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Influence of N-Methylaniline on physicochemical and optical properties of $\gamma$-Al$_2$O$_3$ nanoparticles

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Abstract

Influence of N-Methylaniline (NMA) on the physicochemical and optical properties of $\gamma$-Al$_2$O$_3$ nanoparticles were investigated using XRD, FTIR, UV−vis DRS, TEM, AFM, PL, TGA and the results were reported here. The TEM results were showed that the surface morphology of NMA capped $\gamma$-Al$_2$O$_3$ nanoparticles has sphere like nanostructures. The size of the nanoparticles was controlled by the concentration of NMA and thus modified the geometry of $\gamma$-Al$_2$O$_3$ nanoparticles from primary (~8 nm) to spherical nanoparticles (~30−50 nm). XRD results were confirmed the enhancement of crystallite size of $\gamma$-Al$_2$O$_3$ proportionally with the NMA molar concentration. FTIR results were confirmed the formation of NMA capped $\gamma$-Al$_2$O$_3$ nanospheres by N-H stretching and O-H bending vibrations. The PL results were indicated that, the emission intensities at 589 nm were reduced after the capping of NMA for the defects $F_2^{2+}$ center and non-bridging oxygen hole center (NBOHC).

1. Introduction

Recently, nanostructured materials have garnered enough attention to enhance electrical and mechanical properties of functional materials for catalysis, gas sensing, energy storage and biomedical applications [1−5]. Among various metal oxides, aluminium oxide (Al$_2$O$_3$) is a group III metal oxide which has many applications in various fields such as transparent ceramics [6], thermal conductivity enhancers [7], catalysts support [8], electrical insulator in dielectric devices [9], cosmetic fillers [10], absorbents for water treatment [11], polishing and packaging materials [12] and in drug delivery system [13] due to its remarkable and unique material characteristics like high mechanical strength, high electrical insulation, high chemical and thermal resistance. Captivatingly, by various parameters including calcinations rate [14], particle size and contaminates [15], alumina existing different polymorph phases. The polymorph phases such as gamma (γ-Al$_2$O$_3$), delta (δ-Al$_2$O$_3$), theta (θ-Al$_2$O$_3$) and alpha (α-Al$_2$O$_3$) are have dissimilar energy gap values. It also has defect induced luminescence properties useful in the performance of optical sensors and optoelectronic devices [14]. Among them, γ-Al$_2$O$_3$ nanoparticles are of greatest attention since it has comparatively stable over the temperature range and high specific surface area. Significant enhancements in physicochemical properties of nanoparticles are observed for two important reasons; (i) by altering size with morphology and (ii) type of molecular structure [16, 17]. The modification in structure of metal oxide nanoparticles could be done with phosphonate, sulfate and amine groups [18]. The influences of sulfate capping agents such as sodium dodecyl sulfate (SDS) [19, 20] and sodium dodecyl benzene sulfonate SDBS [21] on the surface of Al$_2$O$_3$ nanoparticles have also been reported. The phosphonate and sulfate groups have bulky nature and their interaction with nanoparticles is only due to electron affinity. But in the case of amine which is strongly basic with high electron donating ability and high capping density [22]. N-Methylaniline (NMA) is a secondary amine which can be used as a good capping agent because the nitrogen atom has a lone pair of electrons and readily forms co-ordination bond with inorganic nanomaterials [23]. Also, recent studies suggest that the prerequisite of presence of hydrogen bonding site (amine) is important for the formation of nanostructure materials [24, 25]. The novelty of this work lies in the...
fact that NMA is used as a capping agent with pure γ-Al2O3 nanoparticles to form the spherical nanostructures and enhanced their physicochemical and optical properties. Archana et al used NMA as passivating agent for ZnSe nanoparticles [26] and for ZnO nanoparticles [27].

In this paper, we demonstrated the formation of anionic hydrogen bonding due to NMA can have a profound influence on the morphology of γ-Al2O3 by forming nanospheres, prepared using a simple wet chemical method. Investigations of NMA effects on crystallite size, morphology, surface roughness, optical, defects and thermal properties of γ-Al2O3 nanoparticles were carried out using powder XRD, FTIR, TEM, AFM, UV–vis DRS, PL and TGA spectra.

