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# New sodium dodecyl sulfate-assisted preparation of Nd<sub>2</sub>O<sub>3</sub> nanostrucrures via a simple route

#### Sobhan Mortazavi-Derazkola, Sahar Zinatloo-Ajabshir, Masoud Salavati-Niasari\*

The effect of the anionic shape- and size- controller on the morphology and size of Nd<sub>2</sub>O<sub>3</sub> prepared by new simple approach was studied. Nanostructured Nd<sub>2</sub>O<sub>3</sub> was synthesized by heat treatment in air at 900 °C for 5 h., employing powder, which was prepared by a solventfree solid-state reaction from [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> (L = bis-( 2-hydroxy-1-naphthaldehyde)-ethanediamine Schiff base compound), as new precursor, in presence of sodium dodecyl sulfate (SDS) as shape- and size- controller. The structural, morphological and optical properties of as-obtained nanostructured Nd<sub>2</sub>O<sub>3</sub> were characterized by thermo-gravimetric analysis (TGA), X-ray diffraction (XRD), energy dispersive X-ray microanalysis (EDX), UV–vis diffuse reflectance spectroscopy, field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), Fourier transform infrared (FT-IR) spectroscopy. The obtained results demonstrated that the amount of SDS play key role in the morphology and particle size controlling of the neodymium oxide. Furthermore, the catalytic properties of the as-prepared nanostructures were evaluated by the photocatalytic degradation of the erythrosine (anionic dye) as water ts contaminant.

#### Introduction

In recent years, the preparation and characterization of the nanostructured materials has drawn much attention owing to their potential applications in various fields as well as excellent and <sup>20</sup> unique characteristics<sup>1-6</sup>. Neodymium oxide (Nd<sub>2</sub>O<sub>3</sub>) is well known as one of the most interesting rare earth metal oxides because of its specific, excellent and unique optical and electrical properties as well as several applications in the thin films, luminescent and thermoluminescent materials, advanced <sup>25</sup> materials, protective coatings, photonic and catalysts<sup>7-12</sup>. So far, neodymium oxide has been prepared by the gel combustion<sup>13</sup>, microemulsion<sup>14</sup>, hydrothermal<sup>15</sup>, thermal decomposition<sup>16</sup>, solution-coprecipitation<sup>17</sup> and hydrogen plasma-metal reaction<sup>18</sup>. The development of a reliable and simple way for preparation of <sup>30</sup> nanostructured Nd<sub>2</sub>O<sub>3</sub> is of great importance to the potential

<sup>30</sup> hardstructured 14d<sub>2</sub>O<sub>3</sub> is of great importance to the potential investigation of its characteristics. It has been reported that particle size and shape have key impact on the characteristics of nanoscale materials<sup>19,20</sup>. So, exploring desirable and appropriate ways to produce nanostructured Nd<sub>2</sub>O<sub>3</sub> and controlling its <sup>35</sup> morphology and particle size seems necessary and noteworthy.

The thermal decomposition of the organometallic molecular way is generally accepted as cost effective, versatile, convenient, applicable, reliable and simple synthetic technique. This way does not need special apparatus, complicated approaches and 40 severe reaction conditions, which can restrict the large-scale production of the nanomaterials. Furthermore, this procedure provides effective and desirable method to the production of pure nanostructured materials with uniform morphology.

Herein, we present a novel simple way for the synthesis of pure <sup>45</sup> nanostructured Nd<sub>2</sub>O<sub>3</sub> via the thermal treatment of powder, which is prepared by a solvent-free solid-state reaction from [Nd  $L(NO_3)_2$ ]NO<sub>3</sub> (L = bis-( 2-hydroxy-1-naphthaldehyde)ethanediamine Schiff base compound), as new precursor, in presence of sodium dodecyl sulfate (SDS) as shape- and size-<sup>50</sup> controller. Furthermore, the influence of the amount of the SDS on the particle size and shape of Nd<sub>2</sub>O<sub>3</sub> via a facile thermal decomposition way is studied.

#### Experimental

#### 55 Materials

In this investigation, all the chemical reagents with analytical grade including sodium dodecyl sulfate (SDS), methanol, ethyl acetate, neodymium nitrate (Nd(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O), 2-Hydroxy-1-naphthaldehyde, ethylenediamine, and chloroform, were bought <sup>60</sup> from Merck Co. and were employed as received.

