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Mehdi Erfani Jazi [α] & Thualfeqar G Mohammed [σ]

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Keywords: Zr-MCM-41, benzyl alcohol, acid acetic, esterification, calcinations.

I. Introduction

Esterification is an industrially important reaction, which is one of the methods used to produce ester compounds. These organic esters are intermediates in the synthesis of fine chemicals, drugs, plasticizers, food preservatives, pharmaceuticals, cosmetics and auxiliaries [1]. Esters are normally produced by a batch process in industries using mineral acid catalysts such as hydrofluoric acid, sulphuric acid or Lewis acid catalysts like $AlCl_3$ or BF_3 [2]. Mineral acids are known as corrosive and virulent, so they need to be neutralized after the completion of the reaction, while in the process catalyzed by metal containing Lewis acid catalysts, the excess water has to be removed carefully after the reaction [3].However, it leads to the formation of large amounts of waste [4, 5]. Moreover, all these catalysts are typically categorized as hazardous substances and hence undesirable from the environmental point of view. Therefore, there is a global effort to replace hazardous and environmentally harmful catalysts with ecofriendly alternatives [1, 5].The purely siliceous Si-MCM-41 is structurally stable towards thermal treatment, hydrothermal treatment with steam at mild conditions, mechanical grinding and also towards acid treatment at mild condition. Since, mesoporous materials do not have efficient catalytic properties due to absence of catalytically active sites, so MCM-41 is often modified by incorporating certain active materials such as metal oxides, metal complexes and strong acid compounds [6].

The aim of this work is to develop a new kind of catalyst containing zirconium with ordered mesoporous structure and strong Lewis and Brönsted acidity for the esterification of benzylalcohol. The surface acidity of the silicate mesoporous molecular sieves wasgreatly enhanced by modification of the framework by the incorporation of Zr^{+4} ions into the Si-MCM-41.

II. Chemicals

The chemicals used for the synthesis of isomorphic substitution of Zr-MCM41 are summarized in the Table 1. All the chemicals were used as received without purification.

Table 1 : List of chemicals used in the synthesis of catalyst

Chemicals	Chemical	Manufacture	Purity (%)
Cetyltrimethylammonium bromide (CTABr)	$C_{19}H_{42}NBr$	Fluka	99
Tetraethyl orthosilicate (TEOS)	$C_8H_{20}O_4Si$	Merck	98
Zirconium propoxide (70% in propanol)	$C1_2H_28O_4Zr$	Aldrich	98
Ammonium hydroxide (27 wt %)	$NH_3.H_2O$	-	-

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III. Experimental

a) Catalyst Synthesis

Direct synthesis or isomorphic substitution of Zr-MCM-41 was carried out by using the method proposed by Chen, L.F. *et al*. with some modifications [7]. The Zr-MCM-41 solids were prepared using tetraethyl orthosilicate (TEOS) as Siprecursor and zirconium propoxide (70% in propanol) as Zr source, along with cetyltrimethylammonium bromide (CTABr) as surfactant template. The typical preparation procedure of a Zr-MCM-41 sample with a molar ratio of Si/Zr =5, 10, and 20 is as follow: first of all, two solutions were prepared, the first solution was made by adding given amount of zirconium tetra-propoxide (0.0475,0.095 and 0.19 mol) into given amount of TEOS (0.95 mol) with stirring; the second solution was made by adding given amount of CTABr (0.35 mol) into 110mL hot water (around 50^{o} C) with stirring, followed by addition of 110 mL $NH_3.H_2O$ (28wt %).

Then, the first solution was added gradually into the second solution. The mixture was strongly stirred during the addition for about 2 hours. After that a gel was formed. The gel is left in the ambient temperature to be cooled. Then, the gel is loaded into stoppered Teflon bottle and it maintained at 100°C for two days. Once the gel is cooled to room temperature, the solid powder was recovered by filtration and it was washed with 500mL of deionized water for several times. The white solid powder was heated in the oven for one day at 70°C. After that, the sample was placed into the furnace and calcined at 600°C for about 6 hours in the air. The heating rate is 1°C/min. It can be seen from Table 2 that the molar composition of the gel mixture is the following: 0.95TEOS : 0.35CTABr : 0.65$NH_3.H_2O$: X-zirconium tetrapropoxide : 0.55H_2O. In this study, the amount of X was varied according to desired Si/Zr molar ratios of 5, 10, 20.

