the Si-MCM-41 materials in large scale from C.F.A. is one of the new and green approach for reducing energy consumption and the waste generated in production process.

Materials And Methods:

Source materials

Coal Fly Ash

The coal fly ash (C.F.A.) was obtained from Thermal Power Stations at Chandrapur, and I (Vaidyanath) Maharashtra (India). The Chemical composition of fly ash collected from both

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and also their XRD resembled that the required Si/Al ratio is more prominent in the coal fly (C.F.A.) obtained from Thermal Power Station at Chandrapur. Thus this C.F.A. is used in the study. The Chemical composition of fly ash collected from T.P.S. at Chandrapur, is given in Table-1:

The amounts of the main components of ash viz. both amorphous (mainly SiO_2 , Al_2O_3) and crystalline components (mainly quartz and mullite) show few variations with the type of coal.

Table-1: CHEMICAL COMPOSITION OF AS COLLECTED COAL FLY ASH

Compound	Content (wt%)	CFA (mol/100g)
SiO_2	67.16	1.117
Al_2O_3	19.82	0.193
Fe_2O_3	5.95	0.039
CaO	3.06	0.053
K_2O	1.12	0.012
TiO_2	1.76	0.023
Na_2O	0.33	0.005
MgO	0.79	0.019

als

The chemicals used are Sodium Hydroxide Fisher Scientific, The surfactant solution Cetyl trimethyl ammonium bromide (CTAB), Spectrochem, ethyl acetate, sulfuric acid, de-ionized water. All chemicals were AR grade; hence they were used without further purification.

is method

The amorphous SiO_2 component in the C.F.A. was used as Si- source for the synthesis of Si-1. The synthesis was carried out as follows:

A known quantity of C.F.A. fused with 2M NaOH solution at 100°C for 4 hrs under stirred (300 rpm) in stirred autoclave. The mixture is allowed to cool. The solution was separated by a filtration process. This obtained supernatant solution was mixed with 1.1 gm of nethyl ammonium bromide (surfactant solution) and kept under stirring condition (300 rpm) After 30 min. ethyl acetate was vigorously mixed and the solution kept under stirring at 600 rpm for next 30 min. The obtained solution was allowed to cool to room temperature by natural convection and was adjusted to the selected pH by adding 5.25N H_2SO_4 solution under slow stirring. The obtained during pH adjustment was kept at room temperature for 18 hrs. The solid material after filtration was washed by deionised water number of times and dried at 100°C for 2 hrs. The dried material was calcined under air from 500°C to 800°C at the interval of 50°C at a heating rate of $1^\circ\text{C}/\text{min}$.

erization techniques

X-ray diffraction:

X-ray diffraction patterns of the collected CFA and synthesized materials were recorded on a $\text{Cu K}\alpha$ MiniFlex2 goniometer with a wave length of $1.540 \text{ \AA}$.

powder patterns of synthesized samples were obtained using $\text{Cu K}\alpha$ radiation on a Rigaku X-ray diffractometer. The samples were scanned for 2θ from 1° to 20° .

pectra:

Infrared (FT-IR) spectra of the collected CFA and synthesized materials were recorded on ALPHA FT-IR spectrometer (ATR eco ZnSe) using dry KBr as standard reference in the range of $500\text{--}4000 \text{ cm}^{-1}$.

face area:

BET surface area was calculated using Surface area Analyzer Model SAA-2000 for all synthesized samples. pH values were measured with a digital pH meter

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RESULTS AND DISCUSSION: FTIR

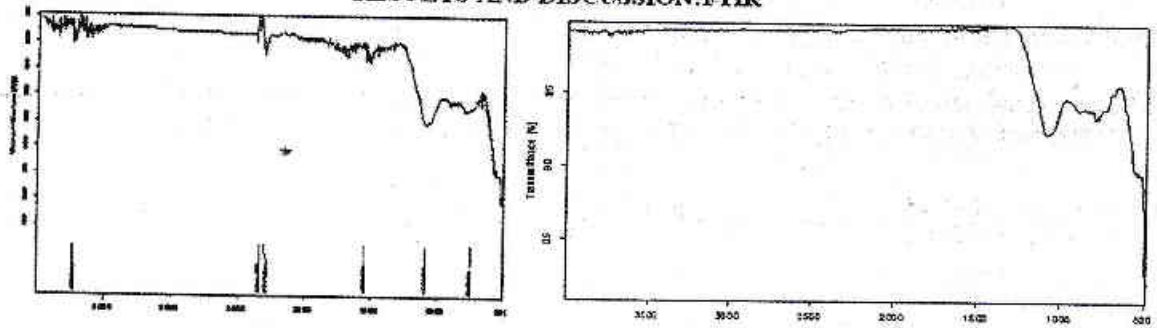


Fig. 1 : FT-IR spectra of CFA collected from Chandrapur (M.S.) India. A) As collected B) calcined at 800 °C

