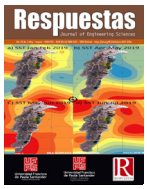

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Color removal of indigo carmine dye solutions using fique fibers modified with ZnO nanoparticles

Eliminación del color de las soluciones de tinte índigo carmín utilizando fibras fique modificadas con nanopartículas de ZnO

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ABSTRACT

Zinc oxide is a useful and recyclable catalyst. In this study, fique fibers were modified with zinc oxide (ZnO) nanoparticles to remove color from indigo carmine (IC) solutions. ZnO nanoparticles were synthesized by precipitation method and the fibers were ex-situ and in-situ modified. The fibers and the nanoparticles were characterized using different techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV–visible spectroscopy and Scanning Electron Microscopy (SEM). The color removal rate was monitored by using an UV/Vis spectrophotometer. Nanoparticles with a mean diameter in the nanoscale and a typical hexagonal structure were obtained, and they were effectively deposited on the fibers. The highest color removal was obtained with the ex-situ fibers (ZnO-Ex/fique) 90 % in 180 minutes. Color removal by in-situ fibers (ZnO-In/fique) was 70 % after 180 min. From the results, ZnO nanoparticles may be an excellent catalyst for removal IC dye aqueous solutions under UV-C light.

Keywords:

color remove,
fique fibers,
zinc oxide nanoparticles,
in-situ synthesis,
ex-situ impregnation,
indigo carmine.

RESUMEN

El óxido de zinc es un catalizador útil y reciclable. En este estudio, las fibras de fique se modificaron con nanopartículas de óxido de zinc (ZnO) para eliminar el color de las soluciones de índigo carmín (IC). Las nanopartículas de ZnO se sintetizaron por método de precipitación y las fibras se modificaron ex situ e in situ. Las fibras y las nanopartículas se caracterizaron utilizando diferentes técnicas como difracción de rayos X (DRX), espectroscopia infrarroja por transformada de Fourier (FTIR), espectroscopía de luz visible (UV-vis) y microscopía electrónica de barrido (MEB). La velocidad de eliminación del color se controló usando un espectrofotómetro UV/Vis. Se obtuvieron nanopartículas con un diámetro medio en la nanoescala y una estructura hexagonal típica, y se depositaron efectivamente en las fibras. La mayor eliminación de color se obtuvo con las fibras ex situ (ZnO-Ex / fique) fue de 90% en 180 minutos. La eliminación del color por fibras in situ (ZnO-In / fique) fue del 70% después de 180 min. A partir de los resultados, las nanopartículas de ZnO pueden ser un excelente catalizador para la eliminación de soluciones acuosas de colorante IC bajo luz UV-C.

Palabras clave:

remoción de color,
fibras de fique,
nanopartículas de óxido de zinc,
síntesis in situ,
impregnación ex situ,
carmín índigo.

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Introducción

Environmental pollution is one of the problems that most countries around the world are facing. The pollution has generated negative effects on human health and nature [1–3]. Wastewater is mainly produced by the textile industry during the dyeing processes. The volume and composition of effluents from the textile industry are important polluting residues in all industrial sectors [4]. During coloration of fibers, the dyes (indigo) and dyes sulfides are also very important for the world market (around 31%). Indigo occupies 7%, representing about 120,000 tons of dyes used annually [5]. Several methods have been described in the literature to remove color: adsorption [6], ion exchange, and membrane filtration [7], chemical coagulation [8], chemical and Fenton oxidation [9], among other. Some of those methods are costly and less effective.

Another common technique in wastewater treatment is the photocatalysis. It can remove color quickly by oxidation caused by some materials (catalyst) [10]. Both homogenous photocatalysis and heterogeneous photocatalysis have been used for dye removal. Homogenous photocatalysis is the ability to absorb photons, so the contaminant and light can also lead to chemical modification of the substrate. Heterogeneous photocatalysis is a process based on the direct or indirect absorption of radiant energy (visible or UV) by a solid (heterogeneous photocatalyst is usually a broadband semiconductor) [11]. Several semiconductor materials have been used for photocatalysis. ZnO [12], SnO_2 [13], TiO_2 [14], Bi_2O_3 [15], Al_2O_3 [16] have been studied to remove color from dye solutions. Among these catalysts, ZnO is a photocatalyst widely used due to its outstanding stability, inexpensiveness, low toxicity and strong photoactivity [17,18].