Table 2 : Sample codes for different Si/Zr **molar** ratio of the materials

Sample	Si/Zr	Si content, mole	Zr content, mole
Si/Zr=20	20	0.95	0.0475
Si/Zr=10	10	0.95	0.095
Si/Zr=5	5	0.95	0.19

b) Characterization of Zr-MCM-41

In this study, comprehensive characterization techniques were utilized in order to elucidate and provide unambiguous structural information and physicochemical properties ofZr-MCM-41. The Powder X-Ray Diffraction (XRD)is Bruker D8 Advance powder diffractometer with Cu-Kα as the radiation sources with λ= 1.5418 Å at 40 kV and 40 mA. The Fourier Transform Infrared Spectroscopy is done using a Perkin Elmer One FTIR Spectrometer. The FTIR spectrums are recorded in a spectral range of 4000-400 cm^{-1}. The Ultraviolet-Visible Diffuse Reflectance Spectroscopy (UV-Vis DR) is a Perkin-Elmer Lambda 900 spectrometer. A spectrum was measured in the wavelength scale of 190-800 nm. The Scanning Electron Microscopy (SEM) is Philip XL40 field emission scanning electron microscope operating at 15kV. The nitrogen adsorption measurements were carried out at 77 K on ASAP 2010 volumetric adsorption analyzer. The evaluation and analysis of the strength and type of acid site, pyridine adsorption on the samples was performed on the 170SX FTIR spectrometer.

c) Catalytic testing

The performance of Zr-MCM-41was measured in terms of conversion of benzyl alcohol and selectivity to benzyl acetate in competition with etherification reaction. The Blank reactions were also carried out in the absence of a catalyst. The main product was found to be benzyl acetate. Experiments were designed by varying the amount of the catalyst, the molar ratios of the reactants, the reaction temperature and the reaction period.

IV. Results & Discussion

a) Characterization of Zr-MCM-41 catalyst

i. *XRD analysis*

XRD patterns of the as-made and calcined Zr-MCM-41(Si/Zr = 20) is displayed in Figure 1a. The Samples have four peaks that are indexed to (100), (110), (200) and (210) reflections, which correspond to well ordered hexagonal pore systems, characteristic of MCM-41-type mesoporous materials. In comparison with the as-made solids, after calcination at 600^{o}C, several variations were observed: the positions of the (110) peak shifted towards larger 2θ values which results a reduction in the spacing distance between the (100) planes (d_{100}) and lattice parameters (a_0). The groups of Si-OH and/or Zr-OH condensate considerably during calcinations which leads to the contraction of unit cell dimension towards fresh solids. There is a remarkable enhancement in the intensities of the diffraction peaks after calcination, which must be corresponded to the surfactant removal molecules in the process of calcination. As shown in the Table 3, the pore wall thickness (t= a_0- d_p) is obtained from lattice cell parameter (a_0= $2d_{100}/3^{1/2}$) and the pore diameter (d_p), is thicker relative to that of Si-MCM-41(t=1.92 nm). The slight increase in pore wall thickness (t) is coincident

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with the enhancement of zirconium content which points out the incorporation of zirconium into the framework. Particularly, after calcination, the intensities of the XRD peaks increase significantly in the Zr-MCM-41(Si/Zr=20), indicating that the structural ordering is increased and ordered hexagonal pore system retained without collapsing. It is remarkable that in the calcined MCM-41 solids, the intensities of the XRD peaks vary with zirconium content. The calcined sample with a Si/Zr=10 shows a lower peak intensity than the calcined sample with a Si/Zr=20 (see Fig. 1b).The intensities of all the peaks decrease as the Si/Zr molar ratio decreases from 20 to 10 and then, decrease with further increase of the zirconium content.

The calcined sample with a Si/Zr=20 shows the highest peak intensity, and the calcined sample with Si/Zr=5 shows the lowest peak intensity, indicating that a too high zirconium content may lower the structural ordering in the resultant material (see Fig. 1c). It is possible that too many zirconium ions incorporated into the framework of Si-MCM-41, might result in a partial collapse of the mesostructure. As shown in Table 3, the wall thickness largely increases by increasing the zirconium content, until the Si/Zr molar ratio decreases to 5, indicating the incorporation of zirconium into the framework.

