Evolution of textural characteristics of surfactant-mediated mesoporous zirconia aerogel powders prepared via ambient pressure drying route

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Abstract

The experimental results in this work represent the preparation of ambient pressure dried zirconia aerogels using surfactant and their textural characteristics studies. Zirconia aerogel powders were prepared by sol–gel process employing Brij-76 as a surfactant. The influence of varying concentrations of Brij-76 (0.01–0.1 M) on the textural characteristics of zirconia powders was studied. During synthesis, the molar ratio of Zirconium *n*-propoxide: *n*-Propanol: Acetic acid: H₂O was kept fix to 1:16.8:2.7:8.7. The structural and morphological observations of these powders were performed through X-ray diffraction analysis and field emission scanning electron microscopy (FESEM). The surface area and average pore diameter of the synthesized powders were estimated using N₂ adsorption–desorption analysis. Zirconia powders obtained using Brij-76 (0.01 M) without any heat treatment exhibited the amorphous nature with a well-ordered mesoporous morphology, specific surface area, and average pore of nearly 177 m²/g and 60 Å, respectively.

Keywords Brij-76 · Sol–gel preparation · Ambient pressure drying · Mesoporous · Zirconia powders · Surface area

Introduction

“Aerogels, the lightest and transparent nanostructured materials, are obtained from a gel by the replacement of liquid present in the pores of gel with air”. They are extremely low dense materials with a wide variety of outstanding properties such as low thermal conductivity, high porosity and large surface area [1–3]. Exhibiting such exceptional properties, aerogels are applicable in various fields such as thermal insulation, catalysis and sensors, etc. [4, 5]. The preparation of aerogels can be performed through supercritical drying (drying above critical temperature (T_c) and

pressure (P_c) of liquid present in the pores of gel) or ambient pressure drying (drying at ambient pressure and temperature > 150 °C) or freeze drying (drying of frozen gel in vacuum) methods. Recent developments have shown great potential of the ambient pressure drying as an alternative and simple method for the synthesis of aerogels replacing the expensive and energy intensive methods of supercritical and freeze drying. Following this, till date the synthesis and characterizations of zirconia (ZrO₂) aerogels via ambient pressure drying was reported by few research groups [6–8]. These reports manifested the studies on the influence of propylene oxide as complexing agent, hydrolysis water, and heat treatment on the pore size and surface area of zirconia aerogels. It is well known that the pore sizes in the materials (bulk or thin film) can be controlled in the mesopore range (2–50 nm) by addition of surfactant (organic additive) which acts as structure directing agents [9, 10]. Because of the small pore sizes, the mesoporous materials have found extensive applications in the field of designing catalyst support, templates, adsorbents and solid oxide fuel cells [11–15]. To obtain mesoporous structures, the surfactants like Brij-76, Brij-58 and Pluronic F127 are being used for the network-formation. The surfactant mediated synthesis of mesoporous materials can be considered as the electrostatic

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interactions between the ions of metal species and the head groups of charged surfactant as well as the hydrogen bonding interactions between the neutral primary amine micelles, and neutral inorganic species [16, 17]. The work on the synthesis of titania- and zirconia-based mesoporous materials has been reported by elsewhere [18, 19]. Much attention is focussed on the ZrO_2 -based catalyst since it has acidic and basic character as well as oxidizing and reducing chemical properties. Moreover, zirconia aerogels exhibit good textural characteristics which help to enhance the catalytic activity, thermal stability, and resistance to poisoning [20]. There are several reports on the catalytic applications of zirconia aerogels related to the modification of their surfaces by metals (copper, iron) or ions (sulfate, phosphate, tungstate) and to the use of surfactant like cetyl trimethyl ammonium bromide (CTAB), octyl trimethyl ammonium bromide (OTAB), etc. The catalytic performances of zirconia xerogels and aerogels were studied in the dehydration of methylpentan-2-ol reaction and $\text{CO}=\text{CO}_2$ hydrogenation by few research groups [21, 22]. Kalies et al. presented the catalytic activity of Pt doped zirconia aerogel for hydrogenation of methoxy moieties [23]. Suh et al. reported the catalysis of surfactant (CTAB and OTAB) mediated zirconia in the skeletal isomerization of 1-butene [24]. Further, the catalytic properties of sulfated zirconia aerogels and xerogels at various calcination temperatures were studied by Ghorbel et al. [25]. The sulfated zirconia aerogels doped with cerium were found to have higher acidity leading to their good activity in *n*-hexane isomerization reaction in the wide temperature range [26]. Interestingly, the iron-doped zirconia solid solution aerogels exhibited an excellent catalytic performance for the solvent-free aerobic oxidation of *n*-hexadecane which can be recycled five times without significant loss of activity [27]. Moreover, mesoporous sulfated zirconia aerogels doped with chromium showed the improvement of *n*-hexane isomerization reaction by burning-off coke [28].