On the other hand, wastes obtained from lignocellulosic materials have been used as a sustainable option for wastewater dye removal. Those residues include peels [19], natural fibers [20], bagasse [21], carbonaceous materials [22], among others. Therefore, a good option to achieve an acceptable level of purification would be used as an ecological and low-cost support. One of the most appropriate options are natural fibers since they have properties flexibility, simplicity of design, ease of operation and low initial cost.

Fique fibers are native to Colombia and they have high contents of cellulose (63–70 %). They also have a heterogeneous surface with high oxygen density that facilitates metal oxide nanoparticle growth and stabilization [23]. Many studies have reported the synthesis of ZnO nanoparticles but only a few shows the synthesis of ZnO nanoparticles onto fiber substrates [23–25]. However, to the best of the authors knowledge there are no applications of fique fibers modified with ZnO to remove color from IC solutions. Accordingly, the aim of this work is to modify fique fibers with ZnO nanoparticles (ZnO-Ex/fique and ZnO-In/fique) to remove color from indigo carmine dye solutions.

Materiales y Métodos

Fique fibers

Raw fique fibers are commercially available in Medellín, Antioquia, Colombia. During cleaning, the fibers were combed to remove vegetable residues (e.g. waxes, chlorophyll). The procedure was performed using water, and the fibers were dried overnight at room temperature. All chemicals used for the experiments are of analytical grade and they were used without any further purification. Aqueous solutions were prepared using deionized (DI) water, with conductivity < 0.4 uS.

Fiber pretreatment

Clean fibers were immersed into a solution of sodium hydroxide (NaOH) (PanReac, 98%). This procedure was done to remove partially the lignin and to expose the cellulose. Fibers were placed in a 6 % W/V NaOH aqueous solution for 3 h at 80 °C, similar to the process reported by Li [26] and Wang [27]. After this treatment, alkali cellulose is obtained. Cationized fibers were washed with deionized water, dried at 60 °C and used as a support for the ZnO synthesis [23].

In-situ synthesis of ZnO nanoparticles

Zinc acetate dehydrate ($Zn(O_2CCH_3)_2$ $(H_2O)_2$), MERCK, ≥ 99.5 %) was used as precursor to obtain the ZnO nanoparticles. The procedure used by Khosravian et al. [24] was modified by adding sonication and modifying the Zn precursor. The cationized fique fibers previously treated were submerge in a solution of NaOH at 15% for 5 min. Afterwards, fibers were immersed into a solution of zinc acetate dehydrate at 2 % W/V. It was heated at 80

°C under sonication. Finally, the fibers were washed with DI and dried at 80 °C for 24 h.

Impregnation (ex-situ) of ZnO nanoparticles on fique fibers

For the impregnation process, 40 mg ZnO of nanoparticles were dispersed in 20 mL of deionized water and sonicated for 30 min at room temperature (24 °C ± 2 °C) to prepare the impregnation solution. Then, the pretreated fique fibers were immersed in this solution for 1 h at room temperature with continuous stirring (100 rpm). Afterwards, the fibers were washed with deionized water and dried at room temperature. The ZnO/fique fibers were analyzed by using SEM and EDS, in order to confirm the incorporation of ZnO nanoparticles on treated fiber [28].

Materials characterization

The morphology of the nanoparticles was characterized by using Scanning Electron Microscopy (SEM, JEOL JSM-7100) and the chemical composition was measured by Energy-Dispersive Spectrometry (EDS). The presence of functional groups in treated and ZnO/ fique fibers was investigated by using Fourier Transform Infrared Spectroscopy (FTIR) in a Shimadzu IR Tracer-100 spectrometer, the Attenuated Total Reflection (ATR) mode was used.