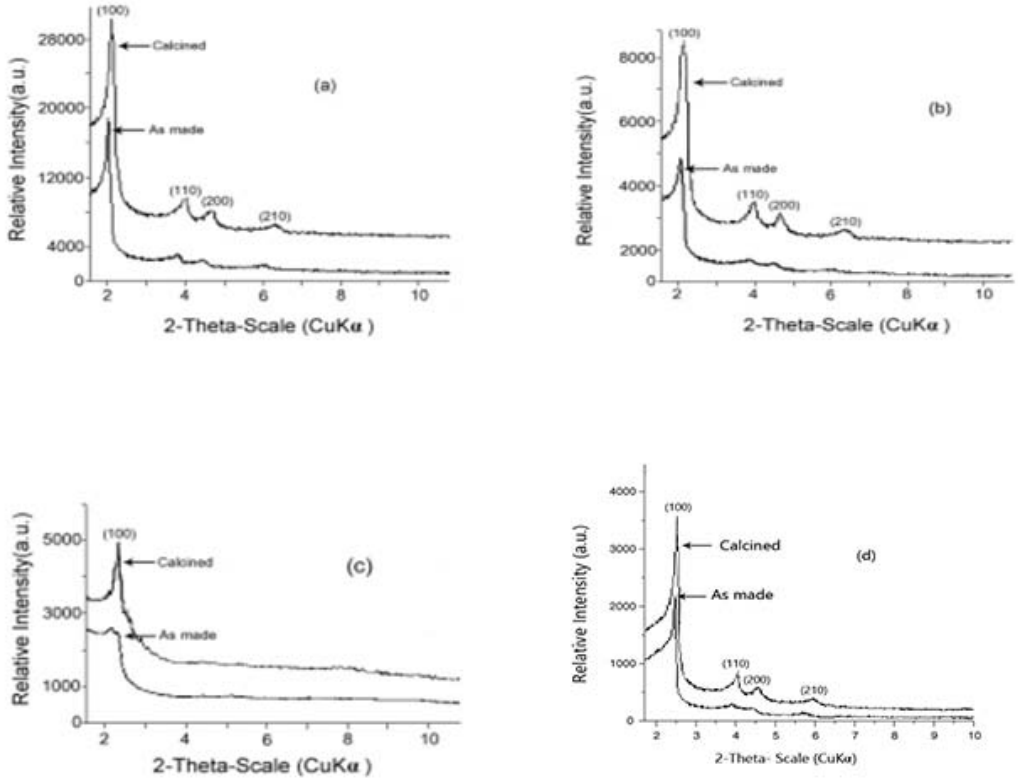

Figure 1 : XRD patterns of the as-made and calcined Zr-MCM-41:(a)Si/Zr=20, (b)Si/Zr=10, (c) Si/Zr=5 and (d) Si-MCM-41

Table 3 : Structural data obtained from N_2 physical sorption isotherms and XRD of Zr-MCM-41

Si/Zr	hkl	2θ(CuKα)	d_{100}(nm)	a_0(nm)	d_b(nm)	t(nm)
20	100	2.38	3.76	4.38	2.45	1.93
10	100	2.34	3.83	4.43	2.48	1.95
5	100	2.32	3.87	4.49	2.51	1.98
∞	100	2.41	4.02	4.64	2.72	1.92

ii. *Textural properties*

Figure2 (a, b, c) shows the loops of the N_2 adsorption-desorption isotherms of the calcined Zr-MCM-41 samples. Four regions are observed: The first stage, at $P/P_0 < 0.2$, is due to a monolayer adsorption of nitrogen molecules on the walls of the mesopores. The second stage, at $0.3 < P/P_0 < 0.4$, determined by a high increase in adsorption, is due to capillary condensation inside the mesopres. The third stage, the adsorption isotherm is the horizontal section beyond the P/P_0 of 0.4,

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which is corresponds to multilayer adsorption on outer surface of the particles. The last stage at $P/P_0 > 0.9$ can be related to capillary condensation in the solid with high zirconium content. The pore diameter distribution of the samples with low zirconium content, Si/Zr=20 and 10, shows only a single peak around 21.90 and 22.5 Å. However, in the sample with high zirconium content, Si/Zr=5, some larger pores appear, which is due to the formation of mesopores between particles. The Si/Zr=5 sample shows a single peak around 24 Å which is higher than single peaks for both samples of Si/Zr= 20 and10. This is due to the collapse of mesopores in the solids with high zirconium content. The high zirconium content in the mesoporous solids reduces the pore volume and the surface area. The determination of the mesopore volume was based on the N_2 adsorbed at $P/P_0=0.4$. The surface area of the mesoporous solids were 997, 811 and 446 m^2/g for Si/Zr= 20, 10 and 5 respectively.

