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Effect of impurities on characteristics of ZrO_2 and ZnO ceramic powders produced by spray pyrolysis

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Abstract Previously it was observed that addition of impurities to a precursor solution may alter the size and morphology of the particles produced by spray pyrolysis. To investigate this further, the spray pyrolysis technique was used to prepare zirconia (ZrO_2) and zinc oxide (ZnO) ceramic powders, with addition of slight amounts of NaCl in various concentrations. The results show an increase in the percentage of nondisrupted particles which corresponds to an increase in the weight percentage of NaCl in the precursor in ZrO_2 powder produced at 400 °C. This effect is not repeated in ZnO powder produced at 400 °C, as the addition of NaCl to the precursor results in the disruption of individual particles into much smaller particles. As far as the morphology and strength of particles are concerned, it is concluded that the addition of NaCl to the precursor solution has a beneficiary effect on the morphology of ZrO_2 particles and an adverse effect on ZnO particles, both of which are negated at a higher reactor temperature of 600 °C.

Introduction

Spray pyrolysis (SP) is a simple and single-step method to produce agglomerate-free complex micro- and nano-powders [1] and thin films [2]. The process starts with atomization of a liquid precursor into small droplets, followed by chemical decomposition of solution droplets, which work as microreactors. For powder production, solution droplets are carried to a hot-wall reactor, where they dry to form particles. In the case of thin film formation, in a process similar to spray forming of metals, solution droplets impact and deposit on a surface to form a thin film [2]. SP is capable of producing simple ceramic powders such as zirconia (ZrO_2) [3] and zinc oxide (ZnO) [4], and also complex powders such as $LiMn_2O_4$ used in lithium batteries [5] and Eu:ZnO [6] and $ZrO_2{:}Pr^{3+}$ [7] phosphors used for display applications. Some of the other methods to produce micro- and nano-ceramic particles include wet chemistry route such as the sol–gel method [8], gas-phase route such as flame spray pyrolysis [9], laser methods such as the laser–liquid–solid interaction technique [10], mechanical methods such as ball milling [11], solvothermal reaction method [12], and self-propagating high-temperature synthesis technique [13]. Poondi et al. [10] have summarized the advantages and disadvantages of various methods. Research trends of powder production via the spray route have been addressed by Eslamian and Ashgriz [14].

Among simple but strategic ceramic powders, ZrO_2 and ZnO have numerous applications. Zirconia powder is a hard white powder, used in manufacturing of pigments, refractories, artificial jewelry, fireproofing materials, fuel cells, oxygen sensors, dental filling, to name a few. Zirconia is considered as a biomaterial ceramic as well [15, 16]. Among several methods that may be employed to produce zirconia, the spray methods (conventional and flame spray pyrolysis) are advantageous, because they do not require the multiple steps, high liquid volumes, and surfactants of wet chemical processes. Spray methods, of course, have disadvantages as well; one being their low production rate.

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Typical applications of ZnO particles are in ink, rubber, oil, greases, ferrites, cosmetics, pharmaceuticals, food additives, UV coatings, lacquers, and textiles. Ultrafine ZnO particles have additional applications such as sunscreen and antibacteria in cosmetics, deodorant, antimicrobial, antiultraviolet in textile, antiaging in paint, coating, plastic and gas sensors, image track record materials, piezoelectricity materials, capacitors, etc. ZnO powder has also been investigated as a possible root canal filling material [17]. ZnO powder is mostly produced by gas-phase synthesis (e.g., oxidation and condensation of zinc vapor). Few percents are made by wet processes. ZnO used for the emerging applications mentioned above have necessitated a precise control of ZnO particle properties [9]. SP is a sophisticated method, which allows *controlled* preparation of ZnO particles [4].

Depending on the operating conditions, the final product particles of spray pyrolysis (and also spray drying) may be fully filled or hollow. Based on a model developed by Jayanthi et al. [18], hollow particles form at the onset of solute precipitation on the droplet surface if the solute concentration at the droplet center is smaller than the solution equilibrium saturation. Otherwise, particles will be fully filled if the condition of the percolation theory is satisfied as well. Although hollow and disrupted ceramic particles are desirable in some applications, such as development of high temperature insulators, in most cases fully filled particles are of great value, because of their high mechanical strength. SP is essentially a high-temperature process (requirement of thermal chemical decomposition); this high temperature results in a high solvent evaporation rate, which is detrimental to the formation of solid fully filled particles. It has been shown that if spray pyrolysis proceeds at reduced pressure, even at high process temperatures, the evaporation rate is lower than that of the atmospheric pressure and the likelihood of the formation of solid fully filled particles increases [3, 19]. In another study, Chau et al. [20] discovered that addition of NaCl solution to zirconium hydroxychloride solution leads to the formation of nondisrupted zirconia particles, whereas disrupted zirconia particles would have been produced otherwise.

Although the first application and modeling of spray pyrolysis date back to 1990s, the mechanism of particle formation particularly for complex and multicomponent particles is not clear yet. The overall objective of this research is to examine the effect of impurities on evolution of solution droplets and mechanism of particle formation. As a secondary objective, the possibility of preparing nondisrupted ZrO_2 and ZnO particles by spray pyrolysis will be studied. This is done by analyzing the effects of process temperature and the amount of impurity (NaCl) added to the precursor solutions on the resulting morphology and chemical composition of the bulk product.