After synthesis, the remaining solution was tested via UV – Vis spectrophotometer to detect ZnO nanoparticles in solution (UV-Vis 8453th) and this solution was centrifuged and dried at 80 °C for 24 h in order to obtain the XRD spectra of ZnO. XRD patterns of the NPs were determined by X-ray diffraction (XRD) (PANalytical X'Pert PRO diffractometer). The samples were scanned in the range of 2Θ = 20 – 80° at a scanning speed of 0.013 °/s, using Cu Kα radiation at 45 kV and 40 mA.

Heterogeneous photocatalysis evaluation for color removal

Experiments were performed in photoreactor equipped with two commercial UV tubes each of 15W UV-C lamps (LUMEK-15 W), the distance between the lamps and the surface of the solution was 10 cm. Dye solution (50 ppm) was distributed in six beakers of 20 ml and 0.1 g of fiber ZnO/fique each sample. The mixture was stirred in the dark for 60 min to obtain an adsorption-desorption equilibrium. Under a regular interval of time, samples were centrifuged (10000 rpm) before examining the dye absorbance by UV–vis spectrophotometer.

Results and discussion

Characterization of ZnO nanoparticles

UV-visible spectroscopy was used to identify the characteristic absorbance of the nanoparticles. In this work, the technique was used to confirm that ZnO nanoparticles were obtained after synthesis process. In figure 1 the absorption band for ZnO was observed. The band was found in the range of 350–410 nm [29,30]. The spectrum confirmed the existence of ZnO with a peak at 357 nm.

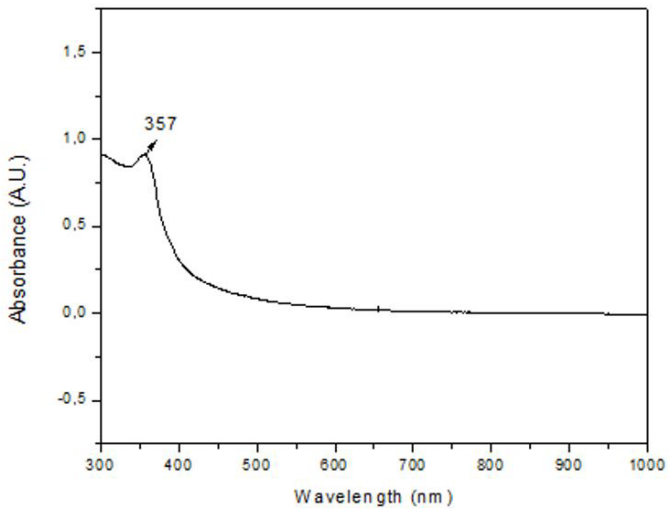

Figure 1. UV–Vis absorption spectrum of ZnO nanoparticles deposited in the fiber.

ZnO nanoparticles were studied by XRD to identify the crystalline structure. Figure 2 shows the X-ray pattern. Diffraction patterns showed the standard diffraction pattern with reference number ICSD (00-036-1451) related to the hexagonal structure of ZnO. The peaks correspond to the reflections from (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) planes were observed at 2θ values around 31.770°, 34.422°, 36.253°, 47.539°, 56.603°, 62.864°, 66.380°, 67.963°, 69.100°, 72.562°, 76.955°, respectively. Moreover, there are no additional peaks that show other phases of the ZnO.

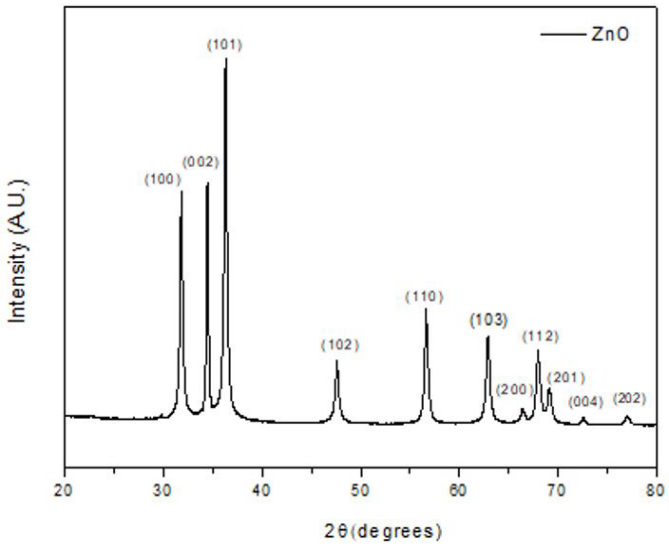

Figure 2. XRD spectra for the ZnO nanoparticles synthesized.