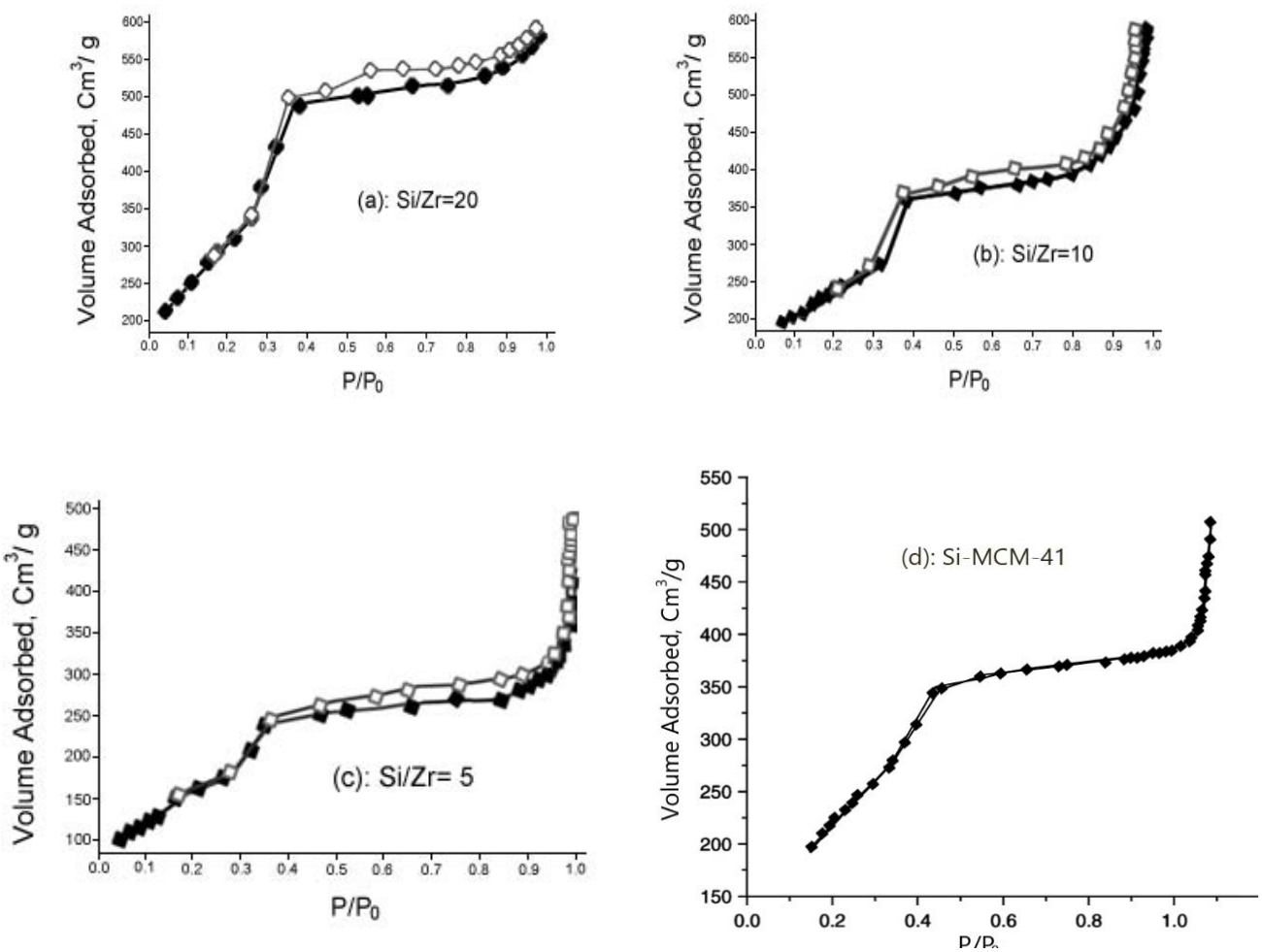

Figure 2 : N2 adsorption-desorption isotherm of the calcined Zr-MCM-41:(a) Si/Zr=20, (b)Si/Zr=10,(c) Si/Zr=5 and (d)Si-MCM-41

iii. *Morphology features*

The morphology of MCM-41 and Zr-MCM-41 materials was studied by scanning electron microscopy. Typical SEM images are given in Figure 3. It can be seen that, whatever the zirconium loading of MCM-41 and Zr-MCM-41 (Si/Zr=10 and 20), the morphology of parent MCM-41 and Zr-modified samples consist of particles less than 1μm with irregular shapes, probably due to agglomeration of particles. It is obvious that smaller particles can be recognized in the sample of Si/Zr=20.

