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Ionic–gelation synthesis of gadolinium doped ceria ($\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.90}$) nanocomposite powder using sodium-alginate

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ABSTRACT

Nanocomposite powders of gadolinium-doped ceria (GDC, $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.9}$) were synthesized via thermal treatment of the gel formed by contacting ionic solutions of sodium alginate as the jelling template and metal (gadolinium/cerium) nitrates as the starting material. The influence of calcination temperature and sodium alginate loading fraction on the properties of the synthesized GDC nanocomposite powders was investigated. Characterization was performed by energy dispersive X-ray spectroscopy, powder X-ray diffraction, thermogravimetric analysis, Field Emission Scanning Electron Microscopy, Fourier transformed infrared spectroscopy and nitrogen adsorption/desorption analysis. It was observed that the particle size and the surface area of the produced GDC nanocomposite powders are dominantly controlled by the calcination temperature, while the effect of sodium alginate loading fraction is limited by the range of the calcination temperature. In this study, the smallest mesoporous GDC nanocomposite powder with cubic fluorite structure (8 nm crystallite size and $3.05 \pm 0.005 \text{ m}^2/\text{g}$ surface area) was synthesized using 2 wt% of sodium alginate at a calcination temperature of 550 °C (for 4 h). The results of this study could help to perceive the influence of the basic processing variables on the particle size and the other physiochemical properties of GDC nanocomposite powders produced by the ionic-gelation method.

1. Introduction

Solid oxide fuel cell (SOFC) is amongst the prospective energy conversion devices with a capability to efficiently generate electricity in an environmental-friendly manner [1,2]. Depending on the operating temperature range, SOFC are classified into low, intermediate, and high temperature categories. The conventional SOFCs operating at ~1000 °C are mainly fabricated using yttria-stabilized zirconia (YSZ) as the electrolyte material [3,4]. The operation of SOFCs at such high temperatures is susceptible to a number of drawbacks such as higher costs, lower flexibility in the fabrication of electrodes, more thermal degradation, and more crack formation due to the larger thermal stress of the cell components [1,5–8]. This has created a considerable interest in replacing high temperature SOFCs with low and intermediate temperature SOFCs (500–700 °C), which provides more flexibility in fabrication of electrodes, higher thermal stability, and wider choice of material selection [9,10]. Therefore, synthesizing alternative electrolyte

powders with a higher ionic conductivity and an improved thermal stability is crucial to fabricate SOFCs operating at reduced temperatures [5]. Recently, Gadolinium doped ceria (GDC) has seen extensive consideration as an attractive electrolyte material suitable for low- and intermediate- temperature SOFCs operating at 500–700 °C [11–13].

In the last decade, with increasing interests in application of GDC powder in SOFC and catalysis sectors, many research papers have reported its synthesis with different methods such as sol-gel [14–16], hydrothermal treatment [17,18], acetylacetonate precursor method [19], combustion [20–22], high energy ball milling [23], co-precipitation [24,25] and polymer pyrolysis [26,27]. Co-precipitation and hydrothermal methods have been widely used for synthesizing GDC powders, but both methods have been reported to show agglomeration of the fine crystallites, non-reproducibility, and a relatively high cost [23]. The precursors used in the sol-gel method such as ethylene glycol [5] and ammonium carbonate [28] are highly degradable and toxic. Similarly, though it has been reported that nanocrystalline powders can

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Table 1
Loading fraction of sodium alginate and calcination temperature for different samples.

Experiment ID	Sodium Alginate loading fraction (wt%)	Calcination temperature (°C)
1	0.5	550
2		600
3		650
4		700
5	1	550
6		600
7		650
8		700
9	2	550
10		600
11		650
12		700

Fig. 1. Thermal analysis of a) as-prepared GDC nano-composite and b) calcined sample with 2% sodium alginate at 700 °C.

Table 2
TGA weight losses for all samples.

