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(54) **Title**: CATALYST COMPOSITION (ICaT-2) COMPRISING OF RARE EARTH METAL

(57) **Abstract**: The water tolerant heterogeneous acid catalyst (ICaT-2) has been disclosed herein. The catalyst of the invention comprises of rare earth metals in the form of trifluromethansulphonate anchored with hexagonal organic- inorganic functionalized mesoporous silica as base metal through organic-inorganic linkages. The said catalyst composition has the specific surface area in the range of 200 m^2/g to 850 m^2/g and the pore diameter in the range of 20-50 Å. The ICaT-2 catalyst was found to be highly active and reusable for biomass based chemicals.

TITLE OF THE INVENTION

"CATALYST COMPOSITION (ICaT-2) COMPRISING OF RARE EARTH METAL"

FIELD OF THE INVENTION

The present invention is related to the process for preparation of a water tolerant heterogeneous acid catalyst (ICaT-2). The catalyst comprises of rare earth metals in the form of trifluromethane sulfonate anchored with hexagonal organic- inorganic functionalized mesoporous silica as a base metal through organic linkage. The said catalyst composition has specific surface area in the range of 200 m^2/g to 850 m^2/g and pore diameter in the range of 20-50 Å. The catalytic activity tests were carried out for dehydration reactions. The catalyst was found to be highly active and reusable for biomass based chemicals.

BACKGROUND OF THE INVENTION

Tightening of legislation for the treatment and disposal of excessive toxic waste, produced during the isolation and work-up of the reaction media to get the product, is driving industry to consider cleaner technologies, including the use of heterogeneous catalysis.

In 1992, researcher at Mobil Corporation discovered M41S family of silicates/alumino silicate mesoporous molecular sieves with exceptionally large uniform pore structure (Kresge, C. T.; et al. *Nature* 1992, 359, 710-712, Beck, I. S.; et al. *J. Am. Chem. Soc.* 1992, 114, 10834-10843). Synthesis of mesoporous material have been reviewed by Tanav, P. T. and Pinnayaia, T. I. (*Science*, 267, 865-867). There are four general methods of preparation of mesoporous materials used in laboratory.

Syntheses of mesoporous material by neutral templating method provide a better approach but it has disadvantage of very low acidity compared with other solid acid catalysts, particularly for the reactions requiring high acidity. Therefore, any

modification which can promote the surface acidity along with their molecular sieving property of these catalysts will be highly desirable. During synthesis of these mesoporous materials, their chemical and physical properties can be modified by incorporating functionalized organic groups, either by grafting or co-condensation techniques by using functionalized substituted trialkoxy silanes during synthesis (Zhao, D.; et al. *Science,* 279, 548 (1998); Stein, A.; et al. *Adv. Materials*, 12, 1403 (2000); Van Rhijn, W. M.; et al.; *Chem. Commun.,* 317 (1995)).

Ordinary Lewis acid catalysts are water sensitive and can be hydrolyzed by water but some Lewis acids, such as group III B and rare earth triflates, are water tolerant and can be used in variety of reactions. Some of the water tolerant Lewis acid catalysts are europium triflates, hafnium triflates, lanthanum triflate and ytterbium triflate etc. These triflates are used as catalyst in several industrially important reactions.

US 6352954 discloses synthesis of Lewis acid triflate, encapsulated in the network of polymer gel. This was used in the variety of organic synthesis such as imino-aldol condensation, Mannich-type reactions, Micheal reactions and Friedel-crafts reactions.

US 6348631 discloses acylation or sulphonation of aromatics with Lewis acid, such as rare earth triflate.

US 6194580 discloses synthesis of esters by reacting a compound containing a tertiary alcohol with acylheteroaromatic ion-based compound in the presence of a lanthanide metal based catalyst.

US 5948696 discloses Aldol condensation of aldehydes in the presence of metal triflate in dichloromethane.

US 5728901 discloses a process for nitrating an aromatics with nitric acid in the presence of metal triflate.

This invention relates to the development of heterogeneous solid acid catalyst ICaT-2 (Institute of Chemical Technology, Mumbai). In which metal trifluromethane sulfonates are coordinated with hexagonal organic – inorganic functionalized mesoporous silica through a chemical bonding. The resulting catalyst has porosity, Bronsted acidic characteristic of organic – inorganic functionalized mesoporous silica, as well as Lewis acidic nature of water tolerant metal triflate.

