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SYNTHESIS AND OPTICAL PROPERTIES OF TITANIUM DIOXIDE NANOPARTICLES USING SOL-GEL METHOD

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Abstract

The main objective of this research work was to synthesize and to characterize titanium dioxide (TiO_2) nanoparticles using sol – gel method with different weight ratio of NaOH. The optical properties and band gap energies of obtained nanoparticles were investigated with UV-Vis spectroscopy. The crystal structural and morphological properties of fabricated TiO_2 nanoparticles were characterized with XRD and SEM techniques. From the results of investigating methods, it was revealed that the fabricated particles were good agreement with standard physical properties of TiO_2 nanoparticles and they could be used in Solar cells, waste water treatment and industrial applications.

Keywords: TiO_2, NPs, NaOH, SEM, XRD, UV-Vis

Introduction

Nowadays nanoparticles are the most widely used in the worldwide. They are like cornerstones of nanotechnology, nanoscale science and the interdisciplinary field of various science area. Nanotechnology is the studying of extremely small structures reducing bulk state materials as a result of size in nanoscale. These nanoparticles are very attractive because of its properties that is very different from bulk material. Nanoparticles have between the range size of 1 nm to 100 nm [Djouadi D., Aksas A.]. Titanium dioxide nanoparticles have one of great attractive significant interesting materials for scientists and physicists due to their properties. Titanium dioxide (TiO_2) is n - type semiconductor and it has wide energy band gap (> 3eV). TiO_2 exists in three different crystalline phases: anatase (tetragonal), rutile (tetragonal), and brookite (orthormbic). Both anatase and rutile have tetragonal crystal structure but belong to different phase. As a bulk material rutile is the stable phase, however solution phase preparation methods for TiO_2 generation favors the anatase structure. The c/a ratio of tetragonal anatase phase has greater than one and rutile phase has less than one [Galioglu S]. TiO_2 nanoparticles were synthesized by various methods. These are hydrothermal method, sol-gel method, chemical method and so on. From these methods, sol-gel method is one of the most promising methods due to low cost, low temperature and ease fabrication than the other method. In this research, TiO_2 nanoparticles were prepared by sol – gel method and they were deposited onto glass substrates using spin coating method. The structural, morphological and optical properties of fabricated particles and thin films were investigated.

Experimental Procedure

In this research, $TiCl_3$ is used as precusor for synthesis of nanoparticles. According to the stoichiometric balance reaction equations were;

$$TiCl_3 + 2HCl + H_2O \rightarrow TiCl_4 + 2H_2O$$

$$TiCl_4 + 2H_2O + C_2H_5O + NaOH \rightarrow TiO_2 + H_2O + NaCl$$

3.5ml of $TiCl_3$ was mixed again with 5 ml of hydrochloric acid and they were starring at 300 rpm under aluminum fume hood for 30 min. After stirring, 0.1g, 0.3 g and 0.5 g amount of

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sodium hydroxide were mixed into the solution. The exothermic reactions were observed and it released heat energy. And then the mixed solutions were continuously stirring at constant speed for 1 h to obtained homogenous solutions. In order to remove the produced impurities of reaction, the obtained solutions were annealed at 300˚C for 1 h in the atmospheric pressure. After heating process was done the white color amorphous TiO_2 were obtained. The obtaining amorphous white TiO_2 were washed 3 times to removed sodium chloride ions by filtration process with de-ionized water. And then they were centrifuged to separate the dregs at speed 1500 rpm for 10 min. Subsequently white TiO_2 were annealed at 300˚C to remove the absorbed water for 2 h. Finally, pure white TiO_2 nanoparticles were obtained. The obtained TiO_2 powders were mixed in 2.5 ml of ethanol and 2.5ml of acetone using magnetic stirrer for 5 min. The 200 µl of TiO_2 colloidal solution was deposited on the glass substrates using spin coating process at 2000 rpm for 20 s. The obtained TiO_2 coated glasses, were annealed on the hot plate at 150˚C and 30 min. the obtained TiO_2 thin films were characterized by UV-Visible spectroscopy to determine its optical properties. The other properties such as structural and morphological properties were also investigated.

