

Comparison the Characterization of Synthesis of TiO₂ NPS Using Citrullus Colocynthis and Ricinus Communis Leaves Extracts

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Abstract:

The present study concluded the synthesis of titanium dioxide (TiO₂) nanoparticles using titanium tetra-isopropoxide (TTIP) as titanium source. Citrullus Colocynthis (CcExt-TiO₂NPs) and Ricinus Communis (RcExt-TiO₂NPs) as leaves extracts X-ray diffraction (XRD), Fourier transform infrared (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), and ultraviolet-visible spectroscopy (UV-VIS) were used to classify the samples. The results of the FT-IR study terpenoids, flavonoids, and proteins were found to be associated in the formation and stability of titanium nanoparticles. The result have been show that the XRD (CcExt-TiO₂NPs) and (RcExt-TiO₂NPs), In nature, they were found to be crystalline, with a mixture of rutile and anatase types. SEM showed that (CcExt-TiO₂NPs) and (RcExt-TiO₂NPs) were spherical in shape and the size ranger from 15 to 32 and 20 to 25 nm respectively. EDX image confirms the presence of titanium and Oxygen element in sample, The results indicated that the photocatalytic activity of the titanium dioxide TiO₂ nanoparticles that the breakdown of the dye Congo Red (CR) was verified by lowering of the absorbance and the degradation in presence of (CcExt-TiO₂NPs) was more than in presence of (RcExt-TiO₂NPs).

Keywords : TiO₂NPs , Citrullus Colocynthis , Ricinus Communis , comparsion

1. Introduction

Nanotechnology is considered a highly developed discipline because of the wide variety of applications it has in medical science, technology, and research. The word nano comes from a Greek word that means too little or infinite little thing [1]. Nanoparticles are particles with lengths ranging from 1 to 100 nanometers in two or three dimensions, according to the ASTM standard definition.[2]

Metal oxide nanoparticles NPs have physical, chemical, and biological characteristics that are distinct. This is due to the small particle size and high surface-to-volume ratio of their particles[3].

Titanium dioxide, Due to its unique properties, is one of the most significant nanomaterials that has attracted a lot of interest.. The optical, dielectric, and catalytic properties of titanium (TiO₂) powders are fascinating. leading to industrial uses such as fillers, pigments and photo-catalysts [4-8].

Titanium dioxide (TiO₂) can be made in a variety of ways, including solvothermal, hydrothermal, sol-gel, chemical vapor deposition, and electrodeposition. Nonetheless, these techniques necessitate a lot of resources as well as dangerous chemicals and solvents [9-12]. The Green synthesis of nanomaterials is an alternative route to

physical and chemical methods that leads to environment-friendly products due to the use of non-toxic materials, and does not require high temperatures and pressure, and at a lower cost. Green synthesis may be by microorganisms, plant extracts, and plant biomass [13-14].

Characteristics of product applications. The characterization is a necessary stage to understand the structure and applications of nanoparticles. The characterization process is achieved using techniques of UV-visible spectroscopy, Energy Dispersive x-ray (EDX) and (SEM) Scanning Electron Microscopy. UV-vis test was considered to understand the electronic transitions of synthesized. $\text{TiO}_2\text{NP}'\text{S}$ The EDX technology was used to illustrate the elemental composition of the synthesized $\text{TiO}_2\text{NP}'\text{S}$ of extract from a plant *Citrullus Colocynthis* and *Ricinus commun*. While the size, morphology and surface charge of $\text{TiO}_2\text{NP}'\text{S}$ was analyzed using Scanning electron microscopy (SEM).

2. Materials and methods

2.1 Collecting and preparing samples

Common plants with relatively high local consumption were selected based on their characteristics from local markets for the sale of plants obtained from the markets of the city of Nasiriyah, Iraq.

The plants were washed with ion-free water and then dried at room temperature, then milled the plant, and sifted with a sieve with a 202 micro. Then save in clean and dry plastic bottles.

2.2 Preparation of plant extracts

The leaves of the plant were collected and dried. Then 5 gm of sheets were weighed and dissolved in 100 ml distilled water. Then The extracts were filtered with a Whatman N. 1 filter Paper, store and use for further analysis.