FTIR spectra shown in the Fig. 1 provide valuable information about the basic characteristic of the molecule, namely, the nature of atoms, their spatial arrangement and their chemical linkage forces. Infrared spectroscopy has been extensively used for identifying the various functional group of the support, as well as identifying the various functional groups of the active component. The mid infrared region of the spectrum contains the fundamental framework vibration of Si(Al)O_4 groupings. The absorption band in between the wave numbers $980\text{--}1320\text{ cm}^{-1}$ in IR spectrum of fly ash are treated fly ash represent the presence of substituted Al atoms in the tetrahedral forms of silica framework.

XRD of collected fly ash :

The XRD pattern of Coal Fly Ash (CFA) collected from Chandrapur TPS shown in Fig. 2. The different minerals have different unit cell composition, therefore XRD technique allows for qualitative identification of the phases present in the collected mineral. The XRD peak information is important to quantify changes in the composition of Quartz and Mullite reactants that affect reaction conditions of hydrothermal synthesis of materials and reaction products. From these XRD patterns it can be seen that the major crystalline phases found in the fly ash are common mineralogical phases such as quartz, mullite and aluminosilicate glass amorphous material forming during the combustion process.

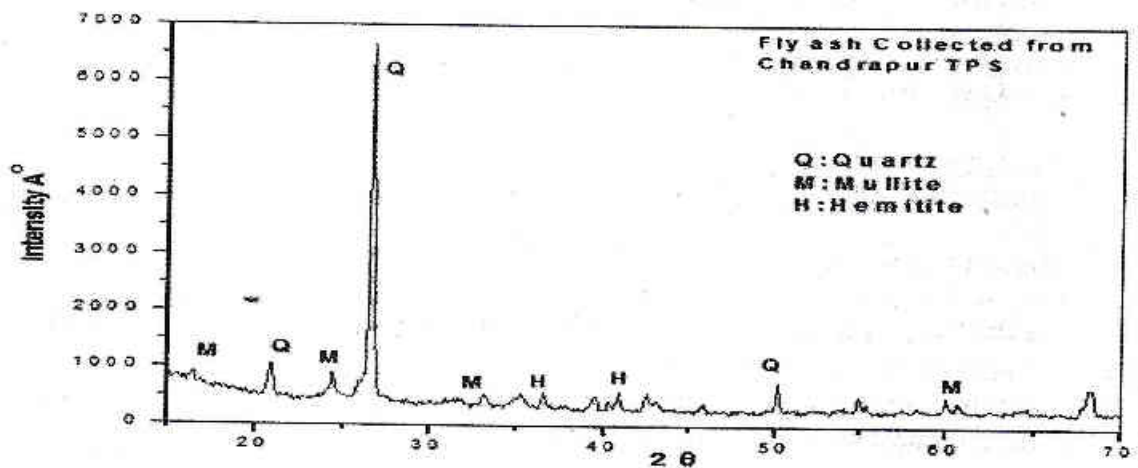
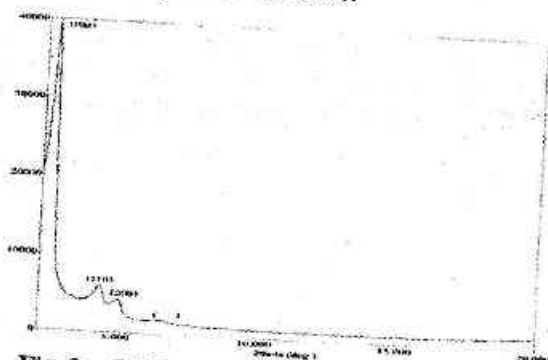


Fig. 2 : XRD pattern of Fly ash obtained from Chandrapur Super Power Station

The X-ray pattern of the synthesized mesoporous silica material is an highly periodic silica phases which is normally reflected by the distinct XRD signatures at low 2θ angles from 0° to 30° as shown in Fig.3. Sharp signal in XRD spectra indicates the presence of long range order of uniform hexagonal phase in the mesoporous materials. The well defined reflections from [100] plane are prime characteristics of the hexagonal lattice symmetry of the MCM-41 structure.

