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FTIR SPECTRA OF ADSORBED PYRIDINE ON H-YAND USY AND TI-USY ZEOLITES AND MEASURE OF BRONSTED AND LEWIS ACIDITY PRODUCED ON THEM

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Abstract:

The FTIR of adsorbed pyridine has been used as a characteristic tool to detect and measure the presence of Bronsted and Lewis acid centers on the solid surfaces. The FTIR bands of pyridine are of interest to study the C-H and N-H stretching as well as the C-C and the C-N stretching in pyridine ring. In fig1-5 FTIR Spectra of adsorbed pyridine at 100,200, 300, 400OCon the samples are listed in the table are depicted. The acidity ,stability as well as catalytic activity of the Na-Y zeolite can be changed to various level by modifying its pore opening, pore volume void volumes as well as frame work Si/Al ratio and it is made suitable as a stable catalyst under sever conditions of catalytic reaction. It can also be used as a molecular sieves due to change in the dimensions of its pore opening due to its modification. The Na –Y synthesized zeolite is modified and made ultra-stable by controlled hydrothermal treatment. It is converted in to H-Y and various types of USY and Ti- USY zeolites. The modified H-Y and USY and Ti-USY samples produced are finally used for FTIR study of adsorbed pyridine in the present paper.

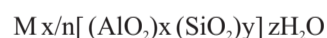
KEYWORDS:

(1) Synthesized highly crystalline Faujasite Na-Y zeolite (2) controlled Steam chamber for hydrothermal dealumination.(3)NH₄NO₃

.INTRODUCTION

What are Zeolites?

All Zeolites are crystalline hydrated alumino- silicates with rigid three dimensional infinitely extended framework structure. It has the cavities and channels of molecular dimensions .and permits the molecules to pass through it of the size smaller than our equal to the pore opening. Hence it acts as molecular sieves. The framework structure contains corner sharing of [SiO₄]⁻⁴ and [AlO₄]⁻⁵ tetrahedral linked through common oxygen atoms as the primary building units. The general empirical crystallographic unit cell formula of zeolite is expressed as follows,



where

M= charge compensating cations of valency. n.

Title:FTIR SPECTRA OF ADSORBED PYRIDINE ON H-YAND USY AND TI-USY ZEOLITES AND MEASURE OF BRONSTED AND LEWIS ACIDITY PRODUCED ON THEM Source:Review of Research [2249-894X] K.U.GORE yr:2013 vol:2 iss:8

x and y denotes the no. of moles of SiO₂ & AlO₂ where y = x and z = represents the no. of water molecules.

Almost all the zeolites are very unstable and collapse their structure and catalytic activity under the severe conditions of catalytic reactions and hydrothermal conditions at high temperatures. Due to large dealumination framework structure of zeolite collapses. To overcome this problem, the easily synthesized Y-zeolites in the industry or in the laboratory are dealuminated in a very controlled manner under hydrothermal conditions for increasing its catalytic activity as well as stability and are called as USY zeolites. The extra lattice aluminum species formed in it are removed by acid leaching. The unit cell of Y type zeolite is cubic with a large dimension of 25 Å and contains 192(Si, Al) O₄ tetrahedral, has remarkably stable and rigid framework structure with largest void space, which amounts to be nearly 40% by volume of the dehydrated crystal. Experimental: Already highly crystalline and synthesized Na-Y zeolite is obtained in pure form from Union Carbide Corporation, USA with Si/Al = 2.4 is used as original sample and modified for further applications. It is ion exchanged by using Ammonium Nitrate (NH₄NO₃) at 100°C temperature by reflux method about 8h. Then the solution is filtered and washed with hot deionized water, several times to remove any traces NO₃ ions from the sample, then dried sample is calcined at 500°C. The ion exchange is repeated three times to convert it into highly crystalline low silica H-Y form of zeolite.

The H-Y zeolite thus prepared is then used for stabilization and conversion into USY zeolite by treating it in steps very carefully under controlled hydrothermal conditions at 550°C for 4h and then gradually cooling on an average at the rate of 2°C/min. and then ion exchanged by using Ammonium Nitrate (NH₄NO₃) at 100°C temperature by reflux method about 8h. Then the solution is filtered and washed with hot de-ionized water, several times to remove any traces NO₃ ions from the sample, then dried sample is calcined at 500°C. The ion exchange is repeated three times to convert it into highly crystalline low silica H-USY-550 sample. This sample is then heated under hydrothermal condition very carefully to 700°C and then to 850°C in steps by keeping it for 4h at each temperature in the steam chamber. By taking the previous sample as a mother sample, for each next higher hydrothermal treatment. The further similar procedure is used like cooling after 550°C temperature. After heating hydrothermally at each temperature for 4h. The final samples prepared are named as MSTP-X-Y-Z respectively. Where X stands for sample prepared at first temperature, Y stands for second steaming temperature and Z stands for third steaming temperature and MSTP stands for multiple steaming temperature procedure. Thus samples prepared are identified as MSTP-550, MSTP-550-700, and MSTP-550-700-850 respectively. These samples are used for study of FTIR Spectra of adsorbed pyridine to know the type of acidity produced on the samples prepared. The above condition of the sample are standardized.

