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Research Article

Synthesis, Characterization, and Evaluation of Mesoporous Silica Catalysts for Biodiesel Production from Rice Bran Oil

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Abstract

This paper describes the synthesis, characterization, and catalytic application of three types of ordered mesoporous silica materials—Si-MCM-41, Si-SBA-15, and Si-KIT-6—for the production of biodiesel through the transesterification of crude rice bran oil (pp. 16, 27). The mesoporous supports were prepared via hydrothermal routes using distinct structure-directing templates. Analytical characterization via UV-Visible spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, Powder X-ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) confirmed high physical regularity and structural integrity. Catalytic screening in the transesterification of rice bran oil demonstrated varying yields, with Si-SBA-15 yielding the highest quantity of biodiesel at 83.91 g under standard laboratory conditions. The synthesized biodiesel fuels were characterized for fuel qualities, including boiling point, iodine value, moisture content, ash content, and acid value, showing favorable performance indices compared to conventional petrodiesel and standard homogeneous NaOH-catalyzed frameworks

Introduction

The rapid depletion of global petroleum reserves combined with deteriorating environmental conditions driven by vehicle emissions has forced an urgent investigation into sustainable and renewable alternative liquid fuels [1-3]. Biodiesel represents an advantageous transportation fuel alternative due to its biodegradability, minimal net greenhouse gas output, low particulate emissions, and lack of sulfur content [3-5]. The chemical synthesis of biodiesel relies heavily on transesterification, where triglycerides present in animal or plant fats are reacted with an alcohol to output fatty acid alkyl esters alongside a glycerol by-product [4].

Historically, homogeneous catalysts like sodium hydroxide (NaOH) or potassium hydroxide (KOH) have dominated commercial biodiesel processes due to short reaction parameters. However, homogeneous lines introduce downstream problems, including equipment corrosion, extensive wastewater production during purification, and catalyst separation limits. Heterogeneous solid catalysts, particularly mesoporous molecular sieves, circumvent these bottlenecks due to easy structural recovery, excellent thermal stability, and large internal active surface areas capable of managing bulky organic reactants. [5-7]

According to International Union of Pure and Applied Chemistry (IUPAC) metrics, mesoporous materials possess regular pore patterns with diameters ranging between 2 nm and 50 nm. Among these, the M41S family (e.g., hexagonal packed MCM-41), Santa Barbara Amorphous-15 (SBA-15), and Korea Advanced Institute of Science and Technology-6 (KIT-6) provide highly tunable, thick-walled silica networks that act as promising heterogeneous frameworks (7-10). Utilizing a non-conventional, inexpensive, low-grade agricultural byproduct like rice bran oil (RBO) as a primary feedstock offers substantial economic advantages, bypassing the food-versus-fuel conflict associated with edible oils. This study details the multi-material synthesis of Si-MCM-41, Si-SBA-15, and Si-KIT-6 to evaluate their physical properties and comparative performance metrics in processing RBO into biodiesel.

Materials and Methods

Catalyst Reagents

The physical precursors employed for material fabrication consisted of Triblock copolymer Pluronic, Tetraethyl orthosilicate (TEOS), Cetyl trimethylammonium bromide (CTAB), Aqueous ammonia (NH_3), n-butanol, hydrochloric acid (HCl), deionized water, crude rice bran oil, and absolute methanol.

Synthesis of Mesoporous materials

The Si-hydrothermal method involves a series of precise steps to achieve the desired material properties. Initially, 10g of a triblock copolymer is combined with 30g of deionized water, followed by heating under 60°C for 6 hours. Next, 4g of tetraethyl orthosilicate (TEOS) is added, with the mixture maintained at 323K for 23 hours. After this period, the resulting gel is dried for 24 hours. For the KIT-6 synthesis, 5g of the triblock copolymer is mixed with 160g of n-butanol and vigorously stirred at 308K for 1 hour. Subsequently, TEOS is introduced, and the mixture is allowed to react for 48 hours at 308K. After this reaction, it is kept overnight using HCl to facilitate the final steps. [11-13]

Results and Discussion

UV-Visible spectral analysis

The solid stateabsorption spectrum of the substance was recorded in the region of 200-800nm. The strong intense band is observed for the catalyst at 260nmand no absorption in the visible region.

