A facile rout to obtain Al2O3 nanopowder via recycling aluminum cans by sol-gel method

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Abstract
Aluminum alkoxide compound was prepared using aluminum cans and used as a precursor in the mesoporous alumina synthesis processes by sol-gel method. The aging temperature of 25, 60, 80 °C and period of 12, 24 and 48 h were investigated as the main parameters in determining the sol particle size and the bonding process during the gel process. The results showed that the aging temperature of 80 °C and duration of 24 h are the optimum conditions for the synthesis of mesoporous alumina with superior properties in terms of specific surface area and porosity. The estimation of calcination temperature showed that the minimum temperature required to reach the alumina phase is 500 °C. However, due to the necessity of the presence of crystalline phase with maximum ordering at atomic plane arrangement, 900 °C temperature and 1 h storage time were selected as the optimum calcination conditions. Scanning and transmission electron microscopy studies of the optimum sample showed that the final product consists of nanoparticles with same appearance, and the particle size distribution was in the range of 20 to 40 nm. Due to the binding of nanoparticles and the formation of larger masses, porosities in the meso-range are created in their microstructure, which introduces the end product to be used as a catalyst sub base.

1. Introduction
Al2O3 powder has wide range of applications such as electronic ceramics, high strength materials and catalysts. Among the polymorphs of transition alumina, the γ form is one of the most extensively used in industrial catalysis owing to its comparatively large surface area, unique surface characteristics, and exceptional structural stability. Conventional γ-alumina formed through the thermal dehydration of a crystalline aluminum oxyhydroxide (boehmite) at a temperature above 450 °C. Recently many researchers are showing interest on the preparation and application of nano-sized alumina considering their diverse properties [1–5]. The property of such alumina particles are depends on particle size, morphology, surface and phase homogeneity and these properties can be controlled by selecting a proper synthetic route. γ-alumina with high surface properties such as high surface area and mesoporous properties is commonly used as a high temperature catalyst or catalytic support and as a membrane [6].

Mesoporous alumina (MA) has been synthesized through several routes including: the sol-gel method [7, 8], nonionic templating [9], a reverse cation-anion double hydrolysis method (CADH) [10], and evaporation-induced self assembly (EISA) with block copolymers [11].

Recently, some approaches have been reported on the use of aluminum cans for alumina synthesis [3, 12–14]. Recycling this type of aluminum scrap can save energy and other raw materials [3, 14]. Chemical synthesis and sol-gel are the most suitable routes for the production of ceramic particles or nanoparticles. Chemical synthesis is a very simple, safe and fast manufacturing process having the most significant benefits, i.e. time and energy saving. This fast and straightforward process can be used to synthesize high purity and homogeneous crystalline oxide ceramic powders, including ultrafine alumina powders in a wide range of particle size.
The sol-gel method involves the formation of a sol from organometallic precursor, whose suspended particles in sol are polymerized at low temperature, then the produced wet gel is dried and heat treated. High-purity particles with large surface area can be obtained by sintering ceramic precursors prepared by chemical synthesis and sol-gel methods [15–20]. There are only a few reports on the sol-gel and template-free methods for the preparation of γ-alumina mesostructure. The template-free approach is simple and suitable for large-scale production [21]. Among many aluminum sources for mesoporous alumina, aluminum alkoxides are expensive and aluminum isopropoxide (AlP, Al(OC₃H₇)₃) is commonly used [22, 23]. Efforts have been made to reduce the cost of producing aluminum alkoxide by using scrap aluminum cans to synthesize aluminum isopropoxide. In addition, aluminate or aluminum hydroxide is formed when scrap aluminum cans react with a hot solution of a strong base and have the potential to be used as an aluminum source for porous alumina. Chotisuwan et al [24], produced and analyzed the properties of mesoporous alumina prepared by the template-free sol-gel method using scrap aluminum cans. Two primary aluminum sources such as AlP and aluminum hydroxide solution were prepared from aluminum scrap cans and then utilized in synthesis. According to the authors, they presented their first report on the synthesis of high-surface area mesoporous alumina using scrap aluminum cans as an aluminum source. The mesoporous alumina produced as nickel oxide (II) catalyst support was used for the oxidation of the liquid phase toluene. Lopez et al [25] have reported the synthesis of α-alumina from aluminum cans. They proposed two methods for the synthesis of α-alumina, the first of which included the deposition of aluminum chloride with NaOH for the production of aluminum hydroxide. The second method involved dissolving aluminum pieces with glacial acetic acid to produce aluminum acetate. XRD analysis showed that the alumina powders were obtained by heat treatment of aluminum acetate at a relatively low temperature (1100 °C), initiating from aluminum hydroxide at 1180 °C. This method is capable of producing nanophase alumina powder, with an average crystallite size of 58 and 54 nm for alumina derived from hydroxide and aluminum acetate, respectively. Therefore, the use of these precursors simplifies the process and provides another alternative to the synthesis of crystalline alumina. Liu et al [14] investigated a method to recycle aluminum beverage cans. Herein, aluminum ethoxide is prepared through the reaction of aluminum cans with ethanol. The ethoxide hydrolysates are converted to alumina phase after 2 h at 900 °C. The average particle diameter of 0.26 mm was observed by scanning electron microscopy.