Furfural is exclusively produced by dehydration of D-xylose. Furfural acts as a renewable feedstock for furfuryl alcohol and tetrahydrofuran, which is obtained through hydrogenation of furfural. Furfural and its derivatives are multipurpose intermediates and can replace petroleum based building blocks that are used to make resins, pharmaceuticals, and fine chemicals. Furfural also has applications in the refining of lubricating oil, removing aromatics from diesel, and as fungicide and nematocide.

US 7572925 B2 discloses process for converting fructose to 5-hydroxymethylfurfural (HMF) using biphasic reactor containing a reactive aqueous phase and an organic extracting phase. Wherein the acid catalyst is selected from the group consisting of heteropolyacids, HCl, HNO_3, H_2SO_4, H_3PO_3, oxalic acid. US 2750394; US 2917520; US 2929823; US 3118912; US 4339387; US 4740605 describe methods to produce HMF.

The catalytic activity and recyclability of the ICaT-2 catalyst was tested for the dehydration of carbohydrates to furfurals.

OBJECTIVE OF THE INVENTION

The objective of the present invention is to provide a heterogeneous solid acid catalyst with high surface area, water tolerant, high acidity and mesoporosity.

Yet further objective of the present invention is to prepare a synergistic heterogeneous solid catalyst having benefit of Bronsted acidic characteristic of

functionalized mesoporous sieve and Lewis acidic characteristic of water tolerant rare earth metal triflates.

One another objective of the present invention is to anchor the metal trifluromethane sulfonate on the hexagonal organic-inorganic functionalized mesoporous silicon material via organic linkage.

One more objective of present invention is based on covalent attachment of rare earth metal trifluromethane sulfonates on the hexagonal organic-inorganic functionalized mesoporous silica support. This attachments is based on adsorption, ion-exchange or tethering of the catalyst.

One more objective of the present invention is to design catalyst composition which can be easily separable and reusable.

One more objective of present invention is to prepare hexagonal organic- inorganic functionalized mesoporous silica material which has been subjected to the surface functionality.

Yet another objective of present invention is to make covalent bond between the water tolerance Lewis acid and hexagonal organic- inorganic functionalized mesoporous silica material.

Thus one of the aspects of the present invention is to provide water tolerance Lewis and Brownsted acidic mesoporous synergistic solid catalyst comprising hexagonal organic- inorganic functionalized mesoporous silica having metal triflate selected from the group consisting of lanthanides, actinides, and IIIB group metals and/or mixture thereof. The rare earth metal is selected from the group comprising La, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and/or mixture thereof.

Yet another aspect of the present invention is to prepare heterogeneous catalyst having a surface area in the range of 200 m^2/g to 850 m^2/g, pore volume in the rage of 0.1 ml/g to 0.5 ml/g and pore diameter in the range of 20 to 50 Å and XRD peak at 2 theta angle being 0-50.

According to the process of the present invention, a water tolerant mesoporous solid catalyst "ICaT-2" (Institute of Chemical Technology, Mumbai) is prepared by in situ anchoring of metal trifluromethane sulfonate in the hexagonal organic- inorganic functionalized mesoporous sieves.

One more objective of the present invention is to check catalytic activity of ICaT-2 catalyst for dehydration of pentose and hexose sugars.

SUMMARY OF INVENTION

According to the aspect of the present invention, a process for preparation of a heterogeneous solid catalyst possessing high surface area, water tolerant, acidity and mesoporosity is disclosed here.

The present invention is directed to provide a synergistic heterogeneous solid catalyst having combination of Bronsted acidic characteristics of functionalized organic-inorganic hexagonal mesoporous sieves and Lewis acidic characteristics of water tolerant rare earth metal triflates.

Further aspect of the invention is to anchor the rare earth metal trifluromethane sulfonate on the functionalized organic- inorganic hexagonal mesoporous silicon material via organic linkages.

The aspect of the present invention is to provide water tolerant Lewis and Brownsted acidic mesoporous, synergistic solid catalyst comprising of functionalized organic-inorganic hexagonal mesoporous silica having metal trifluromethane sulfonate selected from the group consisting of Lanthenides, Actinides, and IIIB group

elements and/or mixture thereof. The catalytically active rare earth metal is selected from the group comprising La, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and/or mixture thereof.

The present invention is based on the development of water tolerant heterogeneous catalysts (ICaT-2) having a surface area in the range of 200 m^2/g to 850 m^2/g, pore volume in the rage of 0.1 ml/g to 0.5 ml/g and pore diameter in the range of 20 to 50 Å.