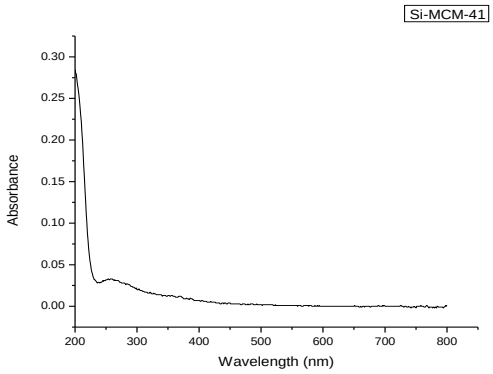


Fig. 1 UV-Spectral analysis of Si-MCM-41

X-ray diffraction spectral analysis

X-ray diffraction (XRD) is one of the most important techniques used to characterize the structural properties of mesoporous materials. The XRD pattern of the Si-MCM-41 mesoporous material synthesized by the hydrothermal method is shown in **Figure 9**. The diffraction pattern exhibits three characteristic peaks corresponding to the (100), (110), and (200) crystallographic planes, confirming the formation of a well-ordered hexagonal mesoporous structure. The crystallite size of the Si-MCM-41 catalyst was calculated using the Scherrer equation, and the average crystallite size was found to be **4.54 nm** [14].

$$D = K\lambda/\beta\cos\theta$$

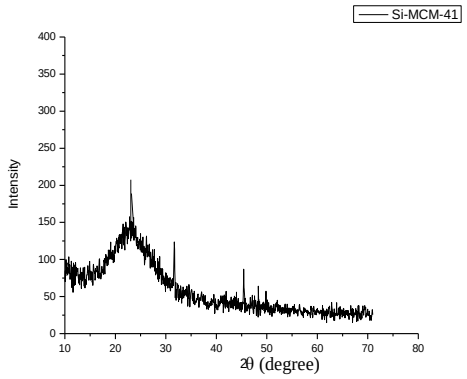


Fig. 2 XRD pattern of Si-MCM-41

Table1: Crystallite size of the Si-MCM-41

2θ	FWHM	Cosθ	nm
21.43	1.378	0.9824	4.61
28.23	1.382	0.9620	4.48

FT – IR spectral analysis

The FTIR spectrum of the synthesized Si-MCM-41 sample was recorded in the range of 4000–500 cm^{-1}. The broad absorption band observed at 1636 cm^{-1} is attributed to the bending vibration of adsorbed water molecules. The strong absorption band at 1060 cm^{-1} corresponds to the asymmetric stretching vibration of the Si–O–Si bridging bonds. The absorption bands in the range of 980–950 cm^{-1} are assigned to the stretching vibrations of Si–OH (silanol) groups present on the surface of the amorphous silica material. These characteristic absorption bands confirm the successful formation of the mesoporous Si-MCM-41 framework and the presence of surface silanol groups [15].

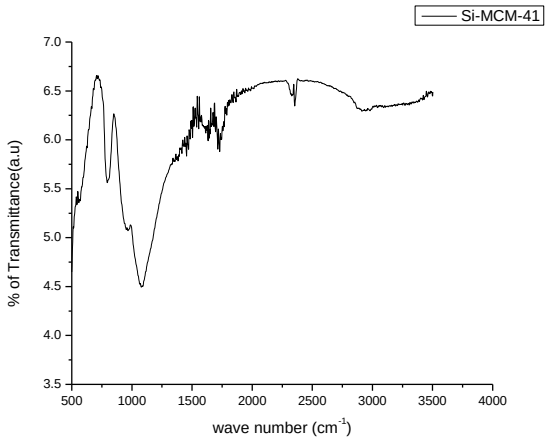


Fig. 3 FTIR spectral analysis of Si-MCM-41

Si-KIT-6

Thesynthesized Si-KIT-6was characterized by powderX-ray diffraction (XRD), Fourier transform infrared (FTIR), ultraviolet-visible (UV-vis), scanning electron microscopic analysis (SEM).

