

A New Template for the synthesis of Nanoporous silicate Material

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ABSTRACT

Now a day's molecular sieves plays a vital role in the field of adsorption, heterogeneous catalysis and material science. Based on the pore size, molecular sieves are classified as macroporous, mesoporous and microporous. The mesoporous (2–5 nm) Aluminosilicate material has the uniform pore size distributions, comparatively large pore openings, huge surface area and superior adsorption capacities. The above properties shows their importance in the area of catalysis. The conventional methods of molecular sieves synthesis require costly templates and source materials, the synthesis procedure also intricate. To rectify these kinds of difficulties, a new phase molecular sieves is synthesized by using dye as a template. And the source materials are Aluminium Chloride and sodium meta silicate. The Aluminium chloride helps to remove sodium from sodium meta silicate. The silicate ion form molecular sieve structure by the direction of Rhodamine B dye molecule. The as synthesized material is characterized by TGA, to find out the Rhodamine B degradation temperature and thermal stability of the nanoporous material. It is find out that the template degraded at 410°C and the material is stable up to 900°C. The synthesized material is calcinated at 450°C and characterized by XRD, IR, BET, TPD and SEM and they proves nanoporous (13nm) molecular sieves formation.

KEY WORDS: Nanoporous, silicate material, dye molecule.

1. INTRODUCTION

Molecular sieves are the most essential material, especially with their pore size, many of the recent applications needs porous materials with the specific pore size distribution. To encourage that kind of requirements, Aluminosilicates and aluminophosphates are the widely used mesoporous molecular sieves in the field of heterogeneous catalysis, separation, adsorption and also possible new applications etc. Molecular sieves attracted much concentration due to their regular pore size, large surface area and pore volume and well ordered structure. The molecular sieves containing various types of pores, these pores are very useful for carry out the reactions. Recently a sequence of works are reported, the mesoporous materials are synthesised in the acidic medium.

This work provides an idea and motivation in the direction of design and synthesis of new nanoporous silicate molecular sieve material. And the novel mesoporous material is synthesized in the dye medium.

2. MATERIALS AND METHODS

Materials: Aluminium Chloride (98% Merck), Sodium silicate (meta) (Loba Chemie), Rhodamine B GR (C28H31ClN2O3 Loba Chemie) were used for the preparation of mesoporous silicate material.

Synthesis: In a typical synthesis, 13.5g of aluminium chloride was added to the 11.97g of Rhodamine B dye solution with vigorous stirring. Then 28.5g of sodium silicate powder was added to the mixture and stirred on a magnetic stirrer for 30 minutes then it will be kept for one day to attain complete precipitation. The gel molar composition was $\text{AlCl}_3 : \text{Na}_2\text{O}_3\text{Si} : 0.25 \text{ Rhodamine B} : 300 \text{ H}_2\text{O}$. The final product was washed repeatedly with distilled water. Then the sample was dried at 120°C for 3h in hot air oven. This as synthesized sample was calcined at 600°C in the presence of air until all the impurities and template were completely removed from the pores of the molecular sieves.

Characterization: The morphology was analyzed with a scanning electron micrograph (SEM) and the images were recorded in Carl Zeiss EVO 18. The experiment, temperature programmed desorption (TPD) were run under helium flow (30ml/ min) and the quantity of desorbed ammonia was measured by TCD detector. The TPD – NH₃ desorption curve was recorded at a rate of 20 deg / min from room temperature to 800°C and the signals were recorded chemisoft TPxV1.02.

3. RESULTS AND DISCUSSION

The synthesised sample is characterized by XRD, FT – IR, TGA, TPD, BET and SEM techniques. These techniques are used to verify the structure, thermal stability, surface area and morphology of the nanoporous silicate. **XRD Measurements:** The XRD pattern of the Fig 1 shows that the material has high thermal stability crystalline nature and also it confirms the various types of pores present in the molecular sieve. The calcined material shows the high intensity of the diffraction peaks at 11.1 deg with the d-spacing of about 7.9nm respectively. The sharp peak around in 11.1 deg of the calcined sample proved the high crystalline nature of the material.

