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**Biosynthesis and Characterization of ZnO/NiFe<sub>2</sub>O<sub>4</sub> Nanocatalyst via Green Route Using *Anacardium occidentale* *linn* (cashew nut) and its photocatalytic activity for the treatment of tannery wastewater under sunlight irradiation**

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**ABSTRACT**

The *Anacardium occidentale* (cashew nut) leaves extract have been excellently utilized as a reducing as well as capping agent for the synthesis of zinc oxide and nickel ferrite nanoparticles using zinc acetate, and iron (III) chloride, nickel chloride as precursor with a different calcined temperature at the furnace and its application in the treatment of tannery wastewater. The generation of zinc oxide was proved by various characterization techniques. XRD studies displayed that a cubic phase and the average crystallite size for the intense peak measured were about 30-56nm was exhibited by the synthesized nanoparticle SEM image showed that synthesized particle are granular around 50nm and roughly spherical in shape. EDS analysis proved the elemental composition of the synthesized sample and disclosed that oxygen as well as zinc are the dominant elements. The synthesized catalyst was used in the tannery wastewater treatment also the quality parameters of tannery wastewater after treatment with the synthesized catalyst under sunlight irradiation, significantly changed and found to be within acceptable range of quality water according to (WHO). The color, odor and appearance where changed to colorless, odorless, and clear. pH, 7.4 total organic carbon (TOC), 32(mg/dm<sup>3</sup>), turbidity, 2.5(NTU) respectively. The highest efficiency of the treatment process was attained under sunlight irradiation conditions in comparison to those which were recorded under dark conditions as revealed by the result of the analysis. This procedure is safe, environmentally friendly and viable due to its fast, low cost compared to other methods.

**Keywords;** Biosynthesis, *Anacardium occidentale*, ZnO, NiFe<sub>2</sub>O<sub>4</sub>, Characterization tannery wastewater treatment

**1.0 INTRODUCTION**

Tannery effluent is among one of the most hazardous pollutants of industry. Major problems caused by tannery wastewater containing heavy metals, nutrients, toxic chemicals, chloride, lime with high dissolved and suspended salts, and other pollutants and this represent a serious challenge and cause considerable ecological imbalances (Hamza, M.F et al., 2022). With the growth of population, the increasing requirement of leather and its products led to the establishment of large commercial tanneries. Tannery operation consists of converting of the raw hide or skin into leather which consume huge amount of water in several stages, generating an enormous amount of liquid

effluents which are hazardous to the environment to which they are discharged, consequently, make it as a potentially pollution intensive industry (Sivagami *et al.*, 2018). Large volume of tannery wastewater containing thousands of organic and inorganic pollutants, high concentration of biological oxygen demand (BOD), chemical oxygen demand (COD), suspended solids (SS), are generated and discharged into the limited water bodies on daily basis without or with partial treatment which is one of the major concerns of environmental pollution (Wosnie and Wondie, 2014). (Fouda, A *et al.*, 2022). The tannery wastewater is highly turbid, coloured and have odoriferous smell with pH value of about 7.5.

Exposures to tannery effluents have been reported to cause several diseases such as skin diseases, kidney problem, disruption of endocrines in humans and aquatic lives (Lam *et al.*, 2018). Several methods have been utilized to remove these toxic substances in tannery wastewater namely, coagulation-flocculation, precipitation, oxidation (Zgura *et al.*, 2018). Ion exchange, membrane filtration and ozonation (Lam *et al.*, 2018). activated carbon adsorption (Vaiano *et al.*, 2018) to mention but a few. But these methods are associated with shortcomings such as high cost of reagents, toxic sludge generation, low efficiency, not only that but also organic pollutant were only transfer from one phase to another without necessarily mineralizing them to harmless substances such as CO<sub>2</sub> and H<sub>2</sub>O water (Velanganni *et al.*, 2018). Urgent required of Remediation of the polluted sites from hazardous pollutants, such as treatment of tannery wastewater using the green synthesized nanoparticles derived from plants extracts through simple, rapid, economical, and environmentally safe methods, (Alhalili, Z., 2022). Nanoparticles are characterized by many physical and chemical properties such as their small sizes (1– 100 nm), that give them a enough surface area-to-volume ratio, together with their mobility, reactivity, strength, solubility, as well as unique interface effects and quantum properties; these properties differentiate them from the bulk of materials. The innovative physicochemical properties of nanomaterials potentially provide uses in several applications such as medical engineering, and biological (Bhavyasree, *et al.*, 2022).

