Eco-Friendly synthesis of Zinc Oxide Nanoparticles Using Aqueous Extracts of Different Types of Dates (*Phoenix Dactyliferal*) Grown in the Oases of Southern Algeria

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Abstract

In this work, biosynthesis of Zinc Oxide nanoparticles (ZnO-NPs) was achieved using the aqueous extract of different types of dates grown in the oases of southern Algeria. They are represented in the type of dates **(*Degla baidha*** and ***Tinicine*)**, which are classified into the same Species *(Phoenix dactylifera.L),*where we obtained a white precipitate of Zinc Oxide nanoparticles, which were identified and characterized using ultraviolet (UV), X-ray (XRD), infrared (FTIR) and energy dispersive X-ray (EDX) analysis, where the average size of the particles that were obtained using both types of dates in the range (21.28 - 21.30 nm) and the morphological study of zinc oxide nanoparticles using Emission Scanning Electron Microscope (SEM) showed the formation of crystals of Zinc Oxide nanoparticles scattered irregularly shaped. With these findings, we have contributed to the realization of green chemistry principles, the evaluation of plants in our environment, and the green synthesis of Zinc nanoparticles in a seamless manner. These manufactured nanoparticles can be used in various biological fields.

Keywords: Zinc Oxide nanoparticles. Green synthesis .Date palm. *Phoenix dactylifera.L* .Degla baidha .Tinicine

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1 Introduction

Nanotechnology is the science concerned with the study of the design, characterization, production, and applications of shapes, devices, and systems that are controlled by shape and size at the nanometer level [1]. Nanoscience deals with the science of materials and technologies with scaled volumes (1-100nm). Through a presentation by Richard Feynman at the meeting of the American Physical Society in 1959 at the California Institute of Technology in his speech entitled (There is Plenty of Room at the Bottom), Feynman excelled in his lecture as he gave a perception of the possibility of changing the properties of any material and maximizing its features [3]. The term "nanotechnology" was first used by Norio Taniguchi in 1974, although it was not widely known [4]. A lot of studies on nanoscience and nanotechnology have been done all over the world [5]. This led to the discovery of different ways to synthesize new nanomaterials that have different physical and chemical properties than their large non-nano particles [6,7,8]. In fact, these nanomaterials exhibit interesting unique properties Essentially, it opened the door to new generation technologies in electronics, computers, and optics. Biotechnology, Medical Imaging, Medicine, Structural Materials, Aerospace, Energy, etc [9-11].

Zinc oxide nanoparticles is an inorganic compound with the formula ZnO. Zinc oxide comes in three forms :(hexagonal wurtzite, cubic zinc blend, cubic rock salt). The wurtzite structure is the most stable and most common in ambient conditions, Zinc oxide is used extensively in many materials and products including rubber, plastics, ceramics, glass, cement, lubricants, ointments, adhesives, sealants, dyes, foods, batteries, and first aid tapes. [12]. Zinc oxide nanoparticles have a wide band gap of (3.37eV) and the conductivity is diamagnetic [13]. It is used in energy efficient windows and in electronics such as thin film transistors. It is extremely useful for the manufacture of devices, such as electromagnetic coupled sensors and actuators. [14]. Effective nanoparticle zinc oxide can be used for water decontamination as an effective biotechnology for water disinfection and wastewater treatment using the most common semiconductors such as ZnO, CuO, TiO_2 and others [15-18].

It is worth noting that zinc oxide nanoparticles are environmentally friendly, non-toxic, and biologically safe. Which makes it desirable in biomedical applications such as drug carriers, cosmetics, antibacterial, and antioxidants [19,20]. Zinc oxide, due to its band gap energy and low production cost, was considered as a suitable alternative instead of TiO2 [21,22]. Zinc oxide nanoparticles have been used as an antibacterial agent against foodborne illnesses to protect food from bacterial contamination. Additives of zinc nanoparticles in packaging materials can lead to interaction with foodborne pathogens, thus releasing nanoparticles onto the surface of food where they come into contact with harmful bacteria and cause bacterial inhibition [23-26].