2. Experimental

γ-Aluminium oxide (γ-Al2O3, 99% Sigma Aldrich, Germany), N-Methylaniline (C6H5NHCH3, 98%, Sigma Aldrich, Germany), acetone and deionized (DI) water were used without additional purification. The NMA capped γ-Al2O3 was synthesized by using simple wet chemical method. In distinctive synthesis, 0.5 g γ-Al2O3 was dispersed in 50 ml of DI water with continuous stirring. 0.1 M of NMA was dissolved in 50 ml of DI water in a drop wise manner and later added to previous mixer while stirring constantly for 5 h. The precipitate was separated by centrifugation with 5000 Rpm for 10 mins, followed by washing with DI water and acetone for several times. Finally the powder was dried at 100 °C for 5 h. The similar procedure was carried out for 0.2 M concentrated NMA. The prepared samples were labeled as AlN0, AlN1 and AlN2 for pure γ-Al2O3 nanoparticles, 0.1 M NMA capped γ-Al2O3 and 0.2 M NMA capped γ-Al2O3 respectively.

X-ray diffraction (XRD) measurements were carried out by using Philips diffractometer of X’pert Company with monochromatized CuKα radiation (λ = 1.5418 Å). The functional groups present in pure and NMA capped γ-Al2O3 samples were identified by Fourier transform infrared (FTIR) spectroscopy (an ALPHA-T spectrometer) using KBr discs over the range of 4000-400 cm⁻¹. The optical properties of the samples were analyzed by using Ultraviolet-Visible diffuse reflectance spectroscopy (UV–vis DRS, Cary-500, Varian Co.). The photoluminescence (PL) spectra were obtained by spectrophotometer (RF-5301 PC) in the range of 200–800 nm by the excitation of Xenon lamp at 390 nm. The micrographs and selected area electron diffraction (SAED) patterns of the samples were recorded by using transmission electron microscopy (TEM, TECHNAI-20-G2). The surface morphological study for the samples was carried out by the atomic force microscopy (AFM) (Digital instruments nanoscope E with Si3N4 100 nm cantilever, 20 nm resolution and 0.58 N m⁻¹ force constant) in a contact mode at room temperature. The thermal stability was investigated by thermogravimetric analysis (TGA) using SDT Q600 V20.9 Build 20 and the samples were heated from room temperature to 800 °C under a nitrogen atmosphere with a heating rate of 10 °C per min.

3. Results and discussion

3.1. XRD analysis

XRD patterns of pure and NMA capped γ-Al2O3 nanospheres were shown in figure 1(a). The characteristic diffraction peaks observed at 2θ values 32.04, 36.97, 39.41, 45.89, 61.26, and 67.06, which correspond to the (220), (311), (222), (400), (511) and (440) planes of the γ-Al2O3. These results were very well matched with JCPDS Card No.29-0063, space group: Fd3m. The results indicated that the samples were belonged to cubic crystal system with lattice constant a = b = c = 7.92 Å. No additional peaks associated with impurity were observed. The crystallite size of γ-Al2O3 was calculated by using Scherrer’s formula,

\[ D = \frac{K \lambda}{\beta \cos \theta} \]

(1)

where \( K \) is the Scherrer constant (\( K = 0.9 \)), \( \lambda \) is wavelength of the x-ray radiation (\( \lambda = 1.5418 \) Å), \( \theta \) is the characteristic peak angle, and \( \beta \) is the FWHM of the (440) plane in radians. The crystallite sizes were calculated as 8.19 nm for pure γ-Al2O3 and 11.78 and 13.05 nm for AlN1 and AlN2 nanospheres respectively [28]. It could be seen that, the intensity of diffraction peaks increased with increasing crystallite size [29] may due to the influence of NMA on the surface of γ-Al2O3 nanoparticles. However, the broadening of XRD peaks mainly depends on the crystallite size (D) and strain (\( \varepsilon \)). Williamson-Hall (W-H) plot as 4sin\( \theta \) (x-axis) against \( \beta \cos \theta \) (y-axis) has been employed to calculate the strain for prepared samples and the plots were shown in figures 1(b)–(d).

The slope of the line gives the strain (\( \varepsilon \)) and the intercept (\( k \lambda / D \)) of the line on y-axis gives crystallite size (D) from the W-H plot based on the equation,

\[ \beta \cos \theta_{hkl} = \frac{0.9 \lambda}{D} + 4 \varepsilon \sin \theta_{hkl} \]

(2)

where \( \beta \), \( \theta_{hkl} \) and \( \lambda \) represent FWHM of XRD reflection, Bragg’s angle and wavelength of x-ray respectively. The FWHM of (440) plane, crystallite size and strain values evaluated by W-H method were shown in table 1.
the W–H plot, the negative value \((-9.3 \times 10^{-3}\)) indicates compressive strain (shrinkage of lattice) of AlN0, whereas the positive values indicated the samples underwent tensile strain (expansion of lattice)\[30\]. The above result confirms the influence of NMA in lattice expansion.