#### Preparation of bis-(2-hydroxy-1-naphthaldehyde)ethanediamine Schiff base compound (L)

In order to synthesize of Schiff base compound (L), 0.16 mol of 2-hydroxy-1-naphthaldehyde dissolved in methanol (80 ml) was <sup>65</sup> added drop-wise to an ethanediamine solution (0.08 mol) in 80 ml of methanol. The mixture was refluxed for 3 h. The yellow precipitate was separated by filtration, washed and air-dried. It was then recrystallized from methanol. The as-synthesized compound was characterized utilizing FT-IR and<sup>1</sup>H-NMR <sup>70</sup> techniques.

#### Preparation of the neodymium precursor

In order to synthesize of the neodymium precursor, 1 mmol neodymium nitrate was dissolved in 20 ml ethyl acetate and then was drop-wise to a solution of 1 mmol compound L in 20 ml 75 chloroform. The mixture was stirred for 2 h. Then, the yellow precipitate was filtered, washed with ethyl acetate and chloroform for three times, and after that air-dried for 24 h at room temperature. The resulting neodymium precursor was characterized utilizing FT-IR technique.

#### Preparation of Nd<sub>2</sub>O<sub>3</sub> micro/nanostructures

Neodymium precursor (NP) and SDS were mixed and ground for 15 min in an agate mortar at room temperature in a NP to SDS molar ratio of 1:0.5, 1:1, 1:2, 1:3, 1:4, 1:5 and 1:6. The so identification of the resulting powders was based on Fourier transform (FT-IR) spectrometer. Nd<sub>2</sub>O<sub>3</sub> micro/nanostructures were synthesized by subjecting 0.12 g of the as-obtained powders to heat treatment at (900 °C) in the air. An average temperature increase of 30 °C is recorded every minute, before the

temperature reached 900 °C, and after keeping the thermal treatment at 900 °C for 5 h, it was allowed to cool to room temperature. Schematic diagram of preparation of  $Nd_2O_3$  micro/nanostructures is depicted in Scheme 1. To investigate the <sup>5</sup> influence of calcination temperature, the thermal treatment process of the as-obtained powder (1:1 molar ratio of NP to SDS) was carried out in the 700, 800 and 900 °C. In order to examine the effect of SDS, a blank test was performed. In the blank test, 0.12 g of NP was subjected to heat treatment at (900 °C) in the

<sup>10</sup> air. To investigate the influence of L, one experiment was carried out without L (blank test1). The preparation conditions of all products were demonstrated in Table 1. The as-obtained nanostructured Nd<sub>2</sub>O<sub>3</sub> was characterized by TEM, XRD, EDS, UV–vis, FESEM, FT-IR analyses.



Scheme. 1 Schematic diagram of the preparation of neodymium oxide micro/nanostructures.

### Characterization

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- <sup>20</sup> The electronic spectra of the nanostructured Nd<sub>2</sub>O<sub>3</sub> were obtained on a Scinco UV–vis scanning spectrometer (Model S-4100). Powder X-ray diffraction (XRD) patterns of products were collected with a diffractometer of Philips Company with X'PertPro monochromatized Cu Ka radiation (l = 1.54 Å). The
- <sup>25</sup> energy dispersive spectrometry (EDS) analysis was performed by XL30, Philips microscope. Thermogravimetric-differential thermal analysis (TG-DTA) was carried out by a thermal gravimetric analysis instrument (Shimadzu TGA-50H) with a flow rate of 20.0 ml min<sup>-1</sup> and a heating rate of 10 °C min<sup>-1</sup>.
- <sup>30</sup> Fourier transform infrared spectra were recorded by utilizing a Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400–4000 cm<sup>-1</sup>. The microscopic morphology of Nd<sub>2</sub>O<sub>3</sub> was visualized by a Tescan mira3 field emission scanning electron microscope (FESEM). Transmission electron microscope (TEM)
- <sup>35</sup> images of nanostructured Nd<sub>2</sub>O<sub>3</sub> were taken by a JEM-2100 with an accelerating voltage of 200 kV equipped with a high resolution CCD Camera.