Experimental procedure

Apparatus

The major equipment used in this study includes a spray generator, an environmental chamber, radiant cylindrical heaters, and temperature controllers. The spray generator is a commercial vibrating mesh nebulizer (Omron NE-U22V), used to create a mist of the precursor solution. The environmental chamber used in this study is a custom-built apparatus that houses the heaters. Each heater (DS Fibertech Co., International Thermoproducts Division, USA) is hollow and cylindrical in shape: 35.5 cm tall with an outer diameter of 20.0 cm and an inner diameter of 10.5 cm. Two heaters were used to achieve the desired height of the mist travel distance through the center of the heaters. The heaters were stacked on each other and attached at the seam. More details may be found elsewhere [3].

Materials

To produce zirconia, zirconium hydroxychloride, also known as zirconium oxychloride ($ZrOCl_2 \cdot 8H_2O$) dissolved in hydrochloric acid solution, abbreviated here as ZHC, is used as the precursor (Magnesium Elektron Inc. Flemington, NJ). It contains 20 wt% of zirconia. To produce zinc oxide, reagent grade zinc nitrate hexahydrate powder ($Zn(NO_3)_2 \cdot 6H_2O$), abbreviated as ZNH, with 98% purity is used (Sigma-Aldrich). The NaCl used is the reagent grade sodium chloride, >99.0% purity (BioShop Canada Inc).

To prepare a solution of ZHC/NaCl, NaCl powder is added to the aqueous solution of ZHC and stirred. To create a homogeneous solution of ZNH/NaCl precursor, the two substances are initially dry mixed and then dissolved in water and stirred. Table 1 summarizes the precursor

Table 1 Sample summary

ZrO_2 to NaCl mass ratio	Grams of (ZrO_2 + NaCl) per liter of solution	Process temperature (°C)
ZHC		
5:1	0.54	400
10:1	0.92	400, 600
15:1	1.26	400, 600
20:1	1.41	400, 600
1:0	1.34	400, 600
ZNH to NaCl mass ratio	**Grams of (ZNH + NaCl) per liter of water**	**Process temperature (°C)**
ZNH		
5:1	60	400, 600
15:1	160	400, 600
1:0	100	400, 600

solution composition of each sample and the conditions of preparation.

Methodology

The powders are produced at 400 and 600 °C reactor bulk temperature. A powder collection plate is placed atop the heaters so as not to fully cover it. A second plate with an opening is attached to the inlet of the bottom heater. The spray enters the heaters through this opening. Air naturally enters the heaters through the bottom opening. Through buoyancy forces of hot air, the droplets rise up the heated reactor shaft. Evaporation of solvent(s) and precipitation of solute(s) cause the droplets to shrink and form the final particles.

Powder characterization

The microstructures and composition of powders are obtained by a Scanning Electron Microscope (Hitachi S-4500). To assess whether the powder is disrupted or not, one SEM picture from each sample is analyzed to establish how much of the powder is disrupted. The ImageJ software is used to manually measure the diameter of the particles. Approximately 250 particles are measured per image. For the samples selected for energy dispersive X-ray spectroscopy (EDS) analysis, carbon coating is applied to the powder. All elements below sodium are disregarded in this analysis. The machine used for X-ray diffraction (XRD) analysis is the Philips PW1050/37 NR729, the Netherlands.

Results

Zirconia

Particle morphology

Figure 1 shows the SEM images of ZrO_2/NaCl powders produced at (a) 400 °C and (b) 600 °C, for various ZrO_2 to NaCl mass ratios. Various particle morphologies and surface topographies are observed. The particles identified as nondisrupted are shown in Fig. 2. These include: (a) smooth, fully round particles with minimal disruptions and (b) rough, fully round particles.

The particles identified as disrupted are shown in Fig. 3. These include: (a) sliced spherical particles, where these particles are the pieces of solid, round particles that seem to have been sliced from the original particle. They reveal an interior that appears to be solid, instead of a shell-like, concave wall; (b) hollow particles with a relatively small opening on the outer surface; (c) peeling particles, which have a fragmented outer wall, revealing a solid, but usually rough, interior surface. The interior surface is convex, indicating a second shell to the particle.

There are a few noticeable trends as the concentration of NaCl is increased. The most obvious trend is the increase in the percentage of nondisrupted particles as the concentration of NaCl increases. Figure 4 illustrates this trend for the powders produced at 400 and 600 °C. These results are consistent with those obtained by Chau et al. [20], who found that the addition of relatively large amounts of NaCl to ZHC solution results in the formation of a mixture of spherical and cubic particles which are nondisrupted. However, no cubic particles are formed in the present study. This is because, in this study, the highest mass ratio of NaCl to ZrO_2 is 1:5, less than those used by Chau et al. [20]. Another observation is that the sample with the highest percentage of nondisrupted particles also exhibited a sharp increase in rough particles at the increased temperature of 600 °C (see Fig. 1).

The temperature increase from 400 to 600 °C appears to dampen the positive effect of adding NaCl to ZHC solution to obtain nondisrupted particles. As can be seen qualitatively in Fig. 1, and quantitatively in Fig. 4, at 600 °C, the increase in NaCl concentration is not associated with a significant corresponding increase in the percentage of nondisrupted particles.