2.3 Synthesis of TiO_2 nanoparticles

Weighed 7.5 mm of (titanium tetra-isopropoxide) and mixed with 250 ml of Distilled water. After that, 80 ml of the preparation was taken with 20 ml of the plant extract

After that, the sample was preserve For monitoring in Shakir in 24 hours. Changes in color are observed, prepared nanoparticles , And the powder was collected by Using a hot air oven

2.4. Characterization of TiO_2 Nanoparticles

The synthesized titanium NP'S were analyzed by Fourier Transform Infrared (FTIR) spectroscopy (FTIR; U.K / MD 1105 PG instrument Ltd) to classify the biochemical compounds involved in phytosynthesis that are responsible for the FT-IR spectra being recorded in the $400\text{--}4000\text{ cm}^{-1}$ range for *Citrullus Colocynthis* and *Ricinus Communis* leaf broth and biosynthesized titanium nanoparticles.

The powdered titanium nanoparticles were subjected to X-ray diffraction (XRD) analysis using an X-ray diffractometer (Pshillip,Xpert ,Holland). XRD measurements were taken at diffraction angles ranging (2θ) from 20° to 80°

Field emission scanning electron microscope (FESEM; Tescan-Mira3, France) and energy-dispersive X-ray spectroscopy were used to examine the morphology, size, and elemental analysis of titanium nanoparticles. The biosynthesized nanoparticles were dried at 40 °C and imaged using a FE-SEM at a magnification of 200 KX.

3. Results and Discussion

3.1 Fourier-Transform Infrared spectroscopy (FT-IR)

The FTIR spectra of CcExt-TiO₂NPs and RcExt-TiO₂NPs are shown in **(Fig.1,2)**. The spectra show peaks at 3299.69 , 1625.21 , 1508.84 , 1396.01 , 1251.45 cm⁻¹ for CcExt-TiO₂NPs and at 3363.59 , 1635.34 , 1456.57 , 1436.28 , 1418.88 , 1375.50 , 1361.82 , 1282.28 cm⁻¹ for RcExt-TiO₂NPs respectively. These peaks correspond to O-H stretching of alcohols/phenols N-H vibration of amines and C=O stretching of carbonyl groups. The results indicate that the plant extracts contain terpenoids, flavonoids and proteins. The Citrullus Colocynthis and Ricinus Communis leaves extracts for the biosynthesis of titanium dioxide nanoparticles, they acted as both reducing and capping agents. [15]