UV-Visible spectral analysis

The Solid state absorption spectrum of Si-KIT-6 was recorded in the region of 200-800nm. The maximum absorption spectrum of the compound occurs at 234nm.

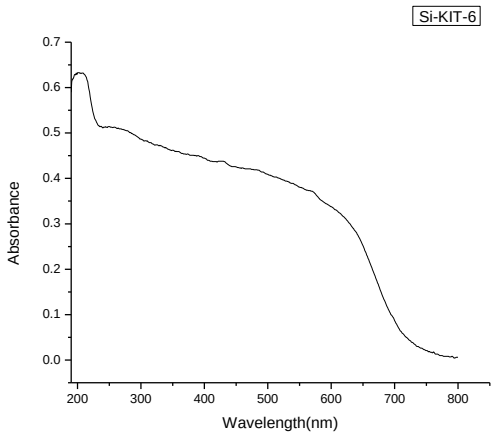


Fig. 4UV-visible spectral analysis of Si-KIT-6

X-ray diffraction analysis

X-ray diffraction pattern of Si-KIT-6 mesoporous materials which is obtained byhydrothermal method is

given in fig 5. The typical XRD pattern of Si-KIT-6 exhibits (001) and (200) planes of the tetragonal structure [16]. Crystallite size of the catalyst was calculated by using the Scherrer's formula. The crystallite size of Si-KIT-6 is 4.8nm.

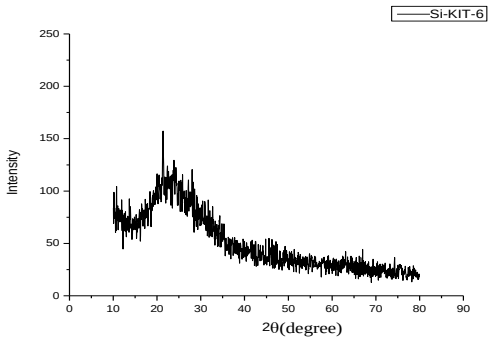


Fig. 5 XRD pattern of Si-KIT-6

Table2: Crystallite size of the Si-KIT-6

2θ	FWHM	Cosθ	Nm
21.53	1.478	0.9964	5.1
28.23	1.478	0.9620	4.6

FT – IR spectral analysis

The FTIR spectrum of the synthesized Si-KIT-6 sample was recorded in the range of 4500–500 cm^{-1}. The observed absorption frequencies and their corresponding vibrational bands confirm the characteristic functional groups present in the material. The absorption band at 2920 cm^{-1} corresponds to the symmetric stretching vibration of the C–H bond. The absorption band at 793 cm^{-1} is attributed to the bending vibration of the Si–O tetrahedral framework, while the band at 958 cm^{-1} is assigned to the framework stretching vibration of Si–O bonds. The broad absorption bands around 3600 cm^{-1} and 1600 cm^{-1} are attributed to the stretching and bending vibrations, respectively, of O–H groups associated with adsorbed water molecules. The intensity of these O–H absorption bands was found to be slightly higher for the Si-KIT-6 catalyst than for the other synthesized catalysts, indicating a comparatively higher surface hydroxyl content [17].