Particle size

Figure 5 shows the effect of NaCl concentration on the size of ZrO_2/NaCl particles produced at 600 °C. At 400 °C, the same trend was observed. As the ratio of ZrO_2 to NaCl increases, the total mass of the solute per liter of the solution decreases (see Table 1). Particle size is a function of solution parameters such as mole or mass of solute per liter of solution, porosity of the final particle, and also process parameters such as temperature, droplet number density, and so on [21, 22]. Introduction of NaCl into ZHC solution may alter particle evolution, crystal structure, and porosity. Comparing the data of pure zirconia powder and the powder with ZrO_2 to NaCl mass ratio of 20:1 reveals that introduction of NaCl into the precursor solution results in a larger mean particle size. Note that the amount of solute per liter of the solution is almost the same for these two powders (see Table 1). Further increase of NaCl results in a decrease in the particle size. This might be due to a decrease in the solution concentration as well. Figure 5 shows that the mean size of the nondisrupted particles is smaller than the overall particle size. This indicates that the disrupted particles are larger than nondisrupted particles, which is expected because larger hollow particles are more susceptible to disruption owing to their thinner walls.

Analysis of the data of Fig. 5 shows that the ratio of nondisrupted particle size to overall particle size generally

Fig. 1 Micrographs of ZrO_2/NaCl particles produced at (**a**) 400 °C and (**b**) 600 °C at different ZrO_2 to NaCl mass ratios. From top to bottom, the amount of NaCl is increased

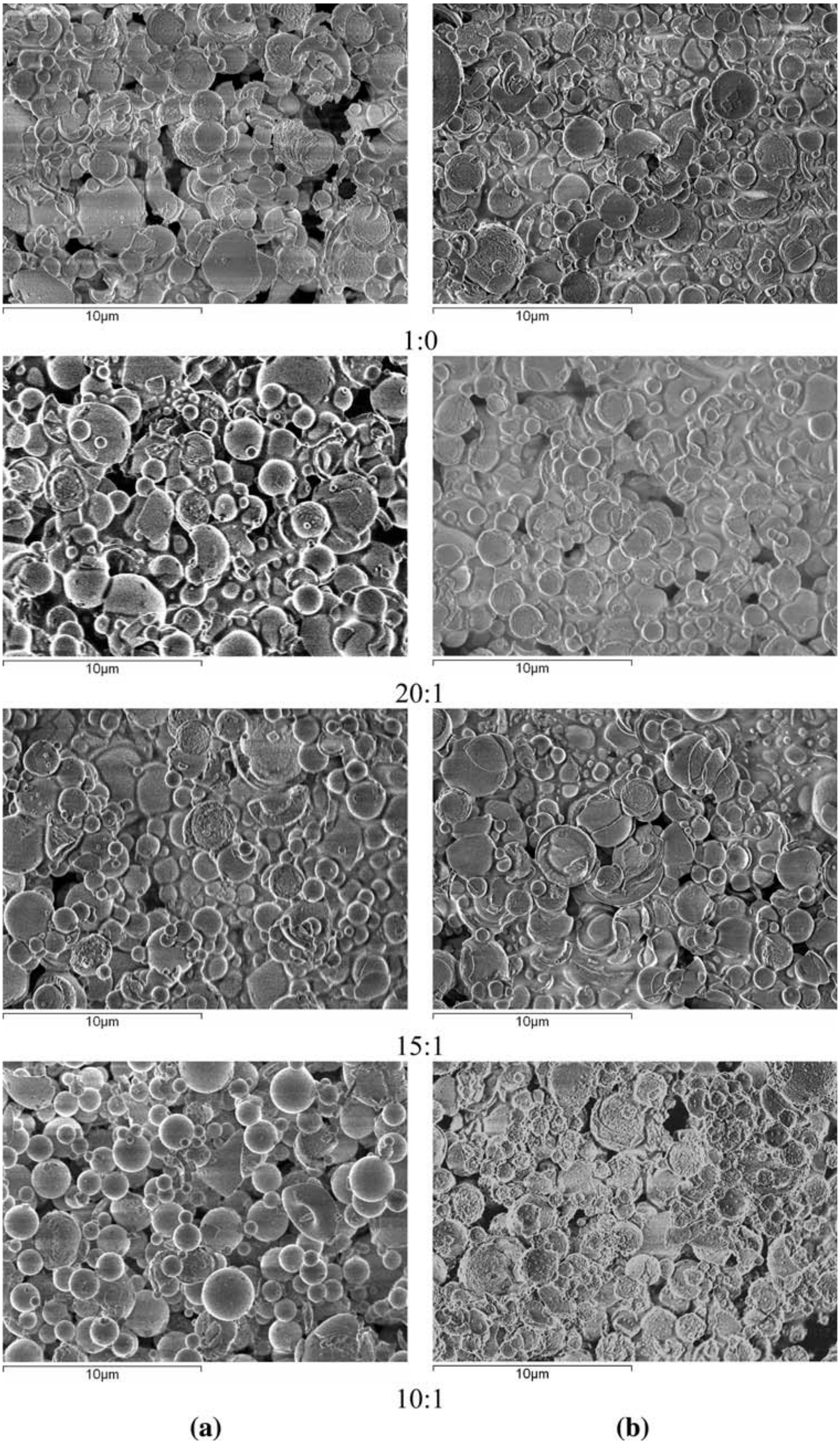


Fig. 2 Types of nondisrupted ZrO_2/NaCl particles. (**a**) Smooth, fully round. (**b**) Rough, fully round

