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Hydrothermal Synthesis of Titanium SBA-16 Mesoporous Materialusing Ultrasonic Treatment and Its Antibacterial Activities

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Abstract: *Mesoporous molecular sieves in the hexagonal phase (SBA16) are synthesized from rice husk ash (R.H.A.) solutions using Pluronic F127 as template and R.H.A as silica source. It is found that R.H.A. effectively transformed into mesoporous materials depending upon the hydrothermal conditions. It is also found that a high concentration of Na^+ ions is not critical in the formation of SBA - 16 when prepared under controlled pH of gel, calcinations temperature and calcinations duration conditions. We provide direct evidence of SBA -16. 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 SBA16 is obtained without hydrothermal treatment in short interval of time. X-ray diffraction (XRD) shows that the highly ordered meso structure was maintained even at the high loading of titanium up to5.5 (bulk molar ratio SiO2/TiO2). The synthesis of Mesoporous TiO2-containing SBA-16 composite with a cubic Im3m structure will open new applications for catalysts. Antibacterial activities of TiO2-containing SBA-16 reveal that it was found that tested Pseudomonas cf. monteilii 9culture found resistant while compared to antibiotic.*

Keywords: *Hydrothermal synthesis, Pluronic127, Rice husk ash (R.H.A.), SBA-16, Si/Ti ratio, Pseudomonas cf. monteilii 9, resistance*

I. Introduction

Rice milling generates a byproduct know as husk .This surrounds the paddy grain. During milling of paddy about 78 % of weight is received as rice, broken rice and burn .Rest 22 % of the weight of paddy is received as husk. This husk is used as fuel in the rice mills to generate steam for the parboiling process. This husk contains about 75 % organic volatile matter and the balance 25 % of the weight of this husk is converted into ash during the firing process, is known as rice husk ash (RHA). This RHA in turn contains around 85 % - 90 % amorphous silica. India is a major rice producing country, and the husk generated during milling is mostly used as a fuel in the boilers for processing paddy, producing energy through direct combustion and / or by gasification. This RHA is a great environment threat causing damage to the land and the surrounding area in which it is dumped. Lots of ways are being thought of for disposing them by making commercial use of this RHA. Mesoporous materials Synthesized of by using hydrothermal method[1, 2, 5.] The properties of these materials make them attractive for adsorption, catalysis, separation, chemical sensing, optical coating, drug delivery and electronic applications. For practical purposes, the overall morphology of a mesoporous material is a necessary requirement in combination with their internal structure. For instance, in application such as high performance liquid chromatography isometric particles are required and spherical particles are preferably used in chromatography for column packing as irregular particles [3,4,6] tend to break down . In this body-centered-cubic structure each mesoporous is connected with its eight nearest neighbors to form a multidirectional system of mesoporous network[7-11] .Due to its large cage, high surface area and high thermal stability, this material appears to be one of the best candidates for catalytic support and packing materials for separation[12-16]. Using F127 as a surfactant is the common way of synthesizing SBA-16 However; there are also reports on alternative surfactants such as F108. a blend of P123 and F127 , and other nonionic surfactants[17-19] . Several studies have been carried out to understand the formation mechanism of this material, for instance[20-25], in the framework of the colloidal phase separation mechanism (CPSM) Yu et al. suggested that, the formation process of mesoporous materials involves three stages: (1) operative self-assembly of inorganic/organic composites, (2) Spherical particles of mesoporous silica SBA-16 structure were synthesized at low pH using Pluronic F127 as template and RHA as silica source[26-29].

Experimental details
Material synthesis

Ti containing **SBA16** composite with different concentrations have been prepared under acidic conditions in the presence of triblock copolymer F127 by using RHA as silica source and Tio_2 as Titanium

source.1.6gm of F127was dissolved in 40gm of 2MHCL under magnetic stirring to obtain homogeneous solution at 30^0c to this solution was added Si/Ti molar ratio of 10 after the mixed solution was further vigorously stirred for 30 min. The mixture solution was further stirred for another 24 hours .Ultrasonic treatment is given at power 70 for 30 min.Then the solution is taken Teflon coated autoclave and heated at 80^0c for one day .The synthesized Sio_2/ Tio_2 mesoporous composite was filtered and dried in air .The sample is calcined at 1.5^oc/ min.at 550^0c for 6hours(Shaodian Shenet al. [30]

Antibacterial Activity of TiO2-containing SBA-16:
Microbial Strain:

To test the antibacterial activity of TiO_2-containing SBA-16, *Pseudomonas cf. monteilii 9* cultures was used [31]which was grown on nutrient broth for 24 hour at 37°C.

Disc Diffusion Method:

Disc Diffusion Method was performed for testing the antibacterial activity of TiO2-containing SBA-16 (Bauer *et al.*, 1959 [31]. First Muller Hinton agar plates were spreaded by bacterial cultures grown earlier in nutrient broth with the help of sterile cotton swabs. Allow the agar surface to dry for 5 minutes. By using the flamed sterile forceps, pick up a sterile filter paper disc and dip the disc in the TiO_2-containing SBA-16 dilutions prepared by ultra sonic treatment. The dipped disc was placed near the edge of the agar surface of the inoculated plate and pressed gently to ensure firm contact of the disc with agar surface. Another disc dipped with another dilution of TiO2-containing SBA-16of different ultrasonic treatment. All the inoculated plates were incubated at 37 °C for 24 to 48 hours in an inverted position. After incubation at 37°C a clear zone around the disc was an evidence for antimicrobial activity.

