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Solvent effect on structural and optical properties of Titania nanoparticles for pH sensing applications

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Abstract. Herein, solvents i.e., methanol, ethanol, propanol, and butanol-assisted titania nanoparticles (TNPs) are synthesized by low-temperature sol-gel method for pH sensing applications. The influence of solvent on the morphology, roughness, porosity, refractive index, and sensing properties are investigated. The relatively high average roughness 2.5 nm and lower refractive index 1.69 is observed for B-TNPs. The sensitivity of butanol-assisted phenol red encapsulated TNPs is observed 2.32 counts/pH.
Keywords: Sol-gel method: fibre optic pH sensing, solvents, structural analysis.

1. Introduction:

Accurate pH measurement is essential in different fields of life such as environmental monitoring, food processing, water analysis, and healthcare monitoring [1]. Mostly, conventional glass electrodes are used due to their sensitivity, stability, and selectivity. Though, a reference electrode is mandatory which limits their miniaturization and quantitative data collection. Therefore, fiber optic is more suitable over conventional pH meters because of miniaturization capability. In the fiber optic sensing mechanism, the optical variations in the fiber coating can be examined by the evanescent field which caused interaction between the guided light and the surroundings [2]. Titania as a host matrix offers unique properties such as smooth morphology with high surface areas, a porosity-dependent desorption behaviour, and the ability to host a multitude of different molecules [3]. The four different solvents i.e., methanol CH_3OH, ethanol C_2H_5OH, propanol C_3H_7OH, and butanol $C_4H_{10}OH$ are used as pores generators. A larger percentage of pores leads to a lower refractive index and a multi-level of pores promotes the transport of electrons which is advantageous for opto-chemical sensing. The titania nanoparticles are synthesized by different techniques such as sputtering, chemical vapor deposition, combustion synthesis, hydrothermal processing, and sol-gel. Among these, the sol-gel route allows homogeneity of nanomaterials, porosity, tuneable grain size, and smooth morphology at low temperatures [4]. Furthermore, for sensing analysis, indicator dye phenol red (PR) is non-toxic, non-carcinogenic, biodegradable material and has a dynamic pH range [5]. Herein, titania nanoparticles (TNPs) are synthesized at a low temperature 100 °C by the sol-gel method. Then these coated TNPs are exposed to different solvents to observe their influence on structural and optical properties. For sensing, phenol red is encapsulated within butanol assisted TNPs. It is worth mentioning here that the synthesis of TNPs at low temperatures is rarely reported.

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2. Experimental

For TNPs sol synthesis, titanium isopropoxide (15 mL) was dissolved in diluted propanol (50 mL of DI water and 50 mL of propanol) followed by 2mL of nitric acid. The mixture was stirred and heated at 100 °C for 1h. The sol was named as TNPs sol. The TNPs sol was coated on the middle region 5 cm of Plastic Clad Silica (PCS) optical fiber 35 cm long. The four fibers were coated at room temperature and kept for 24h to get an adhesive coating. Afterward, the coated fiber was exposed to solvents i.e., methanol, ethanol, propanol, and butanol for 2 min. For sensing analysis, the butanol exposed fiber was coated with phenol red and aged at room temperature for 24 h.

3. Characterization:

The surface texture and average surface roughness of synthesized TNPs were examined through atomic force microscopy (AFM) model SPI 3800 N. The refractive index was measured by J. A. Woollam (Lincoln, NE, USA) variable angle spectrometer M-2000. For sensing evaluation, Ocean Optics USB2000 optical spectrometer was used while white LED is employed as a light source.

4. Results and Discussion:

The surface morphology of M-TNPs, E-TNPs, P-TNPs, and B-TNPs are compared through AFM images, as shown in Fig. 4(a-d), respectively. The 3-D micrographs of all solvent-assisted TNPs exhibited defects-free surfaces. The Ra is observed around 1.4 nm, 1.6 nm, 1.9 nm, and 2.5 nm for M-TNPs, E-TNPs, P-TNPs, and B-TNPs, respectively. The M-TNPs, E-TNPs, P-TNPs, and B-TNPs possessed dispersed nanoparticles with well-defined pores distribution which formed by the evaporation of solvent molecules, also observed by the other researchers [6].