Sodium Alginate loading fraction (wt%)	Dried Sample						Calcined Samples		
	Step 1 Range (°C)	Weight loss (%)	Step 2 Range (°C)	Weight Loss (%)	Step 3 Range (°C)	Weight loss (%)	Calcination Temperature (°C)	Range (°C)	Weight Loss (%)
0.5	25–181	12.75 ± 0.1	182–330	34.65 ± 0.15	355–500	27.85 ± 0.075	550	25–196	4.2 ± 0.1
							600	25–195	4.1 ± 0.1
							650	25–200	4.2 ± 0.1
							700	25–195	4.2 ± 0.1
1	25–186	11.97 ± 0.1	186–333	33.73 ± 0.15	343–500	27.14 ± 0.075	550	25–198	4.6 ± 0.1
							600	25–193	4.6 ± 0.1
							650	25–197	4.6 ± 0.1
							700	25–205	4.8 ± 0.1
2	25–180	10.1 ± 0.1	180–330	35.75 ± 0.15	330–500	24.48 ± 0.075	550	25–198	5.7 ± 0.1
							600	25–216	5.9 ± 0.1
							650	25–191	5.8 ± 0.1
							700	25–200	5.9 ± 0.1

be synthesized with high yield by the polymer pyrolysis route [29], the pretreatment procedure employed by the polymer-pyrolysis technique can create toxic NO [7]. The combustion method is amongst the most commonly used procedures for synthesizing metal oxide and ceramic nanocomposites [30]. However, due to the large volume expansion resulting from the precursors burn-off step, this method is uncontrollable in some extents, particularly for large-scale production of GDC nanocomposites [7]. Therefore, development of a simple, efficient, inexpensive and environmentally-friendly technique for synthesizing GDC nanocomposite powders with high purity is of great interest for both the manufacturing and research communities [31].

“Green synthesis” has been considered as an efficient emerging technique to reduce the generation of hazardous waste for synthesizing high demanding products such as nanocomposites [32–38]. Amongst the “green” materials utilized in “green synthesis” methods, polysaccharides have been found to be a dominant category due to their massive availability, unique organic nature, and less requirements for further purification steps [39]. Synthesis of GDC powder using renewable resources such as maltose and sucrose with pectin have been reported by a sol-gel method [38,40]. Sodium alginate is a nontoxic, anionic, water soluble, and biodegradable polysaccharide extracted from brown seaweed with the main structural blocks of C-5 epimer α -L-guluronate (G) and (1-4)-linked β -D-mannuronate (M) that are covalently bonded together in various sequences [34,41]. One of the most vital characteristics of sodium alginate is the occurrence of carboxylate groups in its structure. The gelation occurs by forming a non-soluble precipitate due to cross-linking of the buckled polyguluronate segments of alginate by exchanging sodium counter-ions with multivalent metal cations through a mechanism known as the ‘egg-box’ model [33]. The existence of the gel structure restraining the metal ions prevents them getting easily close to each other and thus increasing the likelihood of creating smaller nanocomposites [34]. The ionic gelation method has been recently used for synthesizing a range of metal oxide nanoparticles, drug-delivery materials, adsorbents, biopolymers, and biotechnology materials [41–45]. However, as far as our knowledge extends, there has been very little research reported on the application of sodium-alginate for synthesizing solid oxide electrolyte powders. Therefore, this research aims to analyze the effect of synthesizing variables such as the calcination temperature and loading fraction of sodium-alginate on the properties of the GDC nanocomposite powders.

In this study, the ionic-gelation method using sodium alginate as the jelling template has been applied to synthesize high purity GDC ($\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.90}$) nanocomposite powders. The influence of relevant synthesis variables such as loading fraction of sodium alginate and calcination temperature on the purity and particle size of the synthesized GDC nanocomposite has been systematically studied.

(A)

(B)

Fig. 2. FESEM images for loading fraction of 0.5 at calcination temperatures of for A) 550 °C, B) 600 °C C) 650 °C and D) 700 °C.

Characterization of the GDC nanocomposites acquired in this study is performed using thermal analysis (TGA/DSC), X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), Energy-dispersive x-ray spectroscopy (EDX), Fourier transform infrared spectroscopy (FTIR) and Nitrogen adsorption-desorption (BET). This investigation could be helpful to understand the influence of the dominant synthesizing variables on the physiochemical properties of GDC nanocomposite powders produced by the ionic-gelation method using sodium alginate.

2. Materials and methods

2.1. Materials

Gadolinium nitrate hexahydrate ($\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 99.9+% purity) and cerium nitrate hexahydrate ($\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, 99+% purity) from Sigma Aldrich., USA were used as the starting materials without any further purification in this work. Also, Manugel GHB sodium alginate powder ($(\text{C}_6\text{H}_7\text{NaO}_6)_n$, MW: 264 kDa) with an M/G ratio of 0.59 (BioPolymer, UK) was used as the jelling template.

