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Synthesis of Nickel Oxide Nanoparticles by Electrochemical Method, Characterization and Photo degradation of Acetic Acid and Study of Antibacterial Activity of Synthesized Nickel Oxide Nanoparticles

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Abstract: ***The metal oxide (NiO) Nanoparticles is a proficient material with much experimental and industrial application. The nickel oxide Nanoparticle has been synthesized by electrochemical method, which is simple and inexpensive method. The nickel oxide is a p-type Antiferromagnetic oxide semiconductor with band gap 3.6 to 4.0.The synthesized metal oxide (NiO) Nanoparticles is used as a catalyst for the photocatalytic degradation of acetic acid under various experimental conditions by volumetric method. The synthesized Nanoparticles were characterized by SEM-EDAX, UV-VIS spectroscopy, FT-IR spectrum and X-ray diffraction studies. The SEM results showed that the synthesized nickel oxide Nanoparticle is in the needle shape with aggregates. The EDAX spectra showed that the presence of nickel and oxygen. UV-VIS spectra revel that the band gap energy of synthesized nanomaterialis2.91eV calculated using Tauc plot. The FT-IR spectrum shows that the band at 470cm-1 are indicated to Ni-O vibration band. The photocatalytic study for the synthesized nickel oxide nanomaterials was investigated by the kinetics of degradation of acetic acid by volumetric method against NAOH solution. The antibacterial effect of this material against staphylococcus aureus MTCC 7443 and Escherichia coli MTCC 40 was investigated.***
Keywords: Electrochemical method,: Volumetric Method: Nickel oxide Nanoparticles: Acetic acid: Antibacterial activity

I. INTRODUCTION

In recent years the metal oxide is mainly fixed on new types of techniques especially nanomaterials because of their bulk applications. Mainly nanomaterials are interesting because of their improved chemical and physical properties [1].Nanoscience has been advanced extraordinary due to its broad applications [2-3].Nanoparticles are classified as a material in which at least one dimensions. Nanoparticles are not new to the environment and occur naturally in the form of minerals, clays, and products of bacteria [4-6]. Many researchers have reported on the wide range of metal oxide nanopowder applications, they are widely used for textile, medical, water treatment and food packaging applications [7-9]. Metal-based nanotechnology includes metals, metal oxides, metal sulfides, and quantum dots are widely used in various applications [10]. Metal oxide nanopowder is obtained by addition of reducing or oxidizing agents during their synthesis. In the last few years nanomaterials has been used in various industrial and household products [11-13]. Metal oxides nanomaterials acts as an influential role in many research areas of physics, chemistry and material sciences. The metal oxides are used in the sensors, fabrication of microelectronic circuits, fuel cells, coatings for the passivation of surfaces against corrosion, and as catalysts in addition to use as a semiconductor, thermoelectric or electroluminescent material, they also used in biomedical applications as drug delivery systems for treatment and diagnosis. [14-17]. Nickel oxide nanomaterials is an important transition metal oxide with cubic lattice structure. The nickel oxide is used in variety of applications such as: solar cells battery cathodes gas sensors, rechargeable lithium ion batteries, catalysis, magnetic materials and electro chromic films. It can also be used in dye sensitized photocathodes. Nickel oxide semiconductor becomes a inspire topic in the new research areas [18-19]. The nickel oxide nanomaterials has been attracted because of their potential applications and their specific chemical and physical properties [20-21]. In the present paper we synthesized nickel oxide Nanoparticles by electrochemical method which is an environmentally friendly method by passing an electric current between two or more electrodes separated by electrolyte. The synthesized nickel oxide Nanoparticles on photodegradation of acetic acid and the kinetics of degradation of acetic acid were studied by volumetric method.

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II. MATERIALS AND METHODS

All chemicals were used to prepare Nickel oxide nanomaterials were analytical grades of purity. Nickel wire was purchased from Alfa Aesar. Acetic acid from lobachemie, Platinum electrode from Elico pvt.ltd. All solution was prepared in double distilled water. The optical properties for prepared Nickel oxide nanomaterials were studied by uv-visible spectrophotometer (shimadzu-1700 series).The x-ray crystallographic interpretations were performed by x-ray diffrometer (panalytical x-pert) using Cu Ka wavelength (λ=1.54) scanning range from 0 to 70^0 .The morphological feature for the prepared Nickel oxide study as determined by scanning electron microscopy (SEM-EDEX) from quanta-200 FEI, netherlamds. The elemental analysis for the conformation of prepared Nickel and oxygen is confirmed from energy dispersive X-ray analysis (EDAX). Functional groups present in the molecular structure were performed by FT-IR instrument by PerkinElmer Spectrum Version 10.03.09.