BRIEF DISCRIPTION OF DRAWINGS

Drawing 1: N_2-Adsorption/desorption analysis

Drawing 2: Scanning Electron Microscope images of one of catalyst at various magnifications

Drawing 3: Temperature programmed desorption (TPD) data for NH_3 desorption of the catalyst

Drawing 4: XRD image the catalyst

Drawing 5: Thermogravimetric data the catalyst

Drawing 6: Elemental analysis by energy dispersive X-ray spectroscopy (EDX) of the catalyst

STATEMENT OF INVENTION

The present invention is related to catalyst composition (ICaT-2) comprising of rare earth metal in the form of trifluromethane sulfonate anchored with hexagonal organic -inorganic functionalized mesoporous silica having surface area in the range of 200-850 m^2/g; pore volume in the range of 0.1-0.5 ml/g and pore diameter in the range 20-50 Å.

Process for production of catalyst composition comprising the following steps of:

a) The hexagonal organic – inorganic functionalized mesoporous silica is prepared by reacting silicate precursor with organofunctionalised silica in the presence of C_8-C_{14} template.

b) Organic template removed through solvent extraction by using ethyl alcohol and/or by calcination.

c) Ion exchange of rare earth metal with the hexagonal organic – inorganic functionalized mesoporous silica.

d) Conversion of rare earth metal to their corresponding trifluromethane sulphonate salt by reacting with trifluromethane sulfonic acid.

In the catalyst composition, the rare earth metal is selected from the group comprising La, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and/or mixture thereof.

The catalyst shows excellent activity for production of biomass based chemicals and the catalyst is used in an amount of 0.1 to 15 % wt/wt of the reaction mixture. The catalyst is easily separable and has excellent reusability.

DETAILED DESCRIPTION OF THE INVENTION

In accordance with the principle of the present invention, a heterogeneous solid catalyst (ICaT-2) having high surface area, water tolerance, acidity and mesoporosity is prepared.

According to the process in the present invention, heterogeneous solid acid catalyst comprises of silicon metal as basic backbone having hexagonal mesoporosity.

Aforesaid catalyst of the present invention has organic linkage to the silicon backbone.

According to the process of then present invention, the functionality is incorporated either by co-condensation or post grafting techniques by using thio-containing silane.

The mesoporous molecular sieves of the present invention are prepared from alkoxide of silica with primary amine as a templating agent, the said primary amine having carbon atoms from 8 to 14.

One of the embodiments of the present invention is that the heterogeneous catalyst comprises of rare earth trifluromethane sulfonate anchored on organic-inorganic hexagonal mesoporous silica as a support.

The metal incorporated in the catalyst is metal ions, selected from rare earth group consisting of La, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb in the form of chloride or nitrate.

One of the embodiments of the present invention is the pore functionalized with sulfonic acid group, as ion-exchanger with the said rare earth chlorides or nitrates, to form corresponding rare earth incorporated sulfonic acid functionalised organic-inorganic hexagonal mesoporous silica.

The said rare earth incorporated sulfonic acid functionalized organic- inorganic hexagonal silica is treated with trifluromethane sulfonic acid to form corresponding rare earth metal trifluromethane sulfonate anchored on organic- inorganic hexagonal mesoporous silica.

The said ICaT-2 catalyst composition has the specific surface area in the range 200 m^2/g to 850 m^2/g.

The said ICaT-2 catalyst composition has pore diameter in the range of 20-50 Å.

The present invention discloses post grafting and co-condensation methods to prepare hexagonal organic-inorganic functionalized mesoporous silica with sulfonic acid pore fictionalization.

In one of the embodiment of the process of invention the active support is prepared by post grafting method. A primary amine is dissolved in aqueous alcohol. Tetraethyl orthosilicate is added under vigorous stirring. The precipitate is separated and template is removed by calcinations to form the hexagonal mesoporous silica. It was

further grafted with sulfonic group into active hexagonal organic- inorganic functionalized mesoporous silica as support.

In one of the embodiment of the process of invention, active hexagonal organic-inorganic functionalized mesoporous silica is prepared by co-condansation method. A primary amine is dissolved aqueous alcohol. Tetraethyl orthosilicate and thio silane is added under vigorous stirring. The reaction mixture is allowed for aging 5 to 30 h at temperature range 50 to 100 ^{0}C. The template was removed to get the active hexagonal organic- inorganic functionalized mesoporous silica support.

Lewis acidity was incorporated to the active hexagonal organic- inorganic functionalized mesoporous silica support by metal triflates on it. It leads to the ICaT-2 catalyst.

One of the embodiments of the present invention is this ICaT-2 catalyst is characterized by several analytical techniques such as thermogravimetric analysis, NH_3-temperature programmed desorption (NH_3-TPD), BET-surface area and pore volume measurements, elemental analysis by energy dispersive X-ray spectroscopy, surface morphology by scanning electron microscope (SEM), X-ray diffraction analysis (XRD).