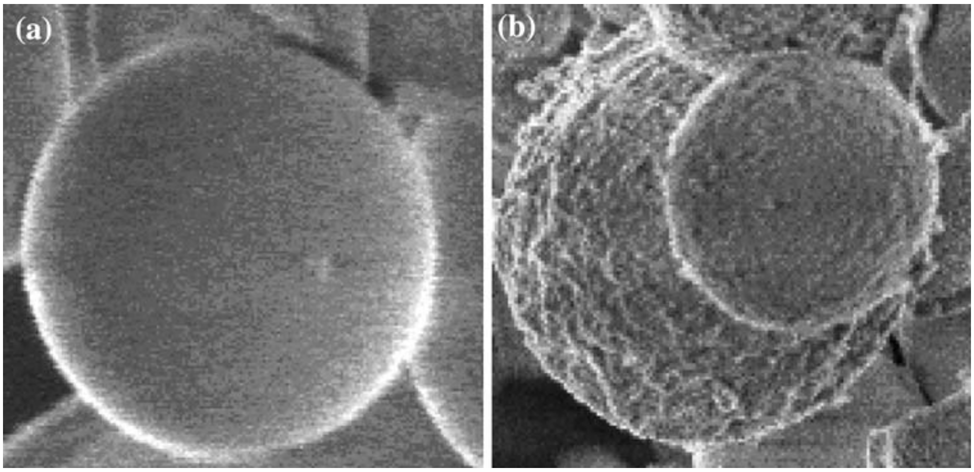
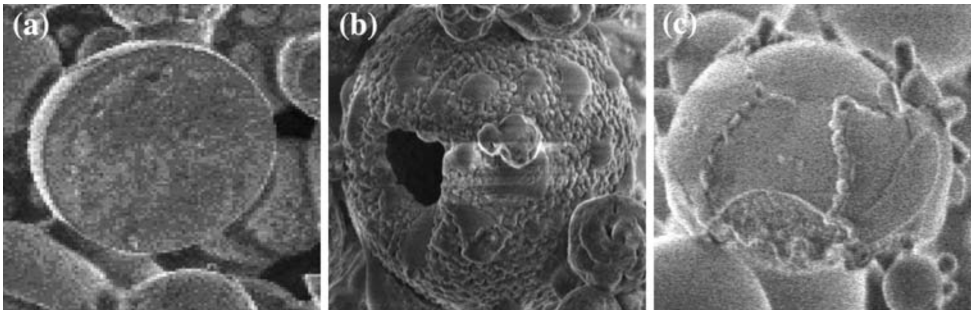


Fig. 3 Types of disrupted ZrO_2/NaCl particles. (**a**) Sliced spherical. (**b**) Hollow. (**c**) Peeling particles



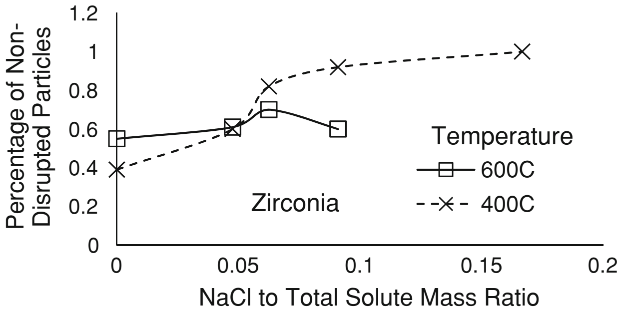


Fig. 4 Percentage of nondisrupted particles vs. the mass ratio of NaCl to total amount of solute for powders produced at 400 and 600 °C

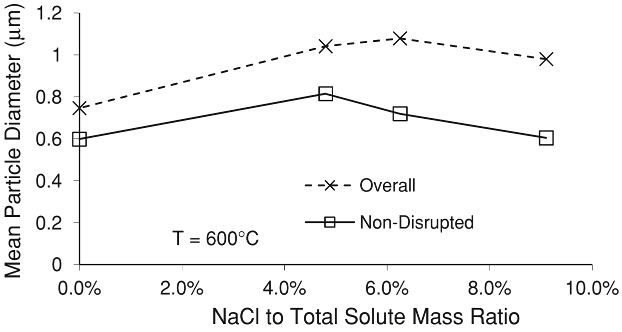


Fig. 5 Effect of NaCl concentration on the size of ZrO_2/NaCl particles produced at 600 °C

remains constant as the concentration of NaCl in the precursor solution varies. However, we found a noticeable difference in this size ratio as the process temperature is increased from 400 to 600 °C. While the average ratio for those powders produced at 400 °C is 0.83, the ratio decreases to 0.72 as the temperature is increased to 600 °C. This indicates that a higher process temperature is associated with the formation of nondisrupted particles with smaller sizes and/or the formation of disrupted particles with larger sizes.

Figure 6 outlines the distribution of the sample with a ZrO_2 to NaCl mass ratio of 15:1 chosen as the representative sample to compare with the base case (pure ZrO_2) produced at 400 °C. The total mass of solute per liter of the solution is similar for both cases (see Table 1). It is observed that the addition of NaCl to the precursor solution leads to an increase in the particle size, as was previously mentioned, by increasing the percentage of larger particles. Similar results and observations were made for powders prepared at 600 °C.