Figure 3a shows the SEM images of the nanoparticles without calcination exhibit an irregular and flat surface structure figure 3a. A spherical morphology was not obtained since the nanoparticles were not calcined to avoid damage of the fibers and this is the probable reason in changing size and morphology, since other authors have shown that a slight change during preparation can affect the characteristics of the nanoparticles [25,31]. In addition, figure 3b shows the calcined nanoparticles. The morphology is very different when they are compared to the nanoparticles without calcination. The calcined nanoparticles have a spherical morphology and a smaller size than the uncalcined nanoparticles. The arrows in the figure 3 indicate some of the nanoparticles impregnated. The mean size measured for ZnO-Ex/fique and ZnO-In/fique nanoparticles was 96.21 nm ± 0.17 nm and 155.58 nm ± 0.28 nm, respectively.

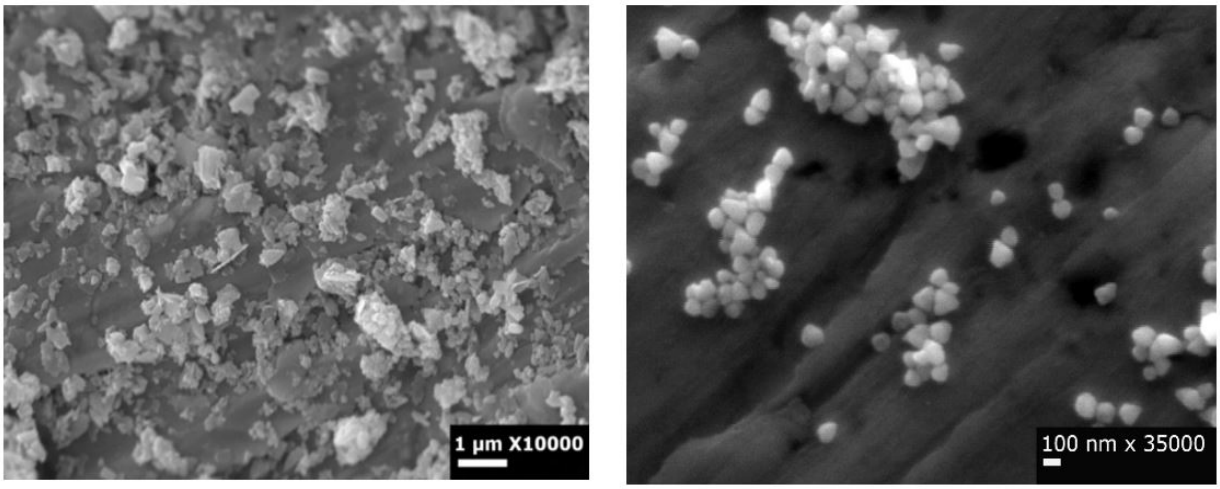

Figure 3. Scanning electron microscope of ZnO, a) Uncalcined nanoparticles (ZnO-In/fique) and b) Calcined nanoparticles (ZnO-Ex/fique).

The nanoparticles are uniformly distributed on the fibers surface figure 4a. The impregnation of ZnO nanoparticles on the treated fiber was further confirmed by the chemical composition analyzed by EDS. Figures 4b-c show that Zn and O are the main elements on the treated fibers; as also, the carbon related to the fiber. The SEM images of ZnO-Ex/fique revealed that the nanoparticles were located on the fiber's surface as was observed in the figure 5.