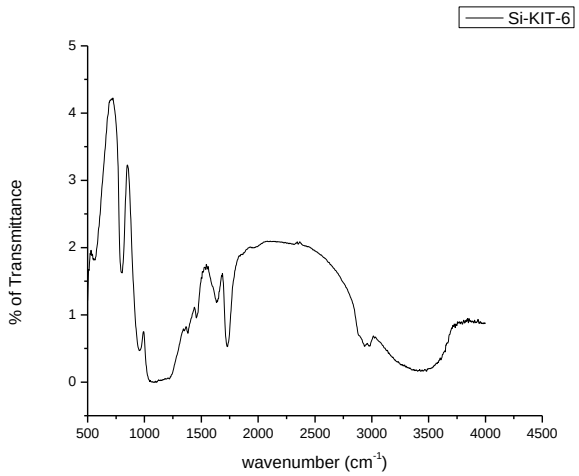


Fig. 6 FTIR spectral analysis of Si-KIT-6

Si-SBA-15

The Si-SBA-15 products were characterized by powderX-ray diffraction (XRD), Fourier transform infrared (FTIR), ultraviolet-visible (UV-vis) scanning electron microscopic analysis (SEM).

UV-Visible spectral analysis

A UV-Visible spectrum of SBA-15 isshown in fig14. The absorption spectrum of the substance was recorded in the region from 200-800nm.There are two strong intense bands observed at 250nm and 360nm.

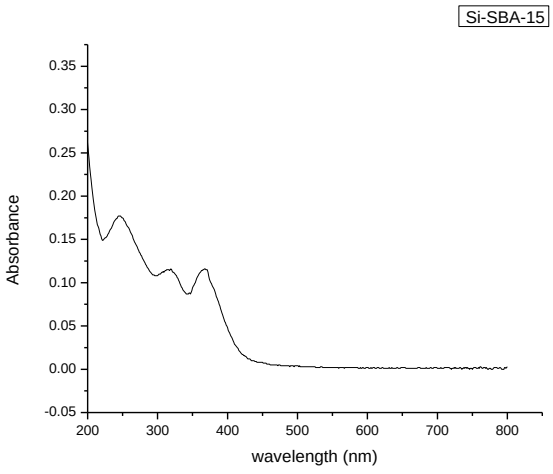


Fig. 7 UV-visible spectral analysis of Si-SBA-15

X-ray diffraction spectrum

The powder X-ray diffraction (XRD) pattern of the synthesized mesoporous silica Si-SBA-15 is shown in Figure 8. The diffraction pattern exhibits one intense peak corresponding to the (100) plane and two weak peaks

corresponding to the (110) and (200) planes, confirming the presence of a highly ordered hexagonal mesoporous silica structure [14]. The crystallite size of the Si-SBA-15 catalyst was calculated using the Scherrer equation, and the average crystallite size was found tobe 3.9nm.