Results and Discussion

XRD analysis

In this XRD analysis, the diffraction angle 2θ was started from 10˚ to 70˚. The XRD profiles of the TiO_2 nanoparticles were shown in Fig 1 (a, b, c and d) respectively. From these figures, all of the diffraction peaks showed anatase phase of TiO_2 tetragonal structure, which were good agreement with the standard library of Join Committee for Powder Diffraction Studies (JCPDS). The crystallite sizes were calculated using Debye-Scherrer's formula the average crystallite size of anatase TiO_2 synthesis nanoparticles was found in the range of 16nm to 35nm.

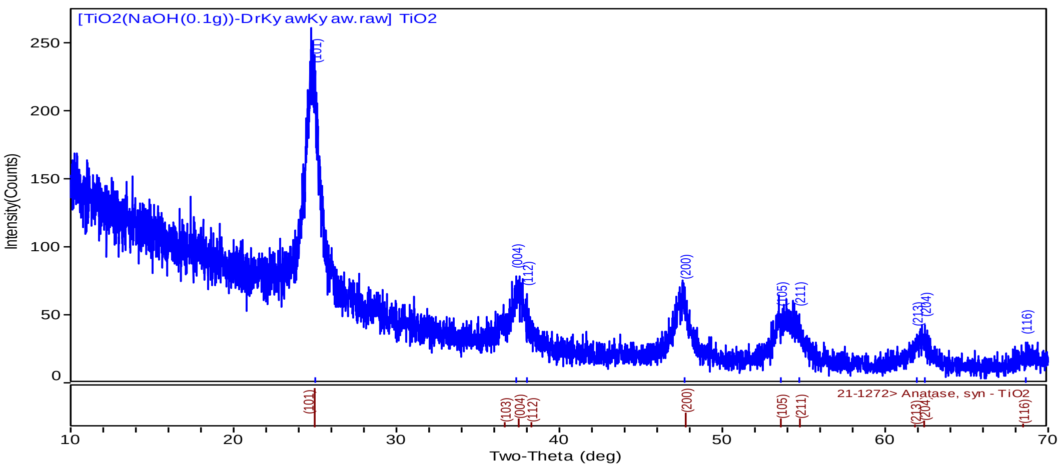

Figure 1 (a) XRD profiles of TiO_2 nanoparticles from NaOH(0.1g).

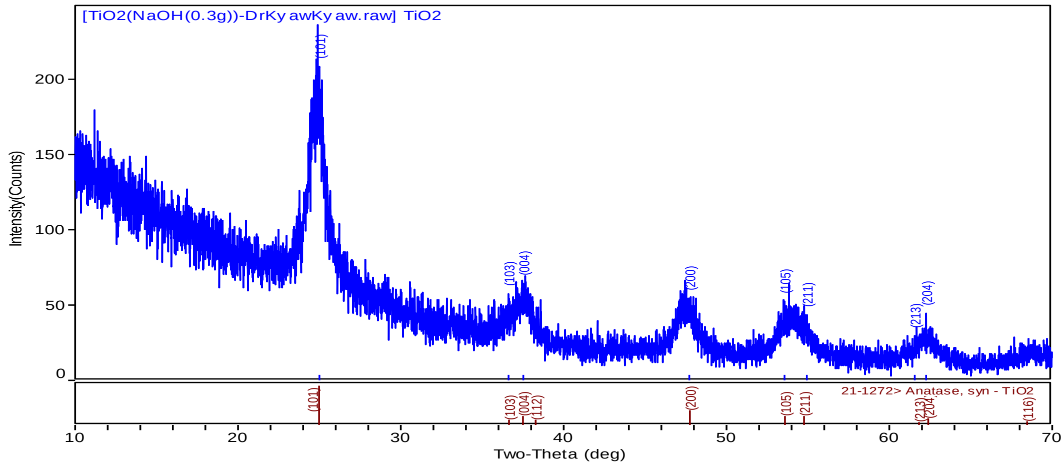

Figure 1 (b) XRD profiles of TiO_2 nanoparticles from NaOH(0.3g).