Themogavimetric analysis (TGA) and Differential thermal analysis (DTA) discloses the thermal stability of the ICaT-2 catalyst (Drawing 5). The said catalyst has the thermal stability in the range of 300 ^{0}C to 400 ^{0}C more preferably around 350 ^{0}C.

The NH_3-TPD profile suggests that ICaT-2 possesses a large number of acid sites with medium acid strength (Drawing 3). Drawing 3 represents the NH_3-TPD profile of fresh and used ICaT-2 after reaction. The used catalyst also possesses the same acid strength after reaction. ICaT-2 catalyst has good reusability and water tolerance.

One more embodiments of the present invention are that the ICaT-2 catalyst, which comprise of rare earth metal ion in the range of 0.1 to 20 mass percentage to the total mass percent of the catalyst.

Another aspect of the present invention is to prepare the aforesaid ICaT-2 ecofriendly heterogeneous catalyst having a surface area in the range of 200m^2/g to 850 m^2/g, pore volume in the rage of 0.1 ml/g to 0.5 ml/g, a pore diameter in the range of 20 to 50 Å and XRD peak at 2 theta angle being 0 to 50.

Surface area and pore volume analysis of the hexagonal organic-inorganic functionalized mesoporous silica, ICaT-2 and used ICaT-2 are carried out by ASAP 2010. It was found that surface area of ICaT-2 was less than hexagonal organic-inorganic functionalized mesoporous, this is because the immobilization of metal trifluromethanesulfonate on it (Table A-C). The fresh and used ICaT-2 catalyst gave almost same surface area and pore diameter; hence the catalyst has good reusability

Table A

Nitrogen adsorption data: (1) Surface area

Catalyst	Sigle point surface area m^2/g	Langmuir surface area m^2/g	BET surface area m^2 /g
Hexagonal organic-inorganic Functionalized mesoporous silica	516.51	792.94	521.43
ICaT-2	423.12	628.93	421.18
Used ICaT-2	427.78	653.13	425.64

ASAP 2010 V 3.00, Analysis Adsorptive: N_2, Analysis Bath: 77.30 K, Low pressure Dose: 5 cm^2/g STP, Equilibrium Interval : 20 secs., Sample weight: 0.2 g.

Table B

(2) Pore volume

Catalyst	Single point total pore cm^3/g	BJH adsorption cumulative pore volume cm^3/g	BJH desorption cumulative surface area cm^3/g
Hexagonal organic-inorganic Functionalized mesoporous silica	0.283	0.106	0.819
ICaT-2	0.230	0.060	0.051
Used ICaT-2	0.233	0.067	0.063

ASAP 2010 V 3.00, Analysis Adsorptive: N_2, Analysis Bath: 77.30 K, Low pressure Dose: 5 cm^2/g STP, Equilibrium Interval : 20 secs., Sample weight: 0.2 g.

Table C

(3) Pore Diameter (A)

catalyst	Average pore diameter (4V/A by Langmuir) Å	BJH adsorption average pore diameter (4V/A) Å	BJH desorption average pore diameter (4V/A) Å
Hexagonal organic-inorganic Functionalized mesoporous silica	21.71	26.57	28.91
ICaT-2	21.88	31.41	34.44
Used ICaT-2	21.68	30.33	30.18

ASAP 2010 V 3.00, Analysis Adsorptive: N_2, Analysis Bath: 77.30 K, Low pressure Dose: 5 cm^2/g STP, Equilibrium Interval : 20 secs., Sample weight: 0.2 g.

To summaries the catalyst ICaT-2 characteristics show,

Surface area	200-850 m^2/g
Pore volume	0.1-0.50 cm^3/g
pore diameter	20-50 Å
XRD (2 theta)	0-50

One more embodiment of the present invention involves checking the catalytic activity ICaT-2 catalyst in the field of biomass based chemicals.

One more embodiment of the present invention is to check the catalyst activity of ICaT-2 by dehydration of xylose to furfural. In this process, furfural is manufactured using ICaT-2 to gives excellent conversion of xylose with high efficiency and selectivity. The ICaT-2 catalyst is easily separable, regenerable and reusable.

One of the embodiment of the present invention is the catalyst activity of the ICaT-2 is done for dehydration of fructose to 5-hydroxymethylfurfural (HMF). HMF is the key chemical for the biomass based chemicals and has very huge industrial potential. The excellent conversion of fructose was observed to get excellent yield of HMF.

