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Research Article

Synthesis and Characterization of Zro_2 and Tio_2 Nanoparticles

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Abstract: The objective of this project is to synthesis ZrO_2 and TiO_2 nanoparticlesby chemical methods. 4.2 g of $ZrCl_4$ was dissolved in 300 mL distilled water, the source solution. 0, 03 M/mL as a molar concentration of sorbitol and ammonia was made as a solution, called a “target” and stirredfor 5 min. The source and target were mixed and stirred slowly. The final solution was heated to 70°C with stirring. Finally The solution was filtered and washed to obtain the nanoparticles; 5 mL of titanium isopropoxide was added to 15 mL of isopropanol. A solution of water, HNO_3 and NH_4OH was added to the first solution in order to tune ph to be 4. Then the mixture was stirred at 60-70°C for 20 h. Thereafter the precipitate was filtered and washed with ethanol and then dried at 100°C for 4 h under vacuum. Finally the powder was annealed at 600°C for 2 h to obtain TiO_2 nanoparticles. UV-VIS, XRD and SEM analyses were made to these powders, compared with other ones and then found that these particles obtained are in Nano range.

Keywords: Chemical method, nanoparticles, titanium dioxide, zirconium dioxide

INTRODUCTION

ZrO2 nanoparticles have many special properties like low thermal conductivity and high thermal stability and its thermal expansion coefficient is very high. So ZrO2 nanoparticles (ZrO2NPs) could be used in thermal barrier cutting, medical uses and as fillers in human bones and tooth (Shukla and Seal, 2005; Liang *et al*., 2003; Chandra *et al*., 2010).

The researchers used several methods in the past to producing ZrO2NPs like sol-gel (Ehrhart *et al*., 2006), hydrothermal (Torres-Huerta *et al*., 2009) and Chemical Vapor Deposition (CVD) (Rozo *et al*., 2008) method. In this study, there is a novel method to produce ZrO_2NPs called "co-precipitation" which was discovered by Alaei *et al*. (2014).

As for TiO_2 nanoparticles (TiO_2NPs), since the beginning of the researches about nanoparticles, scientists attended in formation and applications of TiO_2NPs specially in medicine because they are biocompatible with the human body and used indelivery drugs to its tissues (Wu *et al*., 2014; Liu *et al*., 2009; Hang *et al*., 2011; Miao *et al*., 2012).

TiO_2 has good properties in optics, dielectrics and as catalytic. For these interesting properties, $TiO_2$is used, as a bulk or nanoparticles, as a filler, catalytic and photocatalytic material (Barbé *et al*., 1997; Ahmed and Attia, 1995; Ferroni *et al*., 1996).

So, the formation of TiO_2NPs became very important in industrial and medical applications.

The aim of this study: The aim of this study is to producing ZrO_2 and TiO_2.

EXPERIMENTAL PROCEDURES

Preparation of ZrO_2 nanoparticles:

Chemical used: $ZrCl_4$ as a precursor, sorbitol and ammonia.

The procedure: 4.2 gm of $ZrCl_4$ was dissolved, according to the procedure mentioned in Alaei *et al*. (2014), in 300 mL distilled water, this is called "the source solution".

Thereafter, 0.03 M/mL, as a molar concentration, of sorbitol and ammonia solution were made, called a target and stirred for 5 min. Thereupon, the source and the target were mixed and stirred slowly at room temperature. The final solution were heated to 70°C for few minutes with stirring. The solution was filtered by paper funnel and washed with distilled water many times to obtaining the nanoparticles, then the solution was vaporized with existing of N_2 gas. Thereafter the powder was dried in an oven for six hours and calcined at 550°C for two hours.

During all the operation, Ph was tuned to be 4.

Preparation of TiO_2 nanoparticles:

Chemical used: Titaniumisopropoxide, isopropanol and distilled water.

