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(Organizadores)



**MEIO AMBIENTE,  
SUSTENTABILIDADE  
E AGROECOLOGIA 5**

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**Tayronne de Almeida Rodrigues**  
**João Leandro Neto**  
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(Organizadores)

# **Meio Ambiente, Sustentabilidade e Agroecologia 5**

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## APRESENTAÇÃO

A obra Meio Ambiente, Sustentabilidade e Agroecologia vem tratar de um conjunto de atitudes, de ideias que são viáveis para a sociedade, em busca da preservação dos recursos naturais.

Em sua origem a espécie humana era nômade, e vivia integrada a natureza, sobreviviam da caça e da colheita. Ao perceber o esgotamento de recursos na região onde habitavam, migravam para outra área, permitindo que houvesse uma reposição natural do que foi destruído. Com a chegada da agricultura o ser humano desenvolveu métodos de irrigação, além da domesticação de animais e também descobriu que a natureza oferecia elementos extraídos e trabalhados que podiam ser transformados em diversos utensílios. As pequenas tribos cresceram, formando cidades, reinos e até mesmo impérios e a intervenção do homem embora pareça benéfica, passou a alterar cada vez mais negativamente o meio ambiente.

No século com XIX as máquinas a vapor movidas a carvão mineral, a Revolução Industrial mudaria para sempre a sociedade humana. A produção em grande volume dos itens de consumo começou a gerar demandas e com isso a extração de recursos naturais foi intensificada. Até a agricultura que antes era destinada a subsistência passou a ter larga escala, com cultivos para a venda em diversos mercados do mundo. Atualmente esse modelo de consumo, produção, extração desenfreada ameaça não apenas a natureza, mas sua própria existência. Percebe-se o esgotamento de recursos essenciais para as diversas atividades humanas e a extinção de animais que antes eram abundantes no planeta. Por estes motivos é necessário que o ser humano adote uma postura mais sustentável.

A ONU desenvolveu o conceito de sustentabilidade como desenvolvimento que responde as necessidades do presente sem comprometer as possibilidades das gerações futuras de satisfazer seus próprios anseios. A sustentabilidade possui quatro vertentes principais: ambiental, econômica, social e cultural, que trata do uso consciente dos recursos naturais, bem como planejamento para sua reposição, bem como no reaproveitamento de matérias primas, no desenvolvimento de métodos mais baratos, na integração de todos os indivíduos na sociedade, proporcionando as condições necessárias para que exerçam sua cidadania e a integração do desenvolvimento tecnológico social, perpetuando dessa maneira as heranças culturais de cada povo. Para que isso ocorra as entidades e governos precisam estar juntos, seja utilizando transportes alternativos, reciclando, incentivando a permacultura, o consumo de alimentos orgânicos ou fomentando o uso de energias renováveis.

No âmbito da Agroecologia apresentam-se conceitos e metodologias para estudar os agroecossistemas, cujo objetivo é permitir a implantação e o desenvolvimento de estilos de agricultura com maior sustentabilidade, como bem tratam os autores desta obra. A agroecologia está preocupada com o equilíbrio da natureza e a produção de alimentos sustentáveis, como também é um organismo vivo com sistemas integrados

entre si: solo, árvores, plantas cultivadas e animais.

Ao publicar esta obra a Atena Editora, mostra seu ato de responsabilidade com o planeta quando incentiva estudos nessa área, com a finalidade das sociedades sustentáveis adotarem a preocupação com o futuro.

Tenham uma excelente leitura!

Tayronne de Almeida Rodrigues

João Leandro Neto

Dennyura Oliveira Galvão

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## EXPERIMENTAL VARIABLES IN THE SYNTHESIS OF TiO<sub>2</sub> NANOPARTICLES AND ITS CATALYTIC ACTIVITY

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**ABSTRACT:** Nanoparticles of titanium dioxide (TiO<sub>2</sub>) (average size ~ 5 – 579 nm) with a high surface area (~ 192 m<sup>2</sup> g<sup>-1</sup>) were synthesized in the pure anatase phase, either with or without H<sub>2</sub>O<sub>2</sub>, using isopropanol or acetic acid as a reaction medium. TiO<sub>2</sub> was characterized by using XRD, BET surface area, STEM and UV-

Visible techniques. TiO<sub>2</sub> was obtained differed according to the reaction medium used. Bandgap energy values observed for TiO<sub>2</sub> with H<sub>2</sub>O<sub>2</sub> were 3.10 for TiO<sub>2</sub> with IP, and 2.97 for TiO<sub>2</sub> with HAc. Values when H<sub>2</sub>O<sub>2</sub> was not used were 3.07 for TiO<sub>2</sub> obtained with IP and 3.40 with HAc. The catalytic activity of TiO<sub>2</sub> in solutions of H<sub>2</sub>O<sub>2</sub> was studied by examining the decolorization of methylene blue in the dark. The decolorization in the presence of pretreated TiO<sub>2</sub> of approximately 100% was obtained within 7h in the presence of TiO<sub>2</sub> obtained in HAc and presence of H<sub>2</sub>O<sub>2</sub>.

