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Green Fabrication and Characterization of Zinc Oxide Nanoparticles using Eucalyptus Leaves for Removing Acid Black 210 Dye from an Aqueous Medium

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Abstract

This study uses an environmentally friendly and low-cost synthesis method to manufacture zinc oxide nanoparticles (ZnO NPs) by using zinc sulfate. Eucalyptus leaf extract is an effective chelating and capping agent for synthesizing ZnO NPs. The structure, morphology, thermal behavior, chemical composition, and optical properties of ZnO nanoparticles were studied utilizing FT-IR, FE-SEM, EDAX, AFM, and Zeta potential analysis. The FE-SEM pictures confirmed that the ZnO NPs with a size range of (22-37) nm were crystalline and spherical. Two methods were used to prepare ZnO NPs. The first method involved calcining the resulting ZnO NPs, while the second method did not. The prepared ZnO NPs were used as adsorbents for removing acid black 210 dye (AB210) from simulated wastewater. The removal efficiency using calcinated and uncalcinated ZnO NPs was 57 % and 59 %, respectively.

Keywords: *Green synthesis; ZnO nanoparticles; Adsorption process; Eucalyptus; Acid Black 210 dye (AB210).*

1. Introduction

In the last few decades, several industries like leather, pharmaceuticals, food, and cosmetics have grown a lot, and all of them make dye effluents. Around 70% of dyes used are azo dyes, which have an aromatic ring-bonded N=N character and are pathogenic and carcinogenic. However, these dyes are poisonous and negatively impact the environment and aquatic communities. As a result, considerable attention has appeared to be focused on finding effective remediation methods for treating dye-contaminated wastewater [1].

Water contaminated with dyes has been removed through adsorption, membrane separation, chemical coagulation and precipitation [1], oxidative-reductive degradation, and electrocoagulation [2][3]. Despite the many methods available for removing dyes, adsorption is the preferred method and offers the best results among different dye removal processes because it can be used to remove various coloring components [4]. Due to their high surface areas, the

other catalysts used as adsorbents in wastewater treatment, such as activated carbon, are considered excellent. However, activated carbon is expensive, requires a long time, and is precocious to prepare. Therefore, using nanomaterials prepared from low-cost and available materials is an excellent alternative to overcome problems associated with other adsorbents [4].

Recently, nanotechnologies have been essential in removing environmental pollutants [1]. Nanoadsorbents provide better adsorption capability than conventional adsorbents due to an increase in specific surface area and higher selectivity since the surface of the nanomaterial can be functionalized to target pollutants specifically [5]. Zinc oxide nanoparticles (ZnO NPs) are flexible semiconductors that exhibit notable optical transparency and luminous capabilities in UV-visible (UV-Vis) regions and have excellent chemical and thermal stability [6]. Additionally, ZnO NPs are easy to prepare, with a high specific surface area and non-toxic nature [6].

Various physical and chemical methods have been employed in ZnO NPs preparation, like milling, vacuum sputtering, microwave-assisted techniques, chemical vapor deposition thermal breakdown, and a chemical reduction process using sodium borohydride as a reducing agent [7]. These methods involve using complex equipment, lengthy procedures, and hazardous chemicals. Therefore, the biological approach is promising for resolving these problems and developing a suitable and safe way to produce nanoparticles. The biological method involves preparation technology based on plant extracts or microorganisms [7]. Green synthesis using plant extract has unique advantages such as being environmentally friendly, taking less time, being more affordable, operating steadily, and, most significantly, carrying out the process without any dangerous chemicals [8].

Different plant extracts, including Cassia fistula, Eucalyptus leaf extract [9], Tea leaf extract [10], Trifolium pretense, Ocimum basilicum, and others, have been employed in the green synthesis of ZnO NPs. have also been used in the production of nanoparticles [11]. The eucalyptus plant is a reliable supplier of bioactive compounds. In the green technique, terpenoids, phenols, and their organs have all been expertly utilized for manufacturing nanoparticles. Also, it is a highly available plant in most countries [12].