#### Photocatalytic test

The photocatalytic activity of as-prepared nanostructured <sup>40</sup> Nd<sub>2</sub>O<sub>3</sub> (sample no.3) was studied by monitoring the photodegradation of erythrosine as anionic dye in an aqueous

Sample No.	Neodymium source	Molar ratio of neodymium source/SDS	Calcination temperature (°C)	Figure of FESEM images
1	Nd(III)complex	1:1	700	-
2	Nd(III)complex	1:1	800	-
3	Nd(III)complex	1:1	900	6b
4	Nd(III)complex	1:0.5	900	6a
5	Nd(III)complex	1:2	900	6c
6	Nd(III)complex	1:3	900	7a
7	Nd(III)complex	1:4	900	7b
8	Nd(III)complex	1:5	900	7c
9	Nd(III)complex	1:6	900	8a and b
$10^{a}$	Nd(III)complex	-	900	10a
11 <sup>b</sup>	Nd(NO <sub>3</sub> ) <sub>3</sub> .6H <sub>2</sub> O	1:1	900	10b

<sup>a</sup> Blank test, in the absence of SDS

<sup>b</sup> Blank test 1

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solution. A quartz photocatalytic reactor was employed to carry out the photodegradation reaction. The photocatalytic degradation was performed by employing 0.003 gr of erythrosine solution including 0.05 g of as-obtained nanostructured Nd<sub>2</sub>O<sub>3</sub> at room <sup>50</sup> temperature. For reaching adsorption equilibrium, this mixture was aerated for 30 min. Later, the mixture was placed inside the photoreactor in which the vessel was 40 cm away from the UV source of 400 W mercury lamps. In order to hinder UV leakage, the light source and quartz vessel were placed inside a black box <sup>55</sup> that equipped with a fan. Aliquots of the mixture were taken at definite interval of times during the illumination, and after centrifugation they were analyzed by a UV–vis spectrometer. The erythrosine photocatalytic degradation percentage was calculated

as follow:

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$$D.P.(t) = \frac{C_0 - C_t}{C_0} \times 100$$
(1)

where  $C_t$  and  $C_0$  are the concentration of erythrosine at t and 0 min, respectively.

#### 65 Results and discussion

As described before, in this study the bis-(2-hydroxy-1naphthaldehyde)-ethanediamine Schiff base compound (L) was firstly synthesized, to be employed in the preparation of nanostructured Nd<sub>2</sub>O<sub>3</sub>. For ensuring an adequate purity of the <sup>70</sup> obtained (L), FT-IR and <sup>1</sup>H-NMR analyses were performed. The FT-IR spectrum of the Schiff base compound is illustrated in Fig. 1a. Two absorption bands located at 1644 and 1359 cm<sup>-1</sup> can be ascribed to the v(C=N) of the azomethines and v(Ar-O) of the phenolic hydroxyl substituent, respectively. Fig. 2a reveals the <sup>75</sup> <sup>1</sup>H-NMR spectrum of the prepared (L) compound. <sup>1</sup>H-NMR data of the as-synthesized Schiff base compound (DMSO,  $\delta$ , ppm): 2.3 (s, 4H, 2 × CH<sub>2</sub>), 6.7 (s, 1H, CH aromatic), 7.4 (s, 1H, CH aromatic), 7.6 (s, 1H, CH aromatic), 7.66 (s, 1H, CH aromatic), 7.69 (s, 1H, CH aromatic), 8.29 (s, 1H, CH aromatic), 9.06 (s, 1H, CH), and 14.04 (s, 1H, OH). As observed, the results of the FT-IR and <sup>1</sup>H-NMR analyses illustrated the high purity of the asprepared Schiff base compound.

<sup>5</sup> Fig. 1b-d exhibits the infrared spectra of as-synthesized NP, NP in presence of SDS and nanostructured Nd<sub>2</sub>O<sub>3</sub>, respectively. Two absorption bands seen at 1634 and 1353 cm<sup>-1</sup> can be ascribed to the  $\nu$ (C=N) of the azomethines and  $\nu$ (Ar-O) of the phenolic



Fig. 1 FT-IR spectra of (a) Schiff base compound, (b) neodymium precursor, (c) neodymium precursor in presence of SDS and (d) nanostructured Nd<sub>2</sub>O<sub>3</sub> obtained from 1:1 molar ratio of SDS to neodymium precursor by thermal treatment at 900 °C for 5 h.

hydroxyl substituent, respectively. The FT-IR spectrum of NP <sup>15</sup> illustrates the (L) compound characteristic peaks with several different shifts due to the complex formation. The FT-IR spectrum of the NP reveals (Fig. 1b) an increase in the C–O stretching frequency (10 cm<sup>-1</sup>) compared to the free (L) Schiff base compound. This demonstrates that the coordination to the

<sup>20</sup> neodymium ion happens via the oxygen atoms of the hydroxyl benzene of the as-synthesized (L) Schiff base compound. This was additional indicated by employing the absorption band seen at 470 cm<sup>-1</sup> (Fig. 1b) which may be related to v(Nd–O) vibration.