**KEYWORDS:** *Hydrogen peroxide, solvothermal method, titania nanocrystals.*

## 1 | INTRODUCTION

Titanium dioxide (titania - TiO<sub>2</sub>) is used in different environmental applications, such as photocatalytic degradation for purification of polluted air and in wastewater treatment [CARP; HUISMAN; RELLER, 2004; LI et al., 2003; GARCIA-SEGURA; BRILLAS, 2017, PUEYO et al., 2016]. It also has a wide variety of technological applications, such as pigments, wet-type solar cells [MICHAEL, 2001; AHMAD; PANDEY; RAHIM, 2017], sensors [HIROSHI et al., 2005, ZHANG et al., 2017], photocatalysts [ZHANG et al., 2017, BORA, MEWADA, 2017] and electronic material [LI et al., 2003, DAIA et

all., 2003].

There are three main crystallographic forms of  $\text{TiO}_2$ : rutile (tetragonal), anatase (tetragonal), and brookite (orthorhombic) [LI; WHITE; LIM, 2003, Y. HU; TSAI; HUANG, 2003]. Rutile is thermodynamically stable and the other two forms are metastable [GOPAL; CHAN; DE JONGHE, 1997]. The anatase form has been used in photocatalysis and various other applications [ZAKRZEWSKA; RADECKA, 2017, LOAN et al., 2017].

Methods of synthesizing anatase  $\text{TiO}_2$  crystals and anatase  $\text{TiO}_2$  mesocrystals have been extensively described in scientific literature, including the sol–gel method [PACHECO et al., 2004], the precipitation method [GOPAL; CHAN; DE JONGHE, 1997], aerosol synthesis [AHONEN et al., 2001], and the hydrothermal method [KARTHIKEYAN et al., 2017, BOKHIMI; PEDRAZA, 2004]. In some of these processes, preparation costs can be high, the reaction is time consuming and the process is complex. In order to obtain ultrafine particles, milling and thermal treatment are also needed. In the hydrothermal method, the reaction conditions, such as solution concentration, temperature and pH, have a strong influence on the reaction process but it is relatively easy to control, and the cost is low. Some of the advantages of the hydrothermal method over other fabrication techniques include the purity, homogeneity, and stoichiometry control of the particles obtained. The size of  $\text{TiO}_2$  particles produced depends upon the grain size and composition, but not on the calcination temperature.

However, anatase  $\text{TiO}_2$  crystals or mesocrystals usually prepared by the methods cited above require either complex processing steps or high energy consumption. Some of these methods can be unfavourable for practical and commercial applications in the industry. Goutailler et al. synthesized photocatalytic active nanoparticulate  $\text{TiO}_2$  at a low temperature (100 °C) by an aqueous sol–gel method, but ammonium bromide salts were needed as catalysts [GOUTAILLER et al., 2002]. Hao *et al.* obtained mesoporous titania powder with a mixture of phase (anatase and brookite) framework at low temperatures (80–100 °C) by a modified sol–gel method using dodecylamine as a template [HAO; ZHANG, 2008]. Serrano *et al.* [LI et al., 2017] prepared micro-mesoporous  $\text{TiO}_2$  photocatalysts by a mild crystallization procedure (40 °C), using in the crystallization step four inorganic acids in the refluxing treatment. In this same way Li *et al.* prepared anatase  $\text{TiO}_2$  mesocrystals at low temperature (80 °C) in one-pot synthesis *via* acetic acid (HAc)-induced hydrolysis [LI et al., 2017].

Various parameters such as pH, presence/absence of catalyst, temperature, chelating reagent, nature of precursor, use of organic acid or  $\text{H}_2\text{O}_2$  influence the size, shape and phase of  $\text{TiO}_2$  [SUGIMOTO; ZHOU; MURAMASTU, 2003a, SUGIMOTO; ZHOU; MURAMASTU, 2003b, ATTAR et al., 2008, CHANG et al., 2009]. Chang *et al.* have investigated the effect of pH on the phase and morphology evolution of nano  $\text{TiO}_2$  from peroxo titanium complex in the presence of HAc as chelating agent [CHANG et al., 2009]. Sugimoto *et al.* have studied the influence of pH on the size control and various amines on the shape control of anatase  $\text{TiO}_2$  prepared from sol–gel process [SUGIMOTO; ZHOU; MURAMASTU, 2003a, SUGIMOTO; ZHOU; MURAMASTU,

2003b]. Attar *et al.* have reported the effect of modifier ligands (HAc and acetyl acetone) on the nano TiO<sub>2</sub> formation from TIP by sol–gel method [ATTAR *et al.*, 2008].