3.2. FTIR analysis

The FTIR spectra of AlN0, AlN1 and AlN2 were shown in figure 2. Two broad bands were detected in all spectra around 780 and 600 cm\(^{-1}\) which correspond to Al-O-Al bending mode and Al-O stretching mode, respectively\[14\]. The peaks observed at around 3420 cm\(^{-1}\) and 1380 cm\(^{-1}\) in NMA capped \(\gamma\)-Al\(_2\)O\(_3\) nanospheres attributed to N-H stretching and O-H bending vibrations, respectively\[31, 32\]. The absorption band at 773 cm\(^{-1}\) in (AlN0) spectrum was blue shifted to 779 cm\(^{-1}\) (AlN1) and 782 cm\(^{-1}\) (AlN2) and these shifts were caused due to increased crystallite size. Similarly, the band at 614 cm\(^{-1}\) (AlN0) spectrum was shifted to 602 cm\(^{-1}\) in (AlN1) and further it shifted to 591 cm\(^{-1}\) in (AlN2). This red shift arose from dangling bonds present on the surface of the nanoparticles\[28\]. From the above discussion, it could be cleared that, intensity variation (increases) and shifting of particular bands towards blue and red was due to outcome of the influence of capping agent on the surface of \(\gamma\)-Al\(_2\)O\(_3\) nanoparticles.
3.3. Formation mechanism

On the basis of FTIR results, the formation mechanism of NMA capped $\gamma$-$\text{Al}_2\text{O}_3$ nanoparticles was shown in figure 3. Initially, the $\gamma$-$\text{Al}_2\text{O}_3$ nanoparticles aggregate themselves via self-interaction, which increases its volume. Later, the nitrogen atom of NMA has a lone pair of electrons, which gets attached to the $\gamma$-$\text{Al}_2\text{O}_3$ nanoparticles due to the high electron affinity between the lone pair electrons of nitrogen atoms and $\text{Al}^{3+}$. Also, hydrogen bonding between the partially positively charged hydrogen atom of NH group in NMA and partially negatively charged oxygen atom of pure $\gamma$-$\text{Al}_2\text{O}_3$ nanoparticles. This was the reason for the formation of $\gamma$-$\text{Al}_2\text{O}_3$ nanospheres.

3.4. TEM and SAED analysis

In order to validate the influence of NMA on the formation of nanospheres, TEM measurements were carried out for the samples and corresponding images were depicted in figures 4(a)–(c). The images indicated the presence of NMA bonding via agglomeration of pure $\gamma$-$\text{Al}_2\text{O}_3$ nanoparticles, which was further evidenced by
calculating the particle sizes using Image J [https://imagej.nih.gov/ij/links.html], and found to be 8, 34 and 42 nm for AlN0, AlN1 and AlN2 respectively. The selected area electron diffraction (SAED) patterns of samples correspond to TEM images were shown in figures 4(d)–(f) [33]. The high nano nature of pure sample was clearly indicated by the ring pattern shown in figure 4(d). In figures 4(e) and (f), the superimposition of bright spots indicated the increasing crystalline nature of the samples.

These pattern spots were indexed as the (311), (400) and (440) planes for the cubic structure of $\gamma$-Al$_2$O$_3$ nanoparticles. Thus the SAED patterns were the best evidence for the x-ray diffraction patterns. The corresponding histogram images were shown in figures 4(g)–(i). Commonly the addition agent controls agglomeration by controlling interparticle interaction between nanoparticles by the charged head groups of associated molecules. In the initial stage, (AlN0) nanoparticles were agglomerated via self-interaction process. Further by capping of NMA onto the surface of nanoparticles, lead to aggregation of the particles more promptly and form spherical morphology. This can be attributed with the formation of hydrogen bonding between oxygen atoms of $\gamma$-Al$_2$O$_3$ and the hydrogen atoms of NMA. This type of bonding has a significant role in stabilization of the creation of nanospheres [31].