Photocatalysis is the sequential chemical reaction (oxidation/reduction) which occurred under light irradiation in the presence of oxygen and semiconductor metal oxide (Awual *et al.*, 2019). Catalyst is activated upon the absorption of a photon of light which has the energy equal or higher than the band gap energy of the metal oxide in question. Photocatalytic technology where a photocatalyst harnesses UV radiation from sunlight or artificial light and utilize the energy have been widely applied to break down different substances including organic materials, organic acids, estrogens, pesticides dyes, crude oil, microbes (including viruses) and chlorine resistant organisms

(Goutam *et al.*, 2018), inorganic molecules such as nitrous oxides (NO<sub>x</sub>). In most cases heterogenous photocatalysis can be combined with other technology such as precipitation or filtration to remove metal and other toxic constituents in water (El-Dafrawy, 2016). Due to this universal applicability, photocatalysis with nanoparticles as catalysts have been applied to reduce air pollution for self-cleaning surfaces in addition to wastewater purification and water splitting. Furthermore, ZnO nanoparticles prepared via these methods have high band gap and low surface area thus responsible for its low photocatalytic activity in the visible region (Xue, *et al.*, 2018). Concerted efforts are ongoing towards the reduction of the band gap of ZnO and extend its absorption threshold to visible region (Kumaran and Muraleedharam, 2017). One of such approaches is doping of ZnO with either metals, non-metals, or metal oxides in most cases both to prevent electron hole recombination rate (Snehal *et al.*, 2016). Doping is the anchoring or foreign materials (impurities) onto the ZnO framework in order to suppress electron-movement from valance band to conduction band (Kumaran and Muraleedharam, 2017). One of the dopant considered in this study is Nickel ferrites (NiFe<sub>2</sub>O<sub>4</sub>). Nickel Ferrites are mixed metal oxide of Nickel and iron (III) oxide with Fe as the main component due to its ferromagnetic ordering and magnetism as well as super exchange interaction (Angadi *et al.*, 2016). Mixed spinel structure ferrites are the particularly of interest among researchers all over the world, due to its electric, dielectric and magnetic properties (Xue, *et al.*, 2018). Rajeswari *et al.*, (2017) reported a facile synthesis of hexagonal wurtzite znO nanoparticles through green synthesis method using aqueous leaves extract of *carica papaya* leaf extract. the characterization results revealed that the synthesized znO nanoparticle was hexagonal wurtzite polymorph in spherical shape with particle size of -50 nm. under uv-visible light irradiation, znO nanoparticles successfully degraded the methylene blue dye within 180 min. The utilization of ZnO/NiFe<sub>2</sub>O<sub>4</sub> nanocomposites for the treatment of tannery wastewater will simultaneously reduce the odoriferous smell, colour, turbidity level and raise the solution pH. Combination of green synthesis and hydrothermal technique will help

reduce the possible post contamination of nanoparticles and prevent formation of toxic by-products in comparison to other physical and chemical methods. The present study synthesized zinc oxide and nickel ferrite nanoparticles (ZnO/NiFe<sub>2</sub>O<sub>4</sub>) nano composites using combination of green and hydrothermal method. The photocatalytic degradation of tannery wastewater by synthesized ZnO/NiFe<sub>2</sub>O<sub>4</sub> nanocomposites under natural sunlight irradiation was also investigated.

## 2.0 MATERIALS AND METHOD

All chemicals and reagents used in this study were of analytical grade.