The acid strength distribution over H-Y, USY and Ti-USY samples by FTIR spectra of adsorbed pyridine.

Table-1

S N	Name of the samples & Temperatures in °C	Relative concentration of Bronsted acid sites				Relative concentration of Lewis acid sites				Bronsted / Lewis acid sites (B/L)			
		100°C	200°C	300°C	400°C	100°C	200°C	300°C	400°C	100°C	200°C	300°C	400°C
1	H-Y	45	44	39	33	46	28	21	21	0.98	1.56	1.85	1.56
2	550-MSTP	36	40.6	36	27	42	22	16	15	0.85	1.8	2.2	1.76
3	550-700-850MSTP-10	4	3	3	2	9	6	6	4	0.44	0.5	0.5	0.5
4	550-MSTP-55-0.44- HNO ₃ Ti-USY	5	4.5	4	3.2	6	3	2	1.7	0.83	1.5	0.2	1.88
5	550-MSTP-200-0.44- HNO ₃ Ti-USY	4.3	4.4	3.9	3	4.7	2.5	1.8	1.5	0.91	1.76	2.2	0.2

RESULT AND DISCUSSION:

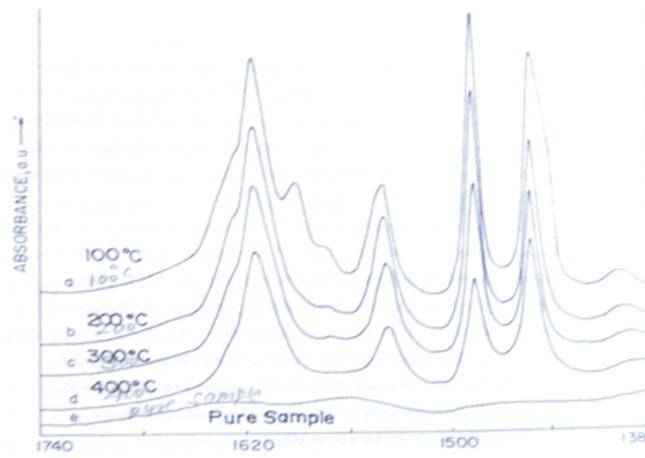
By inspection of the table reveals the following points.

1) Acid site density decreases monotonically with the severity of the hydrothermal treatment due to

dehydroxylation and dealumination.

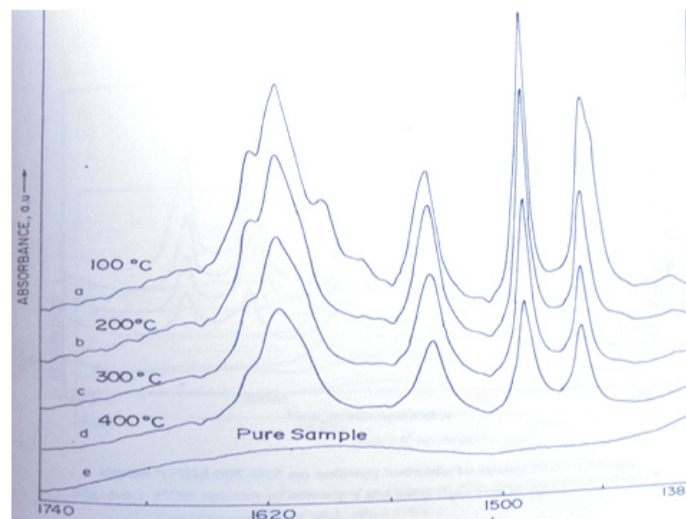
2) Hydrothermally dealuminated H-USY samples have greater number of Bronsted acid sites than Lewis acid sites at all temperatures other than at 100°C, because some amount of loosely physically bound pyridine possibly has remained on the sample at 100°C

Fig-1



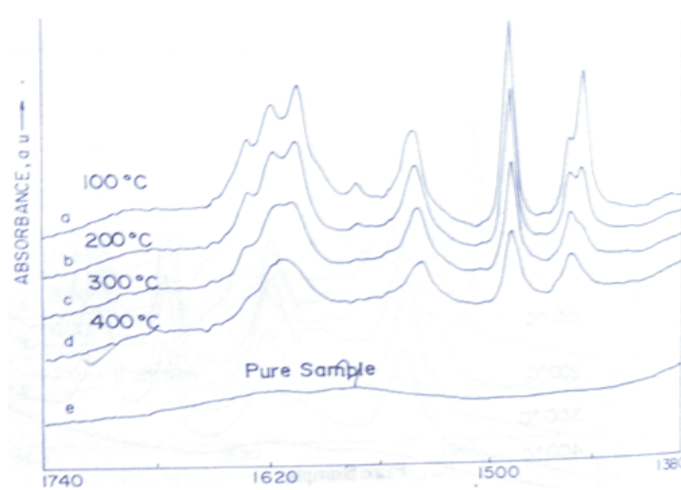
Wave number cm-1
FTIR Spectra of adsorbed pyridine on H-Y Zeolite