2.2. Preparation of nanocomposite GDC powder

Cationic solutions of Ce^{3+} and Gd^{3+} with a total concentration of 30 g/L were prepared by dissolving the calculated quantities of the metal nitrate salts ($\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$) into ultra-pure water. Relevant quantities of sodium alginate powder were dissolved in ultra-pure water using a water-bath at 80 °C to prepare sodium alginate solutions with three different loading fractions (0.5, 1, and 2 wt%).

A gel was formed by adding the prepared sodium alginate solutions slowly to the cationic metal solution that was stirred gently for 24 h by a mechanical stirrer to complete the ion-exchange reactions. The ion-exchange reactions for a single sequential segment of sodium-alginate polymer block could be written as follows:

The resulting gel was then washed thrice with 30 ml ultrapure water

(C)

(D)

Fig. 2. (continued)

to eliminate any impurity (sodium, cerium, or gadolinium nitrates) trapped in the gel structure that was followed by drying in an oven (Memmerth, UF110) at 50 °C for 24 h. Next, the dried samples were calcined at various temperatures (550 °C, 600 °C, 650 °C and 700 °C) with a fixed ramp of 2 °C/min for 4 h. The calcined powders were ground with mortar and pestle to break the agglomerated particles. Table 1 represents a summary of the applied processing variables and the obtained GDC particle sizes for the synthesis experiments in this work.

2.3. Characterization

A thermogravimetric analyzer (TGA, Q50, and USA) was used to study the thermal decomposition behavior of the dried gel and also the calcined samples in air for temperatures ranging from 25 °C to 800 °C with an increasing ramp of 5 °C /minute. An XRD diffractometer (XRD, D8 Discover Bruker., Germany) was used to perform X-ray powder diffraction with $\text{CuK}_{\alpha 1}$ radiation ($\lambda=1.54060 \text{ \AA}$) in the 2θ range

between 10° and 90° and a scanning rate of 0.02°/s using the Bragg-Brentano beam method at 40 kV and 40 mA. All XRD patterns were recorded utilizing ICDD file no. 00–50–0201. The crystallite sizes of the GDC nanocomposite powders were calculated by Scherrer's equation ($D = 0.9\lambda/\beta\cos\theta$, where D denotes the crystallite size in nm, λ denotes the source wavelength in nm, β denotes the full width at half maximum (FWHM) of the peak, and θ is the Bragg angle in radians) [46].

The morphology of GDC nanocomposite were analyzed by the micrograph images obtained by a field emission scanning electron microscope (FESEM, SU8010 Hitachi, Japan). Energy-dispersive x-ray (Oxford Horiba Inca XMax50 EDX) analysis was performed to confirm the presence of cerium, gadolinium and oxygen atoms in all samples. The FTIR spectra of the calcined and the dried powders were obtained by a Fourier transform infrared spectroscopy analyzer (FTIR, NICOLET 1810., USA) over the wavenumber range of 650–4000 cm^{-1} . The surface area of the calcined samples was measured by a BET analyzer (micromeritics, ASAP 2020., USA).

Fig. 3. FESEM images for loading fraction of 1 at calcination temperatures of for A) 550 °C, B) 600 °C) 650 °C and D) 700 °C.

3. Results and discussion

The thermogravimetric analysis (TGA/DTA) of the dried and the calcined samples for 2% sodium alginate at 700 °C are shown as an example in Fig. 1. The TGA data for all remaining samples are provided in detail in Table 2. As could be observed the changes in the results are not significant. However, by looking closer the samples containing more Na-Alg show slightly higher weight loss. This is due to more carbonaceous matters available in the structure of the alginate. Three weight-loss steps corresponding to room temperature ($25\text{ °C} \pm 1\text{ °C}$)–180 °C, 180 °C–330 °C, and 330 °C–500 °C were observed for the dried sample (Fig. 1a). It was found that the decomposition stages in the TGA curves were compatible with the DTA curves represented in Fig. 1a. Approximately, $10.1 \pm 0.10\%$ weight-loss was observed in the first step (25–180 °C) that relates to dehydration of the alginate structure. In the next step (180 °C–330 °C), a weight-loss of $35.75 \pm 0.15\%$ was observed that could be due to the cleavage of alginate segment blocks resulting from the formation of carbonaceous chelated metal oxides (gadolinium and cerium). This is in accordance with the