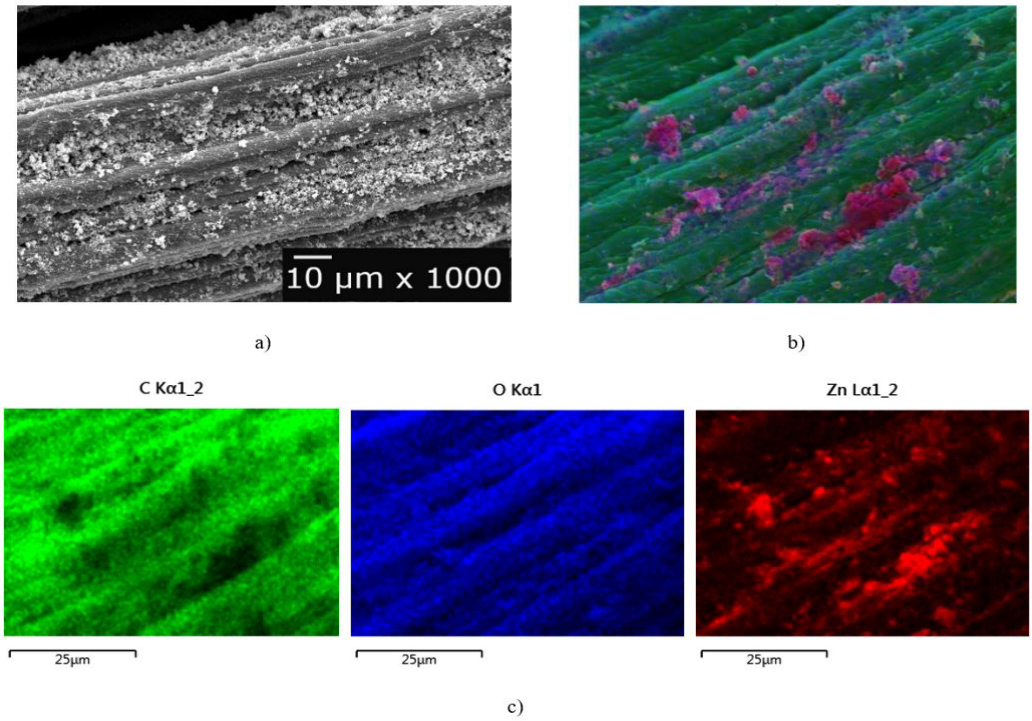

Figure 4. Scanning electron microscope, a) ZnO-In/fique, b) mapping images of ZnO-In/fique and c) EDS spectrum.

Figure 5 shows SEM images of the fibers after impregnating them with the ZnO nanoparticles ZnO-Ex/fique. The nanoparticles impregnated successfully the surface and a uniform distribution in the surface of the fiber was not obtained.

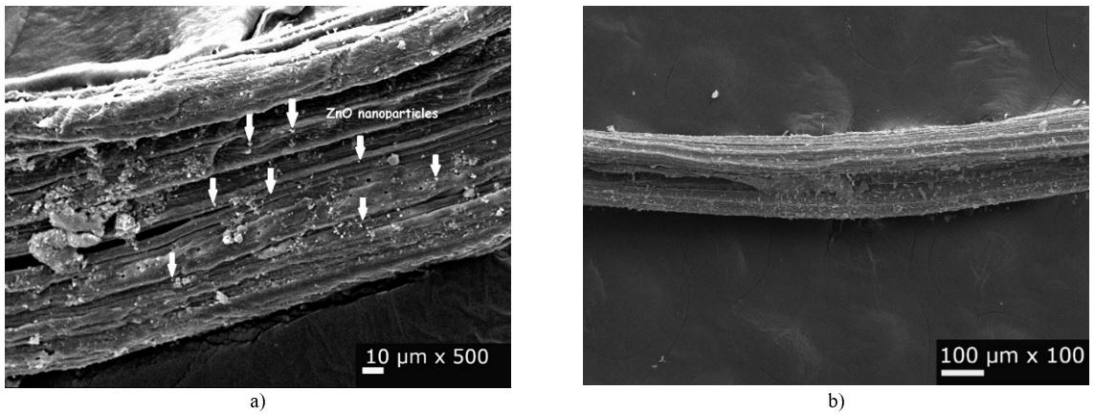

Figure 5. Scanning electron microscope of ZnO-Ex/fique fiber.