### 2.1 Sample Collection and Pretreatment

The leaves of *anacardium occidentale linn* (Cashew nut) were collected from Karal village a location in wanke adc Gusau, Zamfara State, Nigeria. And transported to chemistry Laboratory Federal University Gusau, It was then identified at Herbarium unit of Biological science laboratory Department. The collected leaves were washed to remove dust and air dried at room temperature for about 15 days under dust free condition. Dried leaves were cut into fine pieces, pounded and sieved to get the finest powder and stored in a polyethylene container for further analysis. The raw tannery wastewater was collected from tanning industry located at Marina area of sokoto metropolis using clean dried and transported to chemistry Laboratory Federal University Gusau,

## 2.2 Extraction of the Plant and Green Synthesis of ZnO

### 2.2.1 Extraction of the Plant

A known weight (30g) of powdered plant sample was weighed and put into a 500 ml of beaker followed by the addition of 400ml of distilled water and subsequently heated on a preheated hot plate for 20 minutes. After which an amber colour of extract was obtained and filtered using Whatman filter paper thereby any other particles that might be present was removed. The extract was then put in a 250 cm<sup>3</sup> conical flask and then it was stored in a cold dark place for further analysis.

### 2.2.2 Green Synthesis of NiFe<sub>2</sub>O<sub>4</sub> Nanoparticles

The green synthesis of NiFe<sub>2</sub>O<sub>4</sub> nanoparticle was performed as follows: The mixture of the plant extract was developed by introducing 50ml of plant extract into the 100ml of 0.4MFeCl<sub>3</sub> + 0.2M NiCl<sub>2</sub> solution in 250ml flask. The reaction was performed using reflux mounting continuous stirring at 70°C for 120 minutes. The generation of NiFe<sub>2</sub>O<sub>4</sub> was proved by a colour change to dark brown from green and the product was collected and allowed to age after 24 hours at room temperature and it was washed with distilled water several times and the product was allowed to dry overnight at 80° C and then it was heated in a furnace at 400° C for 2 hours. The powder was considered as the sample of the present study. The resulting sample was studied by changing the temperature at which it was reheated in the furnace at 450 and 500° C as described by (Fouda, A et al., 2021).

### 2.2.3 Green Synthesis of ZnO

The green synthesis of ZnO nanoparticle was performed as follows: The mixture of the plant extract was developed by introducing 50ml of plant extract into the 100ml of 0.5M Zincacetate solution in 250ml flask. The reaction was performed out using reflux mounting continuous stirring at 70°C for 120 minutes. The formation of **Zincoxide** was proved by a colour change to dark brown from green. The brown solid product was collected when it was allowed to age after 24 hours at room temperature and then it was washed with distilled water for several times. The product was allowed to dry overnight at 80° C and then it was heated in a furnace at 400° C for 120 minutes. The powder was considered as the sample of the present study. The resulting sample was studied by changing the temperature at which it was heated in the furnace at 450 and 500° C as described by (Fouda, et al., 2021).

### 2.2.4 Characterization of ZnO Nanoparticle

Characterization of synthesized ZnO/NiFe<sub>2</sub>O<sub>4</sub> nanoparticle was carried out using the following techniques; (HRSEM), (EDS) and X – Ray diffraction (XRD) as described by (Fouda, et al., 2021).

### 2.2.5 Photocatalytic Treatment of Tannery Wastewater

The photocatalytic analysis of Zinc oxide/nickelferrite Nanoparticles was carried using an already characterized Marina tannery wastewater with a known concentration. 150ml of the wastewater was measured and poured into a 250ml beaker and it was repeated for two more beakers. 0.4g of the synthesized Zinc oxide/nickelferrite nanoparticle was then weighed, and poured into two of the beakers and then placed under continues on a magnetic stirrer, one of the stirrer was then placed under sunlight, the second one in a dark room and the last one was placed in an open room without the catalyst. Both beakers were then allowed to stay for 15minutes to attain the absorption /desorption equilibrium after which the samples were collected from the three beakers also after another 15minutes which makes 30minutes of stirring the samples were collected again, analysed and compared with raw tannery

waste water, in order to evaluate the change in physicochemical parameters such as ph, total organic carbon, turbidity, colour, odour, and appearance, and efficiency and activity of used catalyst **as described by** (Rice, E.W., *et al.*, 2021).