HAc is considered to play a key role during the formation of TiO<sub>2</sub> mesocrystals, promoting the transformation of TiO<sub>2</sub> from the amorphous state to the anatase phase at low temperatures (80 °C). The morphology and size of the mesocrystals of TiO<sub>2</sub> anatase prepared in the presence of HAc can be adjusted continuously in the range of 20 nm to 100 nm, adjusting the concentration of HAc [LI *et al.*, 2017, PARRA *et al.*, 2008].

Compared to the hydrothermal method for obtaining TiO<sub>2</sub>, the wet chemical route using the peroxo titanium complex has been little explored [CHANG *et al.*, 2010]. Although some systems have previously investigated. But even not being much explored the peroxide-based route for synthesizing titanium oxide powder is well established [ATTAR *et al.*, 2008, CHANG *et al.*, 2010, MUHLEBACH; MULLER; SCHWARZENBACH, 1970, GAO *et al.*, 2003, GAO; MASUDA; KOUMOTO, 2004, GAO *et al.*, 2007, GAO *et al.*, 2008, JAGADALE *et al.*, 2008].

This study described the synthesis of titanium dioxide nanoparticles which were obtained using the peroxide method. Pure anatase was obtained by the hydrothermal method in the presence of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) using solutions of titanium tetraisopropoxide in isopropanol (IP) or acetic acid (HAc). Hydrogen peroxide is an ideal environmentally friendly solvent and oxidant and used widely to the synthesis of nanostructures of inorganic materials [LI *et al.*, 2006]. The catalytic activity of TiO<sub>2</sub> in solutions of H<sub>2</sub>O<sub>2</sub> was studied by examining the decolorization of methylene blue in the dark.

## 2 | EXPERIMENTAL PROCEDURE

### 2.1 Titania nanoparticles synthesis

Titania nanoparticles (TiO<sub>2</sub>) were synthesized using three methods: *Method A*: by mixing 2.5 mL of titanium isopropoxide (TIP, Ti[OCH(CH<sub>3</sub>)<sub>2</sub>]<sub>4</sub>, > 99%, Acros Organics) in 50 mL of isopropanol (IP, 99.5%, Vetec) or Acetic Acid (HAc, 99.5%, Vetec). The solution was poured into an autoclavable bottle. The bottle was then placed in a regular laboratory oven at a constant 100 °C for 48h.

*Method B*: by mixing 2.5 mL of titanium isopropoxide and 8 mL of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub> 10% V/V, > 99%, Acros Organics), was dissolved in 50 mL of isopropanol. The solution was poured into an autoclavable bottle. The bottle was then placed in a regular laboratory oven at a constant 100 °C for 48h.

*Method C*: TiO<sub>2</sub> was obtained by mixing 2.5 mL of TIP and 8 mL of H<sub>2</sub>O<sub>2</sub>, which was dissolved in 50 mL of Acetic Acid. The solution was poured into an autoclavable bottle. The bottle was then placed in a regular laboratory oven at a constant 100 °C for 48h. A gel was formed and it was hydrolyzed by adding 50 mL of milliq water. The bottle

was then placed again in the oven at a constant 100 °C for 24h.

## 2.2 Characterizations

X-Ray diffraction patterns were carried out with a Rigaku diffractometer, D-Max 2500 2500PC, Japan with Cu K $\alpha$  radiation in the  $2\theta$  range from 5° to 75° in a continuous scan of 0.02°/min, CuK $\alpha$  radiation. Measurements of the surface area, hysteresis curve, and pore size distribution of the TiO<sub>2</sub> samples were obtained by nitrogen adsorption/desorption analysis. A Gaussian function was used to fit the pore size distribution curve. For the HRTEM/TEM (200 kV, model CM200; Philips, Holland) study, a drop of the powder suspension was deposited on a carbon-covered nickel grid. The Raman and infrared spectra were obtained using a Bruker RFS-100/S Raman spectrometer and Bruker Equinox-55. UV–vis spectroscopy for the spectra of optical absorbance in disordered and crystalline TiO<sub>2</sub> powders was taken using Cary 5G equipment. All measurements in this work were taken at room temperature.