Similar to mesoporous zirconia, the pore sizes in zirconia aerogels may also be tuned in mesopore range using surfactant during the sol–gel process and drying at ambient pressure. The as synthesized zirconia aerogels can be applied as a catalyst in replacement to supercritically dried zirconia aerogels. Therefore, it is challenging to synthesize high surface area mesoporous zirconia aerogels via ambient pressure drying. Till date, to the best of our knowledge there are rare reports on the synthesis of ambient pressure dried zirconia aerogels employing surfactant. Therefore, in the present work Brij-76 was used as a surfactant for the synthesis of zirconia aerogels. Brij-76 is also known as stearyl ether with the chemical formula $\text{C}_{18}\text{H}_{37}(\text{OCH}_2\text{CH}_2)_{10}\text{OH}$. Brij-76 is a non-ionic surfactant consisting of hydrophobic tail (long alcohol chain) and hydrophilic head (polyoxyethylene) [29]. So, in the present report, zirconia aerogels were synthesized using Brij-76 as a surfactant and the influence

of varying concentration of Brij-76 (0.01–0.1 M) on their textural characteristics has been studied.

Experimental

For the synthesis of zirconia aerogel powders, zirconium *n*-propoxide ($\text{Zr}(\text{OC}_3\text{H}_7)_4$, 70% in *n*-PrOH, Sigma-Aldrich), *n*-propanol (*n*-PrOH, SD Fine), acetic acid glacial ($\text{CH}_3\text{COOH}/\text{HOAc}$, SD Fine) and Brij-76 ($\text{C}_{58}\text{H}_{118}\text{O}_{21}$, Aldrich, MW 711) were used as precursor, solvent, catalyst/complexing agent and surfactant, respectively. Hexane was used as the exchanging and silylating solvent with hexamethyldisilazane (HMDZ) as a silylating agent. D. I. water was used for hydrolysis and condensation purpose. The flowchart for the synthesis of zirconia aerogel powders using Brij-76 is depicted in Fig. 1. During the synthesis, 0.5 M Zr^{4+} in *n*-PrOH was used [20] along with the Zr^{4+} :Acetic acid (glacial) molar ratio of 1:2.5 [30]. For the given concentration of Zr^{4+} , D.I. H_2O was added till the gel formed. Therefore,

Solution 1 : Propanol + Zirconium propoxide + Acetic acid (glacial) + D.I. H_2O

Solution 2 : Propanol + Brij-76

Fig. 1 Flowchart for the synthesis of zirconia aerogel powders

the final molar ratio of Zirconium *n*-propoxide: *n*-Propanol: Acetic acid: H₂O was kept constant at 1:16.8:2.5:8.7 and the concentration of Brij-76 was varied from 0.01 to 0.1 M with the increment of 0.05 M between each step. To prepare zirconia alcogels, two different solutions were formed and named as solution 1 and solution 2. Solution 1 consisted of the mixture of *n*-propanol, zirconium precursor, acetic acid and D.I. H₂O while solution 2 was made up of *n*-propanol and Brij-76. Both the solutions were stirred for 1 h and mixed which again stirred for 15 min to give alcogels. This alcogel was kept at 50 °C in PID controlled oven to form alcogels. The sol–gel reactions taking place during alcogel formation are given below (reactions 1–5).

These alcogels were subsequently subjected to aging (at 50 °C for 24 h), solvent exchange with hexane (at 50 °C for 24 h), silylation using mixture of 5% HMDZ in hexane, again washing with hexane (at 50 °C for 24 h) and finally drying at 50 °C (for 1 h) and 150 °C (for 2 h) to obtain zirconia aerogels. The obtained zirconia aerogels are in powder form. A set of six zirconia samples was prepared with and without Brij-76 and named as listed below in Table 1. Out of six, four samples were heat treated at 500 °C for 1 h with the rate of 10 °C/min in air atmosphere.