One of the embodiment of the present invention that the ICaT-2 has excellent catalytic activity and only 0.1 to 15 % catalyst required to get furfural and 5-hydroxymethylfurfural in excellent yield.

One of the embodiment of the present invention is the said ICaT-2 catalyst is separable by filtration and regenerated by washing with organic solvent and further used for the next reaction, without any considerable loss in catalytic activity.

Therefore, the foregoing examples are considered as illustrative in terms of principles of the invention.

EXAMPLE 1: Synthesis of ICaT-2 catalyst by post grafting method

The hexagonal organic-inorganic mesoporous silicate was prepared by dissolving 10 g Dodecyl amine in 43 g of ethanol. 60 g of tetraethyl orthosilicte was added under vigorous stirring to it. The reaction mixture was aged for 5 h at 30 ^{0}C. White coloured precipitate was dried. The template was removed either by calcining the resulting material at 250 ^{0}C in air or by washing the material twice in 150 ml ethanol.

5.0 g of dried material was reacted with 3-mercaptopropyl trimethoxy silane in 50 ml toluene for 3 h. It was treated with lanthanum chloride (400 mg) for 2 h. The slurry was filtered and treated with trifluromethanesulfonic acid at 30 ^{0}C for 2 h. The slurry was filtered and washed with water and dried under vacuum to get the active ICaT-2 catalyst.

EXAMPLE 2: Synthesis of ICaT-2 catalyst by co-precipitation method

ICaT-2 is prepared by a co-condensation sol-gel route. Dodecyl amine was dissolved in ethanol. Mixture of tetraethyl orthosilicate and 3-(mercaptopropyl)trimethoxysilane were added to the above solution. It is treated with lanthanum chloride (400 mg) for 2 h. The slurry was filtered and treated with trifluromethanesulfonic acid at 30 ^{0}C for 2 h. The slurry was filtered and washed with water and dried under vacuum to get the active ICaT-2 catalyst.

EXAMPLE 3-6: The ICaT-2 reusability study for fructose dehydration

The reusability of the catalyst is tested by conducting four run (Table E). After the reaction, catalyst is filtered and refluxed in 50 cm^3 of methanol for 30 min, to remove any adsorbed material from the catalyst surface and pores, and then dried at 120 ^{0}C for 2 h. reaction was performed by reacting 0.025 mol of fructose, 0.01 g/cc of used catalyst, 100 ml water as solvent and reaction was conducted at 180 ^{0}C for 30 min.

Table E			
Example	Catalyst	% Conversion of fructose	% Yield of HMF
3	Fresh	96	89
4	1st Reuse	96	88
5	2nd Reuse	94	89
6	3rd Resue	95	88

EXAMPLE 7-10: Reusability study of ICaT-2 for xylose dehydration

The reusability of the catalyst was tested by conducting four run (Table G). After the reaction catalyst was filter and refluxed in 50 cm^3 of methanol for 30 min, to remove any adsorbed material from the catalyst surface and pores, and then dried at 120 ^0C for 2 h. reaction was performed by reacting 0.025 mol of xylose, 0.01 g/cc of used catalyst, 100 ml water as solvent and reaction was performed at 180 ^0C for 2 h. The ICaT-2 catalyst has excellent reusability and can make the process efficient.

Table G			
Example	Catalyst	% Conversion of xylose	% Yield of furfural
7	Fresh	92	67
8	1st Reuse	90	66
9	2nd Reuse	87	64
10	3rd Reuse	86	65