25 Fig. 2 <sup>1</sup>H-NMR spectra of the (a) Schiff base compound (L), (b) NP and (c) the UV-vis absorption spectra of the Schiff base compound (L) and NP.

In comparison with the free (L) compound, the v(C=N) band in <sup>30</sup> the NP shifted to 1634 cm<sup>-1</sup>. This seen blue shift (10 cm<sup>-1</sup>) indicates a stronger double bond characteristic of the iminic bonds as well as contribution of the azomethines nitrogen atoms in the coordination<sup>16</sup>. This result was additional confirmed by employing the absorption band seen at 500  $\text{cm}^{-1}$  (Fig. 1b) which  $_{35}$  might be related to v(Nd-N) vibration. The different coordination modes of the nitrate groups to the neodymium metal were detected by the differences in the two seen bands  $|v_4 - v_1|$ . Several peaks seen at 1479 cm<sup>-1</sup> ( $v_1$ ), 1032 cm<sup>-1</sup> ( $v_2$ ), 843 cm<sup>-1</sup> ( $v_3$ ) and 1311 cm<sup>-1</sup> ( $v_d$ ) in the FT-IR spectrum of the NP (Fig. 1b) can be 40 ascribed to the coordinated nitrate ions. The difference between  $v_4$  and  $v_1$  is nearly 168 cm<sup>-1</sup> illustrating that the coordinated NO<sub>3</sub> ions in the as-prepared NP may play a bidentate compound role<sup>18</sup> In the spectrum of the as-obtained NP, the  $(v_0)$  free nitrate seen at 1382 cm<sup>-1</sup>. In the spectrum of the as-obtained powder (NP in

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presence of SDS), the characteristic peaks of SDS seen at 2920, 2852, 1220 and 1076 cm<sup>-1</sup> (Fig. 1c). In the FT-IR spectrum of the as-synthesized nanostructured Nd<sub>2</sub>O<sub>3</sub>, the peaks observed at 3441 and 1645 cm<sup>-1</sup> ascribed to the v(OH) stretching and bending s vibrations, respectively, which illustrates the presence of physisorbed water molecules linked to as-synthesized nanostructured Nd<sub>2</sub>O<sub>3</sub><sup>21</sup>. The characteristic bands of the Nd<sub>2</sub>O<sub>3</sub> seen at 619 and 512 cm<sup>-1</sup> (Fig. 1d).<sup>22</sup> <sup>1</sup>H-NMR analysis results shown in Fig. 2a and b confirms the formation of the NP.

The UV-vis absorption spectra of the Schiff base compound (L) and NP were performed in DMF solvent (Fig. 2c). The values of the maximum absorption wavelength ( $\lambda_{max}$ ) and the absorbance are listed in Table 2. The absorption spectrum of the Schiff base compound (L) shows four absorption peaks located at  $\lambda_{max}$  361, 15 381, 400 and 426 nm. The two peaks locating at low energy side can be ascribed to  $n \rightarrow \pi^*$  transitions of conjugation between the lone pair of electrons of p orbital of N-atom in C=N group and a conjugated  $\pi$  bond of phenyl and naphthyl rings. The two peaks locating at higher energy arise from  $\pi \rightarrow \pi^*$  transitions within the <sup>20</sup> phenyl and naphthyl rings and the  $\pi \rightarrow \pi^*$  of the C=N group. UVvis spectrum of the NP is different from that of Schiff base compound (L) demonstrating the coordination of the Schiff base compound (L) to the Nd(III) ions. Upon the coordination, the two higher energy absorption peaks of the NP are shifted to longer 25 wavelength region (red shift) compared to those of the free Schiff base compound (L). The two lower energy peaks in the free Schiff base compound (L) are overlapped in a single peak and shifted to shorter wavelength (blue shift). This shift can be ascribed to the donation lone pair of electrons from the Schiff 30 base compound N-atoms to the Nd(III) ions  $(Nd \rightarrow Ln)^{23}$ . As demonstrated, FT-IR, <sup>1</sup>H-NMR and UV-vis analysis results confirm L and NP preparation.