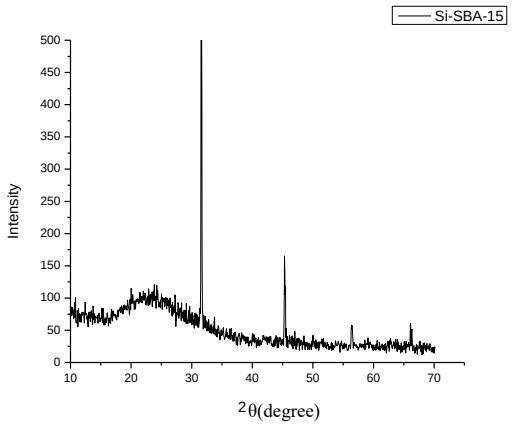


Fig. 8 XRD -Spectrum of Si-SBA-15

Table-3 Crystallite size of the Si-SBA-15

2θ	FWHM	Cosθ	d (nm)
31.67	1.378	0.9620	3.9
42.23	1.398	0.9335	4.1

FT – IR spectral analysis

The FTIR spectrum of the synthesized Si-SBA-15 sample was recorded in the range of 4500–500 cm^{-1}. The observed absorption frequencies and their corresponding vibrational bands indicate the presence of Si–OH, Si–O, Si–H, Si–C, C–O, and O–H functional groups. The broad absorption band around 3450 cm^{-1} is attributed to the stretching vibrations of O–H bonds from adsorbed water molecules and surface Si–OH (silanol) groups. The absorption band observed at approximately 1620 cm^{-1} corresponds to the bending vibration of O–H bonds associated with adsorbed water molecules. The strong absorption band around 1080 cm^{-1} is assigned to the asymmetric stretching vibration of Si–O–Si bridging bonds, confirming the formation of the silica framework. The absorption bands in the range of 980–950 cm^{-1} are attributed to the stretching vibrations of free Si–OH (silanol) groups present on the surface of the amorphous silica material. The symmetric stretching vibration of Si–O–Si bonds associated with the ring structure of the silica framework is observed in the region of 795–790 cm^{-1}. These characteristic FTIR bands confirm the successful formation of the mesoporous Si-SBA-15 framework with abundant surface silanol groups

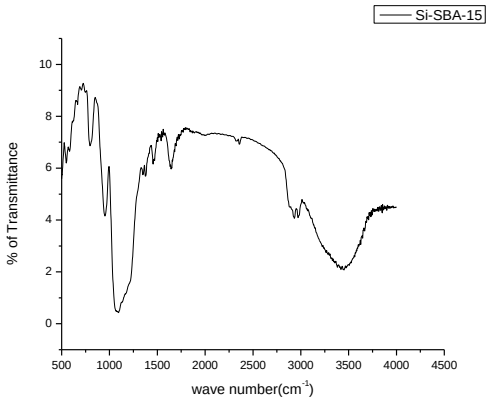


Fig. 9 FTIR spectral analysis of Si-SBA-15

Scanning electron microscopic analysis

The scanning electron micrographs of Si-MCM-41, Si-KIT-6 and Si-SBA-15 are shown in figure 17, 18 and 19. The Si-MCM-41 particles are spherical morphology and aggregate together. KIT-6 particles are well ordered sphere like morphological crystallite and Si-SBA-15 is spherical in shape and some smaller irregular shape particles are observed.

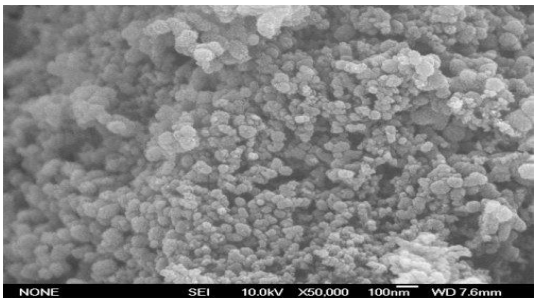


Fig. 10 SEM image of Si-MCM-41

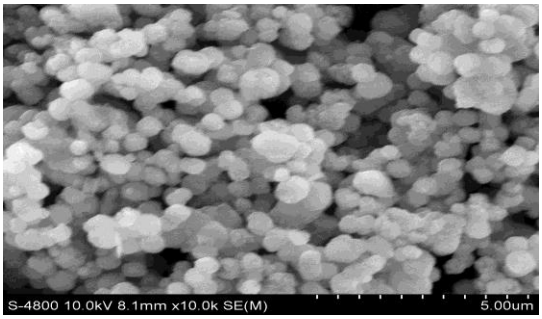


Fig. 11 SEM image of Si-Kit-6



Fig-12 SEM image of Si-SBA-15

CATALYTIC EFFECT OF MESOPOROUS

MATERIAL IN THE SYNTHESIS OF BIODIESEL

Biodiesel, the ester product obtained as the transesterification of fatty acids of vegetable oil, rice barn oil using methanol and $Ca(OH)_2$ or KOH as catalyst. The reaction can be represented as



Methanol Ester of fatty acids of rice barn oil alcohol ester

The role of catalyst for the above reaction is carried out using three mesoporous materials Si-MCM-41, Si-KIT-6, Si-SBA-15 and the yield was compared to find out the efficiency of the catalyst in the transesterification reaction.

DETERMINATION OF BOILING POINT OF BIODIESEL

The boiling point of biodiesel was determined and the data is given in table 4. It is clear that the boiling point of biodiesel is compared to the boiling point of conventional diesel. The boiling point of biodiesel was found to be 156^{o}C, 162^{o}C and 180^{o}C using Si-MCM-41, Si-KIT-6, and Si-SBA-15 respectively. The boiling point of conventional diesel is 149^{o}C.