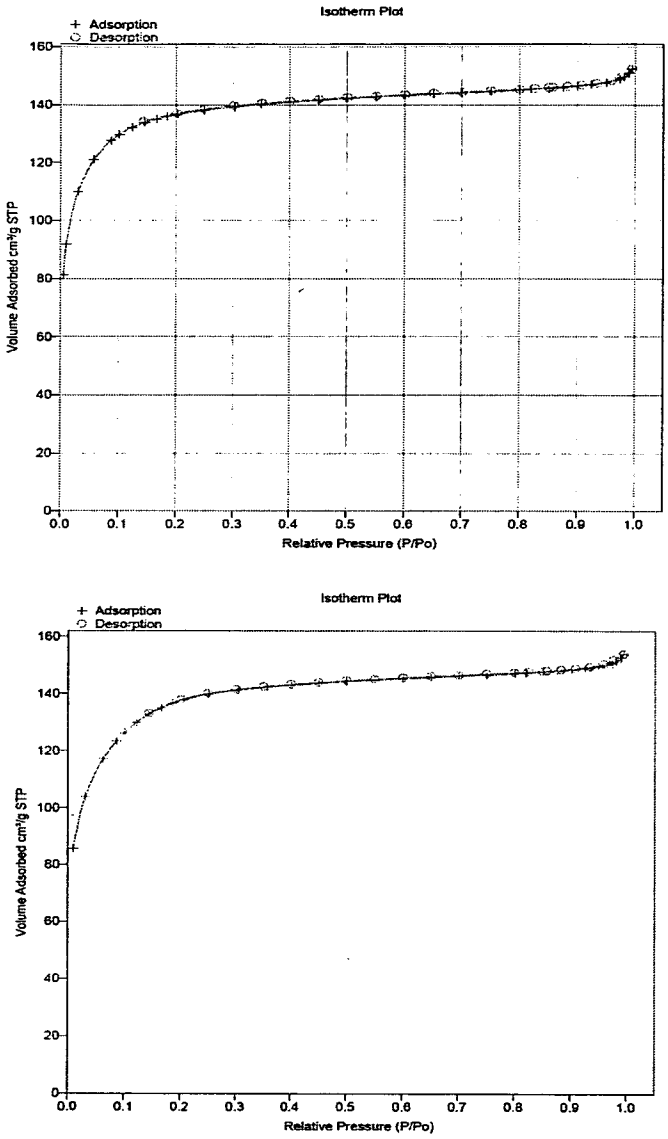
CLAIMS

We Claim:

1. A catalyst composition (ICaT-2) comprising of rare earth metal in the form of trifluromethane sulfonate anchored with hexagonal organic – inorganic functionalized mesoporous silica having surface area in the range of 250-850 m^2/g; pore volume in the range of 0.1-0.5 ml/g and pore diameter in the range 20-50 Å.
2. Process for production of catalyst composition described in claim 1 comprising the following steps of:
    a) The hexagonal organic – inorganic functionalized mesoporous silica is prepared by reacting silicate precursor with organofunctionalised silica in the presence of C_8-C_{14} template.
    b) Organic template removed through solvent extraction by using ethyl alcohol and/or by calcination.
    c) Ion exchange of rare earth metal with the hexagonal organic – inorganic functionalized mesoporous silica.
    d) Conversion of rare earth metal to their corresponding trifluromethane sulphonate salt by reacting with trifluromethane sulfonic acid.
3. The catalyst composition according to claim 1, wherein hexagonal organic – inorganic functionalized mesoporous silica is prepared from tetraethyl orthosilicate with primary amine as a templating agent, the said primary amine having carbon atoms from 8 to 14.
4. The catalyst composition according to claim 1, wherein hexagonal organic – inorganic functionalized mesoporous silica acts as support metal.
5. The catalyst composition according to claims 1, wherein functionalized silica is linked by linking molecule containing a mercapto functional group.
6. The catalyst according to claims 1, silicate precursor with organofunctionalised silica is in the stoichiometric proportions.

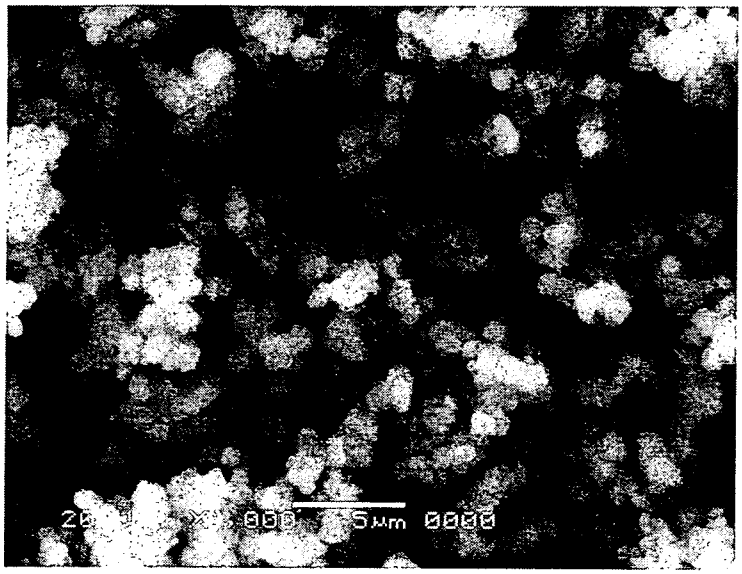
7. The catalyst composition according to claim 1, wherein the rare earth metal is selected from the group comprising of La, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb and/or mixture thereof.
8. The catalyst according to claims 1 to 7, wherein the catalyst shows excellent activity for production of biomass based chemicals.
9. The catalyst according to claims 1 to 7 and 8, wherein the catalyst is used in an amount of 0.1 to 15 % wt/wt of the reaction mixture.
10. The catalyst according to claims 1 to 7 and 9, wherein the catalyst is separated from reaction mass by filtration and reused in subsequent reaction without loss in the catalyst activity.