 $\label{eq:composed} \begin{array}{c} \mbox{Table. 2} The UV-vis absorption peaks ($\lambda_{max}$) and absorbance (Abs.) of Schiff base $35$ compound (L) and NP in DMF solution. $$$ 

Compound.	$\lambda_{max} \ (nm)$	Abs.	Band assignments
	361	1.529	$\pi \rightarrow \pi^*$
т	400	1.933	$\pi \rightarrow \pi^*$
L	426	1.687	$\pi \rightarrow \pi^*$
	381	1.873	$n \rightarrow \pi^*$
	412	2.169	$\pi \rightarrow \pi^*$
NP	425	1.943	$n \rightarrow \pi^*$
	381	2.000	$\pi {\rightarrow} \pi^*$

...In order to study the thermal observation of the as-prepared NP, thermal gravimetric technique (TGA) was employed. Fig. 3 <sup>40</sup> reveals the TGA curve of the as-obtained NP. As illustrated in Fig. 3, the weight loss steps are two. The first weight losses takes place in the temperature range 200–380 °C, and displays 27.73 % weight loss, which corresponds to the loss of two and one nitrate species from the inner and the outer coordination sphere of the <sup>45</sup> as-synthesized NP, respectively. The second step takes place at the temperature range 380–850 °C (illustrating 42 % weight loss),

which may be attributed to the loss of the (L) compound from the inner coordination sphere of the as-prepared NP, and the





Fig. 3 TGA of the neodymium precursor.

XRD technique, which is generally accepted as applicable technique for determination of the crystal structure, was utilized 55 to investigate the composition and purity of the as-synthesized Nd<sub>2</sub>O<sub>3</sub> (Fig. 4 and Table 3). The XRD pattern of products obtained at 700, 800 and 900 °C for 5 h (sample nos. 1-3) are illustrated in Fig. 4a-c. XRD results demonstrate that the products prepared through calcination at 700 and 800 °C consist of the 60 cubic Nd<sub>2</sub>O<sub>3</sub> that the pure hexagonal Nd<sub>2</sub>O<sub>3</sub> has not been synthesized (Fig. 4a and b). By increasing the temperature, pure hexagonal Nd<sub>2</sub>O<sub>3</sub> has been obtained. All diffraction peaks observed in the Fig 4c can be readily indexed to the pure hexagonal phase of Nd<sub>2</sub>O<sub>3</sub> (JCPDS Card file no. 74-2139). 65 Utilizing Scherrer formula<sup>24</sup>, the crystallite size of the obtained Nd<sub>2</sub>O<sub>3</sub> from the XRD results was calculated to be about 32 nm. Therefore, pure hexagonal Nd<sub>2</sub>O<sub>3</sub> can be synthesized by calcining the prepared powder (1:1 molar ratio of NP to SDS) at 900 °C.

The chemical composition and purity of the as-synthesized  $_{70}$  nanostructured Nd<sub>2</sub>O<sub>3</sub> (sample no.3) were additional examined by EDS technique. The illustrated bands in Fig. 5 related to Nd and O. So, the prepared XRD and EDS results demonstrate that hexagonal nanostructured Nd<sub>2</sub>O<sub>3</sub> with high level of purity was successfully produced by the present way.

As mentioned before, in this resaerch, nanostructured Nd<sub>2</sub>O<sub>3</sub> was synthesized by employing [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> (L = bis-( 2-hydroxy-1-naphthaldehyde)-ethanediamine Schiff base compound), as NP in presence of SDS as capping agent via a new simple way. In order to examine the effect of the amount of SDS so on the size and morphology of the Nd<sub>2</sub>O<sub>3</sub>, FESEM images of obtained Nd<sub>2</sub>O<sub>3</sub> micro/nanostructures with SDS:NP molar ratio of 0.5:1, 1:1, 2:1, 3:1, 4:1, 5:1 and 6:1 (Table 1) were taken and illustrated in Fig. 6, 7 and 8, respectively. It can be seen that by employing molar ratio of 0.5:1 (SDS to NP), the nanoparticles swere not form well (Fig. 6a). Therefore, in order to reduce the amount of aggregate and contribute to the formation of nanostructured Nd<sub>2</sub>O<sub>3</sub> greater amount of SDS was added. Fig. 6b

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illustrated that uniform spherical nanoparticles are obtained in the presence of 1 mmol of SDS. When 2:1, 3:1 and 4:1 molar ratio of SDS:NP were employed, nanostructures composed of nanobundles and rod-like nanostructures as well as nanobundles <sup>5</sup> and nanorods were obtained (Fig. 6c and 7a and b). By increasing the mmol of SDS from 2 to 3, the amount of the rod-like nanostructures increased.



10 Fig. 4 XRD pattern of products obtained by thermal treatment of powder in 1:1 molar ratio of SDS to NP at (a) 700, (b) 800 and (c) 900 °C for 5 h.