## 3.0 Results and Discussion

### 3.1. Green Synthesis of ZnO/NiFe<sub>2</sub>O<sub>4</sub>

the formation of a dark brown. color after blending the aqueous extract with Zincacetate indicates the successful formation of ZnO-Nanoparticle. Also, the formation of a dark brown. color after blending the aqueous extract with FeCl<sub>3</sub> and NiCl<sub>2</sub> indicates the successful formation of NiFe<sub>2</sub>O<sub>4</sub>Nanoparticle. Anacadium occidentale *linn* green synthesis of ZnO/NiFe<sub>2</sub>O<sub>4</sub> has numerous advantage over physical, chemical, as well as other biological approaches because of it is economically simplicity, fast, environmental-friendly, nontoxic, as well as efficiency.

## 3.2. Characterization

3.2.1 Morphological Characterization: High Resolution Scanning Electron Microscope (HRSEM) of ZnO Nanoparticle Synthesized at 400°C and 450°C. The morphological characteristics and temperature are presented in plate a and b below

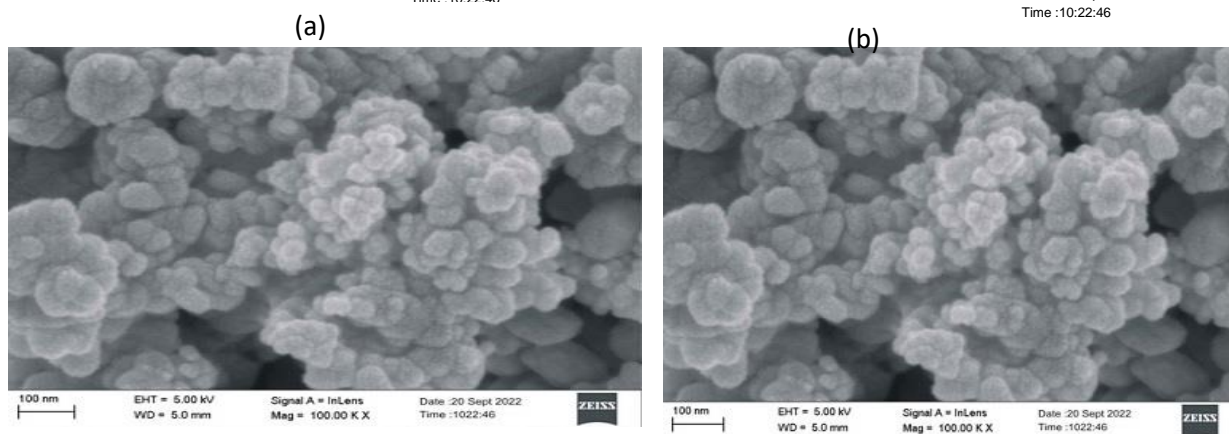
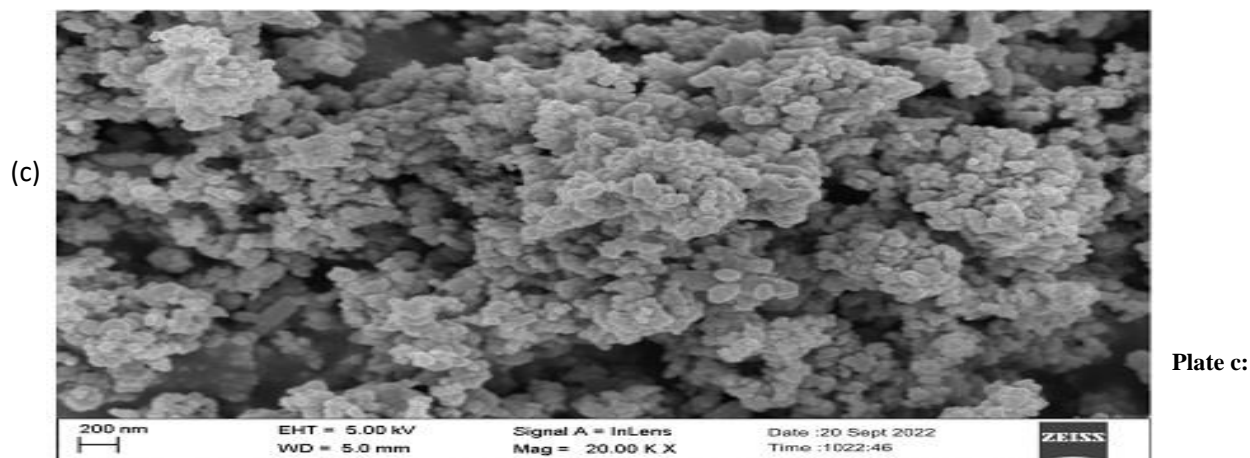