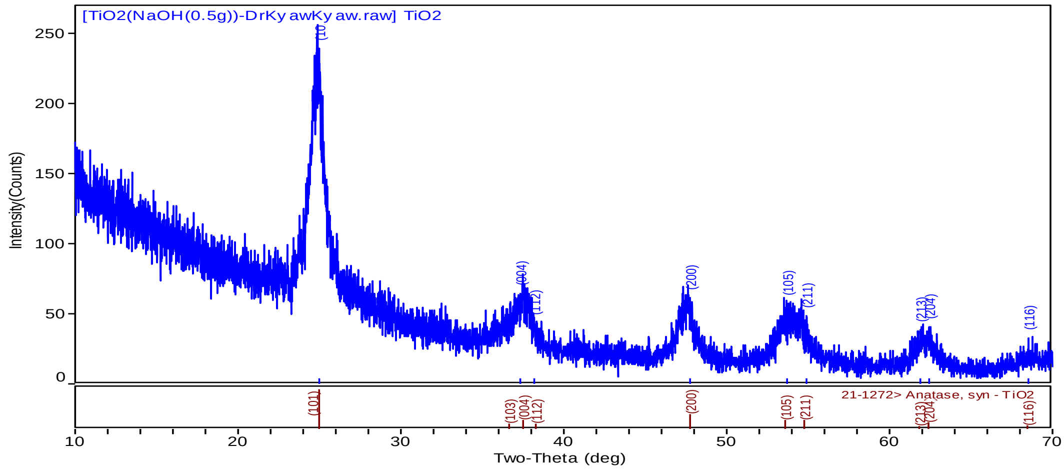

Figure 1 (c) XRD profiles of TiO_2 nanoparticles from NaOH(0.5g).

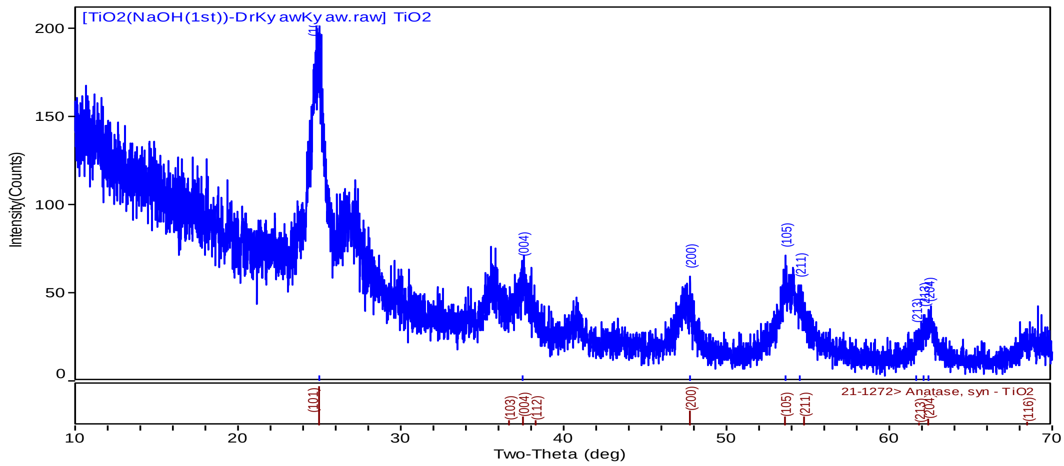

Figure 1 (d) XRD profiles of TiO_2 nanoparticles from NaOH(0.5g) with deionized water.

SEM analysis

The Fig 2(a, b, c and d) showed the SEM images of the TiO_2 nanoparticles prepared by sol-gel method with different amount of NaOH. In the Fig 2(a) the nanoparticles were formed in irregularly shape, overlapped and extremely agglomerated. Fig 2(b) illustrated the SEM result of TiO_2 nanoparticles synthesized with 0.3 g of NaOH. It was observed that the particles became tiny size and spherical shape. It can be observed from the Fig 2(c), some grains were agglomerated in some regions and some grains were separated by pores. Moreover, they were formed in spherical shape and overlapped in some regions. In Fig 2(d), the particles were formed spherical shapes and they had uniform nanoparticles sizes.