Thus, ZnO already has a wide application base, and has huge opportunities in many fields of industry due to its unique properties that have been explored. The future in which ZnO nanodevices become a part of our daily lives is already approaching reality.

ZnO nanoparticles have been synthesized by several methods including vapor-liquid-solid (VLS) [27], hydrothermal synthesis [28], vapor phase precipitation [29,30], chemical vapor deposition [31], Zinc oxidation [32], sol-gel method [33,34], microwave-assisted pyrolysis [35]. Green Synthesis [36] As well as in various morphological forms such nanowires [37], nanobelt [38], nanoflower [39].The traditional methods used to synthesize zinc oxide nanoparticles are expensive and harmful to the environment due to the dangerous chemicals are expensive used and causing problems. health and environmental.

The green method of nanoparticle synthesis is the fastest, most preferred, and environmentally friendly method for nanoparticle synthesis compared to other methods that have unintended effects such as environmental pollution, high energy consumption, and potential health problems. In contrast, green synthesis reduces pollution, improves the environment and human health safety [40]. In our research, our goal was to participate in the promotion of green chemistry principles and to search for safe methods to synthesis zinc oxide nanoparticles (ZnONPs) using the green method. Which use botanical extracts instead of synthetic chemical agents to reduce metal ions.

This was done by using different types of dates that grow in the oases of southern Algeria, represented in the type of dates (***Degla baidha*** and ***Tencine***) **Fig.1**.and classified in the same type (***Phoenix dactylifera. L***)

The date palm (***Phoenix dactylifera L.***) is a monocotyledonous perennial species that is adapted to the local conditions of arid and semi-arid regions. The date palm (Phoenix dactylifera L.) is widely cultivated in tropical and subtropical regions, especially in the Middle East and North Africa [41]. Globally, the total production value of date fruits has increased dramatically since the 20th century, recording the highest value of more than 14 billion US dollars in 2020 [42]. Palm trees contribute to the development of the economic and social aspects of date-producing countries [43,44]. The cultivation of date palms in Algeria is carried out exclusively for the harvesting of date fruits, and is considered a very important social, economic and environmental activity.

The high content of flavonoids, quinones, carotenoids, sterols, phenols and anthocyanins present in these types of dates makes them a good and suitable agent for the synthesis of nanoparticles NPs, particularly ZnONPs[45]. Also, the different high and low molecular weight protein fragments in these types of dates would confer particle stability to the NPs as they were formed [46,47].

As mentioned earlier in this paper, we do the green synthesis of ZnO nanoparticles using different types of dates, especially the fleshy part of dates, and characterize the produced ZnONPs using UV, XRD, SEM, FTIR.

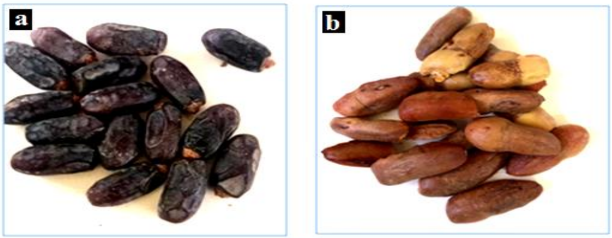

Fig1: a) type of dates (*Tinicine*) , b) type of dates (*Degla baidha*)

2 Experimental

2.1 Materials

The Zinc Acetate Dihydrate [Zn $(CH_3COO)_2.2H_2O$] with a purity of 90% were attained from Sigma Aldrich company. And you get dates (***Degla baidha*** and ***Tinicine***) from the palm forests in the southeastern oases of Algeria, exactly in the autumn season. During all stages of preparation, we used double distilled water to wash the dates, prepare date extracts, and prepare chemical solutions. The formation of ZnO NPs was mainly diagnosed with JASCO UV spectrometer. The vital functional groups in ZnO NPs were determined by SHIMADZU FTIR spectrometer. The crystalline properties of ZnO NPs were confirmed by PROTO XRD BENCHTOP X-ray POWDER diffractometer. The purity of the product was confirmed, and the morphological characteristics were examined in terms of size and shape by an energy dispersive X-ray analyzer (EDX) with SEM equipment a Zeiss Smart EDX type.