IR spectra of treated and ZnO/fique fibers is shown in figure 6. The FTIR spectra of treated fiber shows different signals related to the major constituents of fique fiber (hemicellulose, lignin and cellulose). The peaks ranging from 3250 - 2800 cm-1 are attributed to the vibrations of O-H, H-C-H and C-O-C (glyosidic linkage) groups of cellulose [32,33]. The peak at 1483 cm^{-1} and, the strong and sharp peak at 1100 cm^{-1} corresponded to the C=O vibrations in lignin and hemicellulose [34,35]. The spectra of ZnO/fique shows similar characteristics as treated spectrum except the sharp peak at 453 cm^{-1} corresponding to ZnO nanoparticles [36].

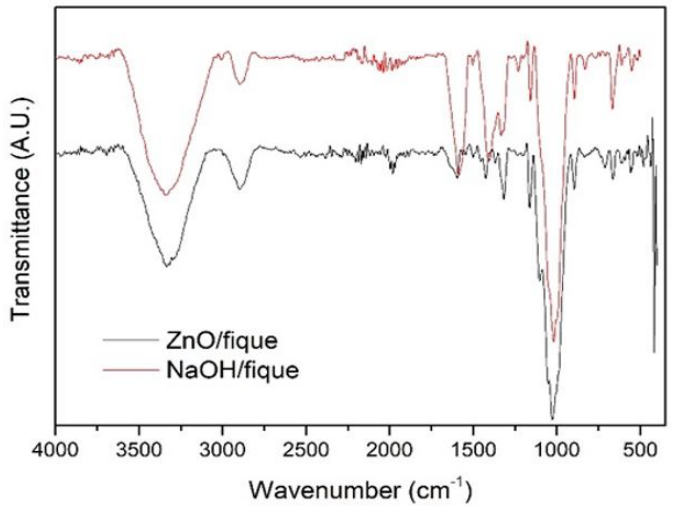

Figure 6. FTIR ZnO/fique and NaOH/fique.

Color removal

In figure 7 the changes in absorbance in the peak related to the IC dye with ZnO-In/fique figure 7a at different times are shown. The control samples were taken at the beginning of the test and then the zero value is represented by 60 min of darkness and the beginning of the irradiation.

Likely, the shift in absorbance during darkness period was caused by an absorption effect. During these 60 min absorption and desorption was happed at the nanoparticles surface. After, a steady period was achieved, and the changes were attributed to the photocatalysis stage. During darkness, no significant color removal was observed for the ZnO nanoparticles. The ZnO-Ex/fique fibers absorbance figure 7b had significant changes compared to the nanoparticles and ZnO-In/fique fibers figure 7c. Accordingly, the highest dye removal was obtained by ZnO-Ex/fique fibers.