The observation of three higher angle reflections other than d_{100} indicates that the product is likely to possess the symmetrical hexagonal pore structure typical of MCM-41. X-ray diffraction data therefore indicates that the supernatant of the fly ash can be successfully used in the synthesis gel to prepare mesoporous materials.



$$d_{100} \text{ value} = 35.3099; \quad 2\theta = 2.500$$

$$\text{Unit cell parameter } a_0 = \frac{2}{\sqrt{3}} d_{100}$$

$$a_0 = 40.8$$

$$\text{Intensity (I): } 14163 \text{ cps} \quad I/I_0 = 1$$

Fig. 3: XRD pattern of synthesized material with parameters (pH=6.9) temperature 550°C and calcination time 4 hrs.
Study of various synthesis parameters for MCM-41 :

Effect of pH of synthesis gel

The pH of reaction mixture of the gel is also plays an important role in synthesis of MCM-41 phase. The effect of change of pH of gel shows that, when pH varies from 1.87 to 6.91 the crystalline nature and phase purity improves to highest level. The synthesis was carried out at constant calcinations temperature 550°C for 4 h. The crystallinity reduces when pH of the gel is below 6.91 and above 6.91.

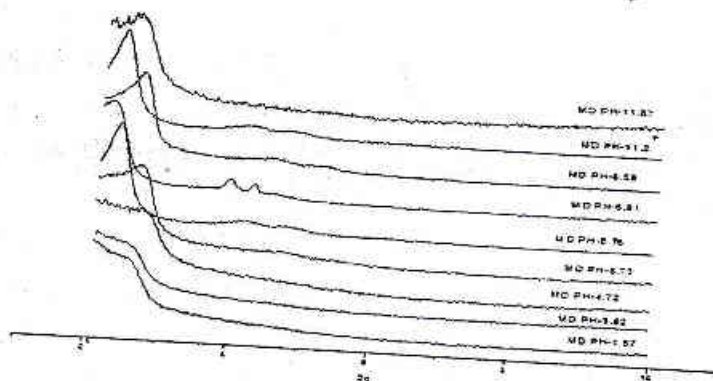


Fig. 4: XRD Patterns of MCM-41 at different pH of synthesis gel.

The XRD data further analyzed to calculate I/I_0 , Unit Cell parameters a_0 , percent crystallinity and obtained data presented in Table 4. The hexagonal unit cell parameter a_0 in the calcined sample increased with pH of synthesis gel value.

Table 2: The effect of pH of gel prepared for synthesis of Si-MCM-41 at 550 °C for 4 h and on its physical properties.

Sample designation	pH of gel	2θ for d_{100}	d_{100}	I/I_0	a_0	% Crystallinity	BET Surface Area $\text{m}^2 \text{g}^{-1}$
MP ₁	1.87	2.64	33.43	0.08	38.8	19	632.161
MP ₂	3.82	2.66	33.18	0.06	38.5	28	---
MP ₃	4.72	2.86	30.86	0.05	35.8	37	---
MP ₄	5.73	2.28	38.71	0.16	44.9	38	1276.181
MP ₅	6.76	2.39	35.91	0.28	41.7	84	---
MP ₆	6.91	2.50	33.30	1	40.9	100	1801.788
MP ₇	8.56	2.68	32.93	0.2	38.2	---	682.871

MP ₈	11.2	2.40	36.78	0.09	42.7	30	368.121
MP ₉	11.87	2.60	33.95	0.03	39.4	25	14.274

Therefore it is observed from presented data in the table 4 that pH of gel solution at 6.9 gives good results. The variation of pH of gel plays an important role in the formation of MCM-41.

Effect of Calcination Time

The crystallization kinetics was also studied by changing calcinations period from 1h to 12 h at constant temperature 550°C. The different powder XRD patterns obtained in this time variation were plotted and given in Figure 7. It shows that at 4 h well crystalline and phase purity of MCM 41 was obtained.

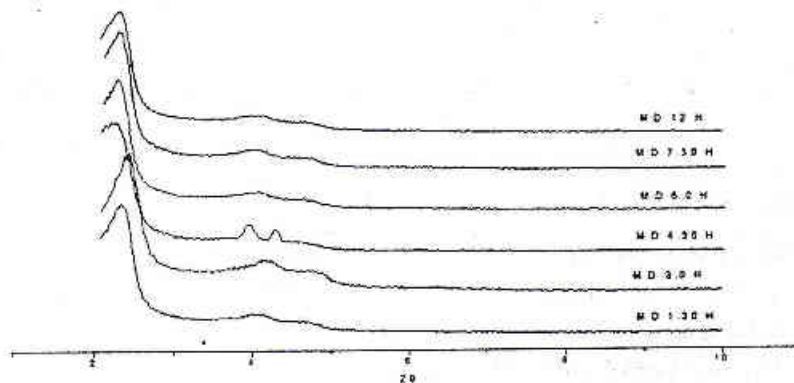


Fig. 5: XRD Patterns of MCM-41 at different calcinations time period

The XRD data further analyzed to calculate I/I_0 , Unit Cell parameters a_0 , percent crystallinity and obtained data presented in Table 3.