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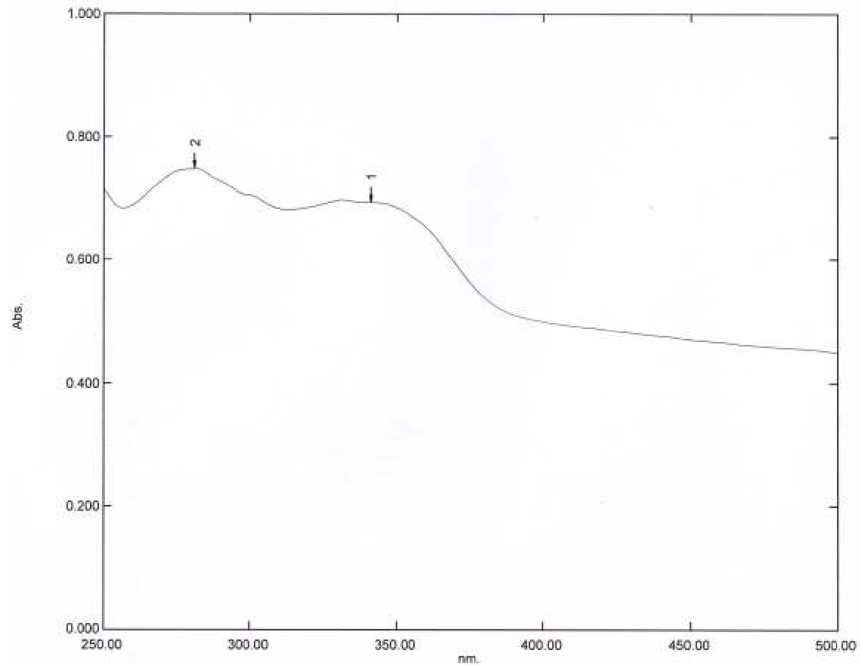

Fig. 1: UV spectrum of ZrO_2-NPs prepared

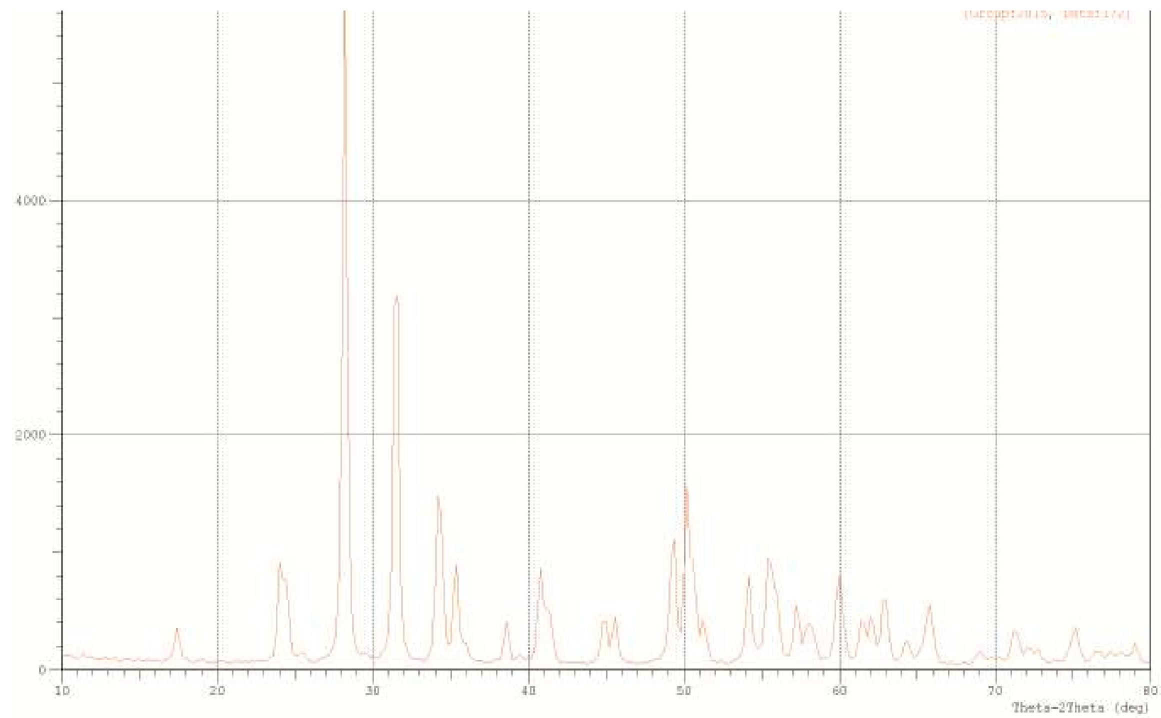

Fig. 2: XRD pattern for ZrO_2-NPs prepared

The procedure: According to the procedure discovered by Mahshad *et al*. (2006), 5 mL of titanium isopropoxide was added to 15 mL. of isopropanol. A solution of water, HNO_3 and NH_4OH was added to the first solution in order to Ph becomes two. Then the mixture was stirred at 60-70°C for 20 h. Finally we obtained a white blue gel suspension. Therefrom the precipitate was filtered and washed with ethanol to get the nanomaterial and then dried at 100°C for 4 h under vacuum, thereon the powder was annealed for 2 h at 600°C to obtain TiO_2 nanoparticles.