2.2 Preparation of aqueous dates extracts

To prepare the extracts, we removed the date seeds, then washed the fleshy part well tap water to remove any dust particles with and dirt, then washed again with de-ionized water. They were dried in a well-ventilated room for ten days. were ground into a fine powder and prepared in concentration 10%, (10g in100 mL double distilled water) and shaking for 2 hours at room temperature. The extract was placed in a water bath at 80 °C for 45 min. The resulting extract were then filtered. The resulting extract was then filtered, then the date extract was allowed to cool down to room temperature **(Fig. 2)** and stored at 4 °C for later use.

Fig 2: a)Aqueous extract of dates(*Tinicine*) , b) Aqueous extracts of dates (*Degla baidha*)

2.3 Green Synthesis of ZnO NPs

We took 100 ml of water extracts of dates, each cultivar separately, and then added 100 ml of zinc acetate solution [$Zn(CH_3COO)_2.2H_20$] prepared by dissolving (5g of zinc acetate in 100 ml double distilled water) each, and heated them to 60 °C using a plate Hot with stirring and continuous heating (within 60 °C) for 3 hours, then we separated the formed precipitate using a centrifuge (4000 rpm for 15 minutes) type (ROTOFIX 32 A – HettichZ entrifugen) and dried the product for 3 hours at 100 °C, the product was calcinated at 500 °C for 4 hours in oven a type Nabiertherm (MORE THAN HEAT 30-3000 °C)to remove all of the impurities, And we got a white precipitate, which was ground in the form of a powder and kept for the necessary analyses.The steps of the experiment are shown in the **fig.3**.

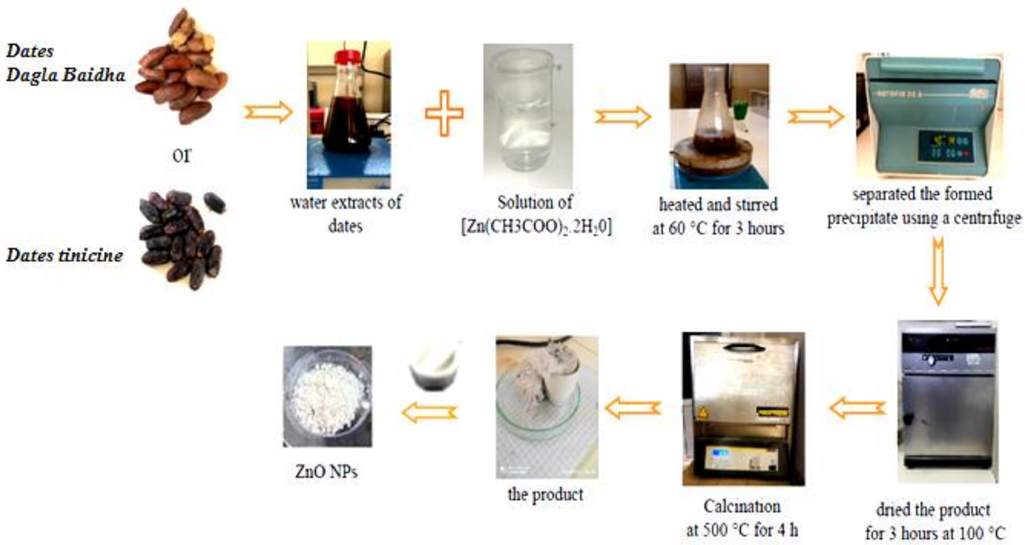

Fig 3: the steps for the synthesis of NPs by aqueous extracts from dates

3. Results and discussion

3.1 Mechanism of green synthesis of nanoparticles by plant extracts

Various plant metabolites, including terpenoids, polyphenols, polysaccharides, alkaloids, phenolic acids, and proteins, play an important role in the biological reduction of metal ions, leading to the production of nanoparticles [48].

