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Synthesis and characterization of Cr-containing silica gel and Ti-containing silica gel

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Silica gel has a lot of advantages in the actual production of life. Here, the main purpose is to use the abundant silicon hydroxyl silicone surface can be modification, surface chemical bonding and operation are understood and applied by experiment. Chromium-containing mesoporous silica gel and Titanium-containing silica gel have been synthesized at ambient temperature using sol-gel method adding different amount of Cr and Ti. In this paper, a lot of measurement used to measure various properties of the chromium silicon gel, such as measurement of Raman spectra, measurement of diffuse reflectance UV-visible spectroscopy, measurement of infrared ray absorption, measurement of surface area, etc. With these measurement, we can come to a conclusion, with the synthesis of silica gel containing chromium in this method, Cr(III) will be excluded and Cr(VI) was left, and the synthesis of silica gel containing titanium, it was possible to prepare materials without forming extra TiO₂. At the same time, other impurities were cleaned in the washing and burning. Also, the product has high activity for the selective oxidation of styrene and large surface area and pore volume. This experiment method compared with other method, the advantage is simple, the synthesis time is shorter, low cost, good repeatability and can be smoothly in the ordinary environment. It is suitable for industrial production.

Key words: Silica gel, Sodium silicate, Chromium nitrate, Titanium sulfate, Framework.

Introduction

Silica gel as a highly active absorbent material, has an important role in this field of research. Because it has no fixed formation, and the material is vary hard, chains or networks of polymer particles, the molecular formula is $mSi_2O_3 \cdot nH_2O$, is a kind of hydrophilic polar adsorbent. Porous silica-based materials containing different transition elements could be used as catalyst or support of bulky molecules because of their tunable nanoscale pore opening and exceptionally high internal surface area.

Microporous and mesoporous materials have attracted widespread attention. The framework of zeolite and mesoporous molecular sieves have unique properties for catalytic reactions, and the use of the surface modification of silica gel, used in the adsorbent to remove heavy metals and harmful substances. Silica gel can act as efficient catalysts for chemical reactions, in some situations, it can take place within the internal cavities.

Silica gel as the carrier of metallocene catalyst, have big pore volume, and the characteristic of surface modification, is a very common solid adsorbent and carrier. But there is a problem, most of silica gel is amorphous porous materials, and they to melt and collapse under the high temperature. So we need to use a certain technology to improve the size of the surface area, realize surface functionalization.

Chromium is a very important catalytic component for a lot of chemical reactions, as chromium compounds have exact nature in selective oxidation. Chromium containing molecular sieve is certainly a very interesting and promising field of research. But the synthesis process of these mesoporous molecular sieves is relatively difficult and complex.

A variety of Ti-containing microporous materials such as TS-1, TS-2, and Ti-, and mesoporous materials such as Ti-MCM-41, Ti-HMS, and Ti-MCM-48, have been developed these years. Academic and industrial have great interest in Ti-containing silica gel in which Si⁴⁺ is isomorphously substituted by Ti⁴⁺ ions in the silica gels framework. Incorporation of Ti⁴⁺ ions in mesoporous materials enlarged the possibilities of solid-catalyzed oxidation reactions in the liquid phase.

In recent years, only few reports are available on the incorporation of chromium ions in silica gel, but the research about Ti-containing silica gel have great concerned and application. On the base of recent work about Ti-containing silica gel and further validation the research results, we use simple method to synthesize Cr-containing and Ti-containing silica gel and analyze the result.

In present work, we describe a novel method to

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synthesize a low surface area as well as high Ti-containing and Cr-containing silica gel with mesostructural units at ambient temperature by a sol-gel procedure. An advantage of these samples was that they could be prepared at ambient temperature, without the help of an autoclave. In this research, we characterize the physicochemical properties of different metallocene silica gel with many methods like Raman spectra, FTIR spectra, UV-vis and Nitrogen adsorption spectra.

Experiment Section

Materials

Experiments applied to the reagent are: in the process of industrial sodium silicate (lanzhou branch of China petroleum and chemical corporation), PVA (Polyvinyl Alcohol), chromium nitrate and titanium sulfate (analytical grade, China pharmaceutical group chemical reagent company), the above materials were greater than 99% purity. Hydrogen peroxide, hydrogen peroxide, sulfuric acid and other chemicals and solution are the analysis of pure (Tianjin chemical reagent co., LTD.). The continued use distilled water in the whole experiment.