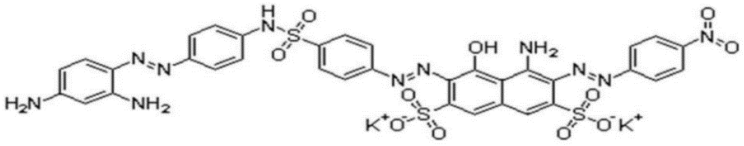
This study focused primarily on two procedures for synthesizing zinc oxide nanoparticles (ZnO NPs) using the extract from the eucalyptus plant; then, it was used to remove an anionic acid black 210 dye (AB210) from wastewater.

2. Materials and Methods

2.1 Chemical and Reagents

CDH company's zinc sulfate hexahydrate (ZnSO4. 7H2O) with a molecular weight of 287.54 g and a purity of >98.0%. (NaOH) was purchased from CDH Company. Absolute ethanol was purchased from Carlo Erba's company. Acid black 210 (AB-210) was taken from a leather factory in Baghdad, Iraq. Table 1 displays the chemical and physical characteristics of the AB210 dye.

Table 1,
Chemical and physical properties of acid black 210 (AB210) [13].

Molecular Formula	$C_{34}H_{25}K_2N_{11}O_{11}S_3$
Melting point	300 ℃
Solublity	Soluble in water
Types of dye	Azo dye (N═N)
Chemical structure	

2.2 Preparation of the leaf extract

The method for the synthesis was taken from [14] with slight modifications. Initially, the eucalyptus leaves were picked up from the yard at Baghdad University and repeatedly washed. For dust and difficult dirt removal, use distilled water. The leaves were then dried for 12 hours at 50 ℃ in the oven. The leaves were ground into a fine powder when thoroughly dried and became

crushable. 150 mL of deionized water was used to wet 10 g of powdered eucalyptus leaves in an Erlenmeyer flask for 30 minutes while stirring at 80 ℃. The resulting extract was cooled and filtered through filter paper; the filtrate was then kept at 4 ℃ until it was used as a reducing and capping agent to reduce zinc ions (Zn+2) into zinc oxide nanoparticles (ZnO).

2.3 Preparation of ZnO NPs

To complete the preparation of ZnO NPs, 1.61 g of solid ZnSO4 was added to 100 mL of deionized water and magnetically stirred at 150 rpm for 10 min. A 0.45 µm membrane filter was used to filter the solution once it had fully dissolved to get rid of contaminants. The ZnSO4 solution was gradually added for 15 minutes at 70 ℃ while being continuously agitated. Eucalyptus extract was fully incorporated before the mixture was stirred at 70 ℃ for 3 hours. The solution's pH was increased to 12. The pH was changed using 1.0 M H2SO4 and 1.0 M NaOH solutions.

The fact that the color of the solution changed from dark brown to light yellow shows that ZnO NPs were made during the reduction process. The yellow ZnO NPs precipitate was taken out with vacuum filtration. Then, it was washed over and over with ethanol and distilled water. In the synthesis, the washing and rinsing procedures are essential because they stop nanoparticles from oxidizing too quickly. The resulting yellow precipitate was dried at 80 ℃ before being crushed using a mortar and pestle into a fine powder. Figure 1 shows below the steps of ZnO NPs preparation.

The other ZnO NPs preparation procedure involved the calcination of the resultant ZnO NPs after filtration and washing. The calcination process was carried out at 400 ℃ for 2 hours [15].