## 2.3 Decolorization of methylene blue

A solution of 0.5 mM methylene blue was prepared and used in this study. The decolorization of methylene blue was first examined in the presence of pretreated TiO<sub>2</sub>. TiO<sub>2</sub> powder (50 mg) was incubated in a 110-mM H<sub>2</sub>O<sub>2</sub> solution (10 mL) in the dark. The suspension was then centrifuged at 5000 rpm for 10 min. All solutions were incubated in the dark with agitation. After this process solutions were analyzed according to the change of time, which was measured with a spectrophotometer (Cary 300) at 661 nm.

## 3 | RESULTS AND DISCUSSION

In this work, it was shown that TiO<sub>2</sub> nanocrystal formation in a highly crystalline phase is strongly dependent on the reaction medium. An increase of crystallinity of the anatase phase of TiO<sub>2</sub> was observed when isopropanol (IP) was replaced by acetic acid (HAc) (as reaction medium), and did not depend upon whether H<sub>2</sub>O<sub>2</sub> was used or not. With IP, the reaction occurs faster than with HAc. With IP, a nanocrystalline powder (anatase) was obtained as a solid in a solution of IP after 48h of reaction (at 100 °C). However, after 48h of reaction with HAc (at 100 °C), the solution turned into a gel, and hydrolysis occurred after the addition of water. The bottle was then placed in the oven at a constant 100 °C for a further 24h. In this case, the hydrolysis rate occurred slowly, which increased the crystallinity of TiO<sub>2</sub>.

The same procedure was carried out to obtain TiO<sub>2</sub> in the presence of IP or HAc, but without H<sub>2</sub>O<sub>2</sub>. The aim of these syntheses was to compare the formed products and discover the real role of H<sub>2</sub>O<sub>2</sub> and whether the solutions had the same behavior as when H<sub>2</sub>O<sub>2</sub> was used.

Fig. 1 shows the patterns of X-ray diffraction of the synthesized TiO<sub>2</sub> samples

obtained in presence and absence of  $H_2O_2$ . Fig. 1 (a and b) shows the patterns of X-ray diffraction of the synthesized  $TiO_2$  samples beginning with titanium isopropoxide, then hydrogen peroxide, isopropanol and acetic acid.

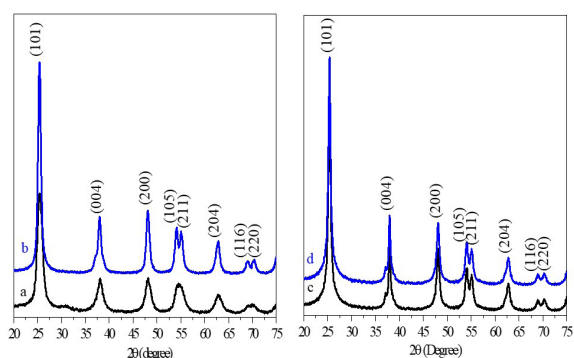


Figure 1. XRD patterns of  $TiO_2$  obtained in the presence of  $H_2O_2$  using (a) isopropanol and (b) acetic acid and in absence of  $H_2O_2$  using (c) isopropanol and (d) acetic acid, as reaction medium.

No peaks corresponding to the rutile or brookite phases were observed, which indicates that the  $TiO_2$  powders obtained had a monophasic anatase structure (PDF #21-1272 anatase  $TiO_2$ ). The crystal structure of the  $TiO_2$  powders was not affected by changing the reaction medium. The narrow diffraction peaks suggest that the  $TiO_2$  that was obtained was nano sized. The average crystalline sizes of anatase phase  $TiO_2$  particles can be calculated by applying Scherrer's equation to the anatase (101) diffraction peak, as shown in Table 1.

	Sample	crystallite size (nm) <sup>a</sup>	Superficial area BET ( $m^2 g^{-1}$ )	Porous volume ( $cm^3$ )	Porous size (nm)
With $H_2O_2$	$TiO_2 - IP$	10.12	192.81	0.46	9.56
	$TiO_2 - HAc$	12.16	157.89	0.29	7.34
Without $H_2O_2$	$TiO_2 - IP$	14.06	148.73	0.22	6.47
	$TiO_2 - HAc$	15.52	129.39	0.21	5.38

Table 1. Texture Data of  $TiO_2$  samples.

<sup>a</sup> calculated using Scherrer's equation

$TiO_2$  obtained without  $H_2O_2$  (Fig. 1 (c and d)) had the same profile as DRX (not shown). The  $TiO_2$  that was obtained was identified as pure anatase  $TiO_2$  (PDF #21-1272 anatase  $TiO_2$ ) and, no peaks corresponding to any other phase were observed, which indicates that the obtained  $TiO_2$  powders also exhibited a monophasic anatase structure. The average crystalline sizes were calculated by applying Scherrer's equation to the anatase (101) diffraction peak as shown in Table 1.