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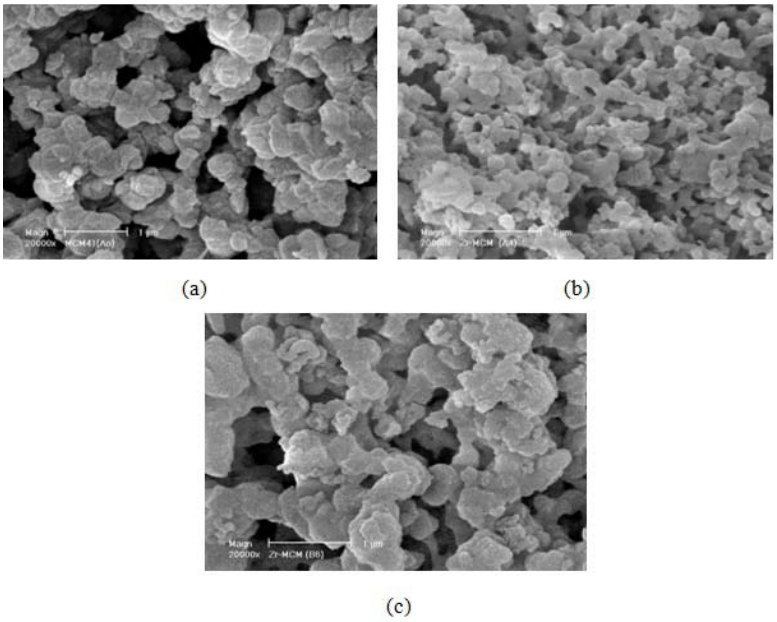

Figure 3 : SEM image of the samples (a)MCM-41, (b) Zr-MCM-41 (Si/Zr=20), (c) Zr-MCM-41(Si/Zr=10)

iv. *UV-Vis DR analysis*

The calcined Zr-MCM-41 solids were also characterized with UV-Vis DR spectroscopy. For the purpose of comparison, the UV-Vis DR analysis of the pure Si-MCM-41 sample was also included as reference. All the Zr-MCM-41 solids show a band around 200 nm which is due to the charge-transfer transition from an oxygen ion to a Zr(IV) ion (see Fig.4), relating to the excitation of electrons from the valence band (2*p* character in O) to the conduction band (4*d* character in Zr).

The Si-MCM-41 sample did not display any peak in the same wavelength range, between 190 and 800 nm. Usually, in the UV-V is spectrum, an absorption band around 250 nm corresponding to Zr^{+4} in monoclinic ZrO_2 phase and an absorption band at around 300 nm of octahedral Zr^{+4} in the perovskite-type $SiZrO_3$ can be observed [8].Our results suggest that the zirconium ions in the Zr-MCM-41 samples are in a different state than in pure ZrO_2 or $SiZrO_3$ (or pure Si-MCM-41 solid) and no separated ZrO_2 phase was formed in our samples. This is another strong evidence of zirconium incorporation into the framework of the mesoporous materials. The intensity of the band increases as the zirconium concentration increases, once again, indicating that more zirconium ions are incorporated into the Si framework at higher zirconium contents.

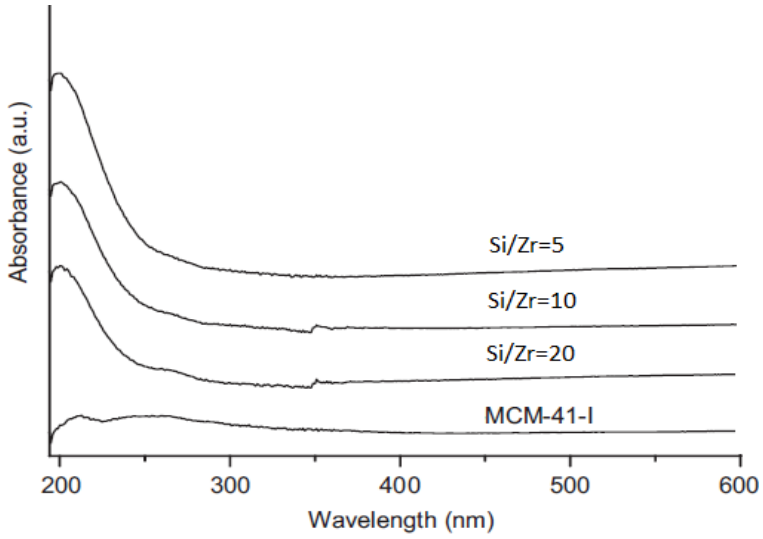

Figure 4 : UV-Vis defuse reflectance spectra of MCM-41 and Zr-MCM-41(Si/Zr=20, 10 and 5)