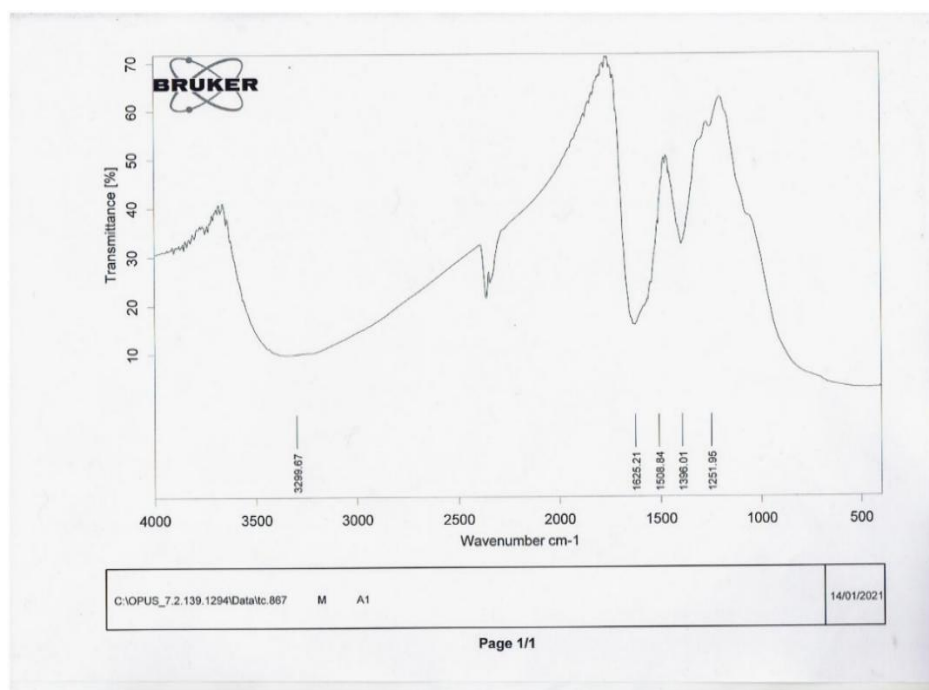


Figure.1: FTIR of TiO₂ NP'S extract from a plant Citrullus Colocynthis

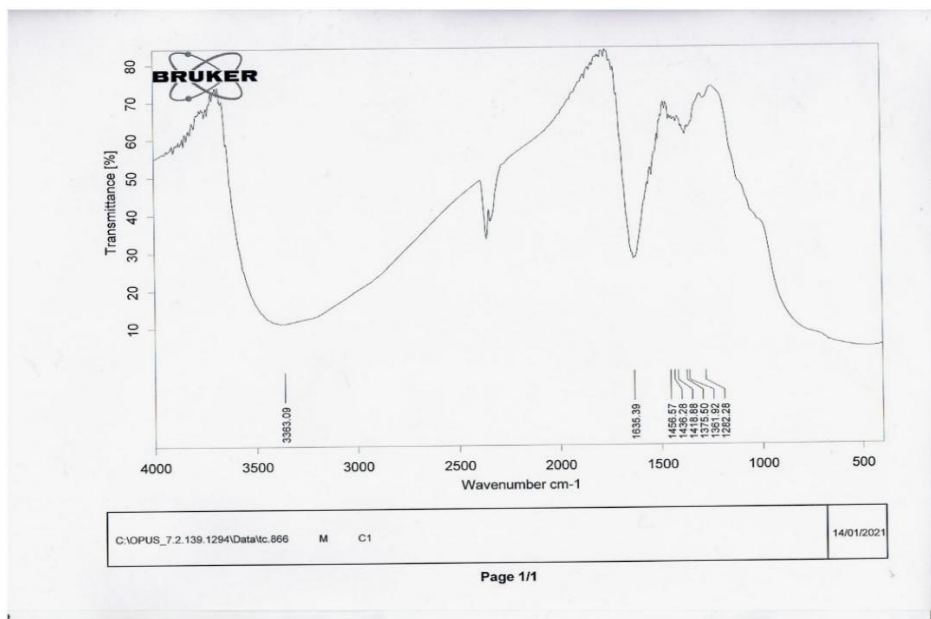


Figure.2: FTIR of TiO₂ NP'S extract from a plant Ricinus Communis

3.2 X-ray diffraction (XRD)

The phase purity and crystallinity of biosynthesized CcExt-TiO₂NPs and RcExt-TiO₂NPs were analysed by X-ray diffractometer XRD (**Fig. 3,4**). The pattern is shown at 25.0, 38.0, 48.0, and 55.5 correspond to the structures of anatase and at 27.0, 47.7, 54.0, 62.0, 69.0 and 76.5° correspond to the structure of the rutile of titanium nanoparticles of CcExt-TiO₂NPs and RcExt-TiO₂NPs respectively, these results confirmed by using JCPDS (No. 21-1276).

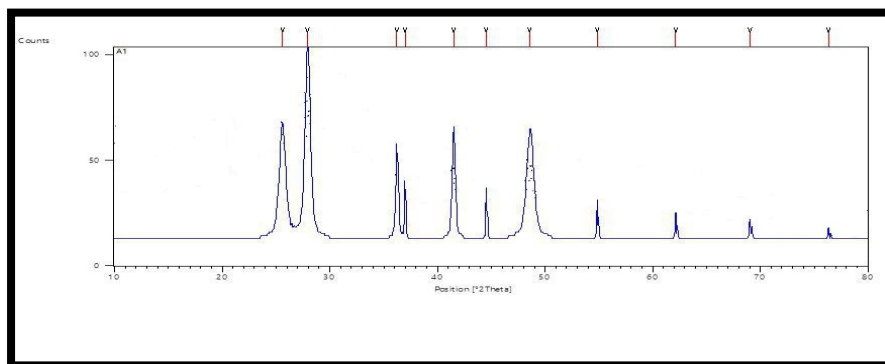


Figure.3 XRD patterns of the synthesized TiO₂ nanoparticles extract from a plant Citrullus Colocynthis