Changing the reaction medium from IP to HAc improved the crystallinity of  $TiO_2$



powders (Fig. 1 and 2). However, the titania obtained without  $H_2O_2$  had a low amount of amorphous titania. This is due to the fast hydrolysis of titanium salt, which results in a fast precipitation of  $TiO_2$ , and a powder with low crystallinity.

In order to further characterize the crystalline phase of the calcined  $TiO_2$ , FT-Raman measurement was performed. The pattern is shown in Fig. 2. The FT-Raman spectrum for  $TiO_2$  obtained with  $H_2O_2$  clearly shows a strong band at  $148\text{ cm}^{-1}$  and four other bands at  $200$ ,  $398$ ,  $516$  and  $638\text{ cm}^{-1}$  which arose from the optical vibration modes represented as  $E_g(\nu_6)$ ,  $E_g(\nu_5)$ ,  $B_{1g}(\nu_4)$ ,  $A_{1g} + B_{1g}(\nu_2 + \nu_3)$  and  $E_g(\nu_1)$  [HAO; ZHANG, 2008]. They can be assigned the characteristic pattern for pure anatase without the presence of rutile or brookite phase [LIU et al., 2006], which is consistent with the XRD analysis.

The FT-Raman spectrum for  $TiO_2$  obtained without  $H_2O_2$  had a similar profile to the one with  $H_2O_2$ .

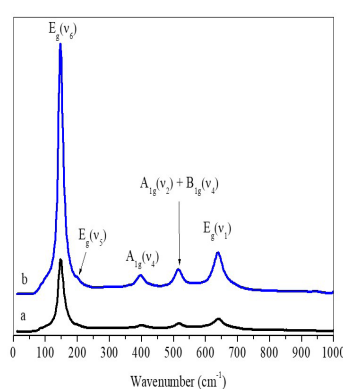


Figure 2. Raman spectra for  $TiO_2$  (at room temperature) obtained in the presence of  $H_2O_2$  using isopropanol (a) or acetic acid (b) as reaction medium.

The morphology and average size of the synthesized pure nanocrystalline anatase  $TiO_2$  as a function of the reaction medium were investigated in detail using electronic microscopy (FEG-STEM). Fig. 3 shows the STEM micrographs of the anatase  $TiO_2$  samples synthesized with hydrogen peroxide in isopropanol as reaction medium for a constant reaction time (48h) (Fig. 3a and 3b) and without  $H_2O_2$  (Fig. 3c and 3d). The STEM images of all the samples show an irregular spherical shape (Fig. 3a) and rod-like structures were obtained in samples, both in the presence (Fig. 3b) and absence (Fig. 3a and 3b) of  $H_2O_2$ .

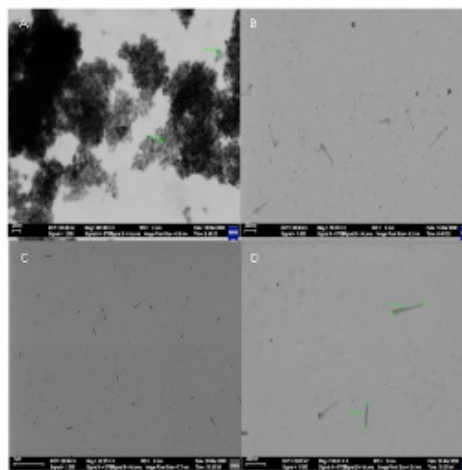


Figure 3. SEM images of  $\text{TiO}_2$  samples obtained in the (a e b) presence and (c and d) absence of  $\text{H}_2\text{O}_2$  and using isopropanol as reaction medium.

The irregular spherical nanocrystals are about 7 and 9 nm in diameter, as shown in Fig. 3a. The rods in the sample obtained with  $\text{H}_2\text{O}_2$  are about 39 nm in diameter and 495 nm long, as shown in Fig. 3b. However, the diameters of the rods in the sample obtained without  $\text{H}_2\text{O}_2$  are much smaller, reaching a maximum of about 27 nm and 156 nm long, as shown in Fig. 3(c and d). The crystallite shapes turn into nanorods with different sizes. We believe that the mechanisms of crystal growth are influenced by the presence of hydrogen peroxide. In its absence, the effect of the starting material can be explained as follows: The titanium salt is surrounded by a large amount of  $\text{OH}^-$  and the nucleation of  $\text{Ti}(\text{OH})_x$  is fast, whereas the particle growth is restricted by surrounding  $\text{OH}^-$ , which results in small  $\text{Ti}(\text{OH})_x$  crystallites. The freshly formed titanium hydroxide decomposes rapidly and thus  $\text{TiO}_2$  nanocrystals are produced. In the presence of  $\text{H}_2\text{O}_2$ , the starting material is titanium peroxide, the  $\text{OH}^-$  concentration around  $\text{Ti}^{4+}$  decreases, and the nucleation of  $\text{Ti}(\text{OH})_x$  slows down. As a result, the  $\text{TiO}_2$  produced is more irregular in shape and much larger in size.