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b) FTIR studies

The FTIR technique was used for the surface characterization of the Zr-MCM-41catalyst [9]. As shown in Figure 5, the bands at 1621–1641 cm^{-1} are aroused by the vibration of the adsorbed water molecules; the band around 811 cm^{-1}corresponds to the bending vibration of Si–O; the band about 460 cm^{-1} is from the bending vibration of Si–O. The band around 3500 cm^{-1} was attributed to the Si-OH stretching bond. As can be observed in Figure 5, for the pure silica MCM-41 sample, the band at 1081 cm^{-1} is from the anti symmetric stretching vibration of Si–O–Si. The Si–O–Si bands of the Zr-MCM-41 shifted to 1079 cm^{-1}, at the same time, we can also observe that the intensity of the band 1081 cm^{-1} is gradually reduced as the zirconium content increases. The red shift in the Si–O–Si band of Zr-MCM-41 sample with the increase in the zirconium content is probably due to the replacement of Si ions in the framework by Zr^{+4} ions.

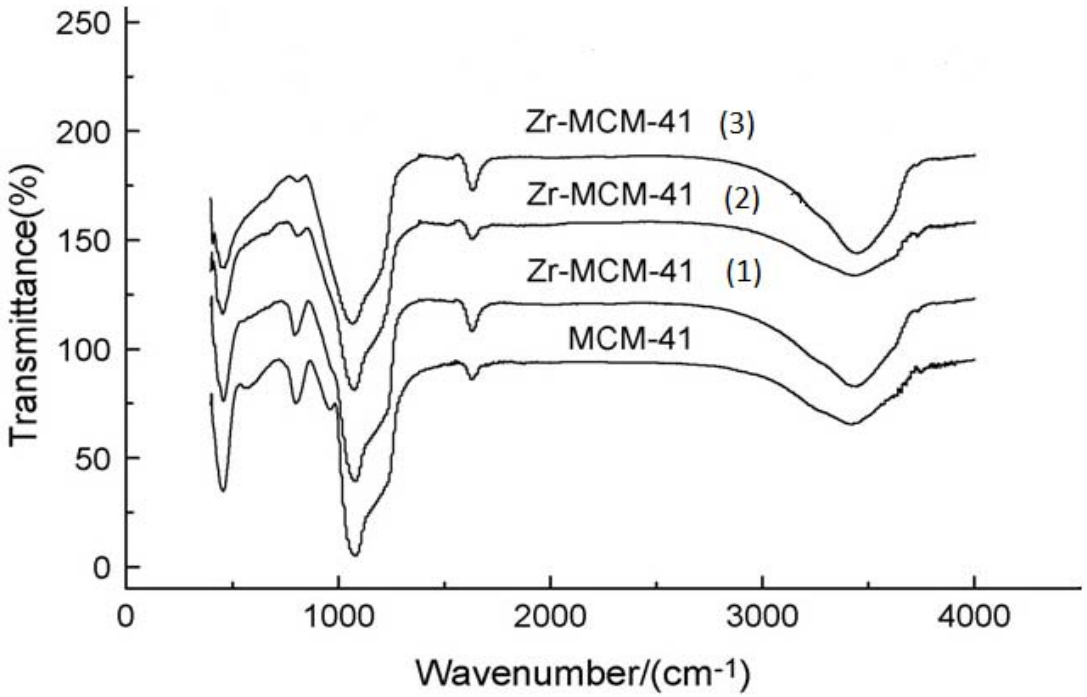

Figure 5 : FTIR Spectrums of Zr-MCM-41(Si/Zr=20 (1),Si/Zr=10 (2),Si/Zr=5 (3)) and MCM-41

c) Surface acidity of the Zr-MCM-41 solids

Figure 6 shows a set of in situ FTIR spectra of pyridine adsorption on the samples of Zr-MCM-41. In the Zr-MCM-41 both Lewis and Brönsted acid sites are formed. These mesoporous solids are heated to 100°C inside IR cell under vacuum. Lewis acid sites formed on the samples with the Si/Zr molar ratios 20, 10 and 5 characterized by the absorption bands at 1445, 1580 and 1598cm^{-1}. The absorption band corresponding to pyridine associated with both Lewis and Brönsted acid sites was observed at 1490 cm^{-1}. Brönsted sites appeared in these solids as indicated by the band at 1540 cm^{-1}. The band at 1640 $cm^{-1}$was attributed to pyridine adsorbed on the hydroxyls ions. The intensities of all the bands corresponding to Lewis acid sites are considerably decreased with the enhancement of zirconium content; simultaneously the intensities of all the bands corresponding to the Brönsted acid sites are almost unchanged. It is known that the surface acidity of mesoporous solids is influenced by incorporation of zirconium ions in the framework. The diameter of Zr^{+4} ions is much larger than that of Si^{+4}.Therefore, the replacement of Zr^{+4} ions with smaller Si^{+4} ions in the framework of solid creates the bond length of Zr-O-Si which is obviously different from that of Si-O-Si that leads to micro-strain within lattice cell. As can be seen from Fig. 6 the presence of silanol groups and small amounts of zirconium may affect the strength of Si-OH bond in the structure. Furthermore, there are several structural defects located in the framework that led to the unbalance situation in the electron charges. The variation of electron density around Si nuclei which can be considered as a result of charge unbalance, variations in electronegativity and local deformation resultig from the introduction of Zr^{+4} ion into the vicinity of the hydroxyls groups carrying silicon can decrease the strength of SiOZr-O-H and SiO-H bonds [10]. This can be taken into account as one of origins which lead to an increase in the number of Brönsted acid sites on the zirconium containing mesoporous materials (Zr-MCM-41). I rewrote this part again and I made several changes and correction to it.