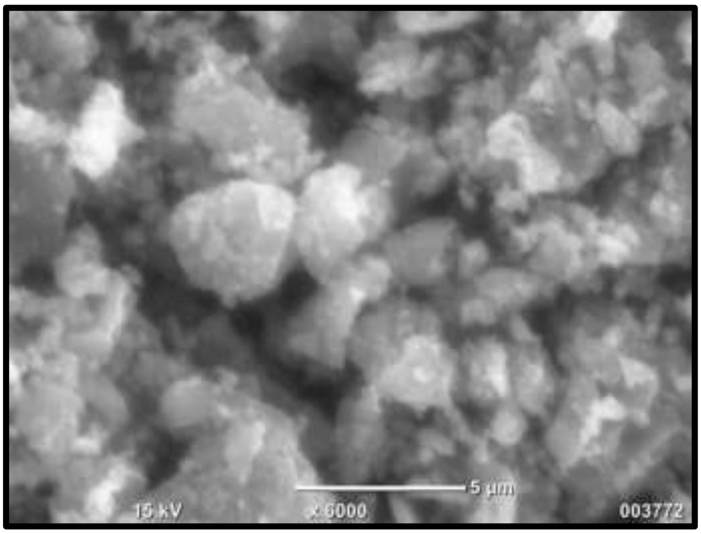

Figure 2 (a) The SEM micrographs of TiO_2 nanoparticles with NaOH (0.1 g).

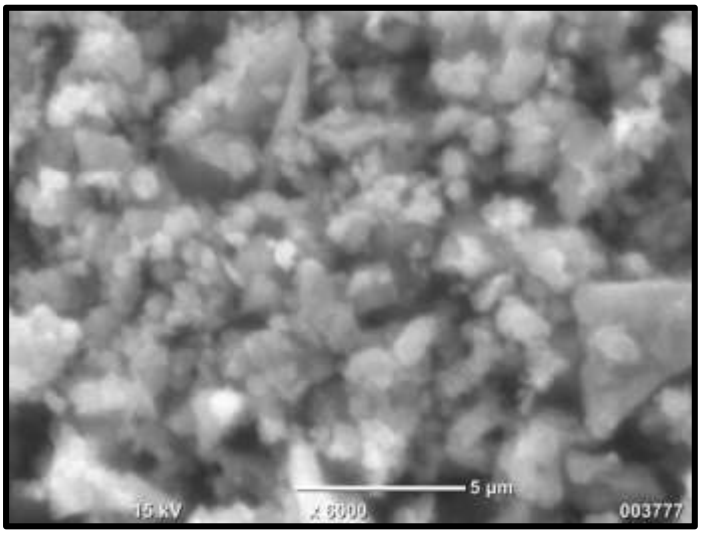

Figure 2 (b) The SEM micrographs of TiO_2 nanoparticles with NaOH (0.3 g).

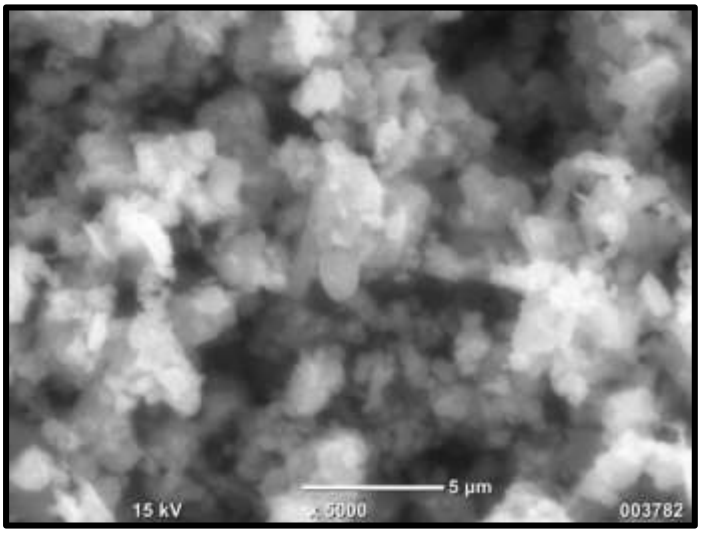

Figure 2 (c) The SEM micrographs of TiO_2 nanoparticles with NaOH (0.5 g).