II. Results And Discussion:

The XRD pattern of rice husk Ash (RHA) shown in Fig.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 quantity changes in the composition of Quartz and Mullite reactants that affecting reaction conditions of hydrothermal synthesis of materials and reaction products.

The X-ray pattern of the synthesized mesoporous silica material is an highly periodic silica phases which is normally reflected by the distinct X-RD signatures at low angles from 1^0 to 30^0 as shown in Fig 1. 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 a prime characteristics of the hexagonal lattice symmetry of the SBA16 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 SBA16. 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

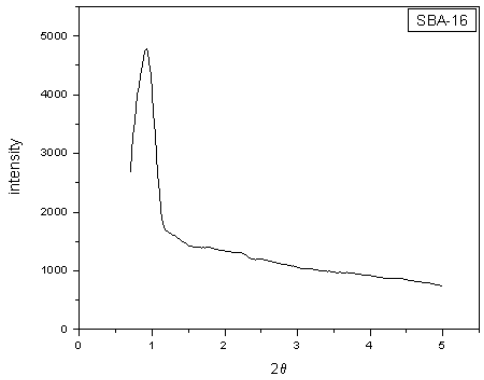

Figure1: X – ray diffraction data Effect of pH of synthesis gel:

The pH of reaction mixture of the gel is also plays an important role in synthesis of SBA16 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 500^0C for 8 h. The crystallinity reduces when pH of the gel is below 6.91 and above 6.91.

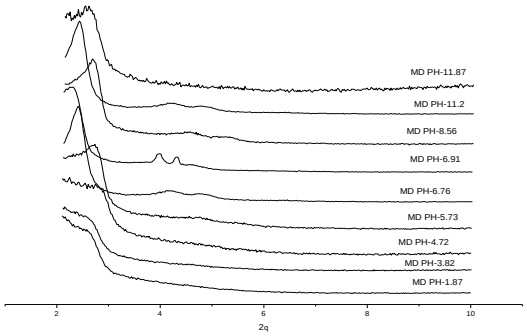

Fig. 2: XRD Patterns of SBA16 at different pH of synthesis gel.The FT-IR spectra of as synthesized SBA16 from Rice husk ash:

The FT-IR spectra of as synthesized from Rice husk ash are shown in Fig. 3. 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.

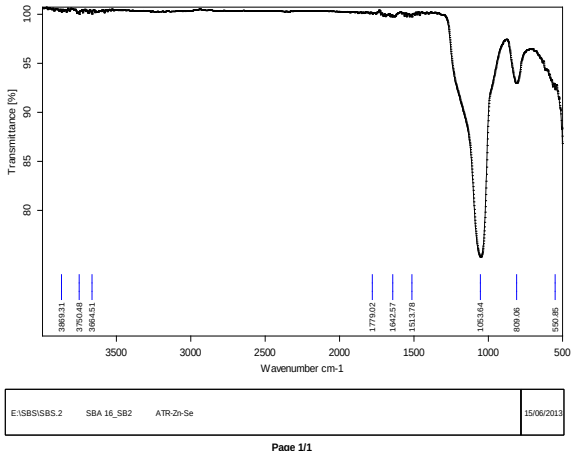

Figure 3: The FT-IR Spectra Of As Synthesized SBA16 From Rice Husk Ash Antibacterial Activity Of Tio_2-Containing SBA-16:

Pseudomonas cf. monteilii 9shown resistance against TiO_2-containing SBA-16, disc diffusion method and compared to antibiotic as shown in figure 4.

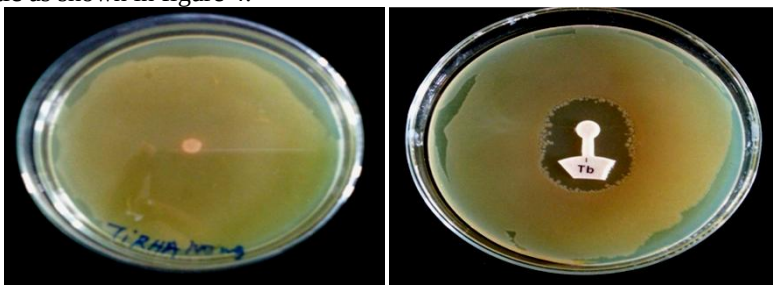

Figure 4: Antibacterial Activity of TiO_2-containing SBA-16

III. Conclusions

Based upon the experimental study it was concluded that pure and ordered SBA16 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 RHA the well ordered mesoporous material SBA16 can be synthesized at 550^0C for 4 hrs. Keeping pH of gel 6.91. The maximum calculated surface area amounts to 1801 m^2/g for the SBA16 materials keeping pH of gel 6.91, calcination time about 4 h. at 550^0C.

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