In general, the mechanism of green synthesis of metals nanoparticles using plant extracts involves three major phases: activation phase, growth phase, and termination phase. **(Fig 5)**

1) The activation phase is the basic step in which metal ions are recovered from their salts by the action of plant metabolites, biomolecules with reductive capabilities. In addition, metal ions change from a mono- or divalent oxidation state to a zero-valence state and transform into nuclei of reduced metal atoms [49].
2) growth phase, which refers to the spontaneous coalescence of nanoparticles in which neighboring small nanoparticles spontaneously combine into larger particles, which is accompanied by an increase in the thermodynamic stability of the nanoparticles [50].
3) The last step of the synthesis process is the termination phase during which the nanoparticles finally reach their maximum possible activity, and this process is affected by the strength and ability of the plant extract to stabilize the metal nanoparticles [51].

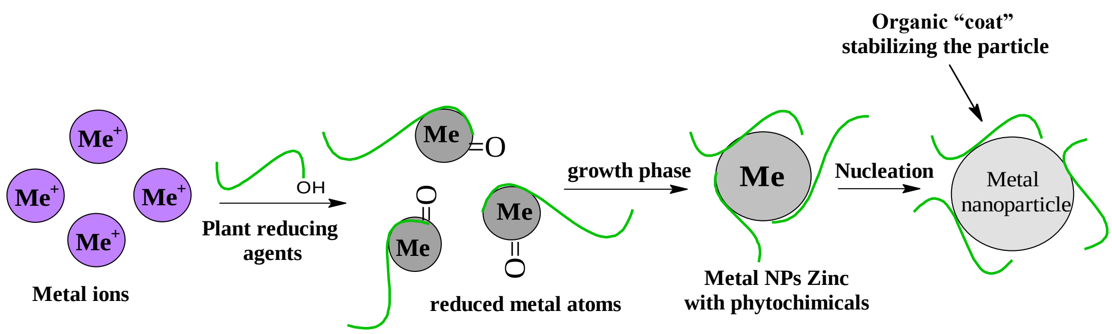

Fig 4: Mechanism of plant mediated synthesis of ZnO nanoparticles.

3.2 Reaction rate of green synthesis of ZnO NPs By aqueous dates extracts

We calculated the reaction rate of green synthesis of ZnO NPs By aqueous dates extracts (**R_1**) before calcination and immediately after drying according to **(relation1**). Then we calculated the reaction rate after calcination (**R_2**) according to **(relation2**) and obtained the results recorded in **Table.1**, where the results show the importance of calcination in removing impurities, which are the remaining organic materials after the reduction reaction.We also notice that the reaction yield in the case of using the aqueous extract of dates type of Tinicine. is high compared to using the aqueous extract of dates type of Degla Bayda.

Table 1: Reaction rate of Green synthesis of ZnO NPs By aqueous dates extracts

dates	m(g) $[Zn(CH_3COO)_2.2H_20]$	m_1(g) The precipitate	R_1(%)	m_2(g) The precipitate	R_2(%)	m_3(g)
Degla baidha	5g	3.80g	76.00	3.30g	66.00	0.50
Tinicine	5g	4.20g	84.00	3.73g	74.60	0.47

3.3 Analytical Characterizations

3.3.a-*Diagnosis of ZnO-NPS by UV-Vis .*

The biosynthesized ZnO NPs were confirmed by UV-Vis spectrum in the range between 200–600 nm **(Fig.5)**. The UV-Vis spectrum identified an absorption peak (λ max) at 374 nm for ZnO NPs. which was synthesised using the extract of the type of dates Tinicine **(Fig.5a)**.and an absorption peak (λ max) at 377 nm for ZnO NPs. which was synthesised using aqueous extract of the type of dates Degla baidha **(Fig.5b)**.