Fig. 4 shows the STEM micrographs of the anatase  $\text{TiO}_2$  samples synthesized with hydrogen peroxide in acetic acid as reaction medium, for a constant reaction time (48h), and for an additional 24h (Fig. 4a and 4b). It also shows the same process when  $\text{H}_2\text{O}_2$  is not used (Fig. 4c and 4d). The STEM images of all the samples have an irregular spherical shape (Fig. 4a and 4b), a rod-like shape (Fig. 4b) and nanotube structures were also obtained too (Fig. 4b). In the absence of  $\text{H}_2\text{O}_2$ , irregular rods were obtained (Fig. 4c and 4d).

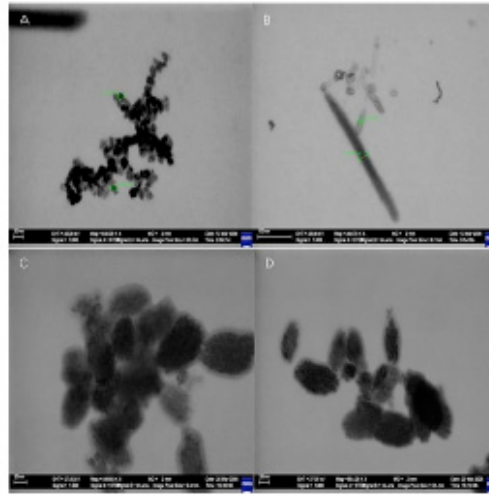


Figure 4. SEM images of  $\text{TiO}_2$  samples obtained in the (a e b) presence and (c and d) absence of  $\text{H}_2\text{O}_2$  and using acetic acid as reaction medium.

The irregular spherical nanocrystals obtained in with  $\text{H}_2\text{O}_2$  were about 10 and 20 nm in diameter, as shown in Fig. 4a. The rods were about 26 nm in diameter and 355 nm long, as shown in Fig. 4b, while the nanotubes were about 5nm in diameter and between 16 to 97 nm long. When HAc was used as a reaction medium, the diameters of the rods obtained without  $\text{H}_2\text{O}_2$  were about 579 nm long (Fig. 4c and 4d). When IP was used as the reaction medium, the crystallite shapes turned into nanorods of different sizes and shapes. Once again, the mechanisms of crystal growth were influenced by the presence of the hydrogen peroxide. The effect of the presence or absence of hydrogen peroxide on the starting material can be explained by the same principle when isopropanol is used as a solvent. But when HAc is used as a solvent, the reaction medium behaves very differently. With the formation of gel, the titanium salt is not surrounded by  $\text{OH}^-$  ions and the nucleation of  $\text{Ti}(\text{OH})_x$  does not occur. After the addition of water, the titanium salt is surrounded by a large amount of  $\text{OH}^-$  that promotes a nucleation of  $\text{Ti}(\text{OH})_x$ . Although the particle growth will be restricted by the surrounding  $\text{OH}^-$ , the slow formation of  $\text{Ti}(\text{OH})_x$  results in crystals of irregular shapes and sizes. As a result, large crystals are obtained, especially in the absence of  $\text{H}_2\text{O}_2$ .

Fig. 5 (a and b) shows the diffuse reflectance spectra of the anatase phase of titania.  $\text{TiO}_2$  is an indirect semiconductor [KOFFYBERG et al., 1979] so that the band gap energy ( $E_g$ ) of the samples can be determined from the tangent lines to the plots of the modified Kubelka-Munk function,  $[F(R^\infty)hn]^{1/2}$ , versus the energy of the exciting light [KIM et al., 1993].