weight-loss reported for alginate by Wang et al. [35] in the same range of temperature, which was justified as result of the cleavage of G-G, M-M and G-M weaker linkages in alginate structure. In the last step, by increasing the temperature from 330 °C to around 500 °C, the residues of carbonaceous material is oxidized by showing approximately $28.48 \pm 0.075\%$ weight-loss, resulting in an exothermic decomposition peak. In other studies, it has been mentioned that the α-L-guluronic acid (G) and β-D-mannuronic acid (M) residues in the alginate structure are completely oxidized in the last step of TGA profile [35,39]. No additional weight loss was noted above 500 °C. Fig. 1a shows the decomposition procedure could finish around 500 °C. For the sample calcined at 700 °C for 4 h (Fig. 1-b), it was observed that there is only a weight loss of $5.9 \pm 0.1\%$ occurred between ambient temperature and around 200 °C that could be due to desorption of its moisture content. No further weight loss was observed over 200 °C, indicating there is no existence of carbonaceous bonds or any other undecomposed residue in the sample.

The FESEM images of GDC nanocomposites synthesized with loading fractions of sodium alginate of 0.5, 1 and 2 are displayed in

(C)

(D)

Fig. 3. (continued)

Fig. 2, Fig. 3 and Fig. 4. Each figure comprises of four FESEM images corresponding to calcination temperatures of 550 °C, 600 °C, 650 °C and 700 °C. The FESEM images were quantitatively analyzed using the publicly available image processing program, ImageJ [47,48]. For this analysis, each image was intensity-thresholded to obtain a binary image, first. Next, a series of morphological operations were performed to carefully eliminate any irregular shapes with smoothing the remaining particles. Finally, the resultant binary images were passed to the automated analysis module for measuring various parameters, including the particle size and circularity. The particle sizes distribution for each image has been embedded in its top right corner, where μ denotes the average particle size with the standard deviation and N denotes the number of particles utilized in the analysis. The relative frequencies of particles corresponding to each circularity range is illustrated using a stacked bar in Fig. 5, where the loading fraction and calcination temperature corresponding to each experiment can be obtained from Table 1. In Fig. 2, Fig. 3, and Fig. 4, it can be observed that, by increasing the calcination temperature (from 550 °C to 700 °C), the particle size gradually becomes larger. This trend may be

a result of the agglomeration of particles that occurs with increasing temperatures due to the 'uncontrolled buildup', 'bridging', or 'lumping' phenomena within the thermal treatment steps [49].

It was found that for a fixed calcination temperature, increasing the loading fraction of sodium alginate produced the nanocomposite powders with smaller particle sizes. This could be explained as a result of forming a denser consolidated cross-linked alginate using the concentrated sodium-alginate solution that consequently increases the biopolymer-to-metal ratio in the resulting precursor (at a fixed concentration of the cationic solution, 30 g/Lit). Therefore, smaller and more segregated metal-oxide particles could be formed by thermal decomposition of this biopolymer-concentrated mixture due to the presence of more degradable material in the precursor structure. This trend can be observed by comparing the representative images for any given calcination temperature across all three figures (e.g. Fig. 2C, Fig. 3C and Fig. 4C).

The results show that increasing the calcination temperature has a more consistent effect on the particle size compared to increasing the loading fraction. As shown in the figures, the particle size consistently

(A)

(B)

Fig. 4. FESEM images for loading fraction of 2 at calcination temperatures of for A) 550 °C, B) 600 °C C) 650 °C and D) 700 °C.

became larger with increasing calcination temperatures (by an increase from 10.09 to 17.87 nm, 9.43–17.53 nm, and 8.27–16.75 nm corresponding to 0.5, 1, and 2 wt% of alginate, respectively), while it is noticeably reduced by increasing the alginate loading fractions at lower calcination temperatures (by a decrease from 10.09 to 8.274 nm at 550 °C observed in Fig. 2A, Fig. 3A and Fig. 4A, and from 12.84 to 9.23 nm at 600 °C in Fig. 2B, Fig. 3B and Fig. 4B). At higher calcination temperatures (650 °C and 700 °C), though a small reduction of the particle size due to increasing the alginate loading fraction could be observed, however it was found to be minimal compared to the lower calcination temperatures (only a decrease from 15.20 nm to 13.97 at 650 °C in Fig. 2C, Fig. 3C and Fig. 4C, and from 17.87 nm to 16.75 at 700 °C in Fig. 2D, Fig. 3D and Fig. 4D). This indicates that the rate of decrement of the particle size (of the calcined nanocomposite powders) with increasing loading fraction is lower at higher temperatures. This observation could be explained by the higher rate of the particles agglomeration occurred by increasing the calcination temperatures, which can influence the powder particle size by concealing the effect of increasing the alginate loading fractions. In conclusion, for preparing

more segregated and smaller GDC nanocomposites, it is recommended to use a concentrated sodium alginate solution (e.g. by 2 wt% loading fraction) followed by calcination of the dried gel at lower temperature range (e.g. 550 °C–600 °C).