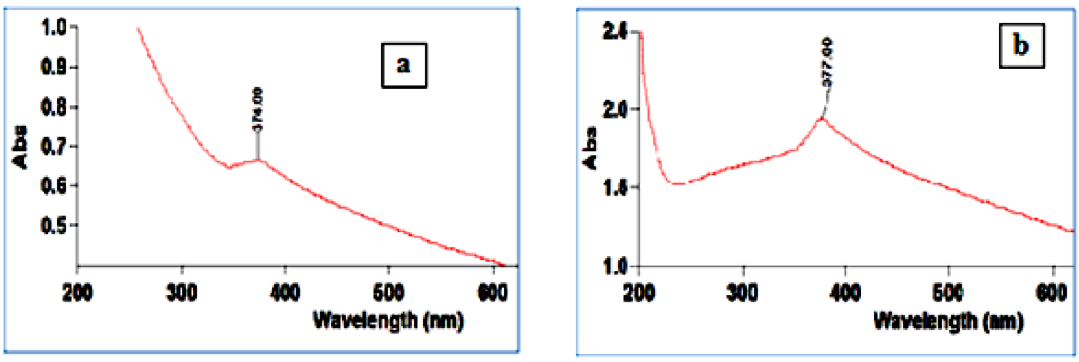

Fig 5: UV-Vis spectrum :a) of synthesized (*Tinicine*-ZnO NPs) ,b) of synthesized (*Degla baidha* -ZnO NPs)

3.3.b-*Diagnosis of ZnO-NPS by FTIR*

We recorded the FT-IR spectrum to confirm the synthesis of zinc nanoparticles using aqueous extracts of dates, and the analysis was carried out in the frequency range of (400–4000 cm^{-1}) at room temperature**(fig.6)**. Through this, it can be seen that several absorption peaks associated with O–H bond expansion do not appear at 3480 cm^{-1} to 3500 cm^{-1} which characterize H_2O and phenolic compounds, flavonoids, phenolic acids and other compounds present in the aqueous extract of dates [45,55,56].and this is evidence of the occurrence of a reduction reaction of zinc salts. We also observe absorption peaks at 875 cm-1 and 882 cm-1 which could be related to the C–H (aromatic) expansion. The observed absorption peak also at 1422 cm-1 can be attributed to the C–C stretching of the aromatic rings, absorption peak also at 1148.89 cm-1 to the C–O stretching.And the emergence of characteristic values at (403 - 674 cm^{-1}).is the characteristic bond signal of Zn-O, which confirms that the material is ZnO.

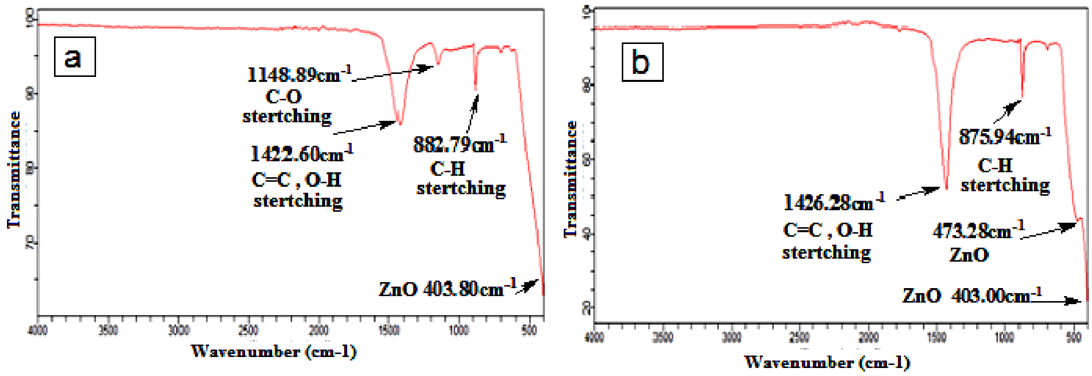

Fig 6: FT-IR spectra :a) of synthesized (*Tinicine*-ZnO NPs) ,b) of synthesized (*Degla baidha* -ZnO NPs)