Characterization: Figure 1 represents the UV-VIS chart, Fig. 2 is for XRD and Fig. 3 is for SEM.

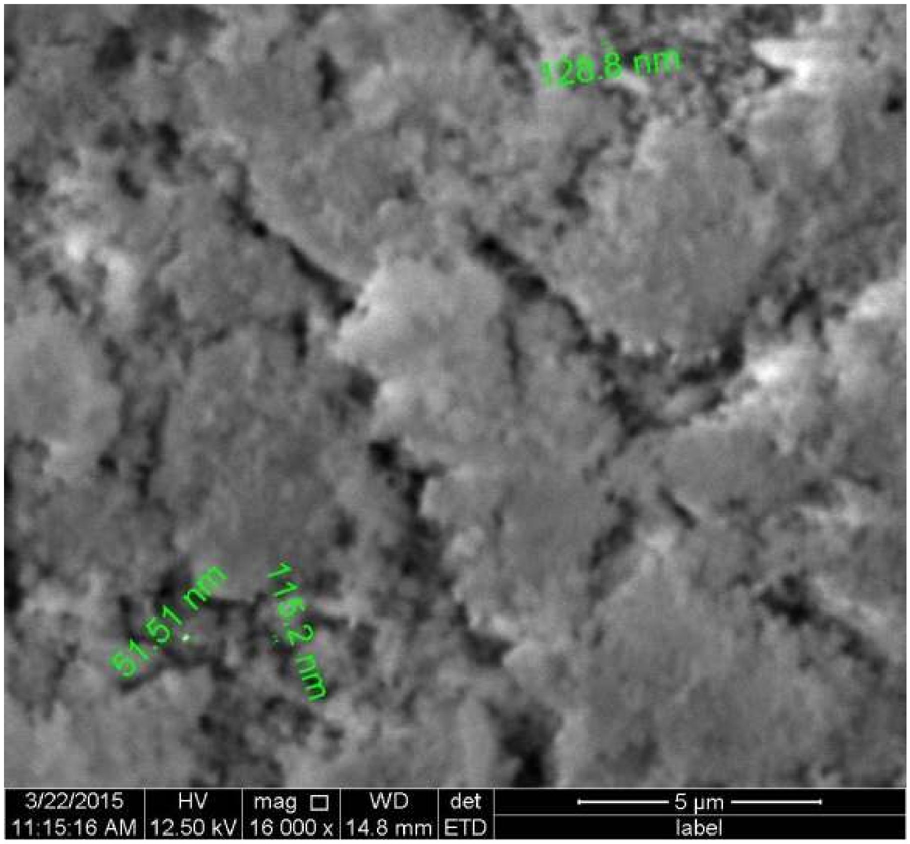

Fig. 3: SEM for ZrO_2-NPs prepared

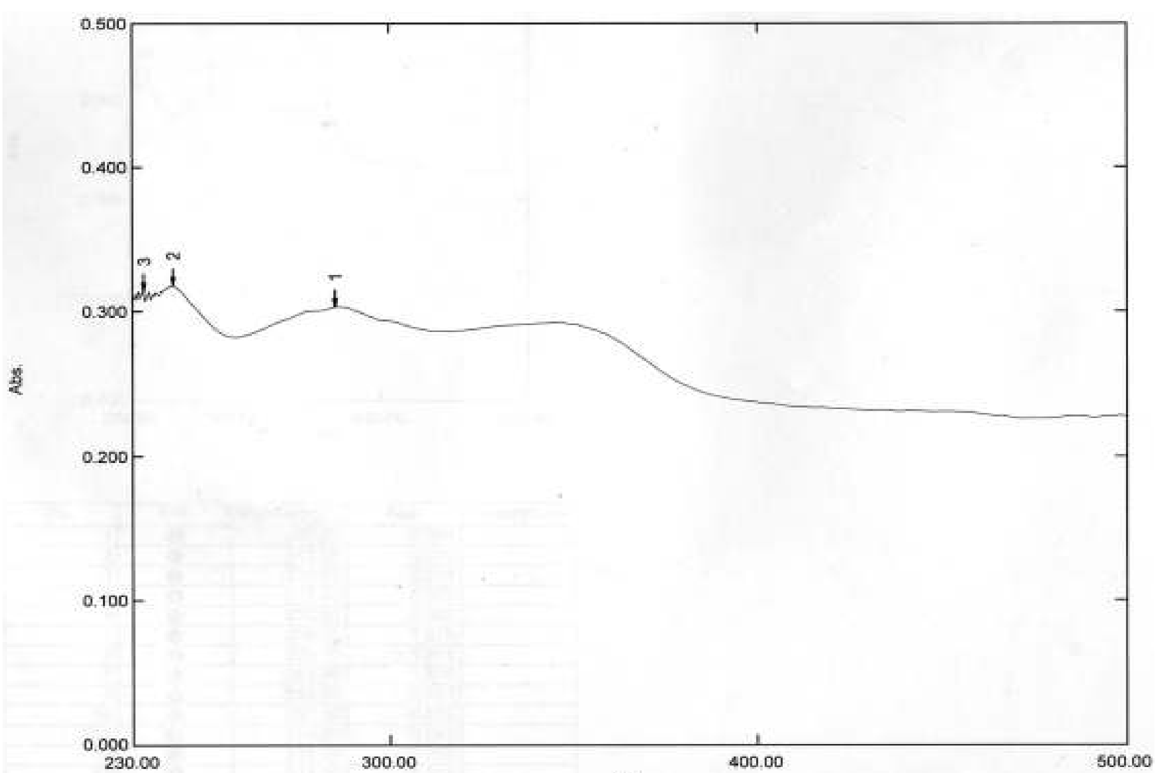

Fig. 4: UV-VIS spectrophotometry for TiO_2 nanoparticles prepared

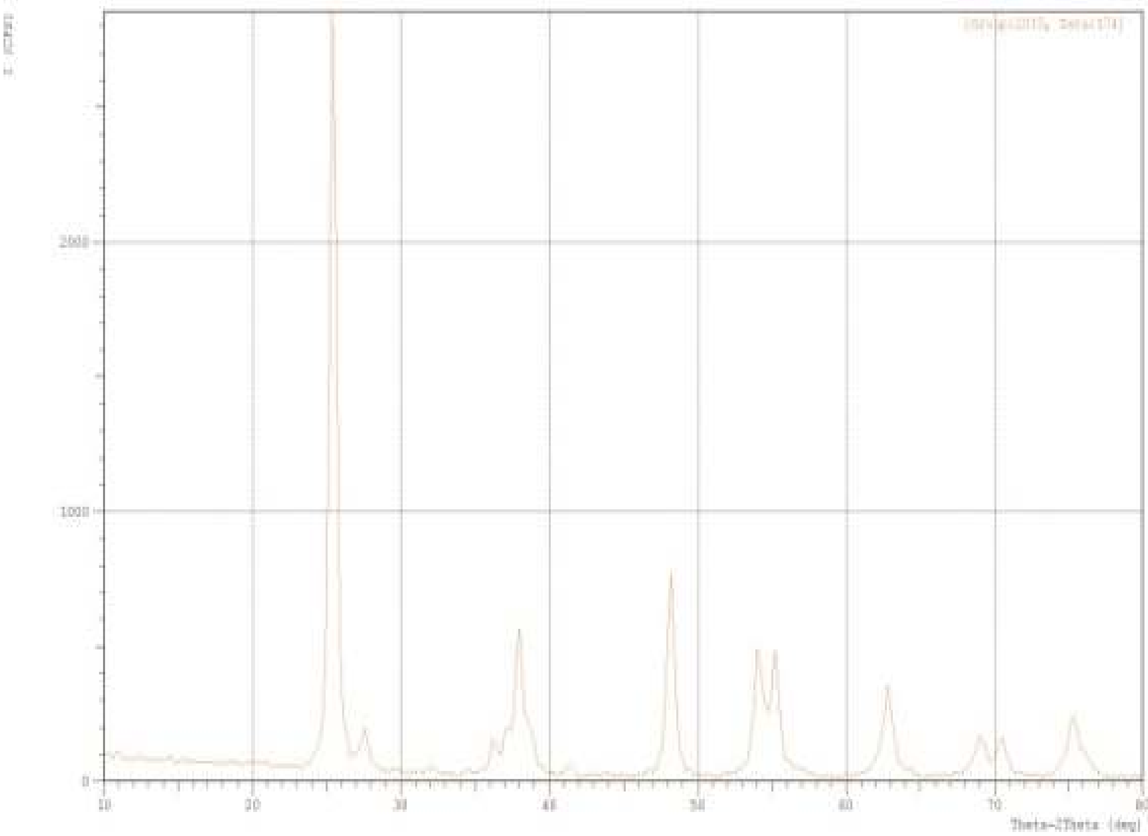

Fig. 5: XRD flow chart of TiO_2-NPs prepared

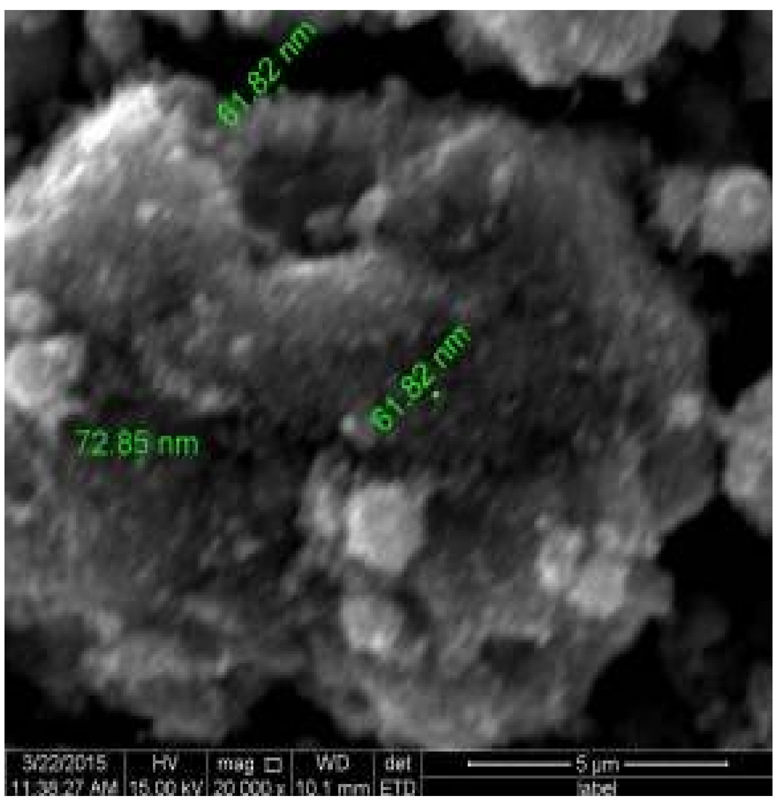

Fig. 6: SEM image of TiO_2-NPs that was prepared

As For TiO_2, UV-VIS spectrophotometry is in Fig. 4, XRD is in Fig 5 and SEM is represented by Fig. 6.

RESULTS AND DISCUSSION

UV-VIS spectrophotometry of ZrO_2NPs prepared, shown in (Fig. 1), tells usthat this powder of ZrO_2 is in nano range since it has a peak at 271 nm as mentioned by Mahmoud *et al*. (2013) (Fig. 