Preparation of silica gel by using sol-gel method

Using sol-gel method to prepare silica gel, its basic principle is, at the start of the experiment, add inorganic salt. Through hydrolysis, the sol was formation. Then the gel formed gradually in the process of aging and reaction. Finally, drying the gel and calcination to remove the unnecessary organic compounds, thus inorganic powder samples can be obtained.

Preparation of Cr-containing silica gel

Chromium-containing silica gel has been synthesized by a simple sol-gel method with cationic surfactant cetyltrimethylammonium bromide as the structure directing agent, chromium nitrate nonahydrate was used as the chromium source and sodium silicate as silica source.

Solution A was prepared by mixing 25 mL sodium silicate solution and 5 mL distilled water into a stainless beaker at ambient temperature and blend fully with a mechanical stirrer. The case no Polyvinyl alcohol, was added to the synthesis mixture.

A second solution (B) contained Polyvinyl alcohol dissolved in distilled water as the templating surfactant. The third solution contained chromium nitrate nonahydrate and distilled water. Sulfuric acid (0.6 mol/L) was used to maintain the pH of the medium.

First, poured solution B into a 250 mL beaker and stirred for 15 min. Second, blended solution B fully with a certain amount of solution A in a transparent beaker and placed in a constant temperature water bath£"ran the blender and adjusted medium-speed, stirred 10 min to make the temperature consistent. Third, added sulfuric acid dripping to the mixture under vigorous stirring which could keep the pH from 8.0 to 10.0, then the gel was formed gradually. Fourth, chromium sources were added into the new forming gel with dropwise addition, stirred at ambient temperature for about 30 min. Reactions proceeded at room temperature and kept the mixture on pH $8.0 \sim 10.0$ with stirring tenderly. And the aging of the gel was performed by using an auto controlling reactor under the conditions of pH $8.0 \sim 10.0$ and a process calefactive from 40 °C to 70 °C in 5 h. Homogeneous gel was further aged in turn for 1 h at 40 °C, 2 h at 50 °C, 1 h at 60 °C, 1 h at 70 °C. After above steps, washed the products with distilled water till the pH of the washing solution was to 7.0, filtered the resulting product and washed $3 \sim 4$ times to neutral. Then, dried the solid products in an oxygen atmosphere for1 h at 50 °C, 1 h at 80 °C, 1 h at 100 °C, and 1 h at 120 °C. Finally, the products were calcined with muffle stove at 650 °C for 6 h. The green as-synthesized samples turned yellow after calcinations, this process also made the surfactant removed from the as-synthesized porous chromium-silica gel.

Preparation of Ti-containing silica gel

Titanium-containing mesoporous silica gel has been synthesized by a simple sol-gel method with cetyltrimethylammonium bromide as the structure directing agent, Titanium sulfate was used as the titanium source and sodium silicate as silica source. Compared with preparation of Cr-containing silica gel, we need to add hydrogen peroxide in this experiment.

Solution A was prepared by mixing 25 mL sodium silicate solution and 5 mL distilled water into a stainless beaker at ambient temperature and blend fully with a mechanical stirrer. The case no Polyvinyl alcohol, was added to the synthesis mixture.

A second solution (B) contained Polyvinyl alcohol dissolved in distilled water as the templating surfactant. The third solution contained titanium sulfate and hydrogen peroxide, hydrogen peroxide is used to dissolve titanium sulfate. Sulfuric acid (0.6 mol/L) was used to maintain the pH of the medium.

First, poured solution B into a 250 mL beaker and stirred for 15 min. Second, blended solution B fully with a certain amount of solution A in a transparent beaker and placed in a constant temperature water bath£"ran the blender and adjusted medium-speed£"stirred 10 min to make the temperature consistent. Third, added sulfuric acid dripping to the mixture under vigorous stirring which could keep the pH from 8.0 to 10.0, then the gel was formed gradually. Fourth, titanium sources were added into the new forming gel with drop-wise addition, stirred at ambient temperature for about 30 min. Reactions proceeded at room temperature and kept the mixture on pH $8.0 \sim 10.0$ with stirring tenderly. And the aging of the gel was performed by using an auto controlling reactor under the conditions of pH $8.0 \sim 10.0$

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Characterization of silica gel

Measurement of Raman spectra

Raman spectra of the Cr-containing silica gel and Ticontaining silica gel were obtained using continuous wave laser lines at 325 nm as the excitation sources for 60 s. The wavelength is $100 \sim 1300 \text{ cm}^{-1}$. In this work, we use Horiba Jobin Yvon LABRAM-HR800.