3.3.c-*Diagnosis of ZnO-NPS by XRD*

Our use in the analysis of nanoparticles resulting from the green synthesis using the aqueous extract of dates type of Tinicine. XRD technique to know the phase purity and the crystalline nature of the nanoparticles. The **fig.7.a** shows the resulting diffraction of Tinicine-ZnO-NPs. The strong crystalline nature of ZnO-NPs obtained by X-ray diffraction peaks, and the characteristic peaks observed at values (31.75°, 34.40°, 36.22°, 47.53°, 56.58°, 62.89°,66.56 , 67.97° and 76.9°) which correspond to the crystalline levels (100), (002), (101), (102), (110), (103), (112), (201) and (202) agree well the standard spectrum of zinc oxide according to the database (ZnO JCPDS 36- 1451), confirming the hexagonal wurtzite structure of the ZnO-NPs [52-54]. Was of a high intensity peak at the (101) level of Diffraction diagram, the mean crystal size of the resulting Tinicine-ZnO-NPs. The average crystallite size for the resultant was evaluated as 21.28 nm using (Debye Scherrer's) equation**(relation3)**

$$D = \frac{K\lambda}{\beta \cos\theta} \ldots\ldots(3)$$

whereβ is the full width at half maximum (FWHM), λ is the X-ray wavelength, θ is the diffraction angle and k is the Scherer's constant of the order of unity.

We note the absence of any other distinctive peaks, which indicates the absence of impurities in the obtained ZnO-NPs system, the phase purity of the resulting ZnO, and the precursor has been completely converted into a NPs product. And it shows the success of the process of green synthesis of particles using an aqueous extract of the type of dates *Tinicine*

On the other hand, in the case of using aqueous extract of dates *Degla baidha* , the crystalline nature of ZnO-NPs obtained by X-ray diffraction peaks, **fig.7.b** at the values (31.78°, 34.44°, 36.26°, 47.67°, 56.61° , 62.89°, 67.99 , 69.10° and 77.00°) which correspond to the crystal planes (100), (002), (101), (102), (110), (103), (112) and (201) and (202) respectively agree well the standard spectrum of zinc oxide according to the database (ZnO JCPDS 36- 1451),. The average crystal size of the result was evaluated to be 21.30 nm using the (Debbie Scherrer) equation.

We also note that there are no other characteristic peaks, indicating that there are no impurities in the obtained ZnO-NPs system, the purity of the phase of the resulting ZnO, and the precursor has been almost completely converted into the NPs product. This further indicates a successful process of manufacturing green particles using an aqueous extract of dates *Degla baidha*

Table 2 and Table 3 shows the FWHM value for each assigned peak for the particle size calculation.