Fig. 5 illustrates the relative frequencies of particles within each circularity range for the experiments. As the FESEM images are 2-dimensional, the circularity is the most relevant measure to report the sphericity (3-dimensional) of the particles. By referring to the legend in Fig. 5, it can be seen that a very small percentage of particles have a circularity value of less than 0.5. In most experiments, more than 90% of the particles have a circularity value of more than 0.6 and in almost every experiment; the circularity range with the largest percentage of particles is 0.8–0.9. These observations confirm that the GDC nanocomposite powders obtained from all experiments are indeed semi-spherical.

Fig. 6 displays the EDX elemental analysis and mapping for the GDC nanocomposite synthesized by 1 wt% of sodium alginate at 650 °C, which confirms the presence of cerium, gadolinium and oxygen atoms in GDC nanocomposite. The sample presented in Fig. 6 was

(C)

(D)

Fig. 4. (continued)

Fig. 5. Average circularity measures of nanocomposites calculated using ImageJ for all experiments.

selected as an exemplar of all samples studied in this paper. Fig. 6 also presents a table of experimental values reported by EDX. The theoretical values for the atomic weight percentage of cerium, oxygen and gadolinium atoms for the synthesized GDC in this work ($\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.90}$) were calculated as $53.28 \pm 0.05\%$, $29.92 \pm 0.05\%$ and $16.74 \pm 0.05\%$ respectively. The percentage error between the theoretical and the experimental values of the atomic weight percentage for cerium, gadolinium and oxygen were calculated as 3.27%, 2.84% and 4.96% respectively that confirms the high purity of the synthesized nanocomposite.

Fig. 7 represents the x-ray diffraction (XRD) patterns of GDC nanocomposite powders synthesized at various calcination temperatures and alginate loading fractions. The XRD analysis of the powders indicated that the samples were crystallized in single phase cubic fluorite structure of $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{1.9}$ with space group $\text{Fm-3m}(225)$, which conforms to the ICDD data (card no.00-50-0201). The average crystallite size of particles determined by the Scherrer's equation, are shown in Table 3. The calculated lattice constant were $5.4205 \text{ \AA}$, which is exactly the same as the theoretical value obtained from ICDD file no. 00-50-0201.

Fig. 6. EDX analysis of GDC oxide, 1% at 650 °C.

The XRD patterns showed that the peaks became sharper by increasing calcination temperature from 550 °C to 700 °C at different loading fractions of sodium alginate that is due to the particles agglomeration and gradual growth of the crystallite size of particles, as reported in literature [49]. The calculated crystallite size given in Table 3 can also confirm the influence of increasing the calcination temperature on growing the particle size for different samples (that has been already discussed in the FESEM results). The calculated crystallite size in Table 3 show good consistency with the results acquired from the quantitative image analysis reported in Fig. 5 that confirms the influence of both the calcination temperature and the alginate loading fraction on the powder particle size. Furthermore, the high similarity between the reported crystal sizes (XRD) and particle sizes (FESEM) in Table 3 indicates that this method is able to produce a nanocrystalline GDC powder. The presence of broader peaks in an XRD pattern at lower temperatures shows the existence of smaller crystallite size for those samples [8]. This could be verified by the broad peaks obtained for the samples calcined at 550 °C and 600 °C (Fig. 7) and the corresponding crystallite size (given in Table 3) as well.

As seen in Table 3, by increasing the weight percentage of sodium alginate from 0.5% to 2%, the particle size decreased consistently at lower calcination temperatures of 550 °C and 600 °C that confirms the trend found by analyzing the FESEM images. According to the results of Table 3, it can be concluded that the calcination temperature of samples has a much more significant influence on the particle size of the synthesized GDC nanocomposite powders as compared to the loading fraction of sodium alginate.