Table. 3 Preparation conditions and corresponding products

Sample No.	Calcination temperature (°C)	Composition of the products (XRD result)
1	700	Hexagonal Nd <sub>2</sub> O <sub>3+</sub> Cubic Nd <sub>2</sub> O <sub>3</sub>
2	800	Hexagonal Nd <sub>2</sub> O <sub>3+</sub> Cubic Nd <sub>2</sub> O <sub>3</sub>
3	900	Hexagonal Nd <sub>2</sub> O <sub>3</sub>

- <sup>15</sup> When 5 and 6 mmol of SDS were employed, corrugated-like and scales-like nanostructures were obtained (Fig. 7c and 8a and b). It seems that when the amount of SDS from 0.5 to 1 mmol increases, the uniform and sphere-like Nd<sub>2</sub>O<sub>3</sub> nanoparticles forms and also the particle size decreases with decreasing NP to SDS
- <sup>20</sup> molar ratio (Scheme 2b). It seems that when the amount of the SDS increases, the aggregation between Nd<sub>2</sub>O<sub>3</sub> nanoparticles decreases and also nucleation to be taken place rather than the particle growth owing to the steric hindrance influence of SDS. By increasing the mmol of SDS from 2 to 6, the SDS as capping <sup>25</sup> agent can provide the different reaction interfaces for NP

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nanorods, corrugated-like and scales-like nanostructures were prepared, respectively. Therefore the molar ratio of SDS:NP has a 30 significant impact on the morphology and particle size control of the Nd<sub>2</sub>O<sub>3</sub>.

(Scheme 2a) and induce the nanoparticles to assemble in the

definite directions, and so rod-like nanostructures, nanobundles,



Fig. 5 EDS pattern of nanostructured  $Nd_2O_3$  obtained from 1:1 molar ratio 35 of SDS to NP by thermal treatment at 900 °C for 5 h.

In order to investigate the detailed particle size and morphology of the as-prepared nanostructured Nd<sub>2</sub>O<sub>3</sub>, TEM technique was utilized. The typical TEM images of the nanostructured Nd<sub>2</sub>O<sub>3</sub> in <sup>40</sup> a molar ratio 1:1 (SDS to NP) is illustrated in Fig. 9a-d. The TEM images of the Nd<sub>2</sub>O<sub>3</sub> demonstrate that nanoparticles with quasisphere-like morphology are sintered together. Furthermore, the images indicate that as-obtained Nd<sub>2</sub>O<sub>3</sub> nanoparticles have diameter in the range of 20-55 nm.

To examine the effect of the SDS on the morphology of the Nd<sub>2</sub>O<sub>3</sub>, sample no. 10 was synthesized as blank product withoututilizing any SDS. Fig. 10a reveals FESEM image of sample no. 10. It can be observed that in the absence of SDS, <sup>50</sup> high agglomerated particle-like and bulk structures were obtained. This result indicates that employing SDS with high steric hindrance effect led to synthesize nanostructured Nd<sub>2</sub>O<sub>3</sub>. SDS as capping agent limits the size of the Nd<sub>2</sub>O<sub>3</sub> nanoparticles and protects them from additional aggregation.

In order to investigate the effect of the neodymium source on the shape of the Nd<sub>2</sub>O<sub>3</sub>, sample no. 11 was prepared as blank 1 product with employing Nd(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O in the presence of the SDS. FESEM image of the sample no. 9 is illustrated in Fig. 10 b 60 and c. It is noteworthy that bulk structures were formed. The bis-(2-hydroxy-1-naphthaldehyde)-ethanediamine compound with high steric hindrance influence in [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub>, can act as a co-capping agent. Obviously, employing [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> as neodymium source in the presence of the SDS results in 65 nanostructured Nd<sub>2</sub>O<sub>3</sub> preparation (Fig. 6, 7 and 8). Therefore, a privilege of employing [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> in the presence of the SDS is that it cause to formed nanostructured Nd<sub>2</sub>O<sub>3</sub>.





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Fig. 6 FESEM images of the products prepared from (a) 0.5:1, (b) 1:1, and (c) 2:1 molar ratios of SDS to NP, by thermal treatment at 900  $\degree$ C for 5 h.

**Fig. 7** FESEM images of the samples obtained from (a) 3:1, (b) 4:1, and (c) 5:1 molar ratios of SDS to NP, by thermal treatment at 900  $^{\circ}$ C for 5 h.



Fig. 8 FESEM images of the product prepared from 6:1 molar ratio of SDS to NP, by thermal treatment at 900 °C for 5 h.