Table-4 Boiling point of the synthesized biodiesel using different catalyst

S.No	Catalyst	Amount of biodiesel taken(ml)	Temperature ^{o}C
Biodiesel-1	Si-MCM-41	2	156
Biodiesel-2	Si-KIT-6	2	162
Biodiesel-3	Si-SBA-15	2	180
Biodiesel-4	NaOH	2	172
Diesel	-	-	149

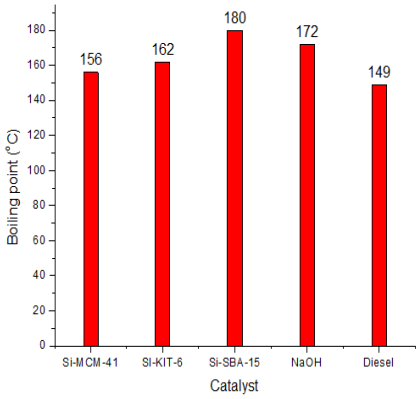


Fig-13 Boiling point of biodiesel with different mesoporous catalyst

Determination of Iodine value

The iodine value depends on the nature of the vegetable oil and is an indicator of the degree of unsaturation present in the fatty acid chains. The biodiesel synthesized from the same feedstock exhibited similar iodine values, as presented in **Table 5**. The iodine value of conventional diesel fuel was found to be approximately **112**

[18]. In comparison, the synthesized biodiesel exhibited a higher iodine value, indicating a significantly greater degree of unsaturation than conventional diesel fuel [18].

Table-5 Iodine value of synthesizedbiodieselusing different mesoporous catalyst

S.No	Catalyst	Amount of Biodiesel taken(ml)	Iodine value
Biodiesel-1	Si-MCM-41	0.2	128.27
Biodiesel-2	Si-KIT-6	0.2	128.27
Biodiesel-3	Si-SBA-15	0.2	128.27
Diesel	-	-	112

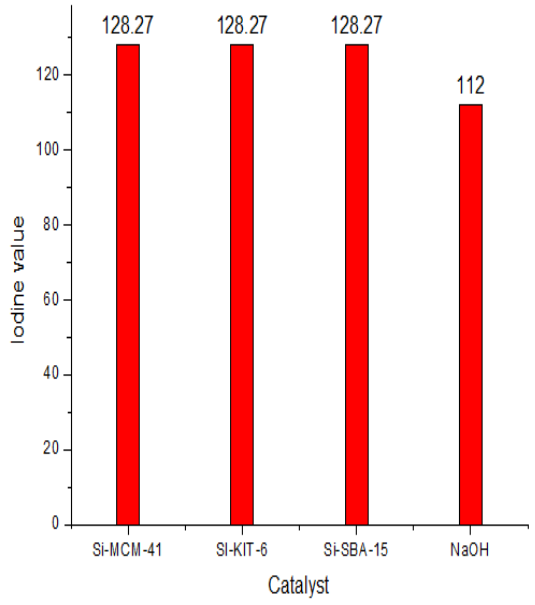


Fig-14 Iodine value of biodiesel with different mesoporous catalyst

Determination of Moisture Content

Biodiesel and its blends are susceptible to microbial growth when water is present in the fuel. Therefore, the moisture content is an important quality parameter that influences the storage stability and performance of biodiesel. The moisture content of the synthesized biodiesel was found to range from 0.0002 to 0.0004, whereas the moisture content of conventional diesel ranged from 0.0006 to 0.0007. The biodiesel synthesized using Si-MCM-41, Si-KIT-6, and Si-SBA-15 catalysts exhibited comparatively lower moisture content, indicating improved fuel quality and enhanced resistance to microbial contamination during storage [19].