3.5. AFM analysis

To identify the influence of NMA agglomeration more accurately, AFM analysis was performed. The morphological alteration of $\gamma$-Al$_2$O$_3$ nanoparticles was validated by measuring the average roughness ($R_a$) and root mean square (RMS) roughness ($R_q$) values of images. Figure 5 shows 2D and 3D surface morphologies of pure and NMA capped $\gamma$-Al$_2$O$_3$ nanoparticles. As depicted in table 2, $R_a$ and $R_q$ values were gradually increased with increasing concentration of NMA, give evidence to the agglomeration of $\gamma$-Al$_2$O$_3$ nanoparticles. The agglomeration geometry is probably due to the interaction between NMA molecules and underlying particle surface through formation of hydrogen bonding [31]. From the above results, it can be predicted that the interactions have been occurred at the solid/liquid interface.
3.6. UV analysis

The measurement of optical parameters of the given material is quite essential to examine its usage optical applications. The optical parameters such as optical band gap, optical conductivity, real and imaginary dielectric constant were obtained from the diffuse reflectance bands of the samples depicted in figure 6. As shown in figure 6, AlN0 peak center was red shifted from 208 nm to 210 nm in AlN1 and to 212 nm in AlN2 due to the increase in concentration of NMA. The absorption ability of the samples was also become stronger, showing the influence of NMA. The calculated (using Kubelka Munk function) direct band gap energies of samples were 5.81, 5.31 and 5.09 eV for AlN0, AlN1 and AlN2 respectively (figure 6, inset) [34]. Here the red shift indicated the $\pi-\pi^*$ transition by the absorption of NMA on $\gamma$-Al$_2$O$_3$ surface. The decrease in band gap values may correspond to increase in particle size which leads to nanospheres formation.

Optical conductivity is a quantity of frequency response of the material when treated with light and the optical conductance was calculated by the equation,

$$\sigma = \alpha n c \varepsilon_0 = \alpha n c / 4\pi$$  \hspace{1cm} (3)

where $c$ is the velocity of the radiation in the space, $n$ is the refractive index and $\alpha$ is the absorption coefficient.

The plot between the optical conductivity and the photon energy was shown in figure 7. The optical conductance of $\gamma$-Al$_2$O$_3$ nanospheres was decreased with increasing photon energy up to 5 eV. For photon energy above 5 eV,

| Samples | R (nm) | Rq (nm) |
|---------|--------|---------|
| AlN0    | 3.96   | 5.39    |
| AlN1    | 4.96   | 6.08    |
| AlN2    | 14.33  | 17.06   |

Figure 5. AFM 2D (a)–(c) and 3D images (d)–(f) of AlN0, AlN1 and AlN2.

Table 2. Average roughness and root mean square roughness of the samples.
the optical conductivity value increases with the increase in photon energy. By comparing with $\gamma$-Al$_2$O$_3$ nanoparticles, the optical conductivity of NMA capped $\gamma$-Al$_2$O$_3$ nanospheres was increased. This was due to the increase in number of charge carriers (from valence band to conduction band) by increasing the NMA concentration $^{[35]}$.

The real ($\varepsilon_r$) and imaginary ($\varepsilon_i$) parts of dielectric constants have been estimated by the relation, $\varepsilon_r = n^2 - k^2$ and $\varepsilon_i = 2nk$, here $k$ is the excitation coefficient. The real part of dielectric constant gives the transfer rate of the speed of light in a material. The imaginary part of dielectric constant gives the absorption energy of light due to the medium. The dependence of $\varepsilon_r$ and $\varepsilon_i$ on photon energy of AlN0, AlN1 and AlN2 were shown in figures 8(a) and (b). The real and imaginary parts of dielectric constant show different patterns with the photon energy. The value of $\varepsilon_r$ is higher than $\varepsilon_i$ $^{[36]}$. The real part of the dielectric constant decreases with increase in energy up to 5 eV. On comparing with AlN0, the dielectric constant of AlN1 and AlN2 increase after 5 eV. This was due to the increase in NMA concentration which increases the number of free charge carriers (lone pair of electrons in nitrogen atom).

3.7. Photoluminescence emission analysis
Photoluminescence emission (PLE) spectra of prepared samples with an excitation at 390 nm were shown in figure 9. In general, $\gamma$-Al$_2$O$_3$ nanoparticles exhibit photoluminescence due to oxygen vacancies. Oxygen
vacancies exist in different forms as singly ionized, doubly ionized and in neutral. An oxygen vacancy occupied by single electron and double electron were referred as $F^{+}_2$ center and $F$ center, respectively [37]. Figure 9 shows the PL spectra has two bands at two different wavelengths $\lambda_{\text{max}} = 589$ nm and $\lambda_{\text{max}} = 713$ nm. The high intense emission band observed in 589 nm (orange-red emission) indicates the presence of $F^{+}_2$ centers (two connected oxygen vacancies engaged by three electrons) [38]. Also a weak emission observed at 713 nm attributes the presence of oxygen dangling bond, which was referred as non-bridging oxygen hole center (NBOHC) [39]. Further, the $\gamma$-Al$_2$O$_3$ capped nanospheres shows blue shift in wavelength from 707 nm in AlN1 and 706 nm in AlN2. This blue shift was due to the change in lattice strain [40] and the decrease in this peak intensity was due to the increase in crystallinity [41]. This result correlates with the XRD measurement.