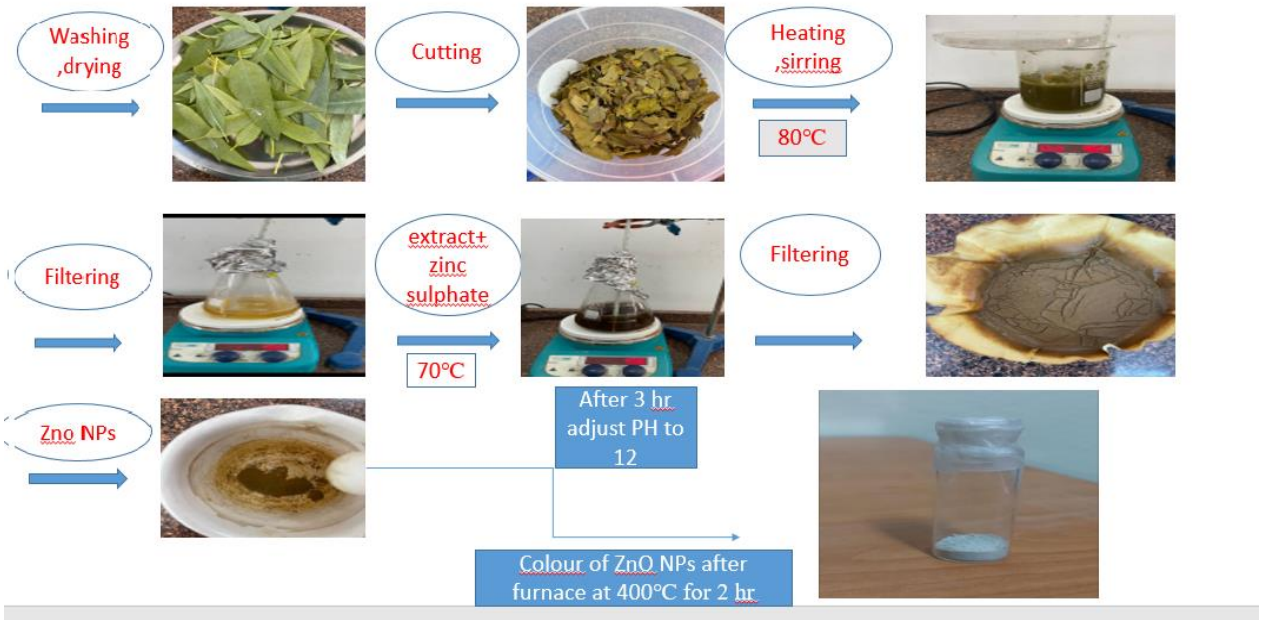


Fig. 1. Schematic diagram of ZnONPs preparation.

3. Results and Discussion

3.1 Characterization of E-ZnO-NPs

Different techniques are used to characterize ZnO NPs to demonstrate the nanoparticles' structure, size, surface area, and chemical constituents. The FE-SEM analysis was used to identify the ZnO NPs structure, morphology, and particle size (Tescan Vega 3, USA). The EDAX technology classifies specific chemicals.

FTIR spectroscopy (Shimadzu,Japan) identifies functional groups of ZnO-NPs. The AFM analysis (TT-2, USA) is used to examine the surface topology, and Zeta potential analyses are an essential tool for evaluating the stability of particles.

3.1.1 Fourier Transform Infrared Spectroscopy (FTIR) analysis

The functional groups of ZnO NPs were characterized by Fourier transform infrared (FT-IR) spectroscopy (Shimadzu, Japan) using a mid-IR spectrum (400-4000cm^{-1}) , as shown in Figure

2. Significant eucalyptus leaf extracts peak to exhibit a broad band at 3414 and 3425.5 cm^{1-} due to the O-H stretching of polyphenols [16]. The following band at 1543 is related to the aromatic C═C stretching [17]. The band at 1481.3 cm^{1-} is caused by the presence of the tertiary alcohol (C-OH) group. A thin, narrow absorption band shows the C-O stretching vibrations of carboxylic acids at 1045.4 cm^{-1} (COOH). Similarly, peaks have been observed at 869.9 cm^{1-} corresponding to the isoprenoids and organic acids in eucalyptus extract [7]. These functional groups show that eucalyptus extract contains a biomolecule that is vital in acting as a capping agent to avoid the aggregation of NPs. The presence of ZnO NPs is demonstrated by the peaks between 439.7 and 416.6 cm^{-1} [18].