Measurement of Diffuse reflectance UV-visible spectroscopy

Diffuse reflectance UV-vis spectroscopy is a very sensitive probe for the presence of Cr(III) and Cr(VI) respective in the as-synthesized and calcined, dehydrated Cr-containing silica gel. Diffuse reflectance UV-vis spectroscopy is very sensitive for the presence of extra framework Ti in zeolites. The measure range is $220 \sim 700$ nm and the apparatus model is ZASCO UV-570.

Measurement of infrared ray absorption

The Fourier transform infrared spectroscopy (FTIR) of the Cr-containing silica gel and Ti-containing silica gel was recorded by using a Nicolet NEXUS 670 FTIR spectrophotometer, and the sample and KBr were pressed to form a tablet.

Measurement of surface area

The average pore size of the Ti-containing silica gel and Cr-containing silica gel synthesized following the above procedures was determined by the Barrett-Joyner-Halenda(BJH) method, While the specific surface area was mensurated by the Brunauer-Emmet-Teller (BET) technique.

Result and Discussion

Synthesis of Ti-containing silicate gel and Crcontaining silicate gel formation

Experiments using sodium silicate is a kind of soluble alkali metal silicate, is through the alkali metal oxide and silicon dioxide reaction synthesis of each other. In this experiment, sodium silicate is formed by the combination of Na_2O and SiO_2 .

To form a silica gel, mainly have three steps. First, the inorganic monomer of SiO_2 is polymerized and

smaller granule formed. Then the small granule gradually grew up, the aggregation between particles is complex to form mutual cross-linked reticular structure.

Sodium silicate solution contains Na^+ and SiO_3^{2-} , after joining the process of sulfuric acid, the monomer chemical polymerization, the main reaction of forming silica gel are as follows:

$$\begin{aligned} \mathrm{SiO_3^{2-}} + 6\mathrm{HCl} &\rightarrow \mathrm{[SiCl_6]^{2-}} + 3\mathrm{H_2O} \\ \mathrm{H_2[SiCl_6]} + 4\mathrm{H_2O} &\rightarrow \mathrm{H_4SiO_4} + 6\mathrm{HCl} \end{aligned}$$

The formation of silica gel is the condensation crosslinking of H_4SiO_4 , after that SiO_2 tetrahedron polymer is formed.

$$\begin{array}{ccc} OH & OH & OH & OH \\ HO-Si-OH + HO-Si-OH \longrightarrow HO-Si-O-Si-OH \\ OH & OH & OH & OH \end{array}$$

In the process of experiment, pay attention to acid amount of added value. If the acid quantity is too little to join, the gel can not fully formed, only partly crosslinking is loose, and unable to achieve ideal effect, if the amount of acid to join too much, easy to agglomerate, hinder the chromium, the titanium and the synthesis of silica gel and chromium ions, the titanium ions into the tetrahedral structure of the gel. PH plays a very important position in the midst of silica polymerization, according to previous research shows that when add acid sodium silicate solution, when the pH value to 10.7, begin to form gel, in order to maintain the stability of gel, the experiment to control the pH of the solution on 9-10. Join the non-ionic surfactant PVA, pay attention to add speed, pay attention to the strong mechanical stirring, otherwise when not at the reaction would be easy to selfassembly, forming polymer, affected the progress of the experiment and data analysis.

Chromium silicone synthetic process, gel appears green, after UV spectral identification, synthesis of chromium silica gel inside the silica structure, form atoms dispersed trivalent chromium ions Cr(III), and only exists in the price of trivalent Cr atoms. But after calcination, trivalent Cr(III) was oxided to hexavalent Cr(VI). The valence changes rendered for the green gel powder, yellow powder.

The complexing mechanism of Titanium and hydrogen peroxide:

$$\begin{array}{ccc} & Framework \ Ti \\ H_2O & \uparrow & H_2O_2 \\ Ti(OC_4H_9) \rightarrow & Ti(OH)_4 & \longleftrightarrow & [TiO_2(OH) \\ \downarrow & & (H_2O_2)_2]OH \\ TiO_2 \end{array}$$

Titanium into the silicon frame:



The hydrolysis of tetrabutyl titanate speed is greater than the hydrolysis rate of sodium silicate, an overdose of Ti(OH₄) can form TiO₂ with a further condensation. If add hydrogen peroxide as a complexing agent, the overdose of Ti(OH₄) will complexing with H₂O₂, left small amount of Ti(OH₄) combinate with silica-oxide bond. With less and less Ti(OH₄), more Ti(OH₄) will be produced through the complexing mechanism, so that the hydrolysis of tetrabutyl titanate speed will be same as the hydrolysis rate of sodium silicate.