DRAWING



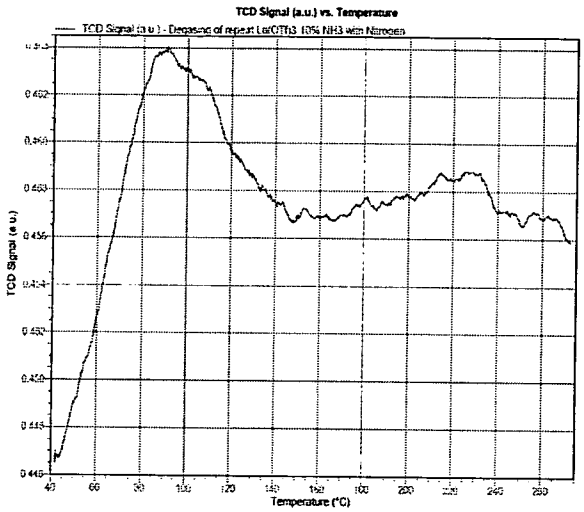
N_2-Adsorption/desorption of ICaT-2 catalyst

DRAWING

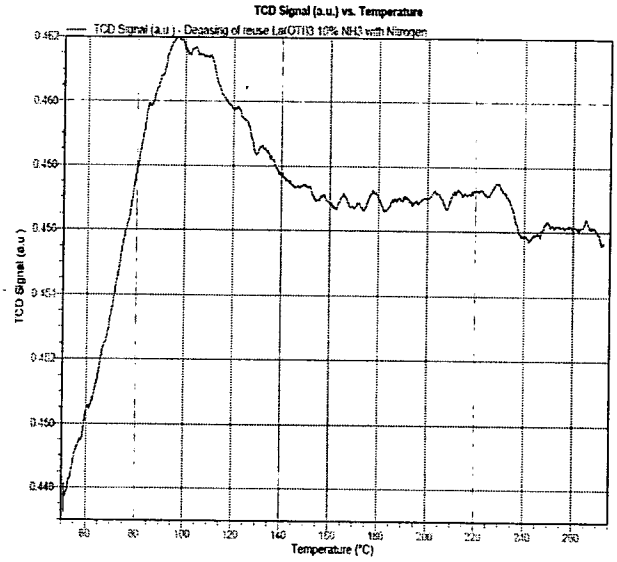


Scanning Electron Microscope images of ICaT-2 catalyst

DRAWING

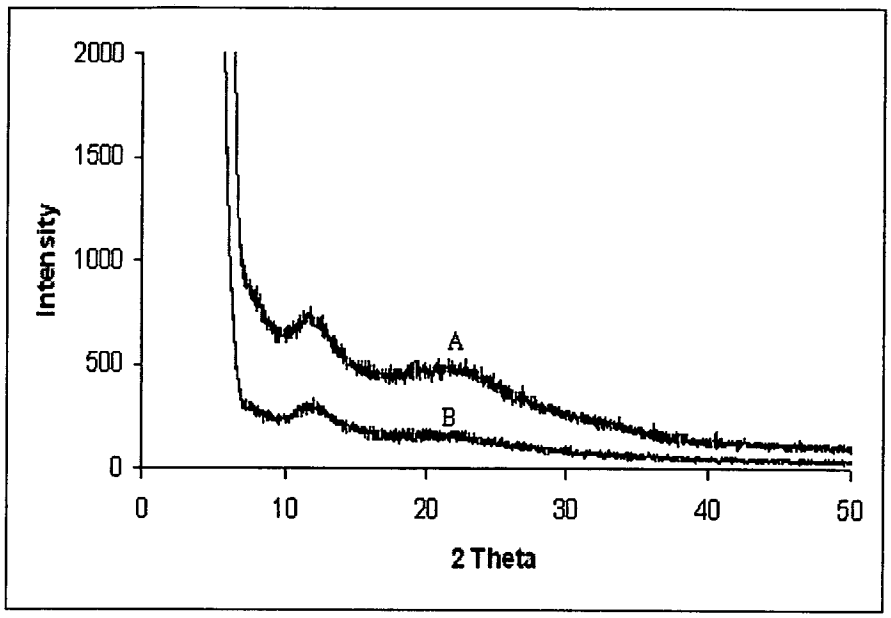


NH_3-TPD profile of ICaT-2 catalyst



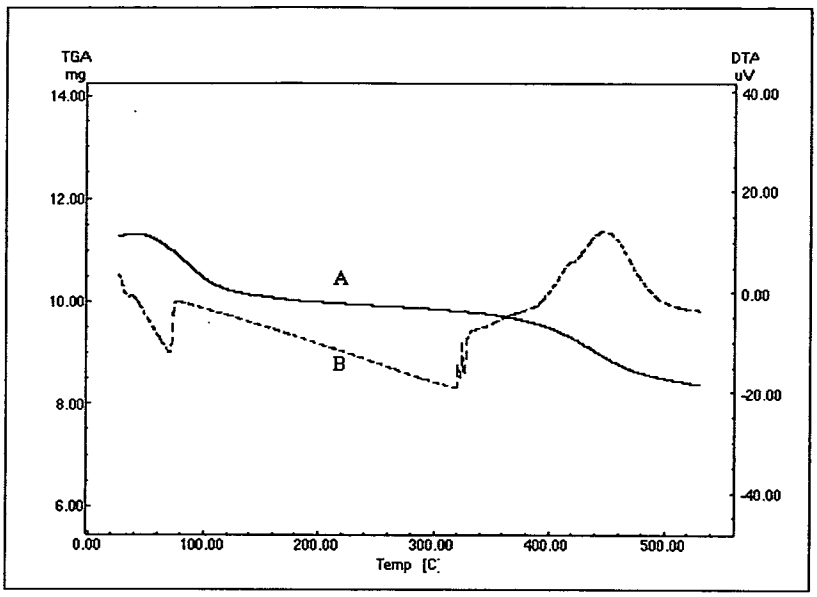