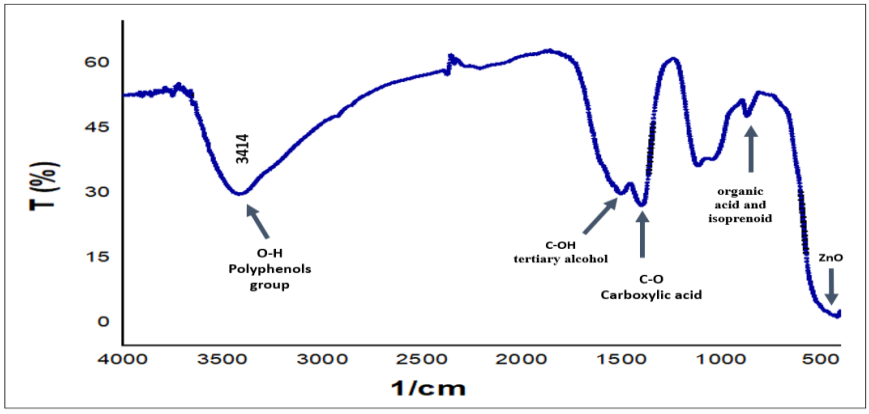


Fig. 2. FTIR of ZnONPs.

3.1.2 Scanning Electron Microscopy (SEM) and Energy Dispersive X- ray Spectroscopy (EDAX) Analysis

SEM analysis of ZnO NPs showed that NPs generally had pores and a spherical shape, with a size ranging from (22 to 37 nm) as shown in Figure 3. Additionally, polyphenol plays a crucial role as a capping agent by covering the surface of ZnO NPs. As a result, only minimal agglomeration is seen in FE-SEM analysis, which prevents the agglomeration of NPs [19]. Moreover, the EDAX analysis illustrated the constituents and chemical composition of E-ZnO-NPs. As shown in Figure 4, according to the ZnO NPs EDAX analysis, the sample comprises zinc, oxygen, and gold. The sample is primarily made up of ZnO, as seen by the zinc and oxygen peaks' high intensities. The weight percents of zinc, oxygen, and gold in the ZnO-NPs sample were 73.4%, 4.619%, and 21.97%, respectively. The atomic percent of zinc was 59.32%, followed by 29.26% for the atomic oxygen percent. However, O indicated the eucalyptus leaf extract's inclusion of polyphenols and other organic compounds [13]. The sample coating used for FESEM imaging caused the gold to be there. Additionally, the EDAX spectrum showed two distinct peaks for zinc at energies of 1 keV and 8.7 keV and a single peak for oxygen at a wavelength of 0.5 keV, indicating the presence of Zn as ZnO-NPs [15].

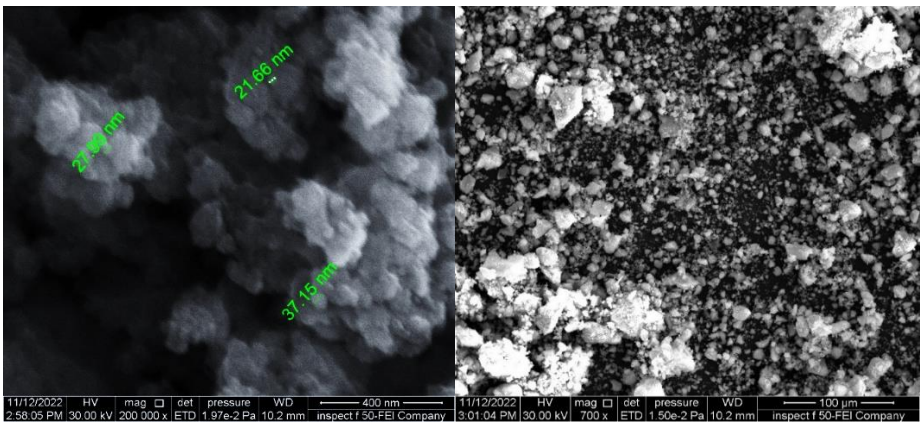


Fig. 3. SEM image of ZnO NPs.