A. Synthesis of Nickel oxide Nanomaterials by Electrochemical Method

The nickel oxide nanomaterial was synthesized by electrochemical method. The experimental preparation is as shown in Figure. 1. The Nickel metal wire is used as anode and platinum electrode is used as cathode. Using 20mA current and potential of 10V the experiment was run for 2.5 hrs with continues stirring. The electrolytic cell is consisting of 5 % of aqueous NaHCO3 solution. The distance of the anode and cathode during electrolysis was 2 cm. During the electrolysis the Nickel wire starts to dissolve and give Nickel ions, which are electrochemically reacted with NaHCO3 to give solid Nickel oxide. The obtained solid Nickel oxide is washed with double distilled water till complete removal of unreacted NaHCO3. The solid Nickel oxide is centrifuged and calcined for 2hr at 750^0C for dehydration and for the removal of hydroxides to get Nickel oxide Nanoparticles. The conduction band energy of synthesized nickel oxide nanomaterials is 1.8eV, the ionization potential is 10. 7eV.The redox potential of Ni is (-0.23) and the ionic radius of Ni^{2+} is $0.72A^0$ respectively. Since the mechanism for the synthesized Nickel oxide Nanoparticles is given in scheme1.

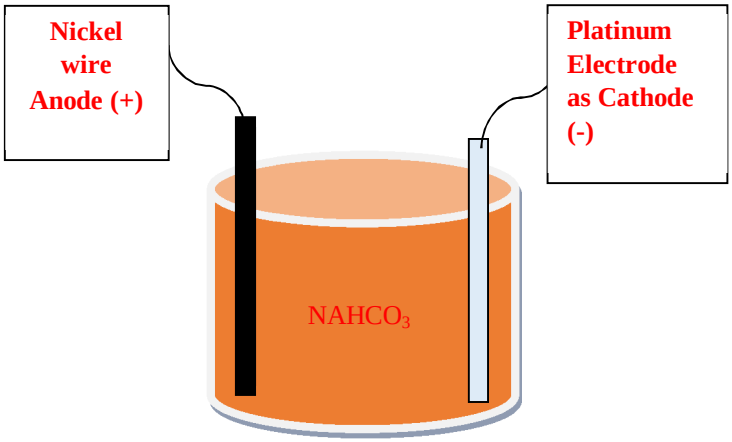

Figure 1. Experimental set up for the electrochemical synthesis of Nickel oxide Nanoparticles

$$Ni \longleftrightarrow Ni^{2+} + 2e^-$$

$$2NaHCO_3 + 2e^- \longrightarrow 2CO_2 + 2Na + 2OH^-$$

$$Ni^{2+} + 2OH^- \longleftrightarrow Ni(OH^-)_2$$

$$Ni(OH)_2 \longleftrightarrow NiO + H_2O$$

$$Ni + 2NaHCO_3 \rightarrow NiO + CO_2 + 2Na + H_2O$$

The overall retroaction $Ni + NaHCO_3 \longleftrightarrow$ NiO Nanoparticles

Scheme 1

B. Determination of Photocatalytic Activities

Acetic acid properties, Molecular formula: $C_2H_4O_2$, Molar mass: 60.052g mol^{-1}, the carboxylic acid solution (1×10^{-3} M) was prepared by dissolving in distilled water. This solution was used as a test contaminant for evaluating photocatalytic activities of the prepared Nickel oxide Nanoparticles. The exploration was carried out under tungsten-halogen UV-light in order to check the effectiveness of Nickel oxide Nanoparticles. The COD has been reported both before degradation and after degradation of the acetic acid solution using dichromate oxidation method [22-23]. COD effect was calculated by the following equations.

$$COD = \frac{(Blank - Sample) \times N_{FAS} \times 8000}{V_{Sample}} \longrightarrow (1)$$

C. Kinetics of Photodegradation by Volumetric Method

Volumetric method involves the measurement of volume of a solution of known concentration which is used to determine the concentration of the analyte. In the present paper the volumetric titration method was used to determine the degradation efficiency. By measuring the concentration of carboxylic acid (Acetic Acid) by the titration against sodium hydroxide solution at different time intervals. After complete degradation of carboxylic acid no colour formation takes place with phenopthaline-NaOH solution. A plot of log V/V_0 versus time was linear up to 60 % of the reaction illustrate the appearance of acetic acid follows first order kinetics.