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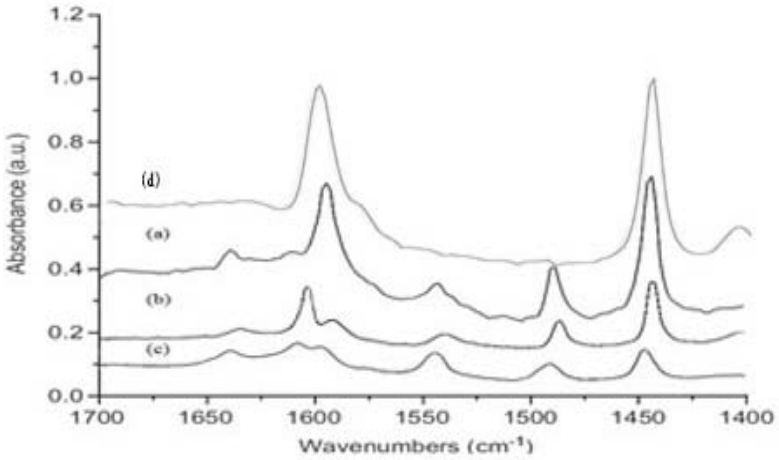

Figure 6 : In situ FTIR spectra of the pyridine adsorption on the various solids. The spectra were recorded at 100°C under vacuum condition; (a): Si/Zr=20, (b): Si/Zr=10, (c): Si/Zr=5 and (d): Si-MCM-41

d) Catalytic Test

In this study, the acidic strength of the silicate molecular sieves (MCM-41) was greatly enhanced by the incorporation of Zr^{+4} ions into the Si-MCM-41 framework. The Zr^{+4} components created Brönsted acid sites on the mesoporous Zr-MCM-41.

Initial investigations of the effect of the catalyst type on the reaction yielded some interesting results (Table 4). Conversion of benzyl alcohol is between 15 and 48% for all different loadings of zirconium, selectivity for benzyl ester product is 100%. It seems that because of the less number of acidic active sites in the samples can inhibit the ether formation, since ether formation required more active acidic sites than ester formation, also the pore size of all Zr-MCM-41 samples, are probably medium, therefore they cannot accommodate the large ether molecule, and thus hinders its formation. The fact that dibenzyl ether is not formed when the reaction is carried out in the absence of any catalyst indicates the involvement of acid sites for the ether formation and the presence of these active sites within the pores of the mesoporous. The deterioration of catalytic activity observed when the Si/Zr molar ratios decreased which in turn increased the zirconium content in the mesoporous solids. The enhancement of zirconium content in the mesoporous materials leads to the variation of electron density around Si nuclei which results to the distortion of mesoporous structures with high zirconium content. Consequently, the incorporation of a proper amount of zirconium can remarkably increase surface acidities and Brönsted type acid sites in the solids.

Table 4 : Esterification of benzyl alcohol with acetic acid: effect of catalyst type. Acetic acid(AA):benzyl Alcohol(BA), 2:1(mol/mol); reaction time 1h; catalyst weight 0.5 g, in 383 K