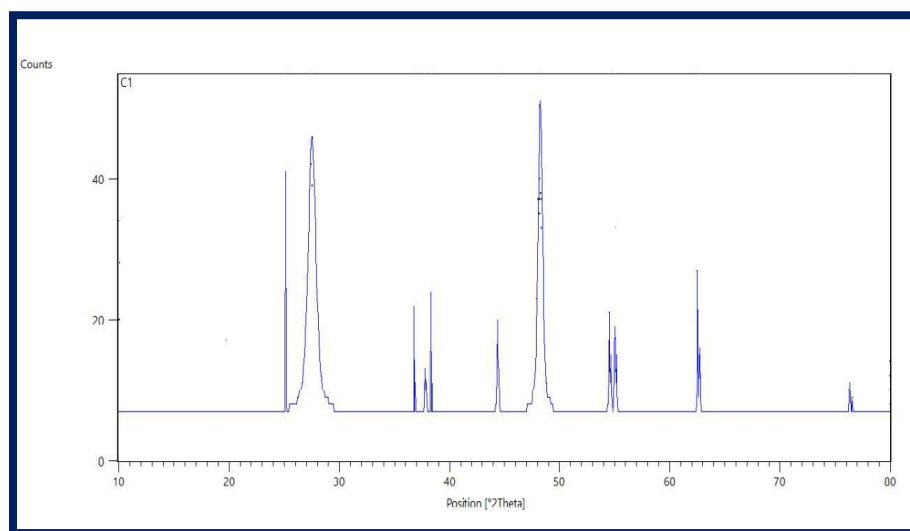


Figure.4 XRD patterns of the synthesized TiO₂ nanoparticles extract from a plant Ricinus Commun

3.3 FE-SEM and EDX analysis of titanium nanoparticles(NP'S)

The results of FESEM (**Fig. 5,6**) images showed that the CcExt-TiO₂NPs and RcExt-TiO₂NPs were in spherical shapes and their sizes were ranged from 15 to 32 and 25 to 20 respectively. Energy Dispersive X-ray spectroscopy (EDX) (**Fig.7: a,b**) analysis showed the strong titanium peaks at 4.5 to 5 KeV , oxygen(O) at 0.5 KeV, Silica(Si) at 1.2 KeV , and carbon (C) at 0.2 KeV. Silica peak was observed as a result of the preparation of the glass substrate film or detector of EDX. The carbon (C) peak indicated the presence of biological molecules on the surface of TiO₂NPs. The results correspond have been reported by anbalagan et al.