- <sup>5</sup> It has been reported that the band gap (*Eg*) has a main impact on the determining the nanostructured materials characteristics utilized in photocatalytic applications and is oftentimes calculated from the UV–vis diffuse reflectance data. The UV–vis diffuse reflectance spectrum of the nanostructured Nd<sub>2</sub>O<sub>3</sub> in a molar ratio <sup>10</sup> 1:1 (SDS to NP) demonstrated in Fig. 11a. In the UV–vis diffuse reflectance spectrum of the as-prepared nanostructured Nd<sub>2</sub>O<sub>3</sub> (Fig. 11a), the eight absorption bands (located at 434, 470, 532, 594, 612, 686, 756 and 800 nm) are ascribed to charge transition from <sup>4</sup>I<sub>9/2</sub> to <sup>2</sup>G<sub>9/2</sub>+<sup>2</sup>D<sub>3/2</sub>+<sup>4</sup>G<sub>11/2</sub>+<sup>2</sup>K<sub>15/2</sub>, <sup>4</sup>G<sub>9/2</sub>+<sup>4</sup>G<sub>7/2</sub>+<sup>2</sup>K<sub>13/2</sub>, <sup>15</sup> <sup>4</sup>G<sub>5/2</sub>+<sup>2</sup>G<sub>7/2</sub>, <sup>2</sup>H<sub>11/2</sub>, <sup>4</sup>F<sub>9/2</sub>, <sup>4</sup>F<sub>7/2</sub>+<sup>4</sup>S<sub>3/2</sub>, <sup>4</sup>F<sub>5/2</sub>+<sup>2</sup>H<sub>9/2</sub> and <sup>4</sup>F<sub>3/2</sub>, respectively<sup>22</sup>. The *Eg* can be determined based on the UV–vis
- respectively<sup>22</sup>. The Eg can be determined based on the UV-vis diffuse reflectance data employing Tauc's equation<sup>25,26</sup>. The energy gap of the nanostructured Nd<sub>2</sub>O<sub>3</sub> has been calculated by extrapolating the linear portion of the plot of  $(\alpha hv)^2$  against hv to the rare<sup>27,28</sup> (Fig. 11b). The Eg amount of the as sumtherized
- $_{20}$  the zero<sup>27,28</sup> (Fig. 11b). The *Eg* amount of the as-synthesized nanostructured Nd<sub>2</sub>O<sub>3</sub> calculated to be 5.12 eV, which illustrated

a blue shift compared with the reported band gap amount of Nd<sub>2</sub>O<sub>3</sub> in previous literatures<sup>29</sup> which this blue shift is attributable to decrease in the grain size which cause alteration in particle <sup>25</sup> energy levels and increase the band gap amount.



**Scheme. 2** Schematic diagram showing (a) the effect of the amount of 30 SDS on the reaction interfaces for NP and (b) the formation of neodymium oxide products at various conditions.



**Fig. 9** TEM images of nanostructured  $Nd_2O_3$  obtained from 1:1 molar ratio of SDS to NP, by thermal treatment at 900 °C for 5 h.

The influence of morphology on photocatalytic activity was studied by monitoring the photodegradation of the erythrosine as anionic dye in an aqueous solution over as-prepared sample nos. 3 (Nd<sub>2</sub>O<sub>3</sub> obtained from 1:1 molar ratio of SDS to NP) and 10 (constrained from 1:2 for a state of the large state of the larg

<sup>10</sup> (prepared in the absence of Schiff base compound) with different morphology under UV light illumination (Fig. 12, Scheme 3). No erythrosine was practically broken down after 120 min without using UV light illumination or Nd<sub>2</sub>O<sub>3</sub>. This observation indicated that the contribution of self-degradation was insignificant. The <sup>15</sup> possible mechanism of the photodegradation of the anionic dye

The possible mechanism of the photodegradation of the anioni may be summarized as follows:  $Nd_2O_3 + hv \rightarrow Nd_2O_3^* + e^- + h^+$ 

 $h^+ + H_2O \rightarrow OH$  $e^- + O_2 \rightarrow O_2$  <sup>20</sup> OH +  $O_2$  + anionic dye  $\rightarrow$  Photodegradation products

According to photocatalytic calculations by Eq. (1), the erythrosine degradation was about 78 and 56 % over sample nos.



Fig. 10 FESEM images of  $Nd_2O_3$  prepared in the absence of (a) SDS and (b and c) Schiff base compound.

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Page 10 of 11 View Article Online DOI: 10.1039/C5RA08394J



**Fig. 11** UV–vis diffuse reflectance spectrum (a) and plot to determine the band gap (b) of the nanostructured  $Nd_2O_3$  obtained from 1:1 molar ratio of SDS to NP, by thermal treatment at 900 °C for 5 h.