NH_3-TPD profile of used ICaT-2 catalyst

DRAWING



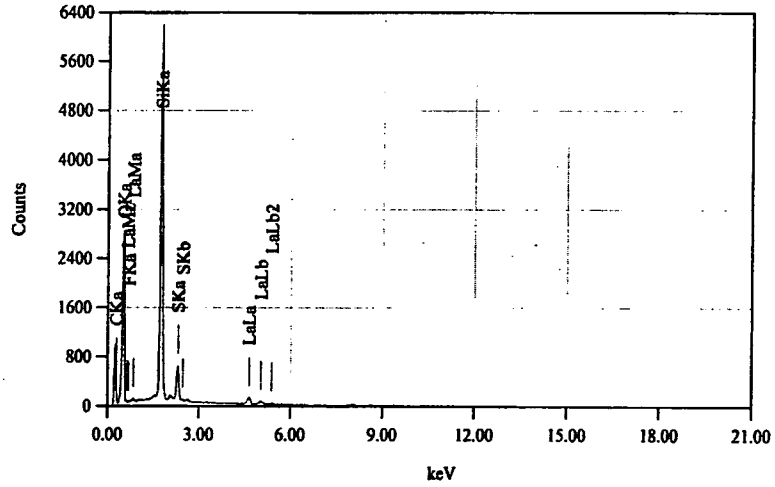
XRD image the ICaT-2 catalyst

DRAWING

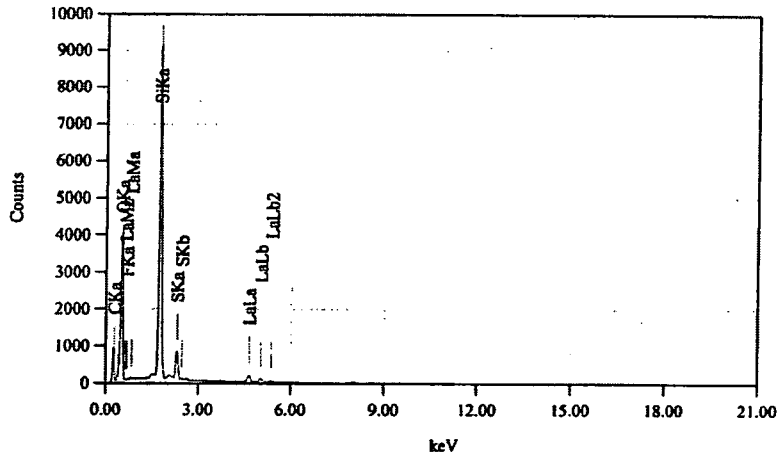


Thermo gravimetric data of ICaT-2 catalyst

DRAWING



EDX Spectra of Fresh ICaT-2 catalyst



EDX spectra of Used ICaT-2 catalyst