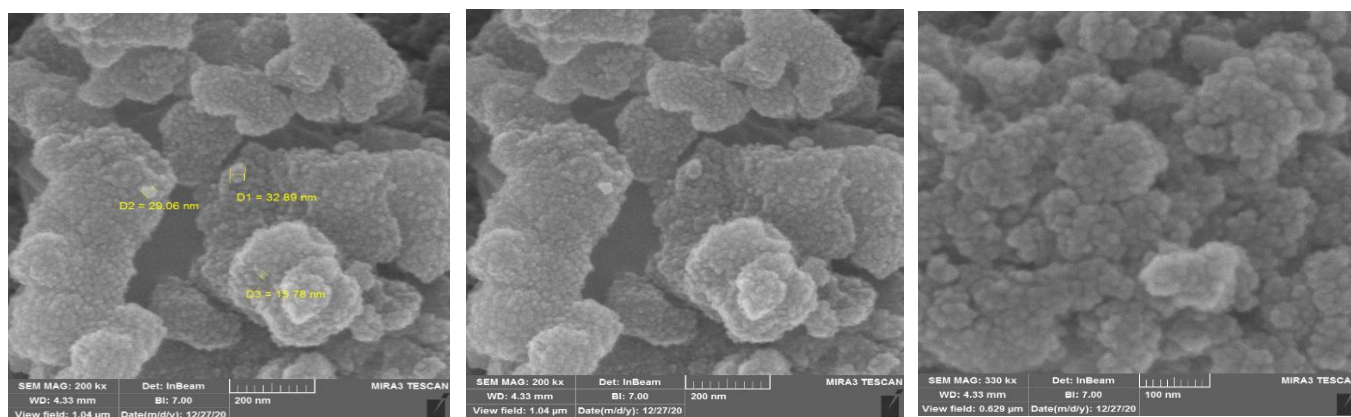


Figure.5 FESEM images of TiO₂ NP'S extract from a plant Citrullus Colocynthis

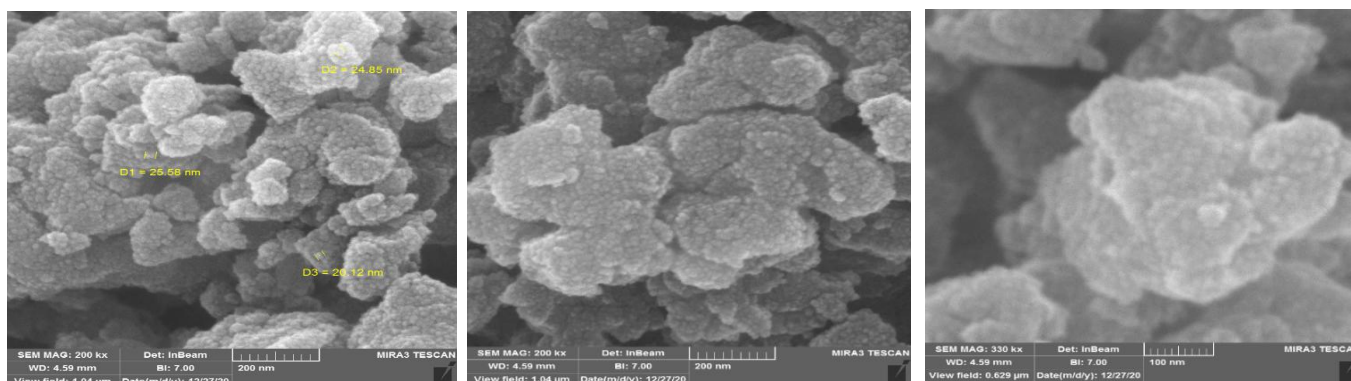


Figure.6 FESEM images of TiO₂ NP'S extract from a plant Ricinus Communis

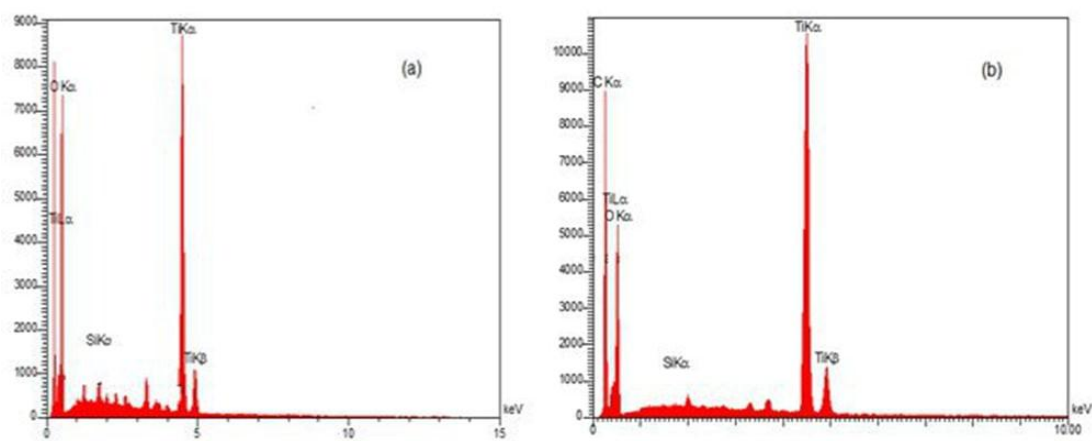


Figure.7: **a** EDX spectrum of TiO₂ NPs extract from a plant Citrullus Colocynthis
b. EDX spectrum of TiO₂ NPs extract from a plant Ricinus Communis

3.4 UV-Vis Spectrophotometer Analysis:

Size, shape and stabilization of nanoparticles were depending on the formation of one or more surface Plasmon resonance (SPR) bands. Formation of single SPR band at short wavelength revealed the presence of small sized spherical nanoparticles in the reaction mixture [16]. **(Fig.8,9)** reveals that the absorption spectrums of (CcExt-TiO₂NPS) and (RcExt-TiO₂NPS) consists of broad intense absorption around 324 and 340 nm respectively.