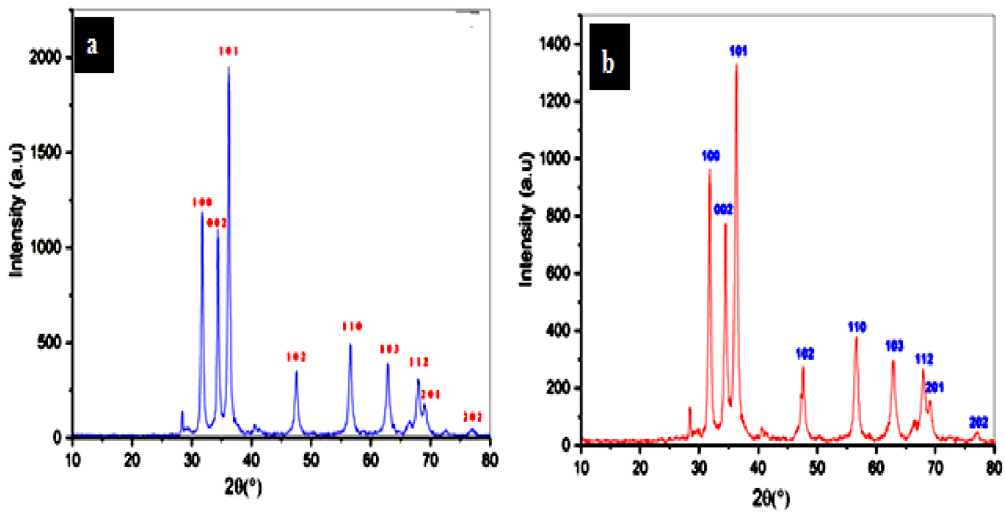

Fig 7: XRD spectra :a) of synthesized (*Tinicine*-ZnO NPs),b) of synthesized (*Degla baidha* -ZnO NPs)

Table2: Parameter Calculation for average crystallite size calculation for (*Tinicine*-ZnO NPs)

2θ (°)	hkl	FWHM (β)	D (nm)
31.75	100	0.2952	29.22
34.4056	002	0.3936	22.07
36.2251	101	0.3936	22.18
47.5309	102	0.3936	23.03
56.5841	110	0.3936	23.94
62.8901	103	0.3936	24.71
66.5643	112	0.7872	12.61
67.9725	201	0.492	20.34
76.993	202	0.7872	13.47
Average D (nm) = 21.28 nm			

2θ (°)	hkl	FWHM (β)	D (nm)
31.7883	100	0.3936	29.43
34.4412	002	0.2952	17.75
36.2592	101	0.492	23.05
47.6721	102	0.3936	15.96
56.6192	110	0.5904	19.77
62.8981	103	0.492	20.34
67.9902	112	0.492	25.59
69.101	201	0.3936	17.96
77.0045	202	0.5904	29.43

Average D (nm) = 21.30 nm

3.3.d-*Diagnosisof ZnO-NPS by Emission Scanning Electron Microscope (SEM) andEnergy Dispersive X-ray (EDX)*

In order to study the morphology of zinc oxide nanoparticles, the topographic images, crystal size and shape of the resulting powder are determined using SEM analysis for the ***Tinicine-ZnO NPs*** sample **(Fig. 8 (a, b))** and for the ***Degla baidha -ZnO NPs*** **(Fig. 8 (c, d))**, which is clearly in the nanoscale. It has an irregular shape with particles having different sized spherical morphology in the form of agglomerates and rods with a smooth surface. This is also probably due to condensation, which leads to a narrow space between the particles. And to further confirm in order to know the elements composition of ZnO NPs synthesized using aqueous extracts of the studied date species, an energy dispersive X-ray analyzer (EDX) was used to analyze the samples. The obtained EDX spectrum analysis of ***Tinicine-ZnONPs*** **(Fig.9.a)**and ***Degla baidha-ZnONPs*** sample **(Fig.9.b)** confirmed the presence of zinc and oxygen elements in the samples in good proportions, which was confirmed by EDX element mapping. The quantitative weight estimation of the elements showed that the nanoparticles samples consisted of 56.64% zinc and 28.59% oxygen for the Tinicine-ZnO NPs sample and 61.14% zinc and 24.79% oxygen for the white Degla baidha-ZnONPs sample, which indicates the purity of the studied nanoparticles. We also noticed the presence of carbon, which may be the result of the sample absorbing carbon dioxide from the atmosphere. Probably from the aqueous extract of the dates used.