III. RESULTS AND DISCUSSION

1) *UV-visible Spectra:* The synthesized Nickel oxide Nanoparticles shows the intensity peak at 350.11nm in the UV region and no absorption peak in the visible region .The UV-Visible spectra of nickel oxide Nanoparticles over the range 200-700nm shows the synthesized Nanoparticles is photoactive under UV radiation .The band gap of Nickel oxide Nanoparticles was calculated using Tauc plot [24].The band gap energy of synthesized Nickel oxide Nanoparticles was found to be 2.91eV.

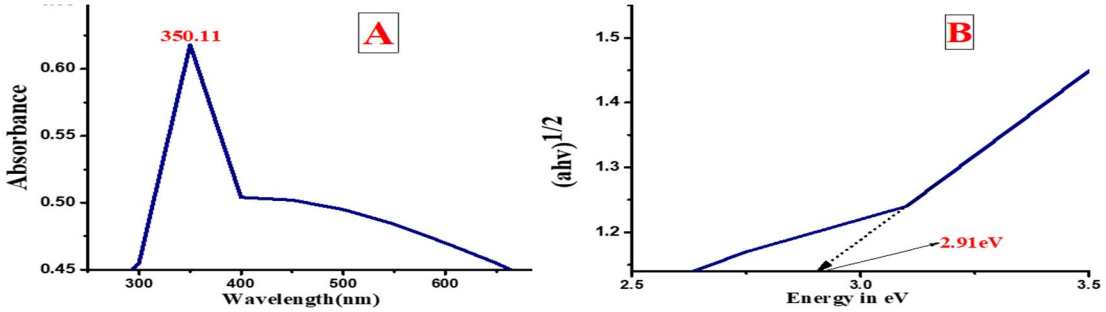

Figure 2.UV-Visible spectra (A) and Tauc plot (B) of Nickel oxide nanoparticles.

2) *X-Ray Diffraction:* The X-Ray diffraction spectra shows that the crystallinity of the synthesized nickel oxide Nanoparticles as shown in fig(3).The peaks at 2theta is 37.36^0,43.46^0 is readily indexed as (111), (200) crystal planes of synthesized nickel oxide Nanoparticles .The diffraction peaks can be readily indexed to be face -centered cubic (FCC) . By using Debye-scherrers formula [25] the X-Ray diffraction pattern and size of the crystal was calculated and it was found to be 21.23nm

$$D=k\lambda/\beta\cos\theta \qquad (2)$$

Where k is an empirical constant equal to 0.9, λ is the wavelength of the X-ray source, β is the full width at half maximum of the diffraction peak and θ is the angular position of the peak

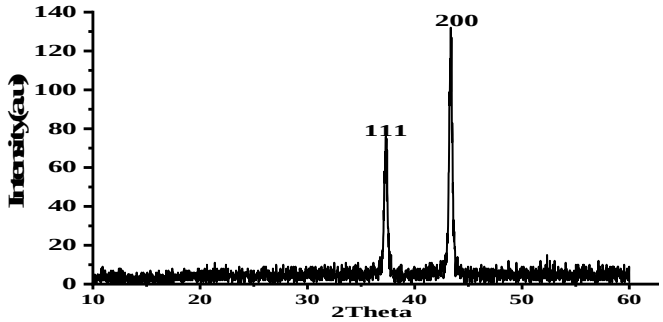

Figure 3. XRD patterns of Nickel oxide Nanoparticles

3) *Scanning Electron Microscopy (SEM):* Scanning electron microscopy (SEM) indicates morphological examination with direct visualization. The surface morphology of the synthesized nickel oxide Nanoparticles was observed by using SEM micrographs. The SEM result shows that the synthesized nickel oxide Nanoparticles are in the needle shape with aggregates. The EDAX spectra show the presence of Ni and O in the Nanoparticles.