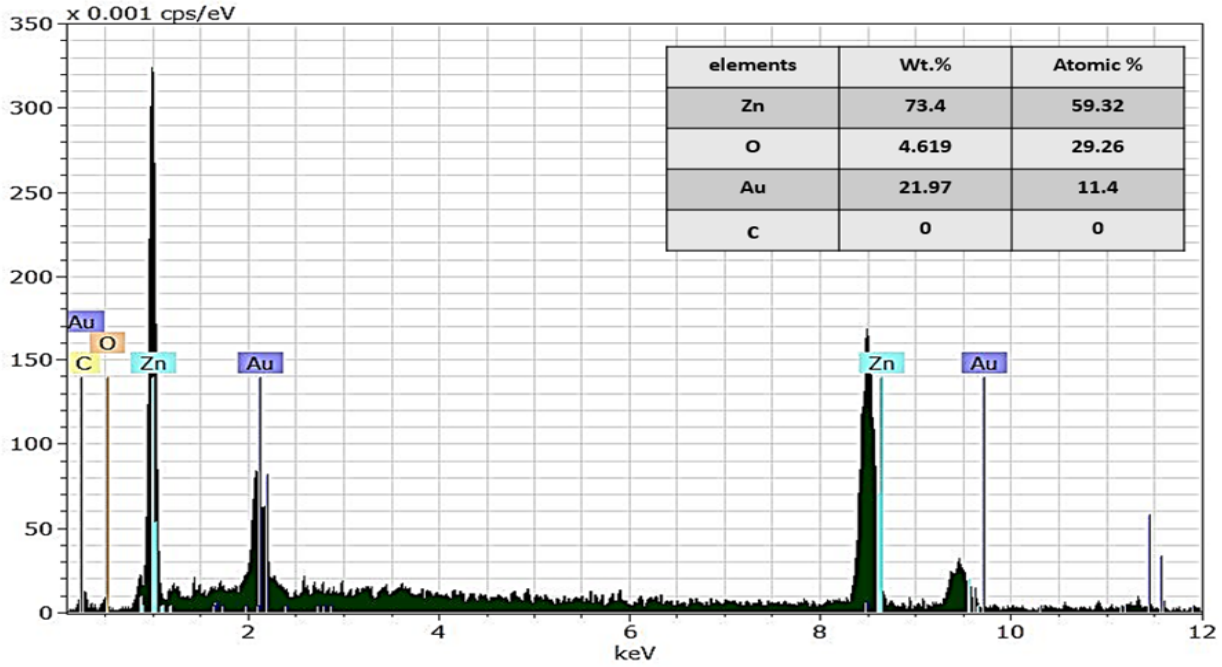


Fig. 4. EDAX analysis of ZnO NPs.

3.1.3 Atomic force microscopy (AFM)

AFM analysis gives us insight into the topography and roughness of nanoparticles [20]. The 3D image of AFM illustrated in Figure 5 demonstrates a smooth surface of nanoparticles with various particle sizes and types. The fabricated nanoparticles are round and triangular and range in size from 14 to 30 nm.