Characterization by Raman spectra, and UV-vis spectra, FT-IR spectra, Nitrogen adsorption

Raman spectra of the Cr-containing and Ticontaining silica gel

This experiment is to use 325 nm laser as excitation light source, by stimulating Cr-containing and Ticontaining silica gel, make the charge transition between Cr-O and Ti-O.

Raman spectra of the Cr-containing silica gel

Fig. 1 shows the Raman spectra of the sample of the calcined, dehydrated Cr-containing silica gel. Despite the sample powder and spectrum measurement is in a normal environment in the air, but since water is considered little into the sample, and diffusion rate is low, further vacuum in processing or absolute dry processing is relatively complex, so in that environment measurement for absolute cases have no effect on



Fig. 1. Raman spectra of calcined, dehydrated Cr-containing silica gel (using continuous wave laser lines at 325 nm as the excitation sources for 60 s).



Fig. 2. Raman spectra of as-synthesized Cr-containing silica gel (using continuous wave laser lines at 325 nm as the excitation sources for 60 s).



Fig. 3. Raman spectra of as-synthesized Ti-containing silica gel (using continuous wave laser lines at 325 nm as the excitation sources for 60 s).

chromium containing silicon gel, and can be used to directly measure without the need for further processing.

Cr-containing silica gel gives a band at 390 cm^{-1} , 899 cm^{-1} and 933 cm^{-1} . The bands at 390 cm^{-1} and 899 cm^{-1} are respectively assigned to the bending and symmetric stretching vibrations of the framework Cr-O-Si species and the band at 933 cm^{-1} is attributed to the asymmetric stretching vibration of the Cr-O-Si.

Fig. 2 shows the raman spectra of not calcined assynthesized Cr-containing silica gel. The Raman band near 536 cm⁻¹ in as-synthesized Cr-containing silica gel is characteristic of a $Cr_2O_3/Cr(III)$, but the band do not appear in calcined, dehydrated Cr-containing silica gel.

Raman spectra of the Ti-containing silica gel

Fig. 3 shows the Raman spectra of Ti-containing silica gel. TI-containing silica gel gives a band at 365 cm^{-1} , 510 cm^{-1} and 640 cm^{-1} . The bands at 365 cm^{-1} , 510 cm^{-1} and 640 cm^{-1} are complicated because the Ti-Si mixed oxide because amorphous silica also possesses Raman



Fig. 4. UV-vis spectra of as-synthesized Cr-containing silica gel (the measurement range is 220-700 nm).



Fig. 5. UV-vis spectra of calcined, dehydrated Cr-containing silica gel (the measurement range is 220-700 nm).

features at 410,487,607,802 cm⁻¹ and the peak band at 150 cm^{-1} is attributed to the sensitive indicator of the presence of anatase. The band at 150 cm^{-1} indicates that most of Ti species have come into the silica gel framework. The bands at 490 and 530 cm⁻¹ are respectively assigned to the bending and symmetric stretching vibrations of the framework Ti-O-Si. The band at 1000 cm^{-1} is most enhanced in the UV resonance Raman spectrum because this vibration mode is more sensitive the charge-transfer transition of Ti-O-Si.

UV-vis spectra of the Cr-containing and Ticontaining silica gel

UV-vis spectra of Cr-containing silicate gel

Fig. 4 and Fig. 5 shows not containing chromium silicon gel and calcined dehydration chromium silica gel of diffuse reflectance UV-vis spectra. The wavelength is 950 nm.

From Fig. 3 we can see, not calcining, dehydration new chromium silica gel. At 438 nm and 608 nm has



Fig. 6. UV-vis spectra of calcined, dehydrated Ti-containing silica gel (the measurement range is 220-700 nm).

obvious absorption peak. It is formed because the two bands are assigned to isolated framework Cr(III) in octahedral coordination, d-d transitions and described as ${}^{4}A_{2g} \rightarrow 4T_{2g}$ and ${}^{4}A_{2g} \rightarrow {}^{4}T_{1g}$ transition. The band at 438 nm in fig. 3 is attribute to a spin forbidden ${}^{4}A_{2g} \rightarrow {}^{2}T_{2g}$ transition of six-coordinate Cr(III) when they are under the tetragonal distortion.