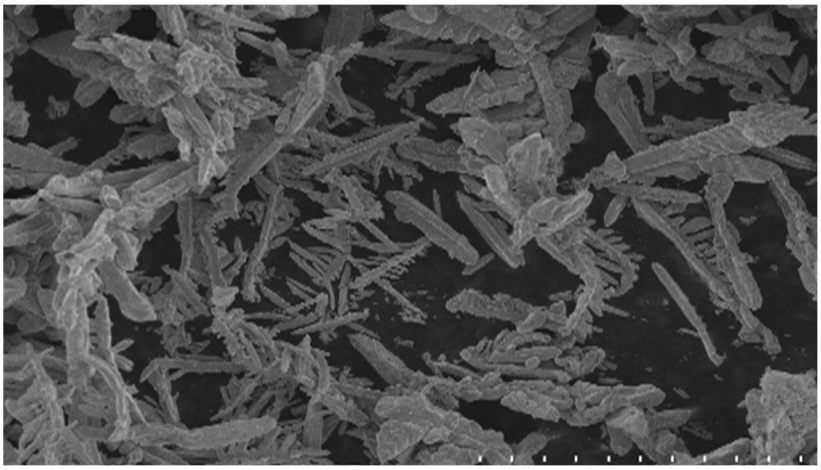

Figure 4. SEM images of electrochemically synthesized Nickel oxide Nanoparticles.

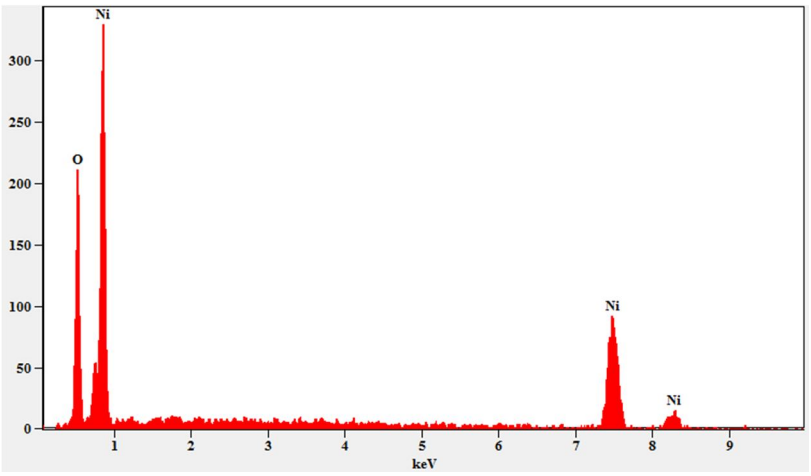

Figure 5. Energy dispersive X-ray analysis spectrum of Nickel oxide Nanoparticles.

4) *FTIR Spectra:* FT-IR transmission spectrum was taken with PerkinElmer Spectrum Version 10.03.09640 infrared spectrometer in the range of 4000–400 cm-1. The FTIR spectrum shows that the bands at 470cm-1 are indicated to Ni-O vibration band. The absorption band at 617cm-1 is indicated to NI-O-H stretching band [26].

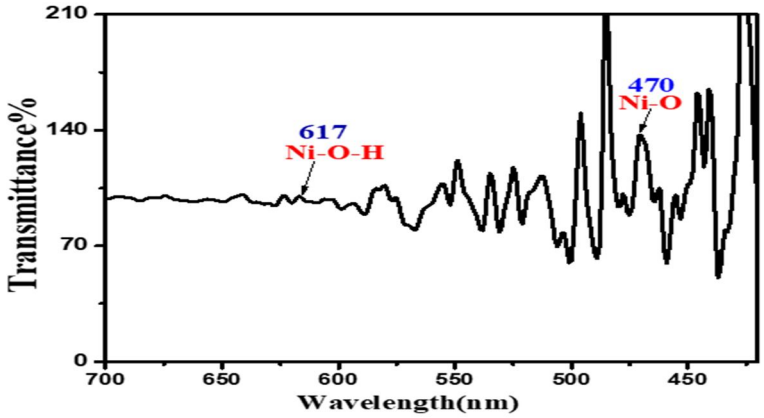

Figure 6.FTIR spectrum of Nickel oxide Nanoparticles

IV. PHOTODEGRADATION KINETICES AND COD MEASUREMENTS

1) *Effect of Concentration of Acetic Acid:* The photodegradation technique was go through with different concentration of carboxylic acid (Acetic acid) solution ($0.5x10^{-3}$to $2x10^{-3}$) with constant weight of Nickel oxide nanomaterials is assistant as a catalyst. The change in concentration of the carboxylic acid was recorded by appearance of colour using sodium hydroxide solution by volumetric method. A plot of log V/V_0 versus Time was linear up to 60 % of the reaction illustrate the appearance of carboxylic acid follows first order kinetics (Figure. 7). The rate constant values are given in Table 1 and the reaction rate decreased with increase in acid solutions. The logic beyond that is with increase in the acid concentration, the solution becomes more intense and the path length of the photons entering the solution is decreased and the few photons reached the catalyst surface. Hence the production of hydroxyl radicals is reduced. Therefore, the Photodegradation efficiency is reduced. The COD for acid solutions before and after degradation were measured and are given in Table 1. Figure. 7. To account for the mineralization of acids solution COD was examined at different stage. The formation of different radical species during photodegradation is given in scheme 2. The Photodegradation efficiency of the photo catalyst was calculated by the following formula,