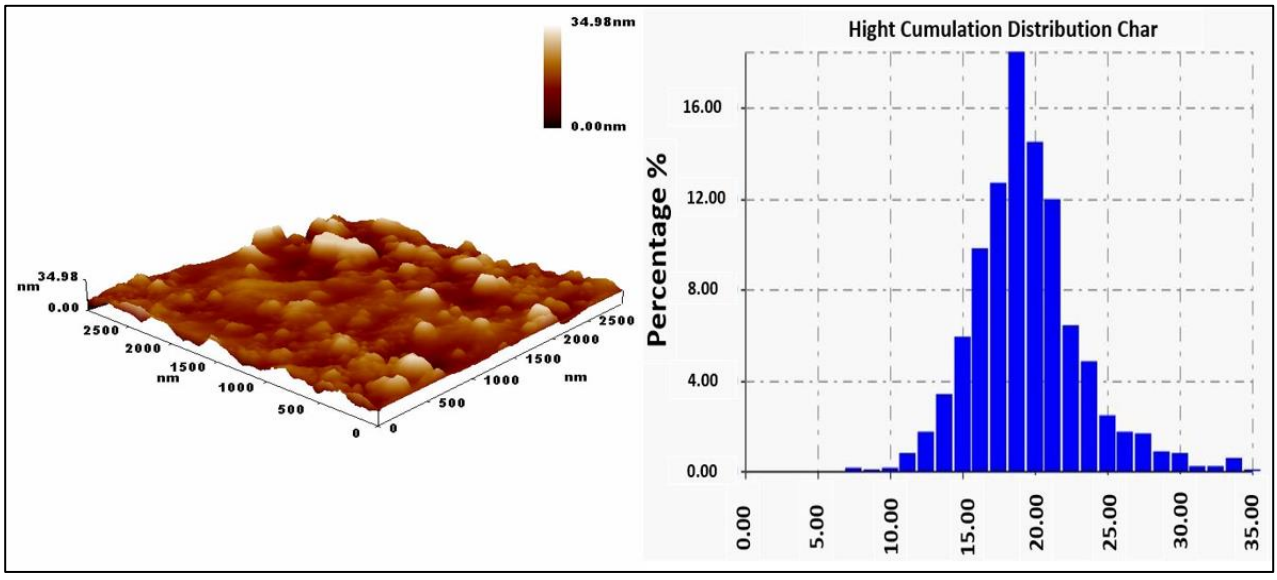


Fig. 5. AFM analysis of ZnO NPs.

3.1.4 Zeta Potential (ZP) Analysis

The determination of zeta potential is a crucial tool for providing stabilization for particles. Nanoparticles are stable because of their high zeta potential, which prevents them from aggregating, while their low potential leads to flocculation. A high negative value of -95.91 mV was obtained from zeta potential analysis, providing good stability of ZnO NPs as illustrated in Figure 6; this

stability derives from existing phenolic substances in the eucalyptus leaf extract [21]. Furthermore, eucalyptus leaf extract biomolecules are responsible for the higher surface charge [9,22].

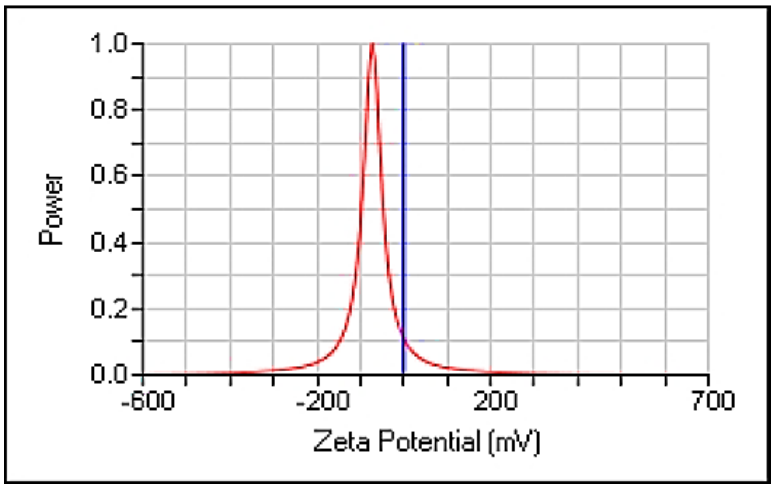


Fig. 6. Zeta potential analysis of ZnO NPs.

3.1.5 Brunauer -Emmett-Teller (BET) Analysis

The specific surface area (SSA), pore size, and pore volume of ZnO-NPs were analyzed by BET analysis. The SSA, pore volume, and pore size were 26.318 m2/g, 0.098 cm3/g, and 11.118 nm, respectively. Because the pore size falls within the range, the results indicate that ZnO-NPs are mesoporous particles [23]. The high SSA proves the ability of these low-cost ZnO-NPs to adsorb pollutants [24].