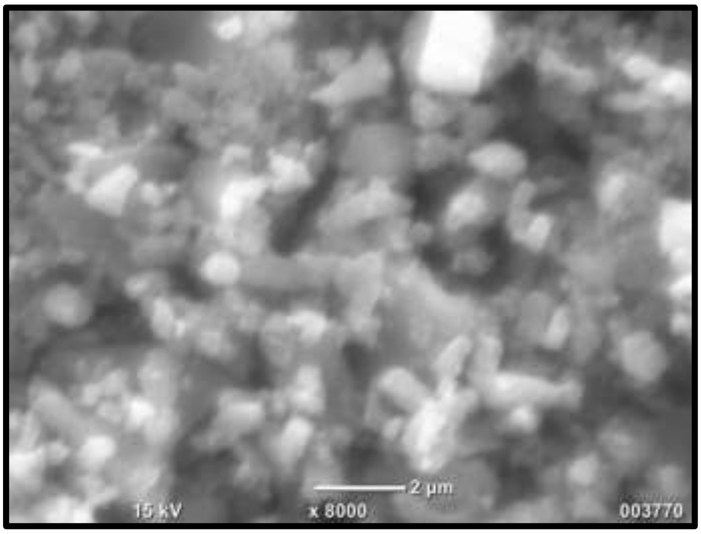

Figure 2 (d) The SEM micrographs of TiO_2 nanoparticles with NaOH (0.5 g) and deionized water.

Energy Band Gap From UV-Vis Analysis

Tauc's relation was used to calculated band gap energies. A plot of variation of $(\alpha h\upsilon)^2$ versus $h\upsilon$ were shown in Fig 3 (a, b, c and d). The band gap energies were evaluated using extrapolation of the linear part of the curve. The obtained band gap energies of TiO_2 nanoparticles were listed in Table 1.

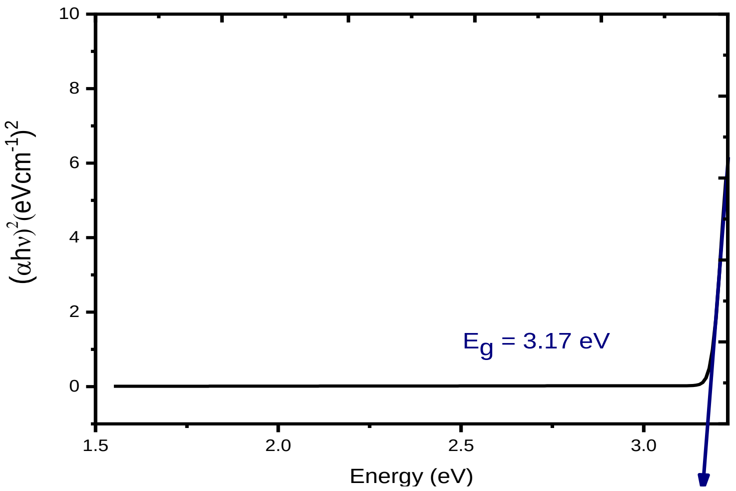

Figure 3 (a) The plot of extrapolation for TiO_2 nanoparticles from NaOH (0.1 g).

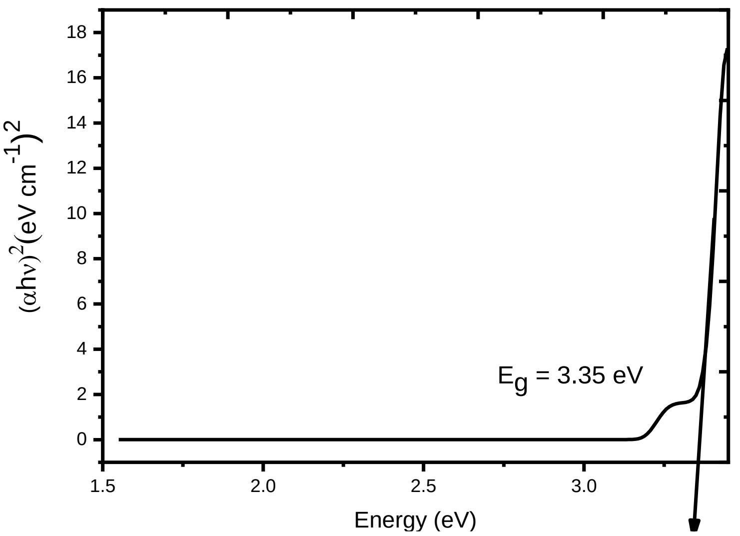

Figure 3 (b) The plot of extrapolation for TiO_2 nanoparticles from NaOH (0.3 g).