Table - 3 : The effect of calcined period of Si-MCM-41 at 550 °C on its physical properties

Sample Name	Time (hours)	2θ for d_{100}	d_{100} a.u.	I/I_0	Unit cell parameter a.u. a_0	% Crystallinity	BET Surface Area $m^2 g^{-1}$
MH ₁	01.30	2.38	37.08	0.5	43.00	76	886.184
MH ₂	03.00	2.46	35.88	0.25	41.60	99	—
MH ₃	04.30	2.50	35.30	1	40.90	100	1801.788
MH ₄	06.00	2.32	38.04	0.36	44.10	89	—
MH ₅	07.30	2.32	38.04	0.36	44.10	70	620.702
MH ₆	12.00	4.10	21.53	0.07	25.00	61	564.092

The tabulated data shows that as calcinations time duration increases, percent crystallinity also increases up to 4.3 h then after decreases for the synthesis system under the study. During the change in calcinations time duration pH of the gel was kept constant at 6.9 and calcinations temperature at 550°C. The highest crystallinity sample was obtained at 4 h.

Effect of Calcination Temperature

The synthesized samples were calcined at different temperature from 500 to 800 °C to study change in crystalline nature of synthesized sample, thermal stability etc. All these samples of MCM-41 were calcined at various temperature for constant period of 4 h.

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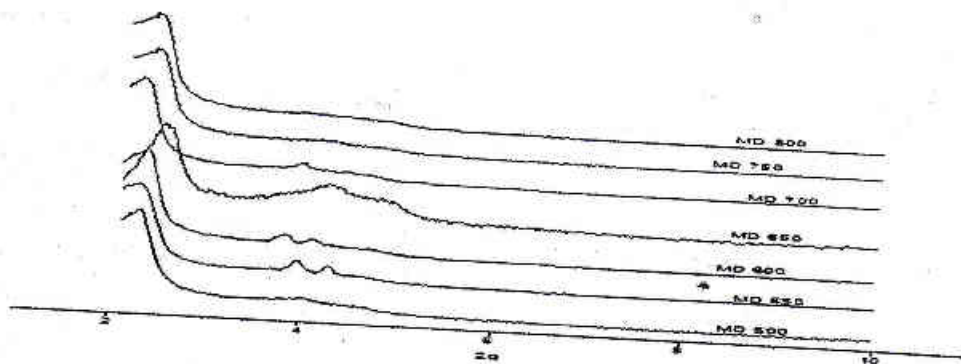


Fig. 6: XRD-Patterns of MCM-41 at different Calcination temperature .

The powder XRD patterns of calcined samples were recorded and presented in figure 4. The XRD data further analyzed to calculate I/I_0 , Unit Cell parameters, percent crystallinity and obtained data presented in Table 2.

Table -4 : The effect of calcined temperature of Si-MCM-41 on its physical properties.

Sample Name	Calcination temp.	2θ for d_{100}	d_{100}	I/I_0	a_0	% Crystallinity	BET Surface Area $m^2 g^{-1}$
MT ₁	500°C	2.32	38.04	0.24	44.10	20	---
MT ₂	550°C	2.50	35.30	1	40.90	100	1801.788
MT ₃	600°C	2.46	38.19	0.26	44.30	76	1276.653
MT ₄	650°C	2.54	34.75	0.31	40.31	40	1101.316
MT ₅	700°C	2.38	37.08	0.35	43.02	29	---
MT ₆	750°C	2.42	36.47	0.33	42.31	19	---
MT ₇	800°C	2.41	36.17	0.33	41.90	15	767.130

The tabulated XRD data shows that at 550 °C the value of I/I_0 is maximum and higher crystalline nature of MCM-41 is obtained. The effect of change of calcination temperature v/s percent crystallinity of MCM-41 was plotted and shown in the Figure 6. It shows that, at initial temperature increases upto 550°C, percent crystallinity also increases and after that it decreases from 100 % to 15 %. Hence 550 °C is optimized temperature to get highest crystalline MCM-41 in the given system of synthesis.