EDS compositional analysis

In a similar study performed by Chau et al. [20], a high percentage of NaCl was added to ZHC solution and, consequently, the powder was a combination of cubic and spherical particles. Their EDS analysis showed that the cubic particles had more chlorine and less zirconium, while

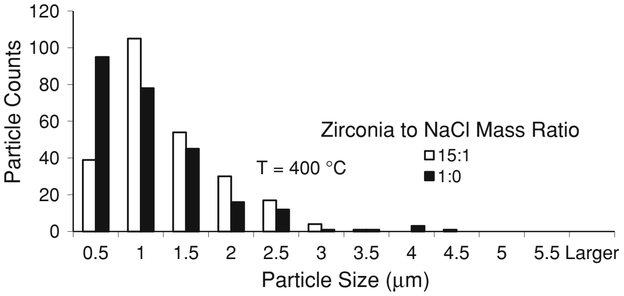


Fig. 6 Particle size distribution of ZrO_2/NaCl powder with and without NaCl in the precursor prepared at 400 °C

for the spherical particles it was the opposite. Here in this study, due to the addition of a slight amount of NaCl, no or very few cubic particles formed, and presumably all particles, which are similar in shape and morphology, have similar composition.

Figure 7 illustrates the qualitative percentage of Zr and Cl in pure ZrO_2 and ZrO_2/NaCl powders with two ZrO_2 to NaCl mass ratios, prepared at (a) 400 °C, and (b) 600 °C. This figure confirms that as the concentration of NaCl in the precursor solution increases, the amount of chlorine found in the resulting powder also increases. Comparison of Fig. 7a and b reveals the effect of temperature on powder composition. As expected, an increase in the process temperature results in less chlorine in the sample powders prepared at both temperatures. Higher process temperatures and longer process times are favorable for the depletion of chlorine.

XRD analysis

Figure 8 shows the XRD spectra for pure ZrO_2 and also ZrO_2/NaCl powders at ZrO_2 to NaCl mass ratios of 15:1, and 5:1, all produced at 400 °C. The base case, containing no NaCl, although only slightly crystalline, matches perfectly with the reference spectrum. The introduction of a slight amount of NaCl into the precursor solution (ZrO_2 to NaCl mass ratio of 15:1) results in a powder that also matches the zirconia database spectrum. An increase in the amount of NaCl (5:1) results in a powder that is not identical to the zirconium oxide characteristic peaks. Instead, it appears that the introduction of a sufficient amount of NaCl, which is crystalline even when produced at low temperatures, results in the formation of a crystal structure similar to that of NaCl. The low crystallinity of the rest of the samples indicates that at a relatively low process temperature, the resulting powder is amorphous.

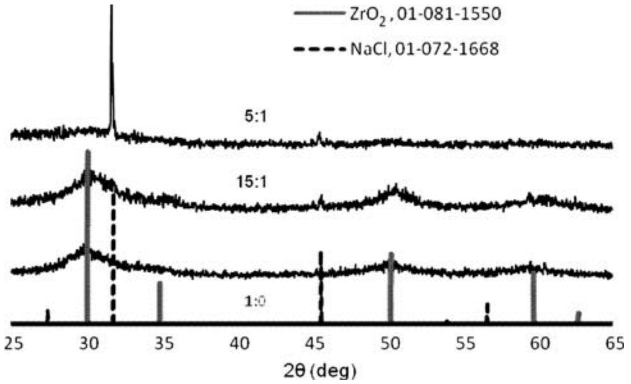


Fig. 8 X-ray diffraction spectra for pure ZrO_2 powder and ZrO_2/NaCl powders with ZrO_2 to NaCl mass ratio of 15:1, and 5:1, all produced at 400 °C. Reference spectra for NaCl and ZrO_2 are also shown

Zinc oxide

Particle morphology

Figure 9 shows images of ZnO/NaCl particles produced at (a) 400 °C and (b) 600 °C at different ZNH to NaCl mass ratios. In contrast to ZrO_2/NaCl, which clearly showed that an increase in the amount of NaCl leads to the formation of fewer numbers of disrupted particles, especially at 400 °C, there are no discernible trends present in the ZnO/NaCl powders; however, some interesting observations can be made. The ZnO/NaCl powders prepared at 400 °C do not have any distinct, spherical, nondisrupted particles. The only sample with distinct noncubic particles is the sample with no NaCl added to the precursor, even though they are all disrupted; however, when NaCl is introduced, nondisrupted cubic particles are formed. These are accompanied

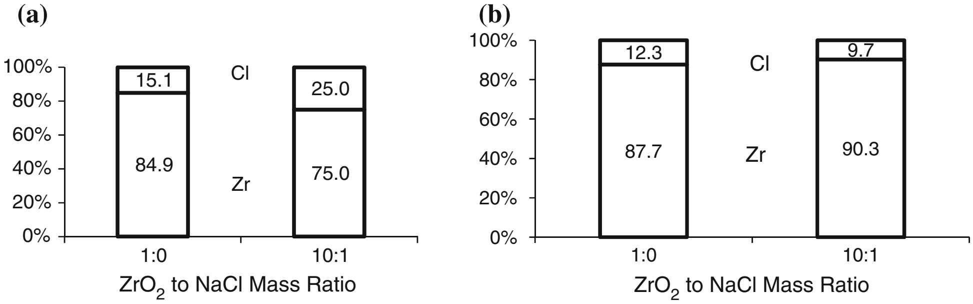


Fig. 7 Composition and weight percentage of elements in pure ZrO_2 and ZrO_2/NaCl powders prepared at (**a**) 400 °C and (**b**) 600 °C