FT – IR Spectrum: FT – IR spectrum of the as – synthesised and calcined samples are shown in Fig.2. The FT – IR was used to confirm the formation of tetrahedral framework of the nanoporous silicate. In Fig 2(b) shows the broad band in 3475cm⁻¹ it confirms the water molecule present in the as synthesised sample. The characteristic C –

H bands at 2900 to 2800 shows the presence of template molecule. After the calcinations the above stretching vibrations are absent. The tetrahedral framework of the nanoporous silicate was observed in 1000 to 1200 cm^{-1} region. The calcined material exhibits the bands at 1090 cm^{-1} (asymmetric stretching) and 790 cm^{-1} (symmetric stretching). These stretching vibrations are due to the fundamental vibrations of the tetrahedral formation, and the bending mode of vibration is observed near the 453 cm^{-1} region. The characteristic peak of 1090 cm^{-1} is confirms the O=Si=O vibration.

Thermal Analysis: Thermogravimetric (TGA) results of as synthesised nanoporous molecular sieves are depicted in Fig 3. The thermal analysis of the as - synthesised sample was studied from 50°C to 900 °C. From the figure we can know about the weight loss starts from 100°C to 230°C it recognized the desorption of water molecule from the surface. The second weight loss occur from 250 to 510°C because the elimination of template molecules from the material. The condensation of adjacent OH group which leads to a weight loss from 830 to 890°C. After that, raising the temperature, it does not show any weight loss in the nanoporous material which is established that the nanoporous silicate material is capable of thermal stability up to 900°C.

Temperature Programmed Desorption: In TPD, 55 mg sample was heated to 500o C@20 deg/min in 30ml High pure Helium flow kept at 500°C for 10 minutes.

N₂ Adsorption: This hysteresis loop is by the reason of porous adsorbents among pores in the range of 1.5 to 100nm.

SEM Analysis: Fig 6 shows the SEM images of calcined nanoporous silicate molecular sieves. The SEM images prove the synthesised molecular sieves are crystalline in nature. And the SEM morphology also proves the formation of pores in the molecular sieves.

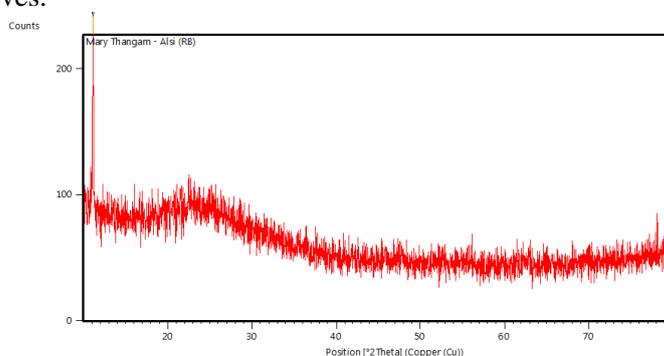


Figure.1.XRD pattern of calcined nanoporous silicate molecular sieves

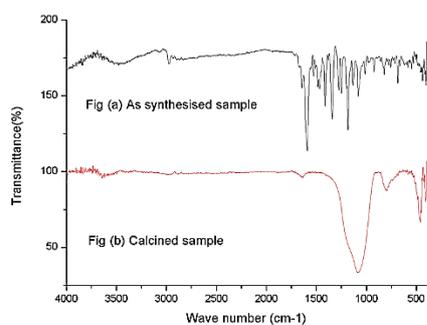


Figure.2.IR spectrum of nanoporous silicate molecular sieves

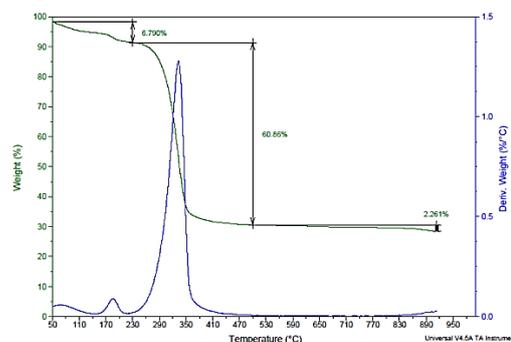


Figure.3.TGA curve for as synthesised nanoporous silicate material

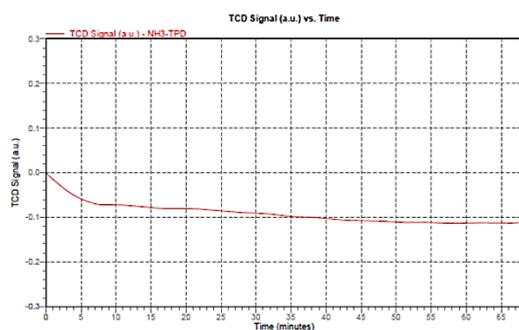
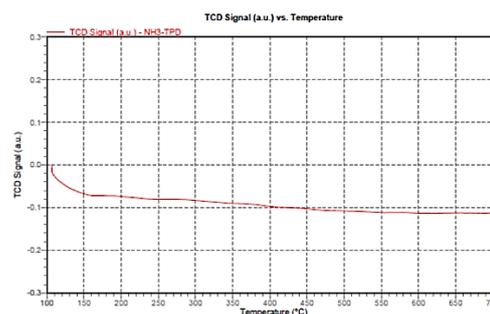


