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Sol-gel Synthesis of Titania Nanoparticles for Photonic and Transformer Applications

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Titania nanoparticles have several industrial applications, including cosmetics, optical, photonic, and electrical devices. However, industrial production of these particles is difficult, complicated, and dependent on a variety of physical characteristics such as temperature and infrastructure availability. This research describes an instant industrial method for producing titania nanoparticles using a wet chemical sol-gel synthesis. X-ray diffractogram (XRD) and Fourier transform infrared spectroscopy (FTIR) analysis of as-synthesized titania nanoparticles revealed a strong diffraction peak at Bragg angle 25°, which can be attributed to the titania anatase phase, and vibration bonds at 463 cm^{-1}, which confirms the presence of titania. The morphology of these titania nanoparticles was examined using a field emission scanning electron microscope (FESEM), which determined the particle size to be around 37 nm. Using diffuse reflectance spectroscopy (DRS), the optical properties of the as-synthesized nanoparticles were studied, and their band gap was determined to be 3.37 eV. At room temperature, the dielectric constant and loss of titania nanoparticles were measured as a function of frequency. Additionally, titania particles were mixed into transformer oil to assess its dielectric breakdown strength for better insulating properties.

Keywords: Sol-gel synthesis, Titania nanoparticles, XRD, FTIR, FESEM, UV-VIS, Transformer applications.

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1. INTRODUCTION

Transformers are generally classed into two types: dry and oil-filled transformers. Oil-filled transformers are of particular importance because to their high temperature with stand and insulating properties. They also control a sizable portion of the electrical equipment industry by playing an important part in electrical substations, accounting for approximately 60 % of overall substation costs. Failure of such systems might result in massively damaging the whole power supply. The transformer oil, which serves several purposes as a coolant and an insulator, is critical in protecting the transformer for optimal usage. For more than a century, transformer oil has been used to insulate transformer electrical components and to transport heat generated in transformer windings [1]. Poor transformer oil monitoring promotes insulation breakdown owing to environmental, electrical, and thermal stresses [2]. According to the current transformer failure study, the average transformer life is 18 years, which is nearly half of the expected life of 36 to 44 years, and one-third of high voltage transformers failed due to insulation failure. Present, mineral oil-based transformers are exhibiting poor cooling performance due to low fire point temperature and increases the risk of fire at elevated temperatures [3]. Hence, it is important to develop a transformer oil that can withstand at elevated temperature and reduces the risk of fire during functioning. The rapid growth of technology in the present generation suggests that nanotechnology, with its constituent nanoparticles having at least one dimension less than 100 nm, is the best choice for optimal transformer performance without power supply interruption [4]. The capability of nanomaterials that improves the thermo-physical property was evidenced by Choi et al in 1995 [5]. Their experiment on the copper nanoparticles when dispersed in water endorsed enhancement in the conductivity by 350 %. The inclusion of nanoparticles to transformer oil predicts reduced insulation breakdown challenges and improved suspension stability. Conducting nanoparticles such as Fe_3O_4, Fe_2O_3, ZnO, and SiC, semiconducting nanoparticles such as TiO_2, CuO, and Cu_2O, and insulating nanoparticles such as Al_2O_3, SiO_2, and BN are examples of such nanoparticles that can be used as nanofillers to increase the dielectric property of transformer liquid.

Titania nanoparticles can be used in cosmetics as sunscreen lotions, but they can also improve the efficiency of numerous photonic [6], optoelectronic [7] and electrical devices [8]. The wide band gap semiconductor material has exceptional optoelectronic and electrical capabilities, allowing it to be used in solar cells and transformer oil. Its high dielectric constant, breakdown strength, and chemical stability led to its selection for a variety of electrical and electronic applications. They are especially utilised for electrical insulation and cooling in high-powered equipment. Titania is a metal oxide that may be found in three distinct phases: anatase, rutile, and brookite. Titania in anatase phase is more stable at lower temperatures than rutile phase, which is stable at higher temperatures. Pillai et al. synthesized magnetite and titania nanoparticles and dispersed in transformer oil for