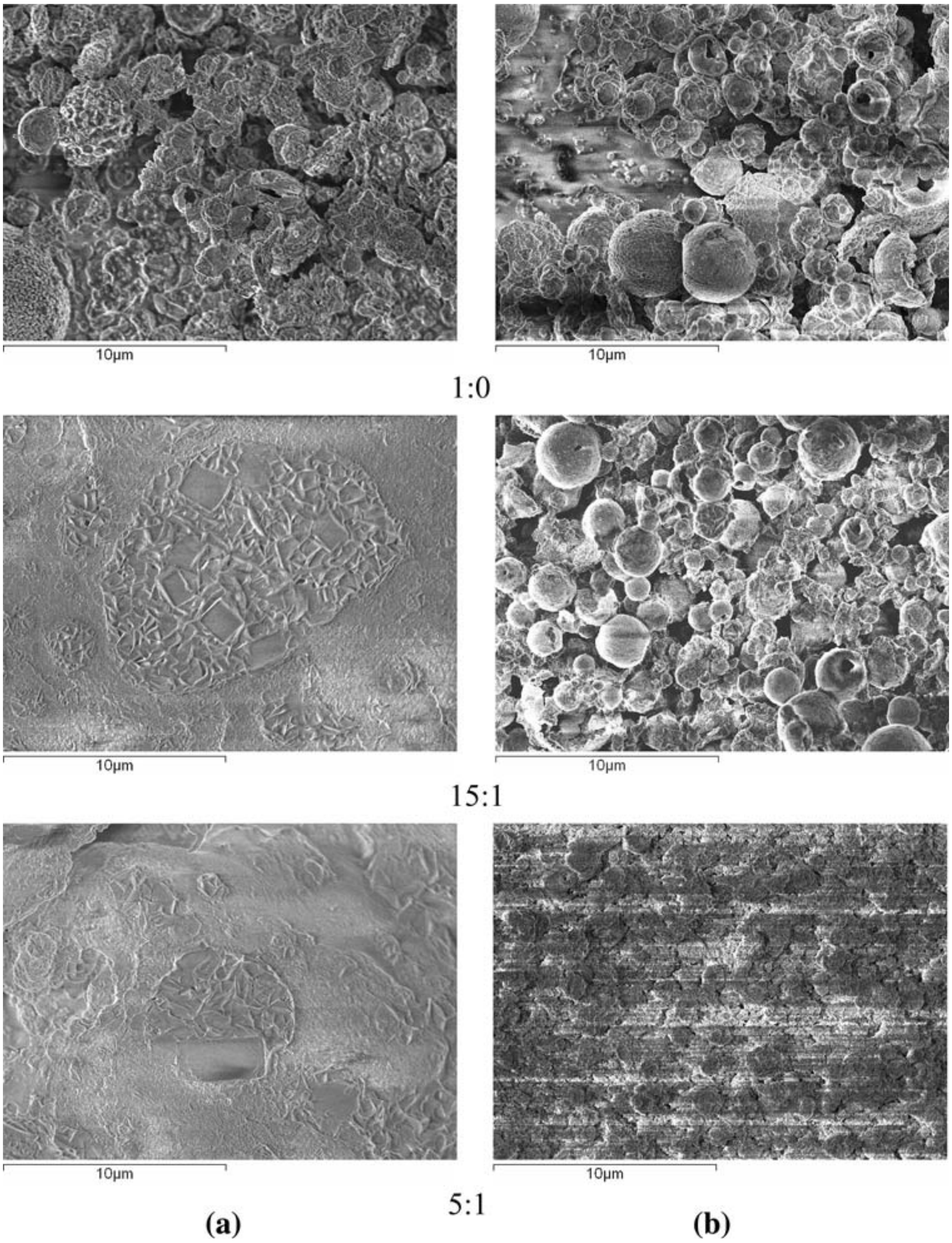


Fig. 9 Micrographs of ZnO particles produced at (**a**) 400 °C and (**b**) 600 °C, at different ZnO to NaCl mass ratios. From top to bottom, the amount of NaCl is increased

by round, jagged areas. Results from the EDS analysis that are provided later show a difference in the chemical composition of jagged areas and cubic particles. The jagged growth mechanism appears similar to those obtained by Milosevic et al. [23], even though they produced their powders at higher temperatures.

Although the ZnO/NaCl powders produced at 600 °C contain more distinct particles (Fig. 9b), there are almost no nondisrupted particles in any of the samples prepared at this temperature. At ZNH to NaCl mass ratio of 5:1, no distinct particles form. Instead, jagged particles and areas form, similar to those observed from the same precursor prepared at 400 °C. These jagged particles were not observed in the preparation of zirconia powders. The addition of NaCl at low concentrations (15:1) does seem to cause more distinct particles to form; however, they are still disrupted. As can be seen in Fig. 9, when particles form, most of them are in a torus shape, while some are hollow.

The most significant effect of a temperature increase on the formation of particles from the ZNH precursor seemed to have suppressed the growth mechanism for cubic particles. This allows the ZnO growth mechanism to dominate and results in the formation of only jagged or disrupted particles in the samples prepared at 600 °C with NaCl in the precursor. At 400 °C, the introduction of NaCl into the precursor solution prevents the formation of distinct particles.

Particle size

Figure 10 shows the effect of NaCl concentration on the particle size of ZnO/NaCl powders produced at 600 °C. It is observed that the introduction of NaCl into the precursor solution results in a larger mean particle size for both nondisrupted and overall population of the particles. Additional increase in the amount of NaCl with respect to ZnO, however, has a minor effect on the particle size. The size of particles produced at 400 °C with NaCl in the precursor solution could not be determined. Therefore, the results presented here are only for those powders produced at 600 °C, and the one powder with no NaCl in the precursor solution produced at 400 °C.