Table-6Moisture content of biodiesel with different mesoporous catalyst

S.No	Catalyst	Amount of biodiesel taken(ml)	Moisture content
Biodiesel-1	Si-MCM-41	2	0.00035
Biodiesel-2	Si-KIT-6	2	0.00043
Biodiesel-3	Si-SBA-15	2	0.00041
Biodiesel-4	NaOH	2	0.0049
Diesel	-	-	0.0007

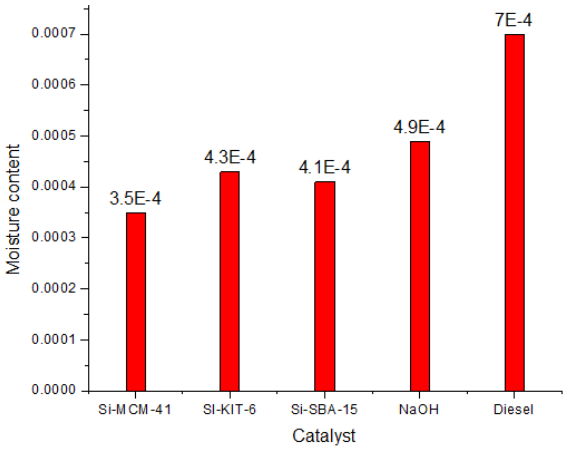


Fig. 15 Moisture content of biodiesel with different mesoporous catalyst

Determination of Ash Content

Ash content is determined to ensure the complete removal of catalysts used during the transesterification process. The presence of ash in biodiesel can lead to filter plugging and the formation of injector deposits, thereby

affecting engine performance. Soluble metallic soaps, residual catalysts, and other solid impurities are the primary sources of ash in biodiesel fuel [20]. The ash content of the synthesized biodiesel was found to range from 0.0052 to 0.0083, whereas the ash content of conventional diesel was 0.01. The lower ash content of the synthesized biodiesel indicates effective catalyst removal and improved fuel quality.

Table-7 Ash content of synthesized biodiesel with different mesoporous catalyst

Sample	Catalyst	Amount of biodiesel taken(g)	Temperature °C
Biodiesel-1	Si-MCM-41	2	0.0063
Biodiesel-2	Si-KIT-6	2	0.0078
Biodiesel-3	Si-SBA-15	2	0.0059
Biodiesel-4	NaOH	2	0.0065
Diesel	-	-	0.01

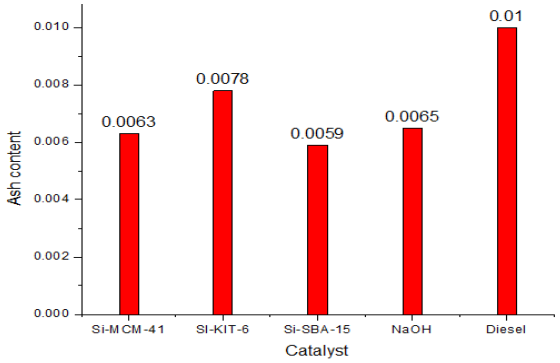


Fig. 16 Ash content of biodiesel with different mesoporous catalyst

Determination of Acid Value

The acid value is an important quality parameter used to assess the aging stability of biodiesel. It indicates the presence of free fatty acids or residual acids used during the biodiesel production process and also reflects the degradation of biodiesel caused by thermal effects. A high acid value may result in injector damage and the formation of deposits in the fuel system, thereby reducing the service life of fuel pumps and filters. The acid value of the synthesized biodiesel was found to range from 0.012 to 0.018, whereas the acid value of conventional diesel was 0.02 [20]. The comparatively lower acid value of the synthesized biodiesel indicates better fuel quality and enhanced storage stability.