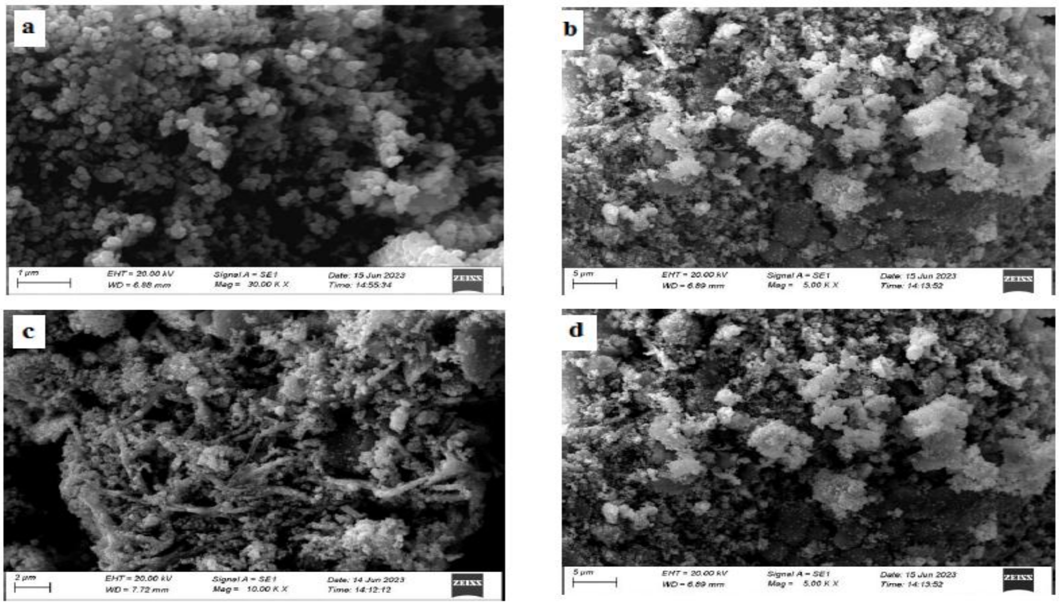

Fig 8: SEM image :a ,b) of synthesized (*Tinicine*-ZnO NPs) ,c ,d) of synthesized (*Degla Baidha*-ZnO NPs)

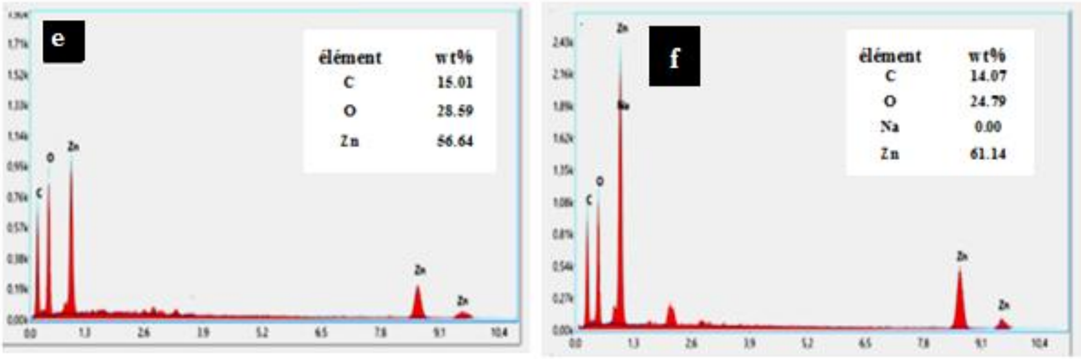

Fig 9: EDX spectrum: e)of synthesized (*Tinicine*-ZnO NPs) ,f) of synthesized (*Degla baidha* ZnO NPs)

4-Conclusions

In this laboratory work, we have achieved a green synthesis of ZnO NPs according to an environmentally friendly method, which is to use aqueous extracts of different types of dates (*Tinicine* and *Degla Bayda*) as a reducing agent for zinc acetate salts. And we obtained a good yield for the reduction reaction ranging between (66% - 74%) and high purity zinc nanoparticles. This synthesis method is simple, inexpensive, and less toxic, and has contributed to the evaluation of our surroundings and the realization of green chemistry principles, green synthesis of ZnO NPs with a low percentage of pollutants and residues. These nanoparticles can be used in many important applications such in optics, magnetism and gas sensing and in the field of catalysis, wastewater treatment Especially biomedical applications because they are safe, cost effective and biocompatible in nature.

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