Plate a and b: HRSEM Images of ZnO Nanoparticles Calcined at (a) 400°C, (b) 450°C.



HRSEM Images of ZnO Nanoparticles Calcined at (a) 400°C, (b) 450°C and (c) 500°C

The plate a, b and c above presented the image of morphological characterization of High Resolution Scanning Electron Microscope (HRSEM) of ZnO Nanoparticle Synthesized at Temperature of 400°C, 450°C and 500°C. From the images it can be seen that the particle size and morphology changes between high and low calcination temperature of the ZnO nanoparticles prepared via green route. Images of nanoparticle were change by change in calcined temperature as it was observed in plate 2 and 3, and it was noticed that the surface morphologies existed as spherical, granular and clusters in the form of assemblies. At 400°C the shape of the particles was not well defined even though tiny spherical particles were noticed as presented by image in plate 1. At 450°C there were partly defined spherical shaped and agglomerated morphology as presented by image in plate 2. However, at temperature of 500°C spherical and more agglomerated particles were observed as presented by image in plate 3.

### 3.2.2 Elemental Composition of ZnO Nanoparticles Synthesized at Different Temperatures.

Energy Dispersive Spectroscopy (EDS) examined the chemical compositions of the synthesized sample. The stoichiometry of EDS spectra at different temperatures are shown in figure 1, 2 and 3 below. The EDS analysis shows the presence of Zinc (Zn) and oxygen (O) as the most dominant

elements. Other elements such as sodium (Na), carbon (C) and iron (Fe) are also present. However, the presence of carbon in trace amount may be attributed to the polyphenolics compound from the plant extract used for the reduction and capping of the synthesized ZnO nanoparticles while sodium originated from the sodium hydroxide (NaOH) used to adjust the pH value.

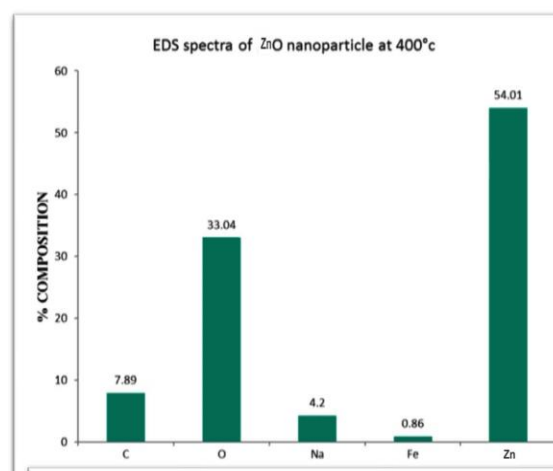


Figure 1. EDS Spectra of ZnO Nanoparticle at 400°C

The atomic percentage of each element obtained from EDS analysis of ZnO nanoparticles produced at 400°C are shown in fig .1 above, which are Ni

(54.01%), O (33.04%), C (7.89%), Na (4.2%) and Fe (0.86%).