Fig. 8 shows the FTIR spectra for the dried gel and the calcined samples at different temperatures. The dried sample FTIR spectrum shows many adsorption bands in the region of 800–1600 cm^{-1} . The peaks around 884 and 955 cm^{-1} could be due to C-C and C-O bands for sodium alginate [50]. Also, the stretch around 3350 cm^{-1} and peaks around 1029 and 1413 cm^{-1} could corresponds to O-H stretching, C-O-C and COO⁻ (symmetric) groups of sodium alginate respectively [51,52]. A strong peak is observed at wavenumber $\sim 1580 \text{ cm}^{-1}$ in the dried samples that could be caused by the formation of M-O bonds following the ion-exchange between the Na⁺ in alginate and the metallic solution cations (Ce⁺³/Gd⁺³) [34,53]. It can be observed that

Fig. 7. XRD pattern of GDC oxide nanocomposite with (a) 2% Sodium alginate, (b) 1% Sodium alginate and (c) 0.5% Sodium alginate at different temperatures.

for the samples calcined at different temperatures, the intensity of bands corresponding to carbon bonds reduces, which confirms that the calcination is effective in completing the thermal decomposition of the biopolymer compound. For all samples at 550 °C, two weak stretches could be detected around 1300–1600 cm^{-1} which are due to presence of C-C, C-O, COO^- and C-O-C groups. Increasing the calcination temperature causes all peaks to disappear, indicating that the samples getting more purity in their metal oxide composition. The absorption band for metal-oxygen (Ce-O and/or Gd-O) is observed at around 565 cm^{-1} [5]; however, due to the presence of noise below 650 cm^{-1} , the measuring range used in these experiments was 650 cm^{-1} to 4000 cm^{-1} .

The BET analysis was carried out by applying Barrett-Joyner-Halenda (BJH) method. Fig. 9 shows nitrogen adsorption-desorption isotherms (hysteresis loops) and pore size distribution of different

samples. The shapes of the adsorption isotherms (Fig. 9) match with type IV according to IUPAC classification which form due to the capillary condensation of the mesoporous materials, confirming the mesoporosity of the synthesized GDC nanocomposite powders [54]. The hysteresis loops match with type H3, suggesting the powders have “non-uniform”, “slit-shaped” channels with “non-rigid” aggregates of the particles [54]. As shown in Fig. 9, it can be observed that for each loading fraction of sodium alginate, the relative pressure (corresponding to the onset of the capillary condensation) has a higher value for the samples calcined at lower temperatures, that indicates the presence of the smaller pore size distribution for that sample [55].

The BJH surface areas of all samples were summarized in Table 3. It can be seen that for each loading fraction of sodium alginate, the largest surface area corresponds to the nanocomposite powders calcined at 550 °C, while it is reduced as the calcination temperature

Table 3

Crystal size of synthesized nanoparticles with Sherrer's formula, particle size and BET surface area^a.

Calcination temperature (°C)	0.50%			1%			2%		
	Crystal Size (nm)	Particle Size (nm)	BET Surface Area (m^2/g)	Crystal Size (nm)	Particle Size (nm)	BET Surface Area (m^2/g)	Crystal Size (nm)	Particle Size (nm)	BET Surface Area (m^2/g)
550	10	10.09	3.050	9	9.43	3.601	8	8.27	3.974
600	13	12.84	2.843	12	12.51	2.912	9	9.23	2.952
650	15	15.20	2.033	14	14.43	2.212	14	13.97	2.548
700	18	17.87	1.908	18	17.53	1.879	17	16.75	1.715

^a %error for crystallite size = ± 0.5 . %error for BET surface area = ± 0.5 .

Fig. 8. FT-IR spectra of: (a) 0.5% Sodium alginate, (b) 1% Sodium alginate and (c) 2% Sodium alginate for dried samples and calcined samples at different temperatures.

is gradually increased to 700 °C. As the particle size increases with reducing the surface area, the above trend also confirms the increase of the particle sizes with increasing the calcination temperature, which is again in good agreement with the particle sizes reported by FESEM and XRD results.

4. Conclusion

In this paper, an ionic gelation method using sodium alginate as the precursor was proposed for synthesizing a high purity GDC nanocomposite powder in an environmentally friendly method. The impact of synthesis variables such as calcination temperature and loading frac-

Fig. 9. Nitrogen adsorption/desorption isotherms of GDC oxide samples with different wt% of sodium alginate at different temperatures. The insets show the pore size distribution of the samples.

tion of sodium alginate in the cationic solution on the properties of the produced GDC nanocomposite powders were studied using various characterization techniques. All produced nanocomposites were found to be semi-spherical in shape. It was found that for a fixed alginate loading fraction, increasing the calcination temperature consistently

increases the particle size and the powder purity, while it can decrease the surface area. However, only at lower calcination temperatures (550–600°), the increase in the alginate loading fraction can noticeably influence particle size and surface area of the synthesized GDC nanocomposite powders.

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