In the UV-vis spectra of calcined, dehydrated Crcontaining silica gel in Fig.5, two intense bands have be obviously seen. The bands around 268 nm and 372 nm indicates that $O \rightarrow Cr(VI)$ charge transfers of a chromate species and also absence of the band around 440 nm which is the characteristic of Cr(VI) polychromate. If in the chromium silicon gel, Cr(VI) contains many kinds of tetrahedral coordination oxide compound, typical Cr₂O₃ and Cr(III) absorption band will appear in the UV-vis spectra. Since there is no observed, contain chromium silicon gel, so after calcination, only a single chromium species that Cr(VI) has left.

The above results show that only a single type of chromium ions Cr(VI), CrO_4^{2-} ion form exists in calcination Cr-containing silica gel, it also conforms to the Raman results.

UV-vis spectra of Ti-containing silicate gel

Mesoporous Ti-containing silica gel UV-vis spectra as shown in Fig. 6, the samples show weak, with center has strong absorption peak at 210 nm, the wavelength should be isolated skeleton titanium tetrahedral coordination mesh structure. Absorption peak at 260 nm may correspond to aggregation state of titanium dioxide nanometer domain. Peak at 330 nm suggests that synthetic silica gel containing titanium did not form a large number of titanium pigment, and the results show that using inorganic titanium sources in the synthesis of silica gel, most of Ti atoms occupy a site-isolate position in the framework of silica gel.



Fig. 7. FTIR spectra of the pure silica gel (recorded by using a Nicolet NEXUS 670 FTIR spectrophotometer).



Fig. 8. FTIR spectra of the Cr-containing silica gel (recorded by using a Nicolet NEXUS 670 FTIR spectrophotometer).

FTIR of the Cr-containing silica gel and Ti-containing silica gel

FTIR of Cr-containing silica gel

Fig. 7 show FTIR spectra of pure silica gel, and Fig. 8 show FTIR spectra of calcined, dehydrated Cr-containing silica gel.

In Fig. 7, we can see the FTIR absorption peaks at 1081 cm^{-1} and 467 cm^{-1} . The two bands are attributed to asymmetric and symmetric stretching vibrations as well bending vibration of framework Si-O-Si bonds of silica gel. And in Fig. 8, the calcined, dehydrated Cr-containing silica gel sample, all these absorption peaks shift to high wave-numbers. The two bands above were shifted to 1101 cm^{-1} and 480 cm^{-1} . This shift of the peaks from lower wave-number to higher wave-number is attributed to the Cr incorporating into the framework of silica tetrahedral.

Compare the absorption phenomenon Fig. 7 and Fig. 8 at 905 cm⁻¹, the optical density of the band is absent in calcined Cr-containing silica gel, which is assigned to a Cr = O or Cr-O vibration from a Cr(VI) species in a dehydrated sample. The band at 960 cm⁻¹ is because



Fig. 9. FTIR spectra of the Ti-containing silica gel (recorded by using a Nicolet NEXUS 670 FTIR spectrophotometer).



Fig. 10. Nitrogen adsorption and desorption isotherms of Crcontaining silica gel.

of a lattice defect because of the presence of chromium ions or other framework ions in a CrS-1 framework.

From Fig. 7 to Fig. 8, a band at 963 cm^{-1} shifted to 931 cm^{-1} . This is suggest in recent works that a Si-O vibration in a Si-OH group, and in this case, we can suggest that this shift is because a replacement of an OH group by O-Cr(VI). And we can came into a conclusion, Cr(VI) atom successfully in the framework of silica gel.

FTIR of Ti-containing silica gel

From Fig. 7 to Fig. 9, a band at 963 cm⁻¹ shifted to 943.6 cm⁻¹. This is suggested in recent works that a Si-O vibration in a Si-OH group, and in this case, we can suggest that this shift is because a replacement of an OH group by O-Ti. And we can come into a conclusion, titanium atom successfully in the framework of silica gel. But there is a problem that the band at 943.6 cm⁻¹ is very weak, that may because this particular sample not have so many titanium atoms in it, but recent works have already proofed the authenticity of this suggestion.

 Table 1. The physicochemical properties of the as-synthesized

 Cr-containing silica gel.

Sample number	Bulk density (g/cm ³)	Specific surface area(m ² /g)	Pore volume (cm ³ /g)	Aperture (nm)
CSG-01	0.387	12.241	0.021	3.390
CSG-02	0.387	5.882	0.013	3.436
CSG-03	0.388	8.930	0.015	3.083
CSG-04	0.388	5.957	0.009	3.382

Nitrogen adsorption and desorption isotherms of the Cr-containing silica gel and Ti-containing silica gel

Nitrogen adsorption and desorption isotherms of the Cr-containing silica gel

From Fig. 10, we can find the Langmuri isotherms is observed, this is characteristic of nitrogen adsorption and desorption isotherms for porous material.