To know the amorphous or crystalline nature of zirconia aerogels, powder X-ray diffraction (XRD, RIGAKU Ultima IV diffractometer, Japan) was carried out using Cu

K α ($\lambda = 1.541 \text{ \AA}$) radiation operated at 40 kV and 30 mA. The morphological study was performed using field emission scanning electron microscopy (FESEM, JEOL JSM-600F, Japan). The surface area and average pore diameter were obtained from the *N*₂ adsorption–desorption analysis recorded at 77 K using a TriStar 3000 V6.05 A, USA. The chemical bonds present in the zirconia samples were analyzed by Fourier transform infra red spectroscopy (FTIR, Thermo Nexus 670).

Results and discussion

The amorphous or crystalline nature of zirconia aerogel powders was confirmed from the X-ray diffractograms as shown in Fig. 2. As seen from Fig. 2a it is observed that sample ZA2 manifested two broad peaks indicating the nanocrystalline amorphous nature of zirconia aerogels which is due to drying at 150 °C only. While sample ZA4 exhibited the tetragonal crystalline nature (Fig. 2b) because of the heat treatment at 500 °C as reported elsewhere [8]. This diffractogram matches with the JCPDS data card number PDF#791,767 with a sharper and stronger peak of (101) indicating phase orientation along $\langle 101 \rangle$. Figure 3 illustrates the FESEM images of the synthesized zirconia samples ZA1–ZA6. From the FESEM micrographs, it is clear that zirconia particles are loosely connected with each other randomly to form a sponge-like shape in case of sample ZA1 (Fig. 3a) while an ordered porous morphology is observed for sample ZA2 due to the presence of Brij-76 (0.01 M) (Fig. 3b). On the other hand for the samples ZA3–ZA6, a dense morphology is observed (Figs. 3c–f). The reason behind this is, the samples ZA1 and ZA2 were dried only at 150 °C and other samples ZA3–ZA6 were heat treated at 500 °C which caused the aggregation of particles. The influence of Brij-76 on the morphology of aerogel (samples ZA1 and ZA2) can be well understood from the pictorial representation as drawn in Fig. 4. In case of zirconia aerogel w/o Brij-76 (sample ZA1), the particles of zirconia in the sol condensate to form porous gel network which after drying form the aerogels with non-uniform larger pores. While in case of zirconia aerogels with Brij-76 (0.01 M) (sample ZA2), the zirconia particles condensate to form gel with uniform pores resulting in aerogels with ordered small pores on drying.

The influence of employing Brij-76 on the surface area and pore size distribution of zirconia samples was studied through *N*₂ adsorption–desorption analysis. Figure 5a, b exhibits the hysteresis isotherms of the zirconia samples ZA1–ZA3 and ZA4–ZA6, respectively. As shown in Fig. 5, all the zirconia samples ZA1–ZA6 exhibited type IV isotherm indicating the capillary condensation taking place in

Table 1 Notation for as synthesized zirconia aerogel powders

Sample id	Zirconia aerogels
ZA1	w/o Brij-76 and w/o heat treatment
ZA2	With Brij-76 (0.01 M) and w/o heat treatment
ZA3	w/o Brij-76 heat treated at 500 °C
ZA4	With Brij-76 (0.01 M) heat treated at 500 °C
ZA5	With Brij-76 (0.05 M) heat treated at 500 °C
ZA6	With Brij-76 (0.1 M) heat treated at 500 °C

Fig. 2 X-ray diffractograms of the zirconia samples **a** ZA2, **b** ZA4

Fig. 3 FESEM images of zirconia samples **a** ZA1, **b** ZA2, **c** ZA3, **d** ZA4, **e** ZA5, **f** ZA6

Fig. 4 Pictorial representation for the formation of zirconia samples ZA1 (w/o Brij-76) and ZA2 (with Brij-76)

the mesopores [31]. The isotherms of samples ZA1 (Fig. 5a) and ZA5 (Fig. 5b) manifested the hysteresis loop of type H_1 indicating the cylindrical pore geometry and facile pore connectivity. And the samples ZA2 and ZA3 (Fig. 5a), ZA4 and ZA6 (Fig. 5b) exhibited hysteresis loop of type H_4 and H_2 related to narrow slit-like pores and pores with narrow mouths, respectively. The pore size distribution of all the zirconia samples is shown in Fig. 6. All of the samples exhibited a broad, asymmetrical pore size distribution in the mesopore range as observed from Fig. 6. In case of sample ZA2, a sharp intense peak is observed at nearly 40 Å along with a broad peak at around 180 Å (Fig. 6a). But in case of