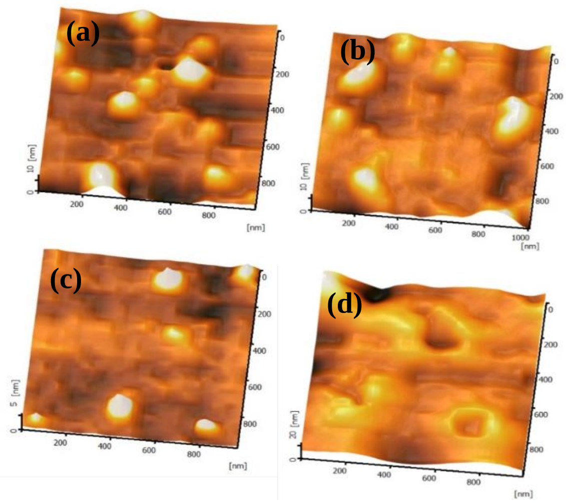

Fig. 1 3-D images of (a) M-TNPs (b) E-TNPs (c) P-TNPs (d) B-TNPs

Fig. 2(a-d) shows the refractive indices dispersion profiles of M-TNPs, E-TNPs, P-TNPs, and B-TNPs. Cauchy's law was applied to estimate the refractive index by increasing the wavelength in the range of 400 nm – 700 nm (visible region). At a constant wavelength (632.8 nm), the refractive index of the M-TNPs 1.72 was decreased to 1.71 for E-TNPs and then increased upto 1.78 after propanol exposure. However, by increasing the carbon chain (butanol) the refractive index was again decreased down to 1.69. The reduction in refractive index was assigned to the presence of the pores which appeared after organic solvent evaporation, as reported earlier [7].

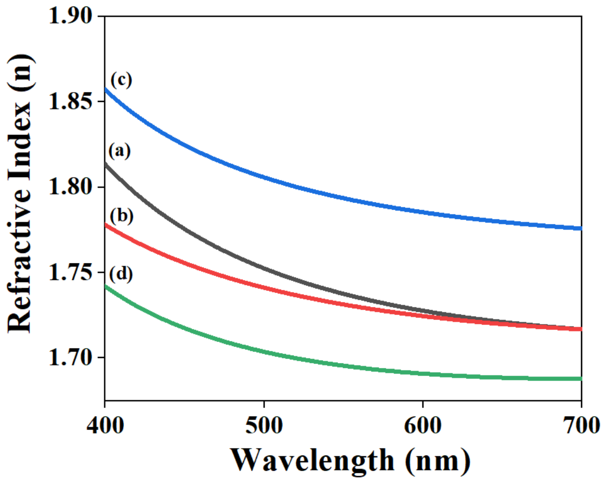

Fig. 2 Refractive index profiles of (a) M-TNPs (b) E-TNPs (c) P-TNPs (d) B-TNPs

Fig. 3(a) shows the optical spectra of PR encapsulated B-TNPs coated optic fiber within different pH solutions from 2-12. The spectra show that intensity in terms of transmittance was increased by increasing the pH [Fig. 3(a)-inset(I)]. The fiber response in pH 12 suggested that there was no leaching/delamination from coating which caused device instability. The sensitivity was measured as 2.32 counts/pH at 454 nm with determination correlation R^2 ~ 88 %. Fig. 3(b) demonstrates the PR encapsulated B-TNPs coated fiber linear response against different pHs 2-12. The variations of nonlinearity and linearity were correlated with the deprotonation and protonation of PR dye species. The dynamic pH range was optimized as 12.

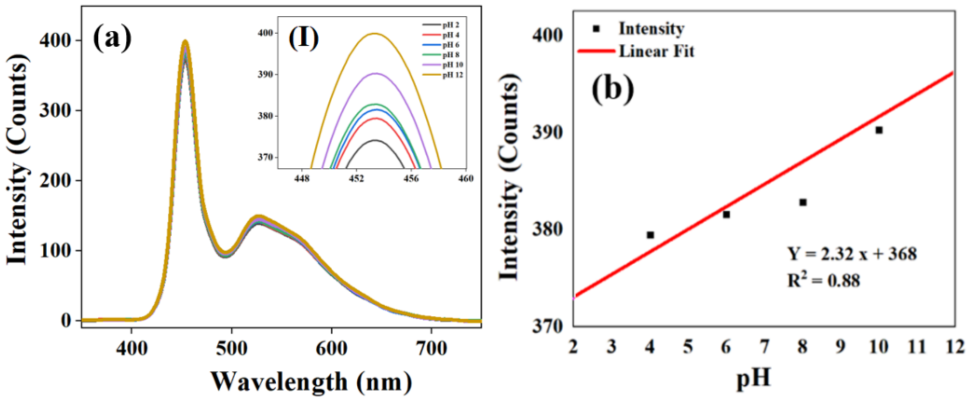

Fig. 3 Optical spectra of butanol-assisted PR encapsulated TNPs coated fiber optic within different pH solutions, inset (I) corresponds to zoomed view of marked area (b) calibration curve at 454 nm

5. Conclusion:

The titania nanoparticles (TNPs) were synthesized at a low temperature 100 ºC by sol-gel method and coated with fiber optic. Afterward, coated fibers were exposed to different solvents i.e., methanol, ethanol, propanol, and butanol. The AFM analysis indicated that the particles are localized and homogeneous without any cracks with distinct pores. The refractive index was observed 1.72, 1.71, 1.69, and 1.78 for M-TNPs, E-TNPs, P-TNPs, and B-TNPs. The sensitivity of B-TNPs assisted PR encapsulated TNPs was 2.32 counts/pH with a determination constant 88 %. Therefore, the P-TNPs have a great potential for opto-chemical pH sensing.

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