Figure.4.NH3 TPD for nanoporous silicate



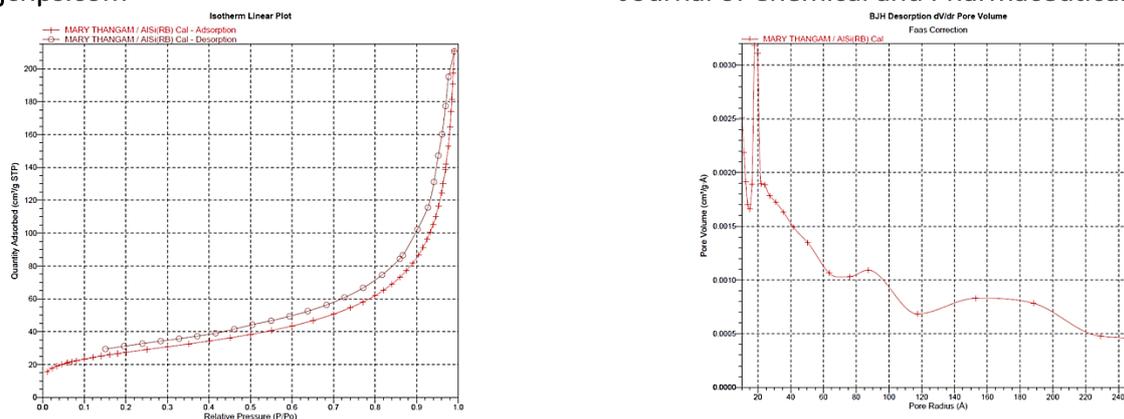


Figure.5.(A) N₂ sorption isotherm and (B) corresponding pore size distribution of calcined Nanoporous Silicate (the open circle represent desorption and the other symbol represent the adsorption)

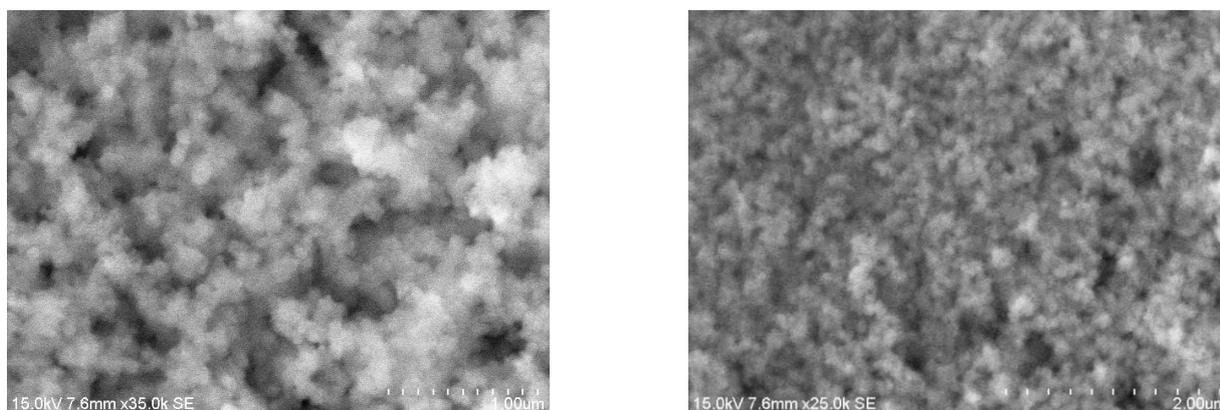


Figure.6.SEM image of nanoporous silicate molecular sieves

4. CONCLUSION

The novel nanoporous silicate molecular sieves are synthesized from inexpensive source material with a good surface area and pore size in a dye medium using Rhodamine B as a template molecule. This nanoporous silicate molecular sieve has various advantages like different types of pores, high thermal stability. And one of the main advantage of the material is, Zero acidity of the molecular sieves. In a dye medium, without acidity of the nanophase material synthesis is great achievement of the present work. This highly effective material synthesis is non-hazardous and eco – friendly. The synthesized nanoporous material is used for separating different kind of porous materials from the mixture of molecules.

5. ACKNOWLEDGEMENT

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