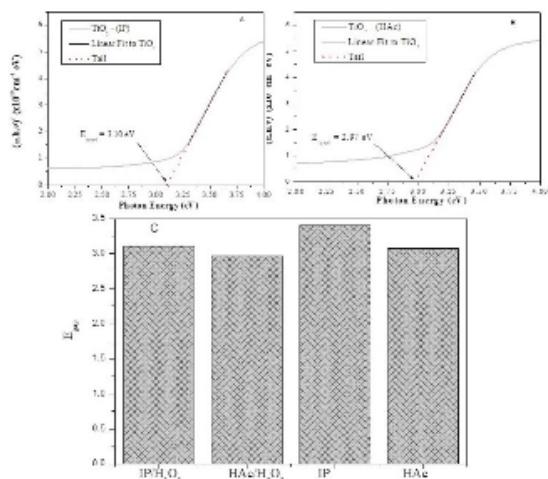


Figure 5. UV-vis absorbance spectra for TiO<sub>2</sub> (at room temperature) obtained in the presence of H<sub>2</sub>O<sub>2</sub> using isopropanol ((a) - TiO<sub>2</sub>-IP) or acetic acid ((b) - TiO<sub>2</sub>-HAC) as reaction medium. And (c) Band gap energies for TiO<sub>2</sub> obtained in the presence and absence of H<sub>2</sub>O<sub>2</sub> using isopropanol (IP) or acetic acid (HAc) as reaction medium.

As shown in Fig. 5c, the E<sub>g</sub> values of TiO<sub>2</sub> were 3.10 and 2.97 eV for TiO<sub>2</sub> obtained in the presence of H<sub>2</sub>O<sub>2</sub>, in isopropanol (IP/H<sub>2</sub>O<sub>2</sub>) and acetic acid (HAc/H<sub>2</sub>O<sub>2</sub>), respectively. The band gaps obtained in this work are in line with data found in scientific literature [KUMAR; BADRINARAYANAN; SASTRY, ZHANG et al., 2007, CHOI et al., 2017] The absorption edge shifts towards shorter wavelengths for the TiO<sub>2</sub> obtained with HAC, which clearly indicates a decrease in the bandgap of the TiO<sub>2</sub> particles obtained with IP. The larger band gap of the TiO<sub>2</sub> - (IP) nanocrystals can be attributed to the quantum size effect. This is because the as-prepared TiO<sub>2</sub> - (IP) nanoparticles have a smaller crystalline size. The E<sub>g</sub> values of TiO<sub>2</sub> obtained without H<sub>2</sub>O<sub>2</sub> (Fig. 5c) were 3.07 and 3.40 eV, for TiO<sub>2</sub> obtained in IP and HAC, respectively

Fig. 6(a, b) shows the nitrogen adsorption–desorption isotherms of TiO<sub>2</sub> – (IP) and TiO<sub>2</sub> – (HAC) samples. It can be seen that both are type IV isotherms with a H1 hysteresis loop (according to IUPAC classification), which is a typical adsorption for mesoporous materials with one-dimensional cylindrical channels [SING et al., 1985].

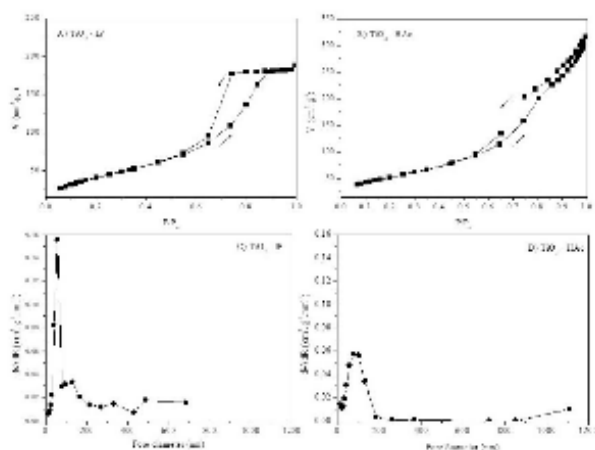


Figure 6. Adsorption-desorption N<sub>2</sub>-isotherms (a and b) and pore size distribution (c and d) from TiO<sub>2</sub> obtained in the presence of H<sub>2</sub>O<sub>2</sub> using isopropanol (a and c) or acetic acid (b and d) as reaction medium.

Furthermore, a well-defined step occurs at the relative pressure of 0.6–0.8, which indicates that the filling of the uniform mesopores is due to capillary condensation [GREGG; SING, 1982]. The position at which the inflection begins is clearly related to the pore size, and the sharpness of these steps indicates the uniformity of the mesopore size distribution [GREGG; SING, 1982]. The specific surface areas and pore parameters of the samples are summarized in Table 1. It can be seen that the  $\text{TiO}_2$  – IP powder shows a large  $S_{\text{BET}}$  value of  $192.8 \text{ m}^2 \text{ g}^{-1}$  and pore volume value of  $0.46 \text{ cm}^3 \text{ g}^{-1}$ . However, the specific surface area, porosity, and pore volume all decrease when the  $\text{TiO}_2$  is obtained with HAc (see Table I). The pore size distribution curves obtained from the desorption branch of the nitrogen isotherm by the BJH (Barrett–Joyner–Halenda) method are shown in Fig. 8c and d. It can be seen that the  $\text{TiO}_2$  – IP has an average pore size of 9.6 nm, and with  $\text{TiO}_2$  – HAc, the average pore size is 7.4 nm.