Type of Catalyst	Conversion %
Blank (MCM-41)	15
Si/Zr=5	19
Si/Zr=10	35
Si/Zr=20	48

i. *Influence of molar ratio of the reactants*

The reaction was carried out over Zr-MCM-41(Si/Zr =10 and 20) using different molar ratios of acetic acid to benzyl alcohol. In all cases, only benzyl acetate was formed. The conversion of benzyl alcohol was found to decrease with the increase in concentration of benzyl alcohol in the reaction mixture. In the all cases, the conversion decreased as the molar ratio of acid: alcohol was varied from 2:1 to 1:3. The selectivity towards the ester was constant. Also, the selectivity towards dibenzyl ether was unchanged. Selectivity towards ester and dibenzylether was always 100% and 0%, respectively. For example, in the case of Si/Zr=20, the conversion of benzyl alcohol was found to decrease with the increase in the concentration of benzyl alcohol in the reaction mixture. In the case of Si/Zr=20, the conversion decreased from 50 to 37% as the molar ratio of acid: alcohol was varied from 2:1 to 1:3. In the case of Si/Zr=10, the conversion decreased from 39 to 10% on varying the acid: alcohol molar ratio from 2:1 to 1:3.

ii. *Influence of catalyst concentration*

In the case of Zr-MCM-41(n=20), the amount of the catalyst was varied from 0.5 to 1.5 g while keeping the molar ratio of acid: alcohol at 2:1. The reaction was carried out at 383 K for 1 h. The conversion of benzyl alcohol increased from around 50 to 80% on increasing the weight of from 0.5 to 1.5 g. In the case of Zr-MCM-

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41(n=10), the conversion increased from around 40 to 68% in the same catalyst range of 0.5–1.5 g.

iii. *Influence of temperature*

The esterification reaction was carried out in the temperature region 383–403 K while keeping the acid: alcohol molar ratio at 2:1 and the catalyst weight at 0.5 g (Table 5). In general, the conversion of benzyl alcohol increases with increase in reaction temperature. The selectivity for the ester was found constant over Zr-MCM-41(Si/Zr=5, 10 and 20).

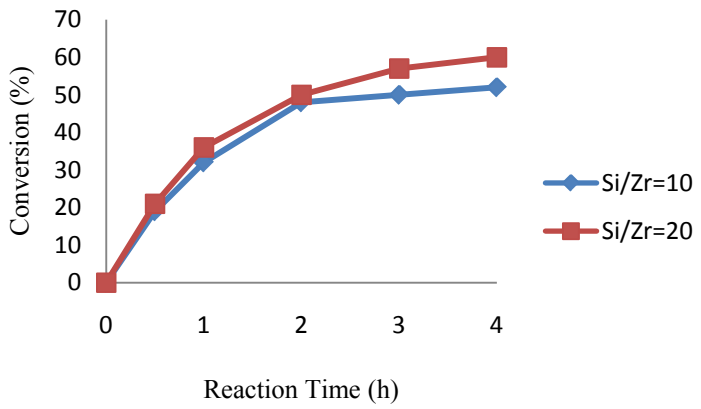
Over Zr-MCM-41(Si/Zr=5, 10 and 20), the selectivity for the ester was found to be 100% regardless of the reaction temperature. This suggests that over all samples of Zr-MCM-41, high temperatures favor the ester formation, whereas such formation is less facile at lower temperatures. Also, the temperature effect on Zr-MCM-41(Si/Zr=20, 10) was higher than Si/Zr molar ratios of 5, because the pores diameter of Zr-MCM-41(Si/Zr=20 and 10) was more regular and smaller than Si/Zr molar ratios of 5 that increased formation of ester in higher temperatures.

Table 5 : Esterification of benzyl alcohol with acetic acid: effect of reaction temperature AA:BA= 2:1(mol/mol); reaction time 1 h

Si/Zr	5			10			20		
T (K)	383	393	403	383	393	403	383	393	403
Conversion%	30	40	50	30	50	65	40	60	77

iv. *Influence of reaction time*

The conversion of benzyl alcohol increases rapidly in the beginning and gradually levels off after 2 h (Figure7). For example, over Zr-MCM-41(Si/Zr=20) the conversion increased from around 21% in the first 30 min to around 57% in 3 h; on increasing the reaction time to 4 h, the conversion increased only to 60%. There is no significant difference in the formation of ether. The ether formation is independent of the esterification reaction.



V. Conclusion

In this study, effort has been devoted to direct synthesis of highly ordered Zr-based mesoporous molecular sieves MCM-41-type through a surfactant-template approach. The molar Si/Zr ratio greatly influences the structural regularity and textural properties. XRD data show that the range ordering of the mesoporous structure of the catalyst decrease extremely as a result of the incorporation of higher amount of zirconium species. The presences of zirconium in tetrahedral coordination were indicated by UV-Vis DR spectra, which show an absorption band around 200 nm in the Zr-MCM-41. Calcination may significantly improve the structural ordering of the resultant materials. This catalyst can be applied in the esterification reaction of benzyl alcohol with acetic acid with a high yield.

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