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their insulating properties [9]. The red shift in emission maxima supported the ageing and nanofilling of the transformer oil, resulting in increased viscosity. The inclusion of these nanoparticles increased the breakdown voltage by 22 %. Elsad et al. synthesized titania nanoparticles with different sizes using simple sol-gel route and sintered them at different temperatures [10]. When compared to the other two particles sintered at 300 and 500 °C, the particles sintered at 400 °C displayed good dispersion in the host oil and resulted in variance in dielectric strength. Wang et al. compared three different nanoparticles (TiO_2, Al_2O_3, and Fe_3O_4) which were prepared using sol-gel synthesis approach [11]. The breakdown strength of the three particles when disseminated in the host liquid improved with increasing concentration up to a critical value and then began to decrease. The work also reported higher dielectric strength for negative impulse breakdown voltages. Vaishnav et al. synthesized Zirconia nano particles using microwave-assisted sol-gel approach to apply them in transformers as coolant and insulating materials [12]. The electrical and thermal properties of the as-synthesised nanoparticles showed that they may be used as an alternative to traditional ester-based transformer oils. The 0.2 g/L concentration provided the best performance of the zirconia particle-based transformer oil. Pourpasha et al. developed titania doped multiwalled carbon nanotubes (MWCNTs) nanoparticles using sol-gel approach and modified them with oleic acid [13]. The heat transfer coefficient of natural and forced was increased by 23.08 % and 24.68 %, respectively, when the synthesised nanoparticles were dispersed in conventional oil at a concentration of 0.2 wt % and an input power of 50.5 W. Meng et al. produced titania nanoparticles with a diameter of 10 nm and dispersed them in host oil using the hydrolysis process to create a nanofluid [14]. The breakdown study on the nanofluid supported lower acid value and increased dielectric breakdown strength by adsorbing water molecules and ageing of by-products. The study also evidenced the enhancement of surface flashover voltage of oil paper insulation at different ageing rates.

The sol-gel method for producing titania nanoparticles is relatively simple and does not require any skilled personnel. The method is cost-effective for producing nanoparticles with good uniformity and composition control. It is also temperature independent and regulates the composition of nanoparticles. Titania nanoparticles with the required form and morphology can be produced using this process. This research explores sol-gel synthesis and rapid production of titania nanoparticles from the hydrolysis of the organometallic precursor titanium isopropoxide, which may be exploited for optical and transformer applications. The particles' morphological, optical, and dielectric characteristics were well studied using XRD, FTIR, FESEM, DRS and LCR meter. Section-2 of this paper deals with the synthesis of titania nanoparticles using sol-gel and their results were discussed in section-3 of the draft. Finally, the conclusion of the entire work was presented in section-4 of the draft.

2. SYNTHESIS OF TITANIA NANOPARTICLES

The present research is to synthesize nanoparticles using sol-gel approach with the analytical chemicals. Titanium isopropoxide (TTIP, Sigma Aldrich) was used as precursor, along with the solvent ethanol (SRL Chemicals limited), catalyst acetic acid (SRL Chemicals limited), and deionized water in the molar ratios 1.5:10:0.5:1.5 in the synthesis process.

All the chemicals used in the synthesis process require no further purification and can use directly in the synthesis. The synthesis of titania nanoparticles using sol-gel synthesis was shown in Fig. 1.