the sample ZA4 (Fig. 6b) most of the volume is occupied by the mesopores as confirmed from the sharp intense peak centred around 40 Å. The BET surface area, pore volume and average pore diameter of aerogel powders obtained from N_2 adsorption–desorption analysis are listed in Table 2. The sample ZA1 has higher surface area of $\sim 204 \text{ m}^2/\text{g}$ and pore volume of 0.37 cc/g compared to the sample ZA2 possessing surface area of around $177 \text{ m}^2/\text{g}$ and pore volume of nearly 0.27 cc/g. The reason behind this may be the occupancy of Brij-76 in the voids of zirconia aerogels [32]. The high surface area and pore volume of the samples ZA1 and ZA2 is due to well-ordered mesostructure, which can be confirmed

Fig. 5 N_2 adsorption–desorption isotherms of the zirconia samples

Fig. 6 Pore size distribution of the zirconia samples

Table 2 BET surface area, pore volume, and average pore diameter of the zirconia samples

Sample id	BET surface area (m ² /g)	Pore volume (cc/g)	Average pore diameter (Å)
ZA1	203.58	0.37	71.71
ZA2	177.87	0.27	60.30
ZA3	27.43	0.04	46.46
ZA4	45.23	0.06	49.60
ZA5	39.74	0.15	147.73
ZA6	33.54	0.08	94.15

by their FESEM images (Fig. 3a, b). In case of the sample ZA3 the surface area suddenly reduced to 27 m²/g because of the heat treatment at 500 °C leading to the collapse of gel network structure. For the samples ZA4 and ZA6 the surface area decreased from 45 to 33 m²/g which may be due to an increase in the concentration of Brij-76 reduces the effective headgroup area and the repulsive forces between hydrophilic chains necessary for the gel network-formation [10]. The gel formation using surfactant can be considered as the electrostatic interactions between the ions of metal species and the head groups of charged surfactant [17]. The high-average pore diameter of 147 Å of the sample ZA5 is in good agreement with the hysteresis loop of type H₁ and increased pore volume [33].

FTIR spectra of the zirconia samples ZA2 and ZA4 are shown in Fig. 7. As seen in Fig. 7, a broad band is observed at around 3300 cm⁻¹ which corresponds to the stretching vibration of the O–H bond (due to physically adsorbed water). The bands at around 2970, 1340–1420 [34] and 845 cm⁻¹ are due to C–H (stretching, bending vibrations) and Si–C bonds, respectively [7]. The bands in between 1000 and 1200 cm⁻¹ correspond to the stretching vibration of C–O bond which may be of Brij-76 [34]. The presence of the absorption bands at around 1260, 950 and 650–750 cm⁻¹ are assigned to the Zr–OPr, Zr–O–Si and Zr–O bonds,

Fig. 7 FTIR spectra of the zirconia samples **a** ZA2, **b** ZA4

respectively [8]. The C–H and Si–C bonds band intensity decreased with increase in drying temperature from 150 to 500 °C indicating the decomposition of methyl groups. Further, there is a decrement in C–O bond band intensity at 1024 cm⁻¹ which may be due to the evaporation of Brij-76.

Amongst all the samples heat treated at 500 °C, sample ZA4 has comparatively high surface area of 45 m²/g and small pores of around 50 Å, with collapse network structure as observed from FESEM image (Fig. 3d). But ZA2 have high surface area of 177 m²/g and average pore diameter of 60 Å with highly ordered particles arrangement. These mesoporous zirconia aerogel powders can be used for catalysis purpose. Thus, the preparation of mesoporous zirconia aerogel powders employing surfactant and the studies on the influence of drying temperature and rate on their textural and mechanical properties will open new challenges in near future.

Conclusions

Brij-76 mediated zirconia aerogel powders were successfully synthesized via ambient pressure drying method. The zirconia aerogel powders synthesized using Brij-76 (0.01 M) without any heat treatment exhibited the amorphous nature as confirmed by XRD analysis. FESEM image of the synthesized aerogel powders clearly demonstrated the regular periodic arrangement of zirconia particles. Further, they possessed the pore sizes of around 60 Å and surface area nearly 177 m²/g as determined by N₂ adsorption–desorption analysis. FTIR spectra of the zirconia aerogels powder clearly showed the decrease of C–H and Si–C peak intensity with increase in the drying temperature from 150 to 500 °C. These aerogel powders can be applied for the catalysis purpose.

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