The specific surface areas and pore parameters of the samples obtained without  $\text{H}_2\text{O}_2$  are summarized in Table I. The  $\text{TiO}_2$  – IP powder shows a high  $S_{\text{BET}}$  value of  $148.7 \text{ m}^2 \text{ g}^{-1}$  and pore volume value of  $0.22 \text{ cm}^3 \text{ g}^{-1}$ .

The methylene blue (MB) decolorization experiments in water, presence of  $\text{H}_2\text{O}_2$  and absence of light were conducted using  $\text{TiO}_2$  as catalytic system Fig. 7.

Fig. 7 show evolution of decolorization of MB in the presence of several catalyst systems.  $\text{TiO}_2$  obtained in HAc and presence of  $\text{H}_2\text{O}_2$  was the better catalytic system. During incubation, approximately 50% of the MB was degraded in the presence of  $\text{TiO}_2$ -HAc (obtained in presence of  $\text{H}_2\text{O}_2$ ) within 26min, and more than 90% decolorization was obtained in the presence within 2h. To investigate the effect of adding  $\text{H}_2\text{O}_2$  well as well  $\text{TiO}_2$  to the catalytic process of MB, two experiments were carried out: 1) MB in the presence of  $\text{H}_2\text{O}_2$  and absence of  $\text{TiO}_2$  and 2) MB in absence of  $\text{H}_2\text{O}_2$  and  $\text{TiO}_2$ . No catalytic activity was observed.

Under the experimental conditions used in this study, we investigated the activity of  $\text{TiO}_2$  obtained from Degussa (P25). Fig. 7 shown that in presence of P25 approximately 40% of MB was decolorized within 2h. All others  $\text{TiO}_2$  systems reported in this work did not present significantly catalytic activity.

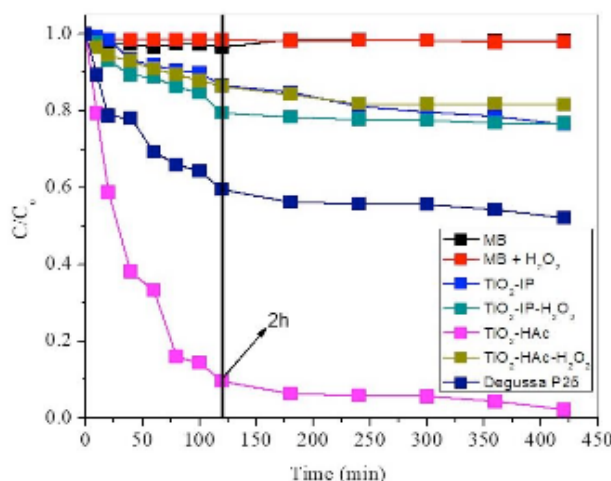


Figura 7. Decoloração do azul de metileno na presença de  $\text{TiO}_2$  e  $\text{H}_2\text{O}_2$ .

## 4 | CONCLUSIONS

The following can be concluded from the results presented in this paper: a method for the preparation of TiO<sub>2</sub> nanoparticles with a large surface area and an exclusively anatase crystal structure has been demonstrated. The successful characterization of the synthesized particles revealed that the nanometer regime and the size of crystals were ~ 7 – 579 nm (~ 5 – 39 nm in diameter and ~16 – 579 in length), estimated using the Scherrer formula. This data is also supported by STEM micrograph evidence. The synthesized TiO<sub>2</sub> has a large surface area, 192 m<sup>2</sup>/g as estimated by the BET method, which is an important property for catalytic, photocatalytic, and gas sensor applications. The particles showed a concomitant blue shift in the absorption spectra. The bandgap energy observed for different TiO<sub>2</sub> was 3.10 and 2.97 for TiO<sub>2</sub> obtained with IP and HAc, respectively, when H<sub>2</sub>O<sub>2</sub> was used; and 3.07 and 3.40 for TiO<sub>2</sub> obtained with IP and HAc, respectively, when H<sub>2</sub>O<sub>2</sub> was not used. A TiO<sub>2</sub> catalyst was activated in a solution of H<sub>2</sub>O<sub>2</sub>, and the decolorization of methylene blue was improved in the presence of these pretreated particles. Decolorizing ratio of approximately 100% was obtained within 7h incubation period in the presence of TiO<sub>2</sub> obtained in HAc and presence of H<sub>2</sub>O<sub>2</sub>.

## 5 | ACKNOWLEDGEMENTS

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