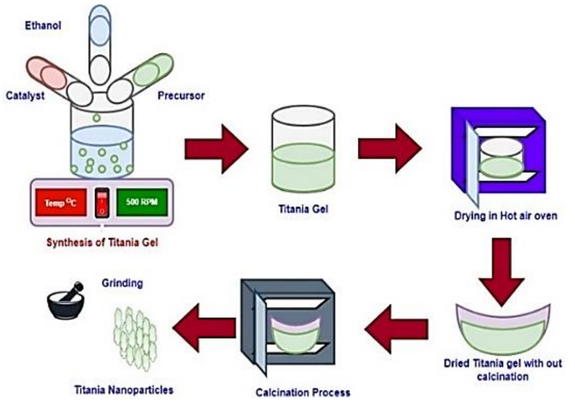

Fig. 1 – Synthesis of titania nanoparticles

It starts with the addition of 10 mL of solvent ethanol to a properly washed beaker. While stirring, 0.5 mL of acetic acid is added to the solvent, followed by 1.5 mL of deionized water. Consequently, the procedure is finished by adding 1.5 ml of TTIP to the resultant and stirring vigorously for 5 minutes. After 5 minutes, a clear and translucent gel was detected, and it was placed in a hot air oven at 100 degrees Celsius for one hour to rapidly dry and solidify. Finally, the produced titania nanoparticles were placed in a silica crucible and calcined at 500 degrees Celsius to eliminate the excess solvent residues and achieve crystallinity. After reaching room temperature in the muffle furnace, the particles were extensively ground with a mortar and pestle setup before being collected and transmitted for analysis.

3. RESULTS AND DISCUSSION

The sol-gel synthesized titania nanoparticles were characterized with X-ray diffractogram (Bruker, D8 Advanced, Germany) to identify the phase for further studies and presented in Fig. 2.

As illustrated in Figure 3, all of the diffraction peaks seen in XRD spectra are consistent with the anatase phase of titania and correspond to JCPDS card No. 21-1272. The absence of rutile phase in the as-synthesized titania nanoparticles was also shown by the spectra. Peaks (101), (103), and (004) (112). (200), (105), (211), and (213) are assigned to the polycrystalline anatase phase in the diffractogram with estimated lattice values *a* = *b* = 3.7830.003 and *c* = 9.4970.003 Å, which is a very little difference from the conventional JCPDS values *a* = *b* = 3.777 and *c* = 9.501 Å. The average crystallite size of the titania nanoparticles was determined to be about 12 nm using the Debye-Scherrer equation [15].

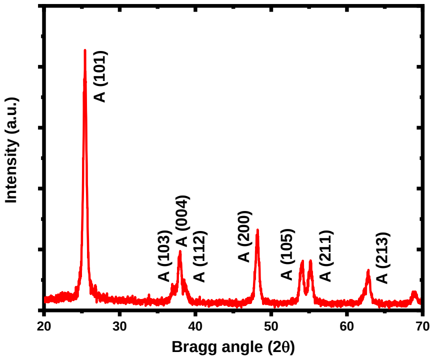

Fig. 2 – XRD spectra of titania nanoparticles

The functional groups of the as-synthesized titania nanoparticles were estimated using Fourier transform infrared spectroscopy (FTIR; Perkin Elmer, Spectrum 1), as shown in Fig. 3. The presence of a hydroxyl (O–H) group, produced by intermolecular interaction, is responsible for the wide band at 3288 cm^{-1}. The peak observed at 1621 cm^{-1} in titania nanoparticles is attributable to the –OH group's distinctive bending vibration and the oxygen-containing functional bond C–O stretching is observed at 1071 cm^{-1}. Furthermore, the broad peak demonstrated at 463 cm^{-1} is attributed to the titania (Ti–O–Ti) bending vibration bonds of the titania lattice.

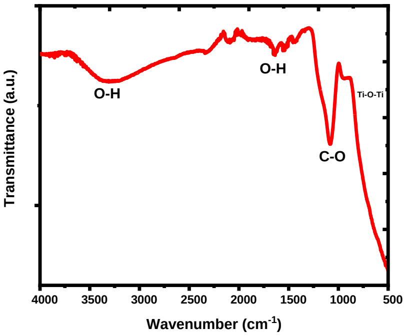

Fig. 3 – FTIR spectra of titania nanoparticles

The optical property of the sol-gel synthesized titania nanoparticles was studied using UV-VIS-NIR spectrophotometer (UV-VIS-NIR, Perkin Elmer, Lambda 750) and presented in Fig. 4 (a) with the morphological analysis shown in Fig. 4(b) investigated using field emission scanning electron microscope (FESEM).