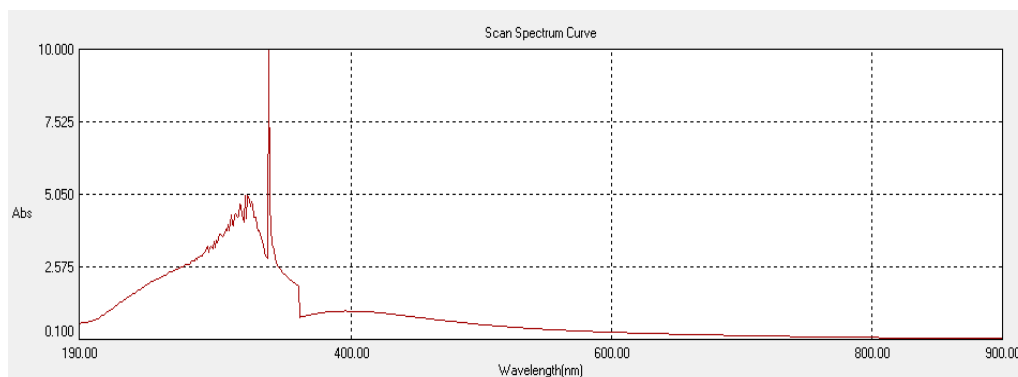


Figure. 8 UV- Vis. Spectrum of TiO₂ in water extract from a plant *Citrullus Colocynthis*

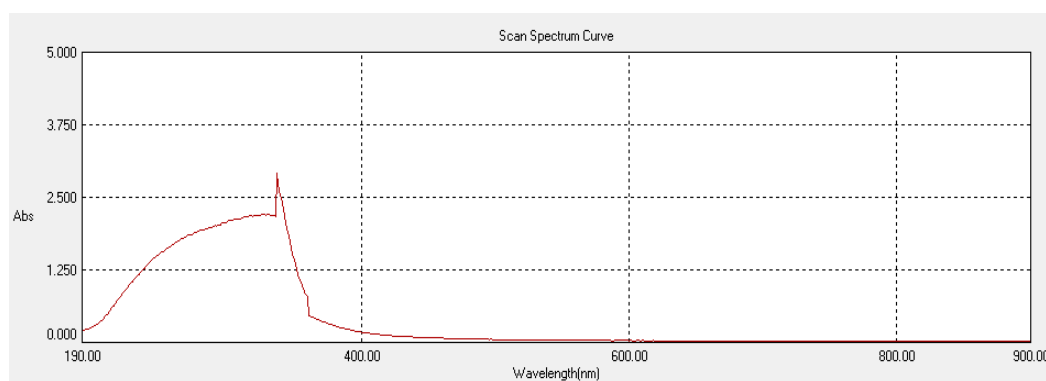


Figure. 9 UV- Vis. Spectrum of TiO₂ in water extract from a plant *Ricinus Communis*

3.5 Photocatalytic activity of CcExt-TiO₂NPs and RcExt-TiO₂NPs

The degradation of Cong Red dye under sunlight irradiation was used to investigate the photocatalytic activity of CcExt-TiO₂NPs and RcExt-TiO₂NPs with respect to time. 10 mg of CcExt-TiO₂NPs and 10 mg of RcExt-TiO₂NPs were added with 50 ml of 0.001%(w/v) dye solution of Cong Red (for each one) and the mixture was magnetically stirred and exposed to sunlight at the time interval 1 , 2 , 3 hours. The clean supernant solution was scanned at ranged wavelength (200-800)nm to study dye the breakdown in the presence of of CcExt-TiO₂NPs and RcExt-TiO₂NPs. [17-18] (**Fig.10,11**) were showed That the breakdown of the dye was verified by lowering of the absorbance and the degradation in presence of CcExt-TiO₂NPs was more than in presence of RcExt-TiO₂NPs.