Size distribution of sample powders with no NaCl added, and the sample with ZNH to NaCl mass ratio of 15:1 showed the same trend in size between particles with and without NaCl in the precursor solution of zirconia (data not shown). The addition of NaCl causes a decrease in the percentage of smaller particles and an increase in the percentage of larger particles. The number of particles close to the mean size remains constant.

EDS compositional analysis

Figure 11a shows the composition of pure ZnO and cubic and jagged ZnO/NaCl powders with ZNH to NaCl mass ratio of 5:1, prepared at 400 °C. Note that the oxygen was not considered in the analysis. As shown in Fig. 9, and discussed before, the ZnO/NaCl powder is composed of cubic and jagged particles. There is a significant difference between the two types of particles. The cubic particles contain considerable amounts of chlorine and sodium, while the jagged particles contain large amounts of zinc. Despite there being no NaCl added to the baseline, sodium is still present in the particles, which might be an artifact of the EDS analysis.

Similarly, Fig. 11b illustrates the composition of pure ZnO and ZnO/NaCl powders with ZNH to NaCl mass ratios of 15:1 and 5:1, prepared at 600 °C. An increase in concentration of NaCl leads to a corresponding increase of chlorine in the final powder. The concentration of sodium, on the other hand, seems to top out when ZNH to NaCl mass ratio is 15:1.

XRD analysis

Figure 12 shows the XRD pattern of ZnO/NaCl powder produced at 600 °C and containing a 15:1 mass ratio of ZNH to NaCl. The spectrum of this sample is well matched to the database spectra of ZnO. However, there are several unidentified peaks present in the XRD spectrum, some of which rather match with those of NaCl. The other two uncorrelated peaks correspond with the intermediate phase of ZNH, $Zn_5(NO_3)_2(OH)_8 \cdot 2H_2O$ [23]. Although Studenikin et al. [24] identified 180 °C as the critical temperature for the thermal decomposition of ZNH precursor into ZnO, Milosevic et al. [23] showed that the

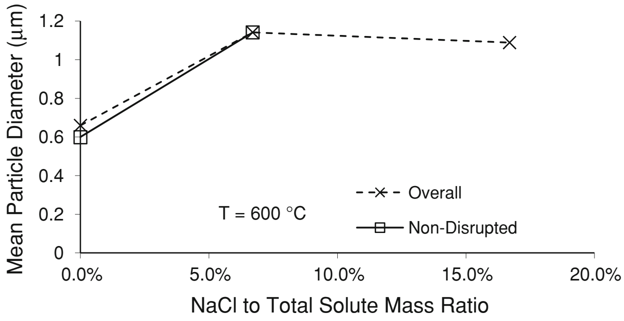


Fig. 10 Effect of NaCl concentration on ZnO particle size prepared at 600 °C

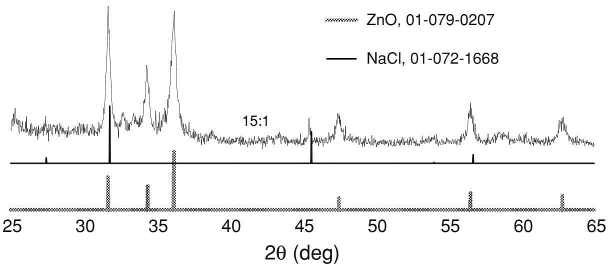
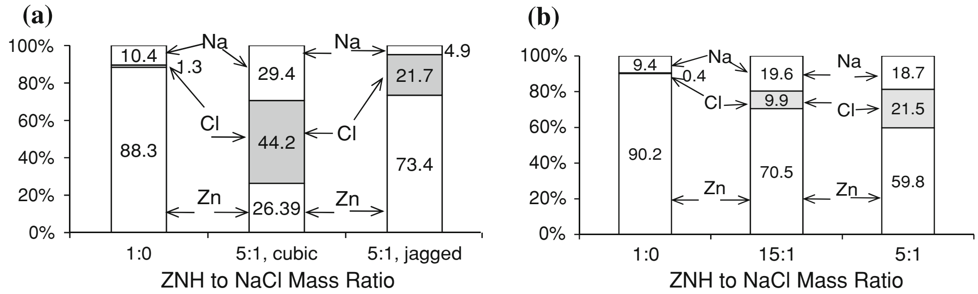


Fig. 12 XRD spectrum for ZnO powder produced at 600 °C with ZNH to NaCl mass ratio of 15:1. The reference spectra for ZnO and NaCl are also shown

Fig. 11 (**a**) Composition and weight percentage of elements in pure ZnO and cubic and jagged ZnO/NaCl powders with ZnO to NaCl mass ratios of 5:1, all prepared at 400 °C. (**b**) Composition of powders at ZnO to NaCl mass ratios of 1:0, 15:1, and 5:1, all prepared at 600 °C



intermediate hydrate is more likely to form if the exhaust gases containing the evaporated water pass over the collected powder, which was the case in our experiments.