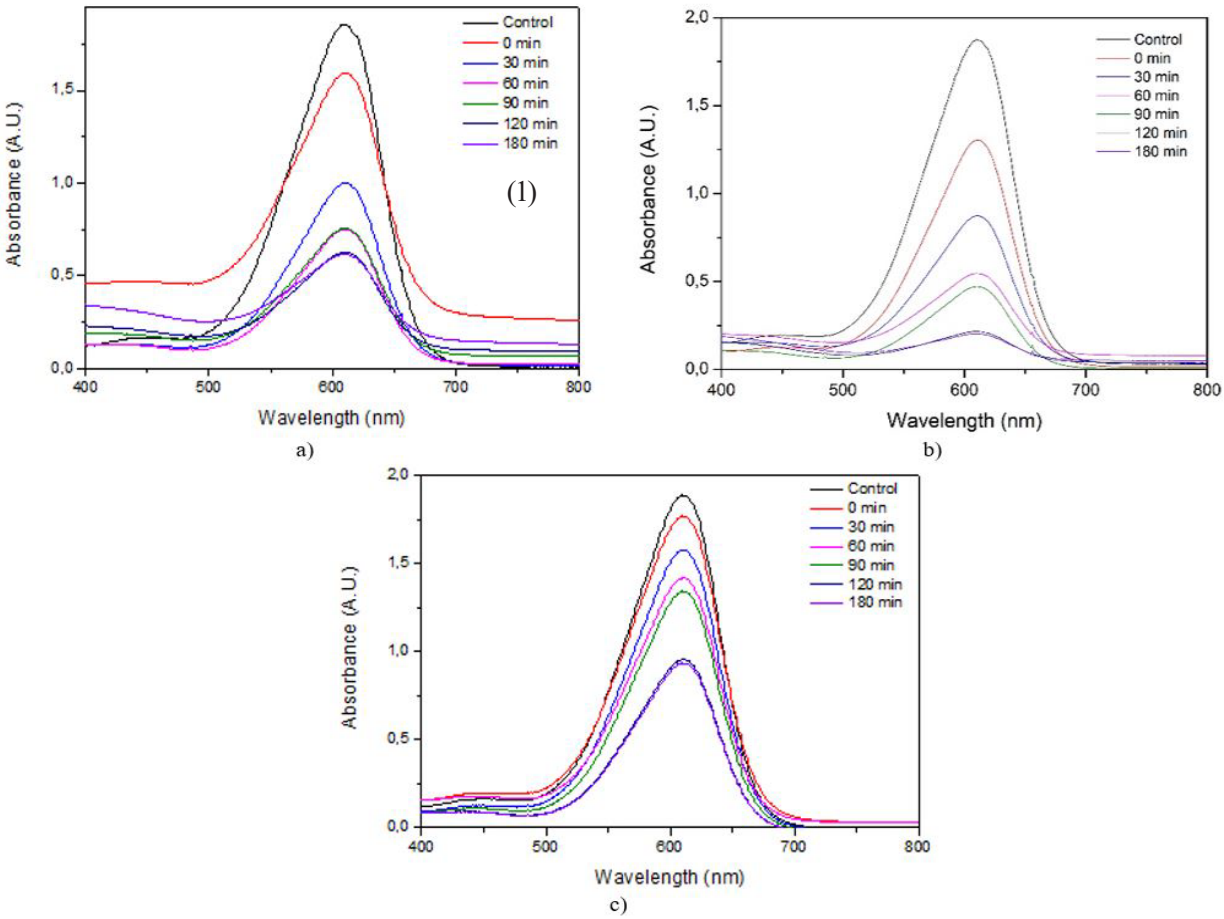

Figure 7. Absorbance spectra of color removal of IC dye with respect to time, a) ZnO-In/fique, b) ZnO-Ex/fique and c) ZnO nanoparticle.

Figure 8 shows the color removal percentage throughout 240 min. After 60 min and the UV lamps were turned on during 180 min (total of 240 min). By the "(1)" was determined the color removal percentage [37]:

$$Color\ removal\ (\%) = \frac{A_0 - A_f}{A_0} \times 100$$

where A_o and A_f are the initial and at pre-set time absorbances of dye indigo carmine, which has an absorption band at 610 nm.

The figure 8a shows that ZnO had high photocatalytic activity to remove color from IC solutions. The nanoparticles (without fibers) could remove the color up to ~ 50% in 180 min. ZnO-In/fique fibers reached 70% and ZnO-Ex/fique fibers remove up to 90% of the color. During the first 60 min, a process of dye absorption by the modified fibers was presented, but then, when the irradiation began, a photo activation of the ZnO was expected to occur.

The ZnO nanoparticles are semiconductor and they can be excited by ultraviolet light. The irradiation allows to obtain photo generated electrons (e−) and holes (h+) at conduction and valence band, respectively. These electric charge carriers can be partially transferred into aqueous solutions to form oxidative radicals. The typical radicals are O2−, OOH and OH. In addition, two mechanisms are important to degrade organic dyes. The first mechanism is the direct decomposition of dye molecules through the oxidative attack of the radicals. Furthermore, the surface-adsorbed dye molecules are degraded through the attack of valence band holes and/or oxidative radicals [38]. The greatest color removal occurred with ZnO-Ex/fique fibers and it was caused by calcined ZnO nanoparticles. In this case, the calcination before impregnation process, improved morphology, size and thereby the surface area of the nanoparticles, and also improved the interactions between the nanoparticles and the dye.