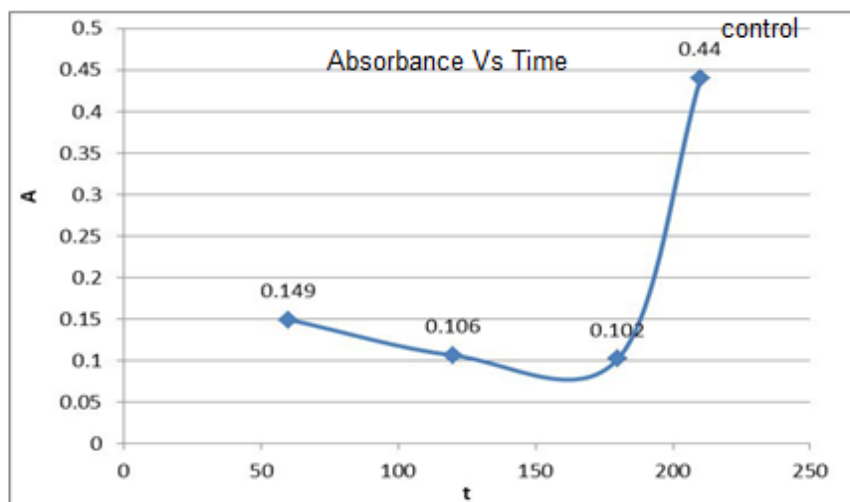


Figure. 10 Cong Red dye Photodegradation of CcExt-TiO₂NPs

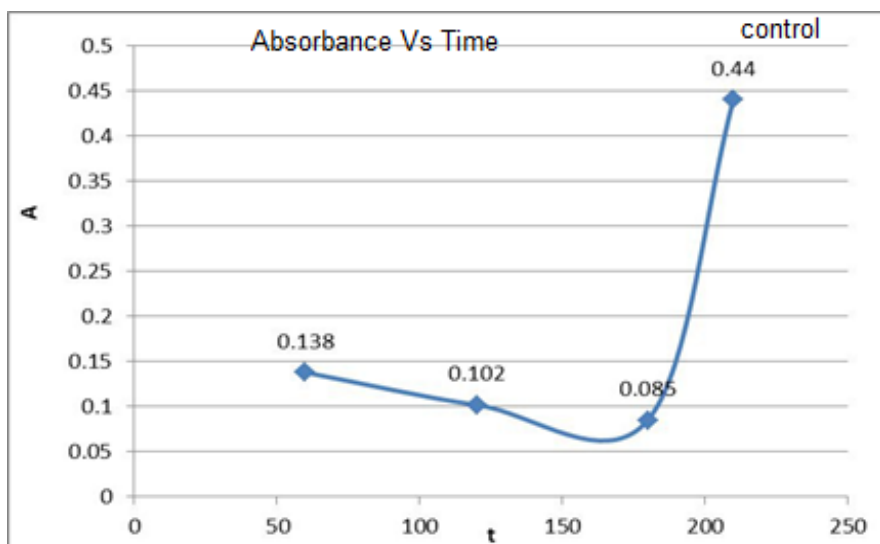


Figure. 11 Cong Red dye Photodegradation of RcExt-TiO₂NPs

Conclusions

Titanium dioxide nanoparticles were prepared using green synthesis, the preparation method is environmentally friendly, that is, it is a method within green chemistry by relying on plant materials with non-toxic and inexpensive chemicals and in one step as it gives an abundant product of titanium oxide particles with homogeneous nanoscale

dimensions, and then it can be used for commercial purposes. From the analysis of X-ray diffraction it was confirmed that the formation of titanium oxide was confirmed and that the data was identical to what is in the standard file JCPDS of titanium oxide. the SEM and EDX results display that it has nearly spherical in shape and the elemental composition TiO₂ NPs in the sample, The EDX image confirms the presence of titanium(Ti) and Oxygen(O) element in sample. The results indicated that the photocatalytic activity of the TiO₂ nanoparticles that the dye's deterioration was verified by the decrease of the absorbance and the degradation in presence of (CcExt-TiO₂NPs) was more than in presence of (RcExt-TiO₂NPs).

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