2. Experimental procedure

2.1. Synthesis of aluminum isopropoxide

The preparation step of aluminum can include the process of removing the logo and all prints on it and eventually removing the surface contaminants. Although removing the logo and color prints on the aluminum surface can be easily accomplished by using solvents such as acetone and chloroform, however, there is a polymer layer (usually polyacrylic) that enhances the surface properties and resistance to environment, and turns the removal of color printing from aluminum cans into a challenge. To solve this problem, the autoclave system should be used to remove the polymer layer on the cans surface in contact with water vapor at high pressure and finally remove the colored logo using organic solvent. The can lid is definitely made of pure aluminum and can therefore be used. Use of the can lid has the advantage of not requiring surface preparation and contaminant removal processes.

In the synthesis of mesoporous alumina by sol-gel method, as mentioned above, it is first necessary to prepare aluminum alkoxide from aluminum raw material. In fact, this part was mainly performed to reduce the supply costs of aluminum oxide and maintain the commercialization capability of mesoporous alumina.

During the sol-gel process, the utilized aluminum alkoxide is converted to gel by hydrolysis control and condensation, which depending on the size of the gel precursor particles and their binding, the microstructure containing porosities and their properties are ultimately specified. This means that if the size of the sol particles can be controlled using controlling compounds and determining their molar ratio to other precursors, the wall thickness and consequently the porosities resulting from the binding of these particles can be controlled eventually.

Aluminum cans were used to prepare aluminum alkoxide with the chemical compound Al(OC₃H₇)₃, known as AlP. Figure 1 illustrates the practical steps of AlP synthesis from aluminum cans. First, the beverage cans were placed in 5 M sodium hydroxide solution for 2 h at 60 °C to eliminate any surface contamination. Also, to remove the label on the cans, the cans were reacted with 2 M sulfuric acid solution at 50 °C for 1 h and finally washed repeatedly with distilled water.

After surface preparation of aluminum cans, the cans were cut to fewer than 3 cm. AlP was synthesized from cut aluminum pieces in a Pyrex chamber of a three-neck flask. For this purpose, 100 c.c. of isopropyl alcohol was poured into a flask with a capacity of 250 c.c. and 10 g of aluminum slices were added. After magnetic stirring of
the mixture, nitrogen gas flow passed through the flask and temperature was gradually increased to 80 °C. At this time, 2 g of mercury iodide was added to the system as a catalyst and held for 24 h to synthesize AlP.