4. Analytical Methods

4.1 Calibration curve

A calibration curve for standard Acid Black 210 (AB210) solution was performed to determine the dye's maximum wavelength (λmax) and get the equation that connects the absorbance with concentration. As seen in Figure 7, the most significant wavelength of AB210 was determined to be 465 nm. There are different peaks in the scan at 316.5, 461.5, and 606 nm. However, the maximum wavelength is 465 nm because it gives the maximum absorbance.

The adsorption capacity at equilibrium q_e is defined as the relation of adsorptive amounts (mg) to adsorbed amounts (g), and it may be calculated using the formula:

$$qe = \frac{C_O - Ce}{W} * V \quad \ldots (1)$$

Where V is the volume of the working solution, Co is the initial concentration, Ce is the equilibrium concentration of the adsorbate ($mg.L^{-1}$), and W is the amount of adsorbent (g).

The removal efficiency (RE) was estimated using the formula below ;

$$RE\% = \frac{C_O - Ct}{C_O} X\ 100 \quad \ldots (2)$$

Whereas C_t is the AB-210 concentration at any time

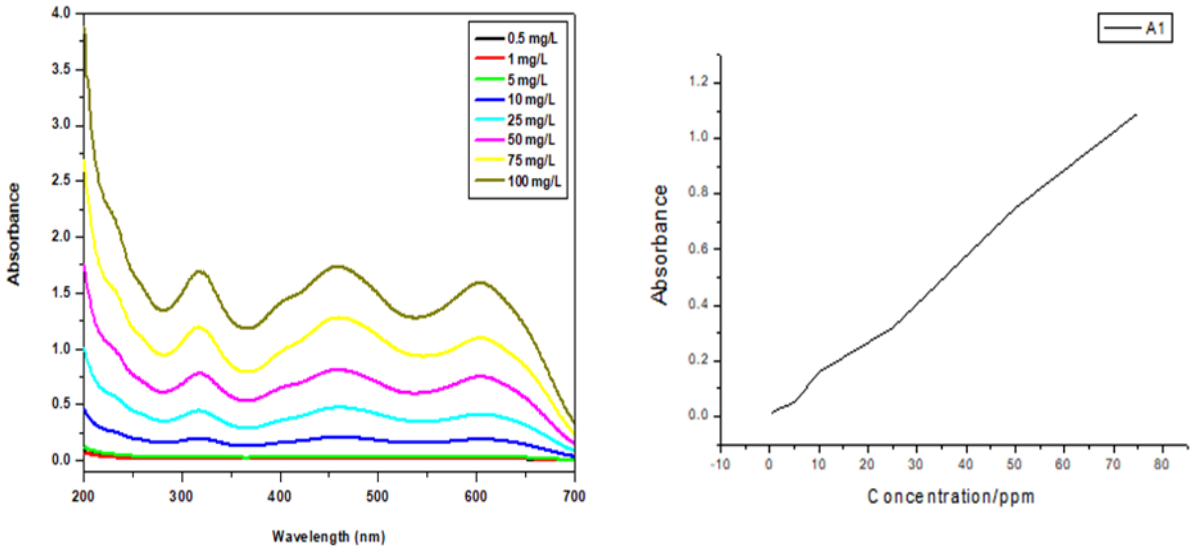


Fig.7. Calbiration curve of AB210.

4.2 Batch adsorption experiments

After the nanoparticles had been fully prepared and characterized, batch adsorption experiments were performed to test the efficiency of calcinated and uncalcinated ZnO NPs in removing AB210 from simulated wastewater. A 1 L of AB210 with a concentration of 25 mg/L was formed, then the pH was regulated, and the temperature was 5 and 45 ℃, respectively. To start the experiment, 0.75 g/L of ZnO NPs was added into AB210 solution at an agitation speed of 150 rpm, and the experiments lasted up to 180 min; λmax of AB210 we chose from calibration curve to be 465 nm; through the experiments, a10 mL samples were withdrawn at regular intervals and filtered with a 0.45 μm trying filter. The concentration of AB210 was determined using a UV-Vis spectrophotometer (Shimadzu-Japan), and the removal rate was utilized using Eq. 2.