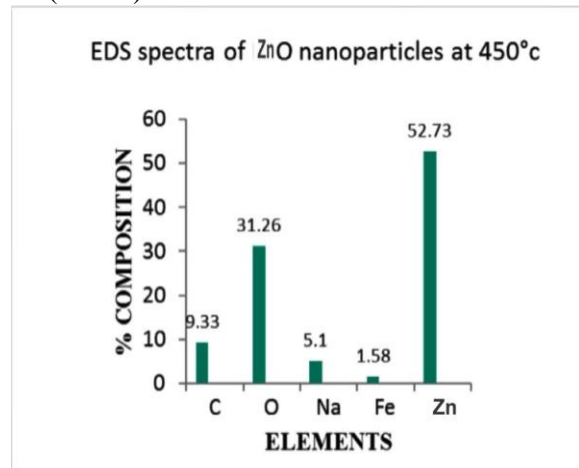


Figure: 2. EDS Spectra of ZnO Nanoparticle at 450°C

The elemental composition obtained from EDS analysis of ZnO nanoparticles produced at 450°C are showed in fig Figure, 2 above which are Zn (52.73%), O (31.26%), C (9.33%), Na (5.1%) and Fe (1.58%), but Zn and O were slightly lower compared to that of 400°C but the levels of impurities were high.

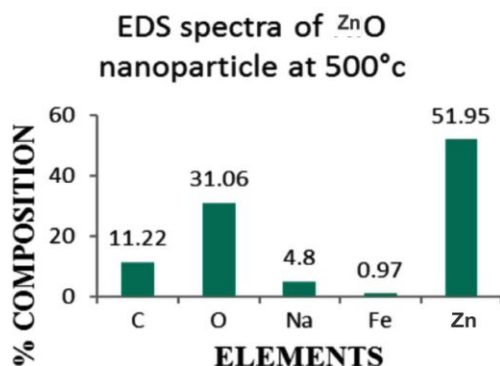


Figure: 3. EDS Spectra of ZnO Nanoparticle at 500°C

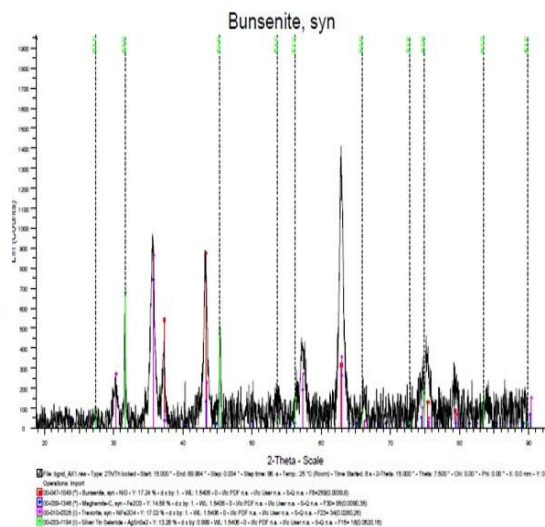
Fig .3 above shows the elemental composition to be at 500°C but Zn and O were slightly lower compared to that of 400°C but the levels of impurities were high.

The elemental composition obtained from EDS analysis of ZnO nanoparticles produced at 500°C

are showed in fig Figure, 3 above which are Zn (51.95%), O (31.06%), C (11.22%), Na (4.80%) and Fe (0.97%). but Zn and O were slightly lower compared to that of 400°C and 450°C but the levels of impurities were high

### 3.2.3 X-ray Diffraction of ZnO Nanoparticles Synthesis.

The obtained XRD patterns were verified using the JCPDS software. ZnO nanoparticles samples have peaks at 31.5°, 37.2°, 43.2°, 57.7° 62.8° and 75.4° which can be attributed to the main characteristic of the (111), (200), (220), (311), (104) and (222) sets of lattice planes were manifested for the X-ray diffraction patterns shown in figure 4 below. The obtained data was balanced with the Joint Committee on Powder