2.2. Synthesize of aluminum hydroxide
To synthesize mesoporous alumina, aluminum hydroxide gel was first prepared according to the method proposed by Kim [26]. For this purpose, along with the synthesized AlP, 2-propanol and acetic acid were used as the solvent and controller of the hydrolysis rate. In this regard, synthesized AlP was firstly solved in 2-propanol solvent and acetic acid and water were added dropwise for the hydrolysis and condensation to take place in the system. Finally, the aluminum hydroxide formed as a gel was aged at 80 °C for 24 h. Molar ratio of Acetic acid to AlP was 0.02, water to AlP molar ratio was 4 and the molar ratio of 2-propanol solvent to AlP was considered equal to 19. Figure 2 illustrates the practical steps for the synthesis of aluminum hydroxide gels.

2.3. Synthesize of mesoporous alumina
After aging of the synthesized aluminum hydroxide gel, the solid portion was first separated by centrifugation at 6,000 rpm and then dried for 8 h under 10 mbar vacuum. The dried powder was calcined at 500 °C for 2 h in air atmosphere. The most important variable examined in this section is temperature and aging time as these two parameters affect the binding kinetics of the hydroxide components to each other and the formation of cavities.
Conditions for the production of AlO

Table 1. The codes for samples prepared at different aging conditions.

| Sample code | AT1 | AT2 | AT3 | BT1 | BT2 | BT3 | CT1 | CT2 | CT3 |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Aging temperature(°C) | 25  | 25  | 25  | 60  | 60  | 80  | 80  | 80  |     |
| Aging time (h)       | 12  | 24  | 48  | 12  | 24  | 48  | 12  | 24  | 48  |

[24]. The aging temperature was 25, 60, 80 °C at 12, 24 and 48 h, respectively. Table 1 shows the codes for samples prepared at different aging conditions.

2.4. Characterization of aluminum isopropoxide and alumina products

X-ray diffraction and phase analysis of the specimens were performed using X’pert equipment using copper lamp and x-ray of Kα = 1.54 Å. The samples were examined at a rate of 2° min⁻¹ in the diffraction angle range of 10 to 90 °. To investigate the morphology and characteristics of the synthesized nanopowders, a Philips scanning electron microscope (SEM) equipped with a tungsten electron gun was used at 25 kV. Transmission electron microscopy (TEM) was used to observe the synthesized mesoporous. In order to conduct the porosity test, nitrogen gas adsorption method was used and results were evaluated based on the relationships developed by BET and BJH models. For this purpose, at first, 0.14 g of mesoporous sample was degassed for 15 h at 120 °C in a vacuum dryer. Then, using Belsorp mini II device, gas adsorption and desorption was performed using nitrogen at 77 K and the corresponding curves were drawn (helium gas was used in the desorption stage). Fourier-Transform Infrared Spectroscopy (FTIR) (Eauniox 55 instrument by Bruker, Germany) was used to observe the functional groups applied to the mesoporous sample.

3. Results and discussion

Process temperature and gel aging time in the sol-gel process are among the most important parameters determining the final product characteristics including particle size and specific surface area. In fact, these two parameters determine the size of the primary alumina particles and how they bond to each other, which ultimately determines the surface and porosity properties. Generally, the temperature and aging time at which uniform initial particle formation with a narrow particle size distribution is provided, is considered as optimal conditions. If the aging conditions are such that the size distribution of the primary particles is wide, as the particles are bonded to each other, fine particles will move to the pores created by the bonding of the larger particles, leading to a significant reduction in the specific surface area and pore volume in the final mesoporous alumina. In this regard, analysis of specific surface area, volume and size of pores was performed on specimens prepared at different temperature and aging times (table 1) to obtain optimum process conditions based on achieving maximum specific surface area and pore volume according to the applications presented in this study. Table 2 shows the results of the porosity analysis by nitrogen gas adsorption and desorption on these samples. As can be seen in the table, the gel samples obtained at different temperatures and aging times had different specific surface areas so that in the AT1 sample prepared at 25 °C and 12 h of aging, the lowest level of specific surface area was obtained to be 285 m² g⁻¹. However, in the case of the CT2 sample, where the process temperature rose to 80 °C and the aging time was set to 24 h, the specific surface area reached the highest level among the samples equaling 520 m² g⁻¹. The results of table 2 show that the minimum process temperature and minimum aging time should be 80 °C and 24 h, respectively. Finally, considering the superiority of the CT2 sample in terms of specific surface area and pore volume, the process conditions of this sample were selected as the optimum conditions for the production of AlO(OH) gels for the synthesis of mesoporous alumina.