Titania nanoparticles demonstrated strong absorption in the ultraviolet and high reflection in the visible and near-infrared spectral ranges, as illustrated in figure 4 (a). The particle band gap was determined using the wavelength value obtained from the diffused reflectance spectra using the formula [16].

$$E_g = \frac{hc}{\lambda}\, eV; E_g = \frac{1240}{\lambda}\, eV$$

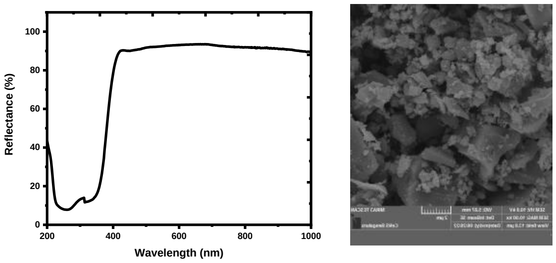

Fig. 4 – (a) Diffuse reflectance spectra, and (b) FESEM image of titania nanoparticles

The total energy gap (*E*$_g$) can be estimated using the above formula. Whereas '*E*$_g$' is the energy band gap (eV), '*h*' is the Planck's constant (6.626 × 10^{-34} m^2 kg/s), '*λ*' is the wavelength (nm) and '*c*' represents the velocity of the light (3 × 10^8 m/s). The absorption edge of the as-synthesized titania nanoparticles was measured to be 367.58 nm, and the band gap was calculated to be 3.37 eV, which can be endorsed to the transfer of charges from valence band to conduction band. It was also in good accord with the published literature 3.2 eV of bulk titania particles [17]. The minor shift in the band gap, which is the difference between the valence and conduction bands, is attributed to the size of the titania nanoparticles synthesized [18]. The titania nanoparticles morphology was studied using FESEM (MIRA3 TESCAN) and presented in Fig. 4(b). The image shows titania particles in irregular sphere shape and agglomerations, with agglomerated particles measuring in the micrometre range, whereas the majority of the particles have a spherical morphology and an average particle size of about 37 nm.

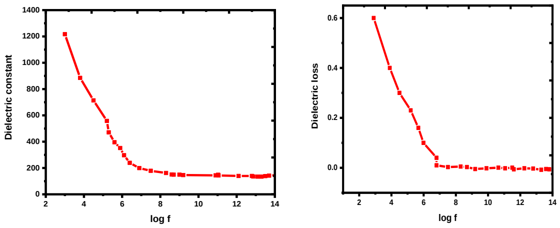

Fig. 5 – (a) Dielectric constant and (b) dielectric loss of titania nanoparticles

The dielectric constant is described as the material's efficiency in accumulating electrical energy, and its loss is expressed as energy dissipation in the form of heat exchange inside the dielectric material, which can occur when internal friction spreads across the dipoles.

The dielectric properties of the sol-gel synthesized titania nanoparticles that were calcined at 500 °C were measured by LCR meter (Wayne Kerr 6500 P) and demonstrated in Fig. 5(a) and (b). Fig. 5(a) shows that the dielectric constant values are increased in the low frequency zone and decrease very fast with increasing frequency until they become constant. Fig. 5(b) demonstrates the dielectric loss behaviour of titania nanoparticles, which exhibits decreasing values due to particle size reduction and an increase in dielectric loss when the applied electric field frequency equals the hopping frequency [19].

The breakdown test was conducted by following the ASTM D1816 standard with different titania concentrations from 0.1 g/L to 0.3 g/L by maintaining electrode gap of 2.7 mm and the voltage ramp rate was 500 V/s. All the measurements performed in the test were carried out under room temperature. The above-mentioned molar concentrations of titania nanoparticles were dispersed into the host oil to make nanofluid and then were tested for the breakdown strengths. The initial breakdown voltage of the transformer was roughly 12 kV without the inclusion of titania nanoparticles, and the addition of titania nanoparticles into the host oil indicated enhanced dielectric strength, as shown in the table below.