The isoelectric point of ZnO is at pH ~ 9.5 [39]. Accordingly, at low pH values (pH < 9.5), the nanoparticles are positively charged, and at high pH values (pH > 9.5) they are negatively charged. Since IC is an anionic dye, at low pH an electrostatic attraction between negatively charged dye surface and positively charged fiber surface is expected. Thus, dye removal is performed at pH 2.5 for all tests. Meanwhile, as shown in the figure 8b, the color of IC solution varied from blue to colorless in 180 min. Therefore, the nanoparticles impregnated onto fibers surface increase the number of active sites, improving the interaction between the dye and nanoparticles, and the color removal performance [40].

The results obtained are comparable with the results reported by Ali Al-Taie and Hiba Majeed Dah [18], who have worked with ZnO nanoparticles to remove of the IC dye. Moreover, they reported that ZnO is a good photocatalyst for dye removal IC.

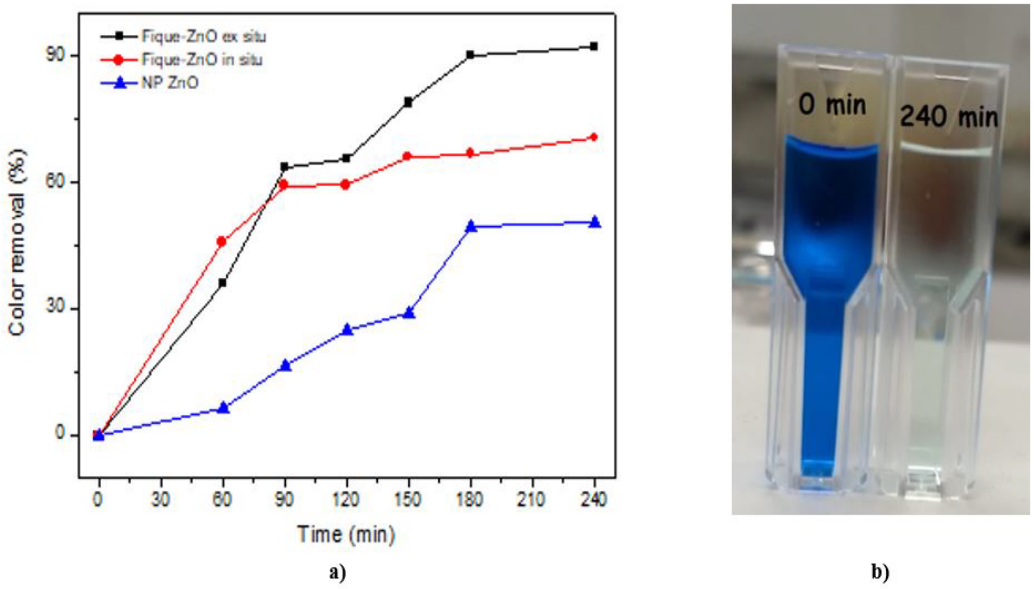

Figure 8. a) Color removal of IC solution at pH 2.5 catalyzed ZnO-Ex/fique fiber and ZnO-In/fique and b) Color removal of IC solution at pH 2.5 catalyzed by ZnO-Ex/fique for 0 min and 240 min.

Conclusions

Two methods were successfully implemented to deposit ZnO nanoparticles on fique fibers. In- situ and ex-situ (impregnation) methods were used and the results were confirmed by the SEM images. The two methods were tested to compare their capacity to color removal from indigo carmine dye solutions.

Fique Fibers modified with ZnO-Ex/fique nanoparticles showed the greatest color removal (90%) and it was attributed to their enhanced crystallinity, morphology and size. It is also known that the smaller the nanoparticles, the better surface area, which provides more active sites for color removal from the dye solution.

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The authors would like to acknowledge to Instituto Tecnológico Metropolitano for financial support and Universidad Nacional for technical support. Programa de Jóvenes Investigadores e Innovadores (Resolution No. 000277 of 04 MAR 2019) and Research group MATyER are also acknowledged.

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