As depicted in the following figure, the removal rate of uncalcinated ZnO NPs was 59%, whereas the calcinated ZnO NPs gave a removal efficiency of 57%. This slight difference in the removal of AB210 could be explained by the loss of some biomolecules from ZnO NPs during the calcination process, which reduces the stability of ZnO NPs.

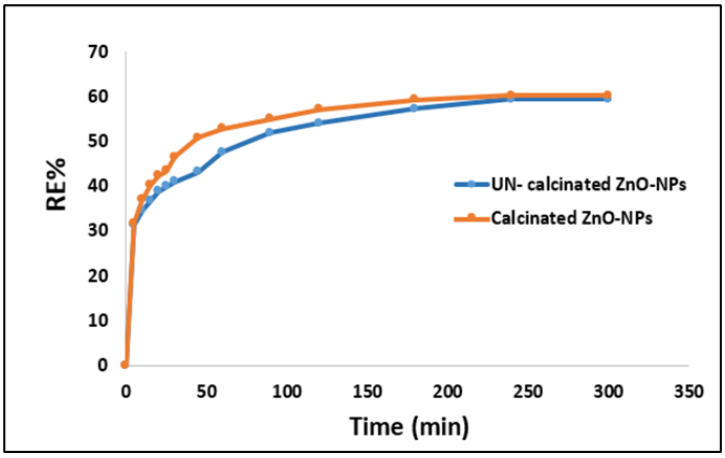


Fig. 8. The AB-210 Removal by E-ZnO-NPs 0.75 g/L dose, PH=5, initial concentration= 5 mg/L, temperature=45℃, agitation speed=150 rpm, and time 180 min.

5. Conclusions

This study involved a green economic approach to producing ZnO NPs created by combining zinc sulfate salt and eucalyptus leaf extract. Eucalyptus leaf extracts contain active components that function as a reducing agent and a capping and stabilizing agent. The characterization of ZnO NPs revealed that they were porous and had a spherical shaper, with nanoparticles ranging in size from 22 to 37 nm. Additionally, ZnO NPs exhibited high stability with a zeta potential of -95.91 mV. Furthermore, the adsorption results indicate that the calcinated ZnO NPs have a lower dye removal efficiency (57%) than the un-calcinated ZnO NPs, which have a removal efficiency of 59%.

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التوليف الأخضر وتوصيف جزيئات أكسيد الزنك النانوية باستخدام أوراق الكالبتوز لإزالة صبغة 210 الحمضية السوداء من الوسط المائي

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الخلاصة

تستخدم هذه الدراسة طريقة تركيب صديقة للبيئة ومنخفضة التكلفة لتصنيع جزيئات أكسيد الزنك النانوية (ZnO NPs) باستخدام كبريتات الزنك. مستخلص أوراق الأوكالبتوس هو عامل تغطية واختزال لتصنيع ZnO NPs. تمت دراسة التركيب والتشكل والسلوك الحراري والتركيب الكيميائي والتركيب الكيميائي والخصائص البصرية للجسيمات النانوية ZnO باستخدام تحليل احتمالية FT-IR و FE-SEM و EDAX و AFM و Zeta. أكدت صور FE-SEM أن ZnO NPs بمدى حجم (22-37) نانومتر كانت بلورية وكروية. تم استخدام طريقتين لإعداد ZnO NPs. تضمنت الطريقة الأولى كلسنة الناتج ZnO NPs والطريقة الثانية بدون تكليس. تم استخدام ZnO NPs المحضرة كمواد ماصة لإزالة الصبغة السوداء الحمضية 210 ((AB210) من مياه الصرف الصحي. كانت كفاءة الإزالة باستخدام ZnO NPs المكلس وغير المكلس 57 ٪ و 59 ٪ على التوالي.