Table-8 Acid value of synthesized biodiesel using different catalyst

S.No	Name of theCatalyst	Amount of biodiesel taken(g)	Acid Value
Biodiesel-1	Si-MCM-41	2	0.015
Biodiesel-2	Si-KIT-6	2	0.013
Biodiesel-3	Si-SBA-15	2	0.016
Biodiesel-4	NaOH	2	0.0165
Diesel	-	-	0.02

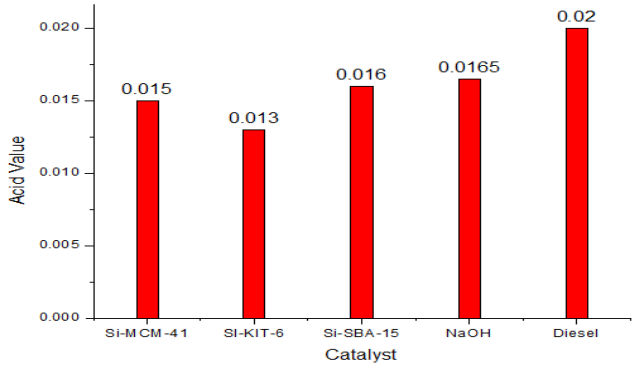


Fig. 17 Acid value of biodiesel with different catalyst

THE EFFECT OF CATALYST ON YIELD OF BIO DIESEL

The effect of catalyst on the yield of biodiesel was studied and it is observed that Si-MCM-41 produced large amount of biodiesel than the Si-KIT-6 and Si-SBA-15.

Table-9 The effect of catalyst on the yield of biodiesel

Catalyst	Yield of the product (g)
Si-MCM-41	75.68
Si-KIT-6	68.12
Si-SBA-15	83.91
NaOH	78.51

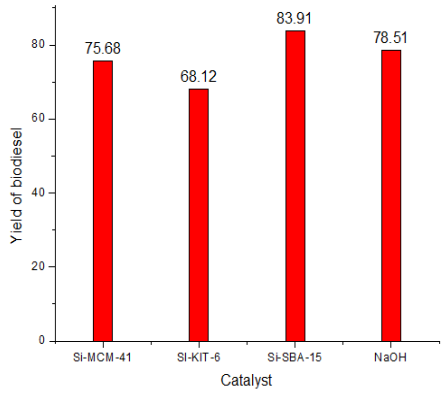


Fig. 18 Yield of biodieselVs catalyst

In this chapter,thecharacterization of three newly synthesized mesoporous materials was discussed. The particle size of the mesoporous materials was determined from XRD data using scherrer's formula as 4.5nm, 4.8nm, and 3.9nm for Si-MCM-41, Si-KIT-6 and Si-SBA-15 respectively.Biodiesel was synthesized by transesterification of ricebarn oil using conventional catalyst, NaOH and by newly synthesized mesoporous catalyst Si-MCM-41, SI-KIT-6, Si-SBA-15. The yield and the quality of biodiesel were compared with petro diesel to highest the efficiency of the catalyst on the transesterification reaction. The pore size of the porous materials (BETanalysis)are yet to be analysed.

SUMMARY AND CONCLUSION

Three mesoporous materials such as Si-MCM-41, Si-KIT-6, Si-SBA-15 were synthesized by hydrothermal method. Si-MCM-41 was synthesized from TEOS (tetraethyl ortho silicate) using CTAB as surfactant.Si-KIT-6was synthesized from TEOS using Pluronic - P123 as template and n-butanol with active sites of OH group. Si-SBA-15 was synthesized from TEOS using pluronic - P123 and HCl with active sites of H^+. The newly synthesized nanomesoporous materials were characterized using UV, XRD, SEM and IR spectral analysis. The particle sizes of mesoporous materials were determined from XRD data using Scherr's formula as 4.54nm, 4.8nmand 3.9nm for Si-MCM-41, Si-KIT-6 andSi-SBA-15 respectively. Biodiesel was synthesized from rice barn oil using conventional catalyst NaOH by transesterification process. Biodiesel was also synthesized using different mesoporous materials as catalyst (Si-MCM-41, Si-KIT-6,Si-SBA-15) and the yield of the formation of biodiesel and the efficiency of the catalyst on the transesterification reaction was also evaluated. Si-SBA-15 catalyst shows high yield in the formation of biodiesel from rice barn oil.

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