Fig-2



Wave number cm-1
FTIR spectra of adsorbed pyridine on 550-MSTP sample (high silica USY)

Fig-3

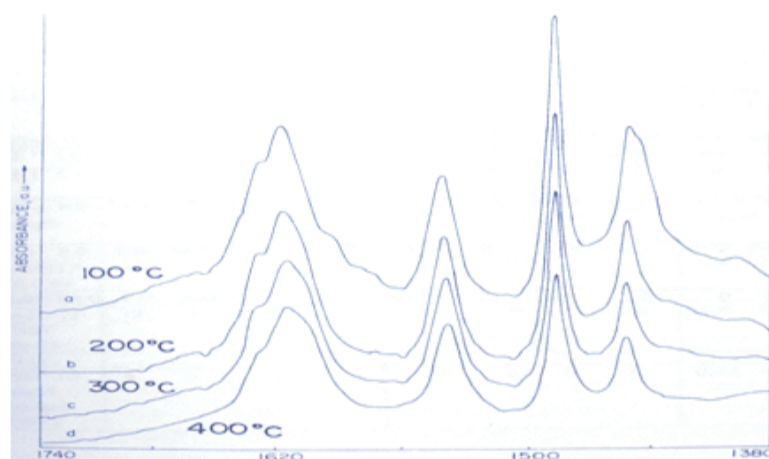


Wave number cm⁻¹

FTIR spectra of adsorbed pyridine on 550-MSTP-55-0.44-HNO₃ sample.
(acid treated high silicate-USY)

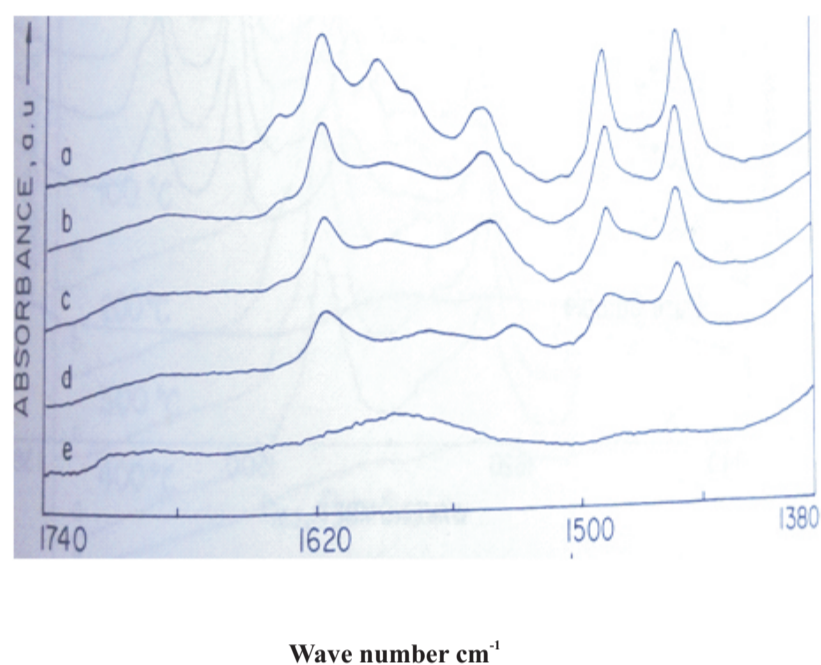
3)USY samples acid extracted many times have very small number of both Bronsted and Lewis acid sites,
but their proportion remains the same at all temperatures up to 400C.

Fig-4 FTIR spectra of adsorbed pyridine on 550-MSTP-200-0.44-HNO₃
(acid treated high silicate Ti-USY)



Wave number cm⁻¹

Fig-5



FTIR spectra of adsorbed pyridine on 550-700-850MSTP-10 sample
(acid treated very high silicate-USY)

4) Ti incorporation in this very high silica USY(Ti-USY) increases both Bronsted and Lewis acid site density and their proportion also remains constant at all temperatures up to 400°C.

CONCLUSIONS:

- 1) The FTIR spectra of adsorbed pyridine over Ti-USY indicates the generation of "super acid" hydroxyl groups and very strong and stable Bronsted and Lewis acidity, richer in Lewis type compared to Bronsted type over USY, Ti-USY zeolites.
- 2) The acid strength distribution and catalytic activity show that USY has very strong and more of Bronsted acid sites whereas Ti-USY has very strong and more of Lewis acid sites.

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