Determining these temperature ranges is crucial for optimal temperature adjustment to produce nanoparticles with superior properties. Figure 3 shows the differential thermal analysis curve (DTA) of the gel sample prepared at 80 °C and 24 h aging time.

As shown in figure 3, the obtained DTA curve has two endothermic peaks in the temperature range of 90 and 450 °C. The first peak, which is relatively less intense, is attributed to the elimination of excess water (free or absorbed water), and the second peak, which has a predominant intensity, is related to the endothermic reaction of AlO(OH) compound decomposition to alumina. Studies have shown that in the Boehmite phase decomposition reaction, the OH groups of the structure leave the system by receiving thermal energy, and thus the alumina phase decomposition reaction is formed according to equation (1):
and according to the DTA curve and the dry gel decomposition reaction, the above reaction occurred in the temperature range of 450 °C – 500 °C.

After determining the optimum conditions for AlO(OH) gel production by sol-gel method, the effect of calcination temperature as the key factor for achieving a mesoporous product is investigated in this section. The calcination temperature for all porous materials is an important factor in determining the final material properties, such that the temperature increases as the pores and porosities in the material are closed, and generally the increase of calcination temperature and sintering progress and grain growth in the material are in contrast with achieving a specific surface area and high porosity volume. However, in most synthesis methods for nano-materials such as sol-gel, the achieved dry material has an amorphous microstructure and it is necessary to use calcination process to achieve the proper properties of the crystalline phase. As shown by the thermal analysis results, the calcination temperature of 500 °C is the minimum temperature required for the progression of the decomposition reaction and production of the aluminum oxide phase. However, the calcination temperature should be chosen above this range because if the material is calcinated in the boundary limit for the hydroxide to oxide decomposition, it will tend to increase hydration tendency, and as a result, re-

| Sample code | Average porosity diameter (nm) | Cavity volume (Vp) (cm³/g) | Special BET Level (m²/g) |
|-------------|--------------------------------|---------------------------|-------------------------|
| AT1         | 3.87                           | 0.64                      | 285                     |
| AT2         | 4.05                           | 0.95                      | 310                     |
| AT3         | 3.9                            | 1.25                      | 320                     |
| BT1         | 3.1                            | 1.36                      | 335                     |
| BT2         | 6.4                            | 1.55                      | 405                     |
| BT3         | 6.5                            | 1.6                       | 420                     |
| CT1         | 4.5                            | 1.25                      | 380                     |
| CT2         | 8                              | 2.63                      | 520                     |
| CT3         | 7.5                            | 1.55                      | 450                     |

**Table 2.** Results of nitrogen gas absorption-desorption porosity analysis in different aging conditions.

![Figure 3. DTA curve of dried gel sample prepared at 80 °C and 24 h aging time.](image)

2AlO(OH) → Al₂O₃ + H₂O
convert to the hydroxide phase by functioning in moisture environments. In this regard, dried samples (synthesized under optimum conditions) were heat treated at temperatures of 500, 700 and 900 °C for 1 h to obtain the optimum calcination temperature based on the minimum temperature in which the sample XRD pattern consisted only by aluminum oxide phase peaks. Figure 4 shows the XRD pattern of these three samples.