**Figure: 4. X-ray Diffraction of ZnO Nanoparticles Synthesis.** Diffraction Standards (JCP)

### 3.3. Results of physiochemical parameters of raw and treated Marina tannery wastewater

the results of physiochemical parameter of local tannery wastewater from marina local tannery, sokoto are presented in the table 3.1 below

**Table 3.1 physiochemical parameters of raw and treated Marina tannery wastewater**

Parameter	Marina tannery wastewater (Raw)	Marina tannery wastewater (Treated)	(WHO) standard									
Colour	Dark-blue	Colourless	Colourless									
Odour	Fishy	Odourless	Odourless									
Appearance	Cloudy	Clear	Clear									
Temperature(°C)	27	25	23.5-30									
pH	6.2	7.4	6.0-9.0									
Turbidity (NTU)	69.47	2.5	5									
Total organic carbon content (mg/dm <sup>3</sup> )	88	<table border="1"> <tr> <td></td> <td>15minutes</td> <td>30minute</td> </tr> <tr> <td>Light</td> <td>40</td> <td>32</td> </tr> <tr> <td>Dark</td> <td>82</td> <td>80</td> </tr> </table>		15minutes	30minute	Light	40	32	Dark	82	80	<75
	15minutes	30minute										
Light	40	32										
Dark	82	80										

#### 3.3.1 Photocatalytic study of the synthesized (ZnO/NiFe<sub>2</sub>O<sub>4</sub>) in the tannery wastewater treatment

In the present research, the photocatalytic activity of the synthesized ZnO/NiFe<sub>2</sub>O<sub>4</sub> nanoparticle was evaluated via tannery wastewater treatment and pollutants removal in the presence and absence of sunlight, two different reaction time were used to check the effect of reaction time on the photocatalytic functioning of ZnO catalyst and NiFe<sub>2</sub>O<sub>4</sub> as catalyst supporter. Turbidity, Total organic carbon (TOC), ph, color, appearance and odor, was used as a testing and evaluation parameters to know the extent and efficiency of the catalyst under sunlight in the removal of pollutant from the tannery wastewater as shown in table 3.1 above, and the results of the physicochemical properties of raw and treated Marina tannery wastewater indicated that the ph, level was changed to 7.4 from 6.2, total organic carbon (TOC) of the wastewater decreased significantly to 32(mg/dm<sup>3</sup>), after the treatment from from 88(mg/dm<sup>3</sup>) in the untreated wastewater. turbidity, of the wastewater decreased significantly after photocatalytic treatment from from 69.47(NTU), in the raw tannery wastewater to 2.5(NTU), color, changed to colourless from dark blue, odour, changed to odourless from fishy odour, and

appearance changed to clear respectively. This results indicated the efficiency of the catalyst used in the treatment of tannery wastewater under sunlight irradiation, significantly the physicochemical parameters of treated tannery wastewater were found to be within acceptable range of quality water recommended by world health organisation (WHO) standards.

#### 4.0 CONCLUSION

The leaves of *anacardium occidentale linn* leaves extract have been effectively used for the synthesis of zinc oxide and nickel ferrite nanoparticles using zincacetate, and iron (III) chloride, nickel chloride as precursor. This method utilized the natural, renewable and lowcost biomaterial for the synthesis of ZnO/ NiFe<sub>2</sub>O<sub>4</sub> nanoparticles. The generation of zinc oxide was proved by different characterization techniques. XRD studies displayed that the synthesized nanoparticle exhibits a cubic phase and the average crystallite size for the intense peak measured were about 30-56nm. SEM image showed that synthesized particles are granular around 50nm and roughly spherical in shape. EDS analysis confirmed the elemental composition of the synthesized sample and revealed zinc and oxygen to be the dominant element. also the photocatalytic activity of the synthesized

ZnO/NiFe<sub>2</sub>O<sub>4</sub> nanoparticle was investigated via tannery wastewater treatment and pollutants removal and result obtained from the study indicated the efficiency of the catalyst used for the treatment of tannery wastewater, significantly the physicochemical parameters of treated tannery wastewater were found to be within acceptable range of quality water recommended by world health organization (WHO) standards.

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