The emission band at 589 nm was convoluted into multi peaks using Gaussian fitting which was shown in figures 10(a)–(c). Figure 10(a) shows the pure $\gamma$-Al$_2$O$_3$ with two distinctive peaks appearing at 588 and 591 nm, and three peaks appearing at 588, 591 and 592 nm correspond to AlN1 (figure 10(b)). The peaks observed at 587, 590 and 594 nm corresponds to AlN2 (figure 10(c)). The above result confirms the decreased defect in AlN1 and AlN2. Not only the $F^{+}_2$ centers but by analyzing the NBOHC also influence the luminescence, which was due to the lone pair of electron in nonbonding orbital of the dangling oxygen.

Figures 10(d)–(f) shows the Gaussian fitting peaks for NBOHC of AlN0, AlN1 and AlN2 nanospheres. The Gaussian peaks gets broadened in AlN1 and AlN2 due to the change in geometry of the nanoparticles [42]. So it is evident that the emission notably loses their intensity due to the less defect states in the geometry of NMA capped
γ-Al₂O₃ nanospheres. This result was also confirmed from XRD, i.e. as the electron/hole recombination rate get reduced due to enhanced crystallinity [43].

3.8. TGA analysis
The thermal behavior and decomposition process of AlN0, AlN1 and AlN2 nanospheres were depicted in figure 11. In figure 11(a) an exothermic peak was observed at 280 °C was due to the weight loss associated with the removal of water molecules adsorbed in the pores of the γ-Al₂O₃(OH), as well as an organic waste release. In AlN1 and AlN2 nanospheres two stages of weight losses was observed at 142 °C–220 °C and 230 °C–248 °C which was shown in figures 11(b) and (c). The weight loss occurred between 142 °C–220 °C was due to the decomposition of NMA, since the boiling point of NMA is about 195 °C [44].

Figure 10. PLE Gaussian peak fitting of AlN0, AlN1 and AlN2.
The second step of weight loss between 230 °C–248 °C was due to the instantaneous vaporization of NMA molecules from the surface of \( \gamma \)-Al\(_2\)O\(_3\). The derivative weight loss curve reveals the apparent weight loss at particular temperature. In AlN1 the weight loss at 218 °C was associated with the degradation of NMA molecules. Another weight loss at 239 °C was attributed to the complete decomposition of the NMA content leaving pure \( \gamma \)-Al\(_2\)O\(_3\) nanospheres. The same result was observed in AlN2. The TGA plots display the total weight loss percentage as 4.43, 1.44 and 1.36 for AlN0, AlN1 and AlN2 respectively. Thus the above result ensures that AlN1 and AlN2 have enhanced thermal stability than AlN0.

4. Conclusions

This work demonstrates the influence of NMA on the stabilization of pure \( \gamma \)-Al\(_2\)O\(_3\) nanoparticles to form nanospheres via agglomeration. Experimental techniques such as XRD, FTIR, UV–vis DRS and TGA were studied to confirm the changes in the physicochemical properties of \( \gamma \)-Al\(_2\)O\(_3\). In addition, the impact of NMA molecules on optical properties, such as optical conductance, real and imaginary dielectric constants of \( \gamma \)-Al\(_2\)O\(_3\) nanospheres were also predicted by using UV–vis DRS analysis. The PLE spectra of all the three samples showed a sharp band at 589 nm (yellow emission) assigned to \( F\text{\textsuperscript{2+}} \) defect center and the band at 713 nm assigned to NBOHC and validated using Gaussian fitting. By an increase in the NMA concentration of the prepared sample, average particle size and agglomeration tendency increased while the band gap energies decreased. Also, the addition of NMA molecules formulates capping via formation of hydrogen bonding, which may lead to the stabilization effect in the course on \( \gamma \)-Al\(_2\)O\(_3\) nanosphere formation. Furthermore the change in geometry due to interaction of NMA molecules on \( \gamma \)-Al\(_2\)O\(_3\) nanospheres has been evaluated with increased \( R_q \) values by using AFM analysis. This study might not only provide a new perception to understand the capping mechanism of NMA on \( \gamma \)-Al\(_2\)O\(_3\), also offers a simplistic and versatile way to promptly synthesize well controlled nanomaterials for ceramic and glass industry.

Figure 11. TGA and derivative thermogravimetric plots of (a) AlN0 (b) AlN1 and (c) AlN2.
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