Table 1 – Effect of titania nanoparticle concentration on transformer breakdown voltage

S. No	Titania nanoparticles concentration (g/L)	Breakdown voltage evidenced (kV)	Breakdown voltage reported elsewhere (kV)
[1]	0	12	12
[2]	0.1	15	14
[3]	0.2	21	19
[4]	0.3	29	26

It is clear from Table 1 that titania nanoparticles are potential materials for distributing in the host oil to increase its dielectric strength. The capacity of titania nanoparticles to trap electrons is attributed to the increase in dielectric strength values. Because breakdown strength is created by fast-moving electrons, the titania nanoparticles produced can slow down the trapping process and so improve breakdown strength. Titania nanoparticles with lower molar concentration have greater particle spacing, which causes electron acceleration and ionization of oil molecules before they are captured by titania nanoparticles, increasing the breakdown strength. Thus, the host oil with higher molar concentration resulted in increased breakdown strength values than the reported values [20] and suggested the as-synthesized titania nanoparticles as fillers in the host oils to improve the insulating and dielectric properties.

4. CONCLUSION

The sol-gel method is used to rapidly produce titania nanoparticles. The XRD analysis of the produced titania nanoparticles revealed anatase phase with the strongest peak at Bragg angle 25° and approved the spectra to JCPDS card No. 21-1272. The presence of titania nanoparticles was established by FTIR analysis, which revealed a wide peak at wave number 463 cm^{-1}. The optical characteristics of the particle were studied using diffused reflectance spectroscopy, which revealed a band gap of 3.37 eV. FESEM morphological analyses confirmed that the particles were agglomerated and estimated the particle size to be around 37 nm. Titania nanoparticles' dielectric characteristics supported enhanced dielectric constant values at lower frequencies, and respective dielectric losses were also lowered due to particle size reduction. Furthermore, the use of as-synthesized titania nanoparticles revealed that when the molar concentration of titania nanoparticles in the host oil rose, the dielectric breakdown strength could be enhanced, and the nanoparticles may be used as fillers in potential transformers.

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Золь-гель синтез наночастинок оксиду титану для фотонних і трансформаторних застосувань

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Наночастинки титану мають кілька промислових застосувань, включаючи косметику, оптичні, фотонні та електричні пристрої. Однак промислове виробництво цих частинок є важким і складним процесом і залежить від різноманітних фізичних характеристик, таких, наприклад, як температура. Це дослідження описує миттєвий промисловий метод виробництва наночастинок оксиду титану за допомогою вологого хімічного золь-гель синтезу. Аналіз синтезованих наночастинок оксиду титану за допомогою рентгенівської дифрактонограми (XRD) та інфрачервоної спектроскопії з перетворенням Фур'є (FTIR) виявив сильний дифракційний пік під кутом Брегга 25°, який можна віднести до фази анатазу оксиду титану, і вібраційні зв'язки при 463 см$^{-1}$, що підтверджує наявність оксиду титану. Морфологію цих наночастинок діоксиду титану досліджували за допомогою скануючого електронного мікроскопа з емісією поля (FESEM), який визначив розмір частинок приблизно 37 нм. За допомогою спектроскопії дифузного відбиття (DRS) було вивчено оптичні властивості синтезованих наночастинок і визначено, що їх ширина забороненої зони становить 3,37 еВ. За кімнатної температури вимірювали діелектричну проникність і втрати наночастинок оксиду титану як функцію частоти. Крім того, частинки оксиду титану були змішані з трансформаторним маслом для оцінки його діелектричної міцності на пробій для кращих ізоляційних властивостей.

Ключові слова: Золь-гель синтез, Наночастинки оксиду титану, XRD, FTIR, FESEM, UV-VIS, Трансформатор.