3 and 10, respectively, after 120 min illumination of UV light, and Nd<sub>2</sub>O<sub>3</sub> with uniform spherical morphology demonstrated high photocatalytic activity. It is well known that the heterogeneous <sup>10</sup> photocatalytic processes comprise the diffusion, adsorption and reaction steps, and the desirable distribution of the pore is helpful and advantageous to diffusion of reactants and products, which prefer the photocatalytic reaction. In this research, the enhanced photocatalytic activity of sample no. 3 with uniform spherical <sup>15</sup> morphology can be related to desirable distribution of the pore, high hydroxyl amount and high separation rate of charge

- In this study, we described a new way to prepare nanostructured  $^{20}$  Nd<sub>2</sub>O<sub>3</sub> with the aid of [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> (L = bis-( 2-hydroxy-1-naphthaldehyde)-ethanediamine Schiff base compound), as neodymium source, in presence of sodium dodecyl sulfate (SDS) as capping agent. The novelty of this research compared to other reports is that for the preparation of Nd<sub>2</sub>O<sub>3</sub>, [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> as
- <sup>25</sup> new neodymium source in presence of SDS were used. The bis-(2-hydroxy-1-naphthaldehyde)-ethanediamine compound with high steric hindrance influence in [Nd L(NO<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub>, can act as a



Fig. 12 Photocatalytic erythrosine degradation of sample nos. 3 and 10 30 under UV light.



**Scheme. 3** Reaction mechanism of erythrosine photodegradation over Nd<sub>2</sub>O<sub>3</sub> under UV light illumination.

Table. 4 Characterization	comparison o	of Nd <sub>2</sub> O <sub>3</sub> with	other works.
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Method	Precursors	Morphology, Size (nm)	Calcination temperature (°C)	Ref.
Thermal treatment	[NdL(NO <sub>3</sub> ) <sub>2</sub> ]NO 3 (L = bis-( 2- hydroxy-1- naphthaldehyde) -ethanediamine, SDS	Sphere-like, 20-55 nm	900	This work
Thermal treatment	Neodymium tartrate	Nearly spherical, 200-3000 nm	800	[31]
Thermal decomposition	Nd(NO <sub>3</sub> ) <sub>3</sub> .5.2HO , Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub> , PEG400	Irregular shapes, 300- 1000 nm	950	[13]
Microemulsion	N-butanol, n- octane, Nd(NO <sub>3</sub> ) <sub>3</sub> , Nd(NO <sub>3</sub> ) <sub>3</sub> , CTAB, (needed stirring for 24 h)	Spherical, 40–80nm	900	[12]
Combustion	Nd(NO <sub>3)3</sub> , oxalyl dihydrazide	Spherical, 20-100 nm.	900	[32]

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carriers<sup>30</sup>.

co-capping agent. In Table 4 some of the precursors utilized in the other works are compared with the present investigation. According to these results, by employing the  $[Nd L(NO_3)_2]NO_3$  in presence of SDS,  $Nd_2O_3$  was synthesized successfully with the <sup>5</sup> smallest size and narrowest size distributions.

#### Conclusions

This work describe a new facile way for the large-scale preparation of nanostructured Nd<sub>2</sub>O<sub>3</sub> with different morphologies <sup>10</sup> via the thermal treatment of the powder, which was obtained by a solvent-free solid-state reaction from  $[Nd L(NO_3)_2]NO_3$  (L = bis-( 2-hydroxy-1-naphthaldehyde)-ethanediamine Schiff base compound), as neodymium source, in presence of sodium dodecyl sulfate (SDS) as capping agent. Employing of [Nd  $15 L(NO_3)_2$ NO<sub>3</sub> as a neodymium source in presence of SDS is the novelty of this research. When as-synthesized nanostructured Nd<sub>2</sub>O<sub>3</sub> was employed as photocatalyst, the percentage of erythrosine photodegradation was about 78 after 120 min illumination of UV light. This result suggests that as-obtained 20 nanostructured Nd<sub>2</sub>O<sub>3</sub> as desirable material has high potential to be employed for photocatalytic applications under UV light. High purity of the as-synthesized Nd<sub>2</sub>O<sub>3</sub> was confirmed by EDS, XRD and FT-IR techniques. The optical characteristics of as-formed Nd<sub>2</sub>O<sub>3</sub> were also investigated.

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#### Notes and references

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