7). This peak results from the transition of the electron from valance band to conduction band across the band gap which equals to 3.8 to 6.1 e.v.

XRD pattern of the ZrO_2 powder prepared (Fig. 2), was compared with the standard one available in the study of Zakeri *et al*. (2013) (Fig. 8) and found that these nanoparticles are for monoclinic ZrO_2.

From Fig. 3, the average particle size of ZrO_2NPs prepared was measured by debye-sherrer equation:

$$D = \frac{0.89\,\lambda}{B\ COS\ \omega}$$

where,
D = The grain size
λ = The wavelength at kα, which is intrinsic value for Cu, λ = 0.154 nm
B = The full wave half maximum of every peak
ω = The diffraction angle

It was found to be 14.14 nm.

SEM in Fig. 3 reveals that ZrO_2 powder prepared is in nano size, but the particles are aggregated.

The agglomeration is attributed to the high surface activity of the nanoparticles so that the nanoparticles react with the other materials easily or agglomerat about themselves especially if the preparation method was sol-powder (Scholz *et al*., 1998; Jiang *et al*., 2009; Maynard and Pui, 2007; Faure *et al*., 2013; Simard, 2007).

Sauter *et al*. (2008) found a solution for this phenomenon by projecting an ultrasonic waves on these nanoparticles agglomerated in the last step of prepation and succeeded in collapsing the agglomeration.

As to TiO_2NPs, from UV-VIS spectrometry (Fig. 4), it could be concluded that these particles are in nano size because it has two peaks at nearly 244 and 285 nm as mentioned in research of Li *et al*. (2009) (Fig. 9).