As illustrated in figure 4, for the sample prepared at 500 °C, primary nuclei of mesoporous alumina are forming. By increasing the temperature to 700 °C the characteristic peaks in the XRD pattern increase, all of which belong to the Al₂O₃ phase, and the large width peaks in this pattern indicate the small size of the crystallites forming the sample particles. As the calcination temperature increases from 700 to 900 °C, more peaks appear in the XRD pattern, leading to the peak characteristics of the crystalline microstructure. Considering the Scherrer equation and determination of the peak widths and half the maximum intensity, the size of alumina phase crystals in these samples was calculated, the results of which are shown in figure 5.

As shown in figure 5, the crystallite size exceeds 1000 Å above 800 °C. At 500 °C this value is set to about 15 nm, which is close to the crystallite size of the dry powder (5 nm). According to the XRD pattern results, temperature of 700 °C and above is selected as the optimum calcination temperature for the gels obtained from the sol-gel process. However, the effect of calcination temperature on the specific surface area of the specimens should also be examined before final confirmation.

Table 3 shows the results of the porosity analysis by nitrogen gas adsorption and the determination of specific surface area based on the BJH model. It can be seen from the table results that with the heat treatment of the samples and through the calcination process, the specific surface area is significantly reduced compared to the original dry gel. In the case of samples calcinated at 500 °C, the specific surface area of the sample decreased by about 40% from the initial dry state, reaching 305 m² g⁻¹. However, in the case of samples calcinated at temperatures of 700 °C and above, this drop in the specific surface area number is very significant, so that in the case of samples calcinated at 700 °C the specific surface area of the specimen is reduced about 72% relative to the initial sample. This specific surface area drop was 77% and 83%, for the samples calcinated at two temperatures of 900 and 1000 °C, respectively.

It should be noted that although the specific surface area drop in the calcinated sample at 500 °C is lower than that of the dry gel sample, this temperature cannot be selected as the appropriate calcination temperature because of the inadequate conditions for complete phase transformations and crystallization of the lattice.
Therefore, the optimum calcination temperature should be selected in the temperature range of 700 to 1000 °C considering the importance of high specific surface area or high crystalline structure in each application. In general, for catalytic applications, due to the harsh working conditions, it is generally recommended to use crystalline structures with minimal structural defects. Although this will reduce the specific surface area, it will also result in a longer life span and higher resistance of the catalyst substrate. Based on the results of the above table, it seems that the temperature of 900 °C is suitable for maintaining high specific surface area and crystal structure and this temperature was selected as the optimum temperature for final product preparation.

Therefore, complementary analyzes were performed on the CT2 sample, which was calcinated at 900 °C for 1 h.

In the first step of complementary analysis on the mesoporous alumina sample (synthesized under optimum conditions) the adsorption-desorption isotherm of nitrogen gas was investigated for this sample (figure 6(a)). As can be seen in these conditions, the adsorption-desorption isotherm of the mesoporous alumina synthesized in the present study follows the type IV isotherm. In fact, the isotherms obtained do not fit the adsorption and desorption curves and create a hysteresis loop at high relative pressures. The existence of this type of hysteresis loop and type IV isotherm in this sample indicates the meso-porous nature of the synthesized mesoporous alumina. It should be noted that in this isotherm, the y-intercept is negligible, indicating the absence of micrometer-sized cavities (below 2 nm). Also in figure 6(b), the pore size distribution curve based on BJH in this analysis, shows the presence of a high intensity peak in the range of 10 nm, confirming the meso-porous nature of the sample being synthesized [24].

Scanning electron microscopy was used to investigate the morphology and bonding behavior of aluminum oxide nanoparticles in the optimum sample. Scanning electron microscopy images of the mesoporous alumina sample are shown in figures 7(a) to (d) at various magnifications. As can be seen in the figures, the sample synthesized under optimum process conditions has a high uniformity in the constituents so that the bonding of nanoparticles with a narrow particle size distribution leads to the formation of similar masses with uniform appearance. Upon closer examination, it was found that there was a uniformity in all parts of components along with no significant change in their size and morphology. This result demonstrates the establishment of uniform and appropriate conditions for the synthesis of nanoparticles at all reaction points during the sol-gel process [27–30].
Gathering of alumina nanoparticles and the formation of pores with a certain size (in the meso range) in this sample confirms achieving the present study goal and the achievement of a product that in addition to being within the desired nano range, can be used in catalytic applications. Figure 8 shows the sequence of microscopic images from low to high magnification to better understand the above explanation.