From the figure, because of the N_2 at pore face, the N_2 capillary coacervation in pore will make the quantity of nitrogen adsorption and desorption change sharply, when P/PO lower than 0.2, the nitrogen adsorption and desorption will increase with the increase of P/PO, and this is the reason which will make isotherms have obvious tantivy between $0.3 \sim 0.45$. And then the long adsorption and desorption indicate the saturation of nitrogen adsorption in capillary.

All of the samples reaction temperature is between $30{\sim}45$ °C, pH is between $8 \sim 10$ and the wt% of Cr is $0.2{\sim}1.0\%$.

From Table 1, it can be concluded that in this group of samples, most of the surface area and pore volume are small, only sample 1 have a large surface area and pore volume. The low pore volume is due to the replacement of an OH group by O-Cr(VI) in the pore of silica gel, since a OH group is smaller than O-Cr(VI), the pore volume become smaller. Beside this reason, if there is some surfactant remain when calcined the sample, or during the experiment, the surfactant is not enough, they all make pore volume of the sample small, and the surface area is small at the same time.

Computational formula of specific surface area:

$S_g = V_m N\sigma/22400W$

In this computational formula, V_m is the value of saturation adsorption in sample monolayer surface, N is Avogadro constant number, σ is the cross-sectional area of a single nitrogen molecule, W is the weight of sample.

Nitrogen adsorption and desorption isotherms of the Ti-containing silica gel

The data and information in Fig. 11 and Table 2 came from an experiment our group did it before. From the discussion above and Fig. 11, we can came into a conclusion that when P/PO lower than 0.2, the nitrogen



Fig. 11. Nitrogen adsorption and desorption isotherms and BJH of Ti-containing silica gel.

 Table 2. The physicochemical properties of the as-synthesized Ti-containing silica gel,

Sample number	Bulk density (g/cm ³)	Specific surface area (m ² /g)	Pore volume (cm ³ /g)	Aperture (nm)
TSG-1	0.379	300.965	1.638	19.585

adsorption and desorption will increase with the increase of P/PO is reasonable, because of the N₂ at pore face, the N₂ capillary coacervation in pore will make the quantity of nitrogen adsorption and desorption change sharply, and which will make isotherms have obvious tantivy between $0.3 \sim 0.45$. And then the long adsorption and desorption indicate the saturation of nitrogen adsorption in capillary.

From Table 2, it can be concluded that the specific surface area and pore volume of as-synthesized Ticontaining silica gel is obviously better than properties of Cr-containing silica gel. Ti-containing silica gel has a large specific surface area and pore volume, so it can make properties, such as the catalytic properties of silica gel better than Cr-containing silica gel.

Conclusions

Silica gel has a widely used in research and petrochemical industry applications. Analyses this kind of silica gel catalyst, can offer an more efficient specific surface area for reactions, make proper pore volumes, and constitute the active center to facilitate reactions, etc. In this study, Cr-containing silica gel framework and Ti-containing silica gel framework are proposed, analyzed and compared. The Cr-containing and Ti-containing silica gel charactered by the Raman spectra, FTIR spectra and UV-vis spectra, and then analyses the properties of these atoms containing silica gel.

For Cr-containing silica gel, Raman, FTIR and UV-

vis spectra show that Cr(VI) monochromate exists in calcined Cr-containing silica gel, and UV-vis spectra show silica gel porous structure and contain atomically dispersed Cr(III). The nitrogen adsorption shows that Cr-containing silica gel has large surface and welldefined pore sizes. Raman, FTIR and UV-vis spectra analyses Ti-containing silica gel can be concluded that most of Ti species enter the silica gel framework, and nitrogen adsorption shows that Ti-containing silica gels have larger surface areas and pore-size. From the results of the study above, we can draw the following conclusion. This synthesis method performed in a short period of time at ambient temperature and pressure, is a valid alternative to obtain Cr-containing silica gel materials. The properties of Cr-containing are well and valuable. But the Ti-containing silica gel have larger specific surface area and pore volume than Cr-containing silica gel, Ti-containing silica gel is more efficient and have better properties and more valuable in application as polyolefin catalyst carriers. Further research work about hetero atom silica gel is progressing in our group. The synthesis regime of the hetero atom silica gel catalytic materials will be lead to a new path and importance for foundational and practical research.

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