N₂-Adsorption Desorption Analysis Of Synthesized Mcm-41 Material:

Nitrogen physisorption probes the textural properties of materials i.e. surface area, pore volume, pore size and pore geometry. At very low relative pressures (p/p_0) a very large amount of nitrogen becomes physisorbed which assigns to condensation of nitrogen inside and outside on the surface of MCM-41. As the surface area is very high this corresponds monolayer adsorption. Upon monolayer adsorption multilayer of nitrogen starts to developed at higher relative pressures. Also in this case both the external surface area and mesopores contributes to the physisorption process.

The collected data from the N₂ adsorption desorption graph is used to calculate the surface of the material using BET method. (Tables 2, 3 & 4). From the above tabular data it is confirmed that the maximum calculated surface area amounts to 1801 m²/g. for the Si-MCM-41 materials keeping pH of gel 6.91, calcination time about 4 h. at 550°C.

Ir Analysis :

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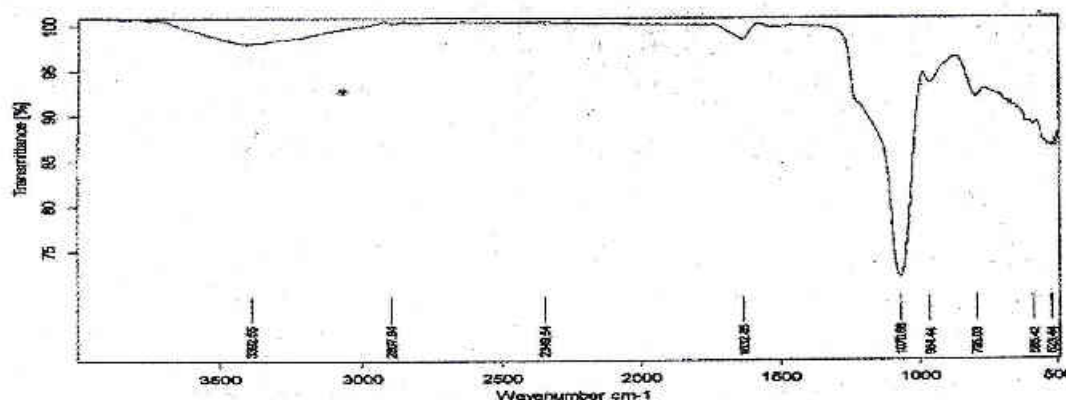


Fig. 7: The FT-IR spectra of as synthesized MCM-41 from coal fly ash

The FT-IR spectra of as synthesized MCM-41 from coal fly ash are shown in Fig. 10. From FT-IR spectra, the absorption bands around 2921 and 2851 cm^{-1} correspond to n-C-H and d-C-H vibrations of the surfactant molecules, such bands disappeared in the calcined sample indicating the total removal of organic material during calcinations. The broad band around 3392.65 cm^{-1} as observed due to surface silanols and O-H stretching frequency of adsorbed water molecule. Moreover the peaks in the range of 1500-1600 cm^{-1} are because of the deformation mode of surface hydroxyl group. A peak at 1070.63 cm^{-1} and 964.44 cm^{-1} corresponds to the asymmetric and symmetric Si-O groups, respectively. The peaks in the range 1010-1079 cm^{-1} are assigned to M-O-M bonding, the bands from 960 to 990 cm^{-1} appeared due to Si-O-M (M=metal ions) vibrations in metal incorporated silanols. The shift in the lattice vibration bands to lower wave numbers is due to the substitution of silicon by other metal ions.

Conclusions:

Based upon the experimental study it was concluded that pure and ordered MCM-41 material could be successfully synthesized from coal fly ash at room temperature during 18 hrs of reaction. The parametric variations such as change of calcination temperature, the change of calcination time duration and the change of initial pH value of gel suggested that from CFA the well ordered mesoporous material MCM-41 can be synthesized at 550°C for 4 hrs. keeping pH of gel 6.91. The maximum calculated surface area amounts to 1801 m^2/g for the MCM-41 materials keeping pH of gel 6.91, calcination time about 4 h. at 550°C.

This study demonstrates that converting CFA into mesoporous materials not only eliminates the disposal problem of CFA but also turns a waste material into a value added product. The synthesized MCM-41 materials may find applications in the waste water treatment, Chemical industries and Agriculture industry etc. The proposed method provides one of the ways of recycling CFA.

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Shri Guru Buddhiswami Mahavidyalaya
Purna (Jn) Dist. Parbhani - 431511 (M.S.)



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