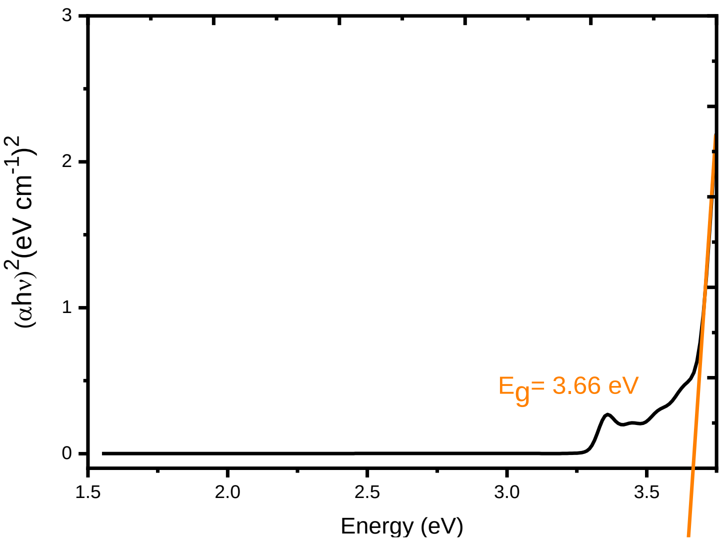

Figure 3 (c) The plot of extrapolation for TiO_2 nanoparticles from NaOH (0.5 g).

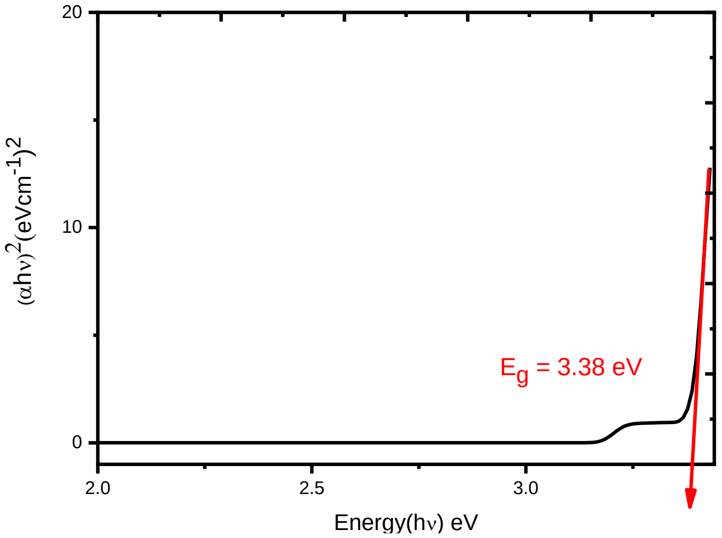

Figure 3 (d) The plot of extrapolation for TiO_2 nanoparticles from NaOH (0.5 g) with deionized water.

Table 1 The band gap energies of TiO_2 nanoparticles.

No	NaOH (g)	Band gap energy (eV)
1	0.1	3.17
2	0.3	3.35
3	0.5	3.66
4	0.5 with H_2O	3.38

Conclusion

Titanium dioxide (TiO_2) nanoparticles using sol – gel method with different mass ratio of NaOH from 0.1 g to 0.5 g were successfully synthesized. UV-Vis analysis showed that the maximum absorption peaks for TiO2 nanoparticles were found between 300 nm and 400 nm. The band gap energies of nanoparticles were varied from 3.17 eV to 3.66 eV due to the effect of NaOH content increase. The crystal structural and morphological properties of fabricated TiO_2 nanoparticles were characterized with XRD and SEM techniques. From XRD analysis, it was observed that the fabricated TiO_2 nanoparticles had polycrystalline nature and it's crystal structure were tetragonal with anatase phase. The grain formation was obviously formed in SEM micrographs. Some grains were agglomerated in some region and some were separated by pores. It could also be noted that one grain composed of approximately 20 crystallites. From this study it was concluded that the obtained TiO_2 nanoparticles were quite suitable to be used in photovoltaic applications such as solar cells.

Acknowledgments

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