Discussion

Zirconia

In previous studies, it was found that micron-sized NaCl particles (used in asthma treatment) produced at 400 °C were fully formed, with minimal disruption [3]. This was explained by the fact that in NaCl a growth mechanism different from most other materials, such as ZrO_2 is followed. Spray drying/pyrolysis of most solution droplets at high temperatures results in the formation of *disrupted spherical* particles; however, spray drying/pyrolysis of some other solution droplets such as NaCl produces *nondisrupted cubic* particles. NaCl particles have either very strong walls that help them withstand the pressure build-up within the particle [25] and/or have porous surfaces to facilitate the release of pressure build-up caused by solvent vapor entrapment. This characteristic of NaCl was utilized by Chau et al. [20] to produce nondisrupted particles of ZrO_2 at 400 °C by introducing NaCl impurities into the precursor. They found that the addition of relatively large amounts of NaCl to precursor solution results in the formation of nondisrupted ZrO_2/NaCl particles, but no clear conclusion was drawn on how the *variation* of the concentration of NaCl in the solution might affect the powder characteristics such as its morphology and crystallinity. The results of the current study indicate an increase in the fraction of nondisrupted particles of zirconia as the NaCl content is increased, for both process temperatures of 400 and 600 °C. Furthermore, if the amount of NaCl is low, no cubic particles from. This may indicate that the growth mechanism of ZrO_2 prevails.

The increase in crystallinity of the powder is evident in the samples containing more NaCl. However, the absence of cubic particles in the samples with high crystallinity suggests, as mentioned above, that the growth mechanism favored is that of ZrO_2. The explanation of the increase in crystallinity with the increase in NaCl concentration may be as simple as the NaCl not reacting and simply forming rather pure particles in the final powder, and adding to the crystallinity of the entire sample. However, if it is not reacting, then NaCl should form cubic particles at this temperature, and the fact that it does not, suggests that all elements have been engaged in the formation of each individual particle. The SEM/EDS results, which do not indicate the presence of cubic NaCl particles, favor the latter hypothesis.

Zinc oxide

In contrast to what was observed for ZHC solution, introduction of NaCl into ZNH did not lead to the production of nondisrupted ZnO particles; it rather had an opposite effect. The process of adding NaCl or other salts into the precursor in SP, resembles the salt-assisted aerosol decomposition process (SAD) described by Okuyama and Lengorro [26]. During the SAD process, provided the droplet/particle temperature exceeds the melting point of the salts, the salts melt and act as high-temperature solvents. The material or its components can then dissolve, undergo reactions, and upon exceeding the solubility limit, precipitate in the solvent. These processes, are absent in conventional SP. In SAD, within a particle, the dissolution/precipitation cycle can lead to the dissolution of some nanocrystallites and the growth of other crystallites by precipitation. This may break down the particle and lead to the disintegration of the individual nanocrystallites to form nanoparticles. The melting point of NaCl is 801 °C and the melting point of ZNH, which is basically its decomposition point to zinc nitrate and water, is 36 °C. Thus, for the two process temperatures used in this research (400 and 600 °C), zinc nitrate and sodium chloride are merely *dissolved* in water and not melted. Therefore, the formation of a particle from a droplet is expected to be by precipitation of the solute within the droplet, similar to the conventional SP, as proposed by Jayanthi et al. [18]. This hypothesis is supported by the SEM images of all ZrO_2/NaCl powders prepared at 400 and 600 °C (Fig. 1), and also ZnO/NaCl powders prepared at 600 °C (Fig. 9), which clearly indicates that each single droplet has formed a single particle. However, the SEM images show that ZnO/NaCl powders produced at 400 °C are not composed of distinct particles. Instead each droplet has been severely broken down into smaller particles, similar to the SAD process. These observations are against the model developed by Jayanthi et al. [18] which states that one solution droplet is converted to a spherical particle, at least for multicomponent solutions. Further research is required to address these intraparticle phenomena. Primarily, it is suggested that the method of adding salts to the precursor solution be extended to other powders, as was done with zinc oxide and zirconia here. Also, employing higher process temperatures so that the added salt can melt within the droplet may help examine and explore the salt-assisted spray pyrolysis method. These are in the direction of discovering new methods to produce nanoparticles due to the destruction of single droplets/particles, and production of nondisrupted strong particles, desirable for many emerging technologies.

Conclusions

This study revealed the effects of process temperature and introducing NaCl and varying its concentration in the precursor solutions on morphology and other characteristics of zirconia and zinc oxide powders. Several significant findings were discovered.

First, the introduction of NaCl into the zinc nitrate precursor solution has an adverse affect on particle morphology, i.e., it results in the disruption of a particle into smaller particles. This effect is observed at both 400 and 600 °C, although at the higher temperature this effect only becomes apparent when the amount of NaCl in the precursor is increased to a higher concentration. This effect is similar to that observed in the salt-assisted spray pyrolysis method. In contrast, when NaCl is added to zirconium oxychloride, with an increase in the NaCl concentration, the number of disrupted particles decreases.

The effect of increasing the temperature during the spray pyrolysis process of both zirconia and zinc oxide negates the effects of increasing NaCl concentration in the precursor solutions that are observed at 400 °C. In the production of zirconia powder prepared at 600 °C, increasing the concentration of NaCl does not improve the morphology of the final particles produced. Similarly, for the zinc oxide, the powder prepared at 600 °C does not exhibit the same destructive effects that are observed when NaCl is added to the precursor of the powder prepared at a lower temperature. It is observed that the addition of NaCl to the precursor solutions leads to an increase in the final particle size at both temperatures.

One last conclusion is that the available spray pyrolysis models may fail to predict the morphology of particles if more than one solute is involved. Development of more sophisticated particle formation models is needed.

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