Continuing the characterization of the optimum mesoporous alumina sample, transmission electron microscopy and diffraction pattern (DP) images were implemented. Figures 9(a)–(d) shows the TEM images of this sample and the diffraction pattern, respectively.

As shown in these images, the optimum mesoporous alumina sample consists of particles in the size range of 20 to 40 nm, which the particles do not differ much in terms of appearance and size. By examining these images it is well known that there is no inhomogeneity or change in the properties of the constituent and also purity of the sample particles and it can be said that no secondary or impurity phase (as an independent phase) is observed in this sample, and the microstructure consists of alumina nanoparticles entirely.

The diffraction pattern obtained from this sample also shows the presence of loops and bright spots present on it. This characteristic in the diffraction pattern is due to the nature of the polycrystalline nanoparticles and their very small size of their crystallites. In fact, the very small size of crystallites in this sample has caused the shape of the loops to change from a continuous loop to a loop containing bright spots, which confirms the small
size of the crystallites. Figure 10 shows the EDX analysis results obtained during TEM imaging. This analysis accurately expresses the purity of the nanoparticles and reveals the presence of very minor impurities. As can be seen in this sample analysis, only two peaks of Al and O elements are present for this sample and no other.

Figure 7. Scanning electron microscopy images of the mesoporous alumina sample at various magnifications.

Figure 8. Sequence of microscopic images from low to high magnification.
Figure 9. (a)–(c) The TEM images and (d) the diffraction pattern of optimum mesoporous alumina sample.

Figure 10. EDX analysis results obtained during TEM imaging.
elements (even in very small amounts) are present. This investigation truly concludes the very high purity of alumina nanoparticles synthesized in the present study.

Another important point realized from examining TEM images at very high magnifications is the presence of specific facets in the synthesized nanoparticles (figure 9(b)). In fact, unlike the amorphous and semi-crystalline nature of the nanoparticles in which the atomic arrangement and formation of atomic planes are not complete, atomic planes have fully formed in this sample and have formed specific facets on the particle surface.

4. Conclusion

1. Aluminum alkoxide compound was successfully prepared from aluminum can lids (after initial preparation) and selected as a precursor for the process of mesoporous alumina synthesis by sol-gel method.

2. Examination of different samples showed that the aging temperature of 80 °C and 24 h time are the optimal conditions for the synthesis of mesoporous alumina with superior properties in terms of specific surface area and porosity.

3. The investigation of calcination temperature showed that the minimum temperature required to reach the alumina phase is 500 °C. However, due to the necessity of the presence of crystalline phase with maximum atomic arrangement order, temperature of 900 °C and storage time of 1 h were selected as the optimum calcination conditions.

4. The results of the adsorption–desorption analysis of nitrogen gas showed that the synthesized mesoporous alumina sample has optimum surface area of 115 m$^2$ g$^{-1}$ and porosity of 0.93 m$^3$ g$^{-1}$. The maximum specific surface area was also obtained for calcinated samples at 700 °C to be 145 m$^2$ g$^{-1}$.

5. Scanning and transmission electron microscopy studies of the optimum sample showed that the final product included similar appearance nanoparticles, and the particle size distribution was in the range of 20 to 40 nm. It was also found that the synthesized nanoparticles have very distinct crystalline facets, and as a result of the nanoparticles bonding to each other and forming larger masses, porosities were formed in their microstructure in the meso-range, which highly nominates the final product to be used as a catalyst substrate.

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