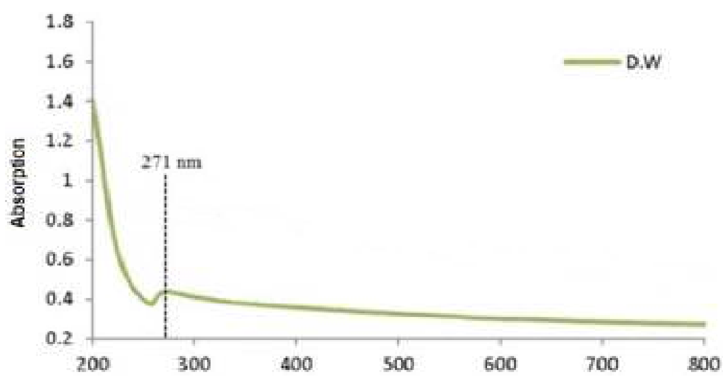

Fig. 7: UV spectrum of ZrO_2NPs (Mahmoud *et al*., 2013)

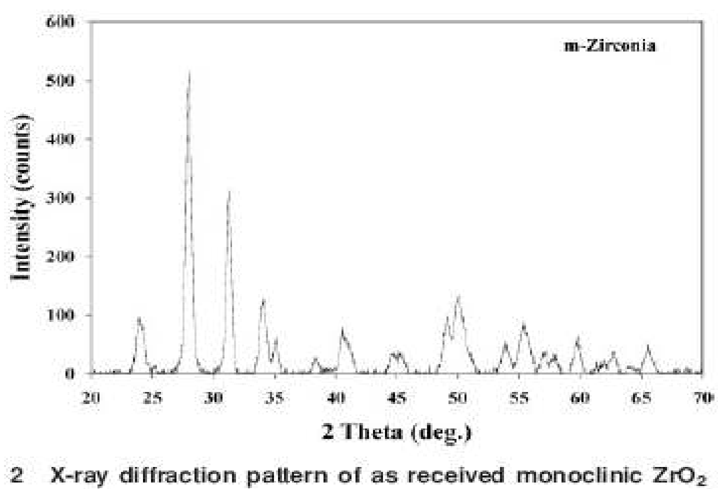

Fig. 8: Standard XRD graph of ZrO_2 nanoparticles

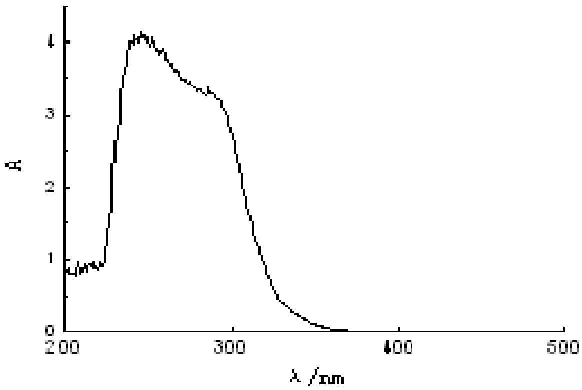

Fig. 9: The UV-VIS fpectrophotometry of TiO_2 nanoparticles done by Li *et al*. (2009)

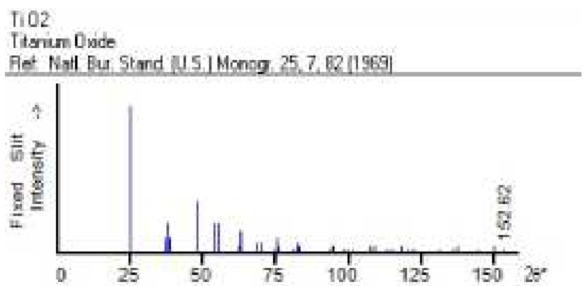

Fig. 10: The standard XRD pattern of TiO_2

The first peak is attributed to the electron transition, in a nanoparticle, from the valance band of oxygen O_{2p} to the conduction band of titanium Ti_{3d} when absorbing UV photon. The second peak is attributed to the absorbing a UV photon by an electron and transformation to a vibrational wave and then the vibration wave comes back to UV photon in less energy and in another direction or what is called scattering (Li *et al*., 2009).

The XRD pattern of TiO_2-NPs prepared, (Fig. 5), was compared with the standard XRD of TiO_2 (Fig. 10) and found that they are correspondent.

The grain size was calculated and found to be of nearly 10.29 nm.

As concerns SEM, we see from its image (Fig. 6), that the grain size is of about 67 nm and the nanoparticles are aggregated because of the reasons mentioned already.

CONCLUSION

ZrO_2 and TiO_2 nanoparticles have been obtained by chemical (sol-powder) method with the presence of the agglomeration.

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