$$\text{Photodegradation efficiency} = \frac{\text{Initial COD} - \text{Final COD}}{\text{Initial COD}} \times 100$$

$$e^-_{cb} \longrightarrow {}^{+}O_2O^-_2$$
$$h^+_{vb} + H_2O \longrightarrow H^+ + {}^{\cdot}OH$$
$$O^-_2 + H^+ \longrightarrow HO^{\cdot}_2$$
$$HO^{\cdot}_2 + e^-_{cb} + H^+ \longrightarrow H_2O_2$$
$$H_2O_2 + {}^{\cdot}O^-_2 \longrightarrow OH + {}^{-}OH + O_2$$
$$OH + \text{Acid} \longrightarrow CO_2 + H_2O + \text{Simple inorganic salts}$$

Scheme 2
Mechanism for the photodegradation of carboxylic acid

Table 1. Effect of Photodegradation at different concentration of Acetic acid under UV light
Nickel oxide Nanoparticle = 0.01g
Temperature = 308K

Acetic acid concentration in [N]	10^3k Sec^{-1}	Time taken for 95% Degradation in min	COD Values in mg/l	
			Before degradation	After degradation
0.0005	4.79	15	256	16
0.001	2.94	35	544	16
0.002	0.45	160	704	48

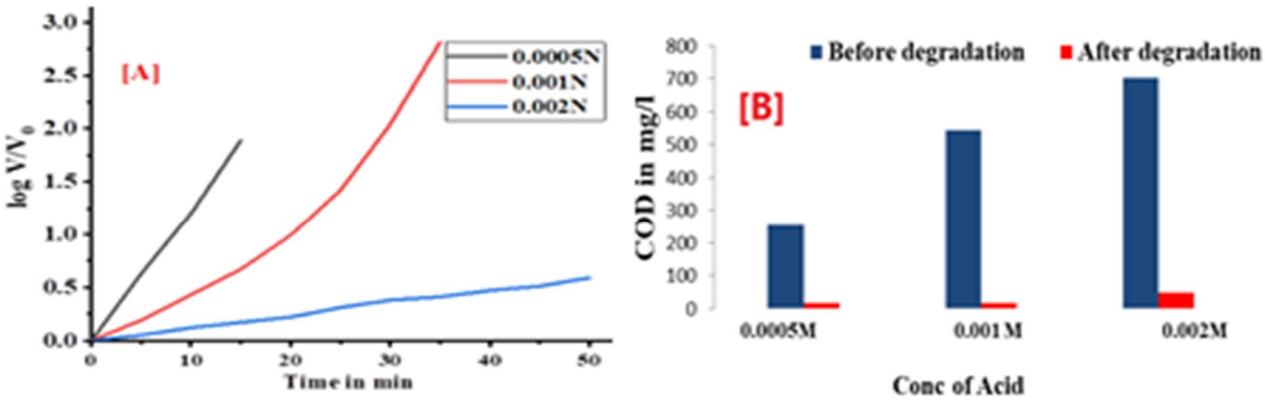

Figure7. Effect of concentration of Acetic acid on the rate of degradation and COD values under UV light

2) *Effect of Catalyst Loading:* The experiments were carried out by taking different amount of catalyst varying from 0.005 to 0.02g keeping acid concentration constant in order to study the effect of catalyst loading. The study showed that increase in catalyst loading from 0.005 to 0.02g increased degradation efficiency. Further increase in catalyst above 0.02g decreased the photo activity of the catalyst, due to aggregation of nickel oxide nanomaterials at higher concentration causing a decrease in the number of active sites on catalyst surface & increase in the light scattering of nickel oxide Nanoparticles at high concentration. This tends to decrease the passage of light through the sample. Further, the present study indicated, from economic point of view, the optimized photocatalyst loading is 0.01g/20 ml (Figure. 8and Table 2). The COD effect as been reported in Figure. 8

Table 2. Effect of catalyst loading on the photodegradation of Acetic acid under UV light
Acetic acid = 0.001N
Temperature = 308K

Catalyst Nickel oxide in mg	10^3k Sec^{-1}	COD Values in mg/l	
		Before degradation	After degradation
0.005	0.52	544	32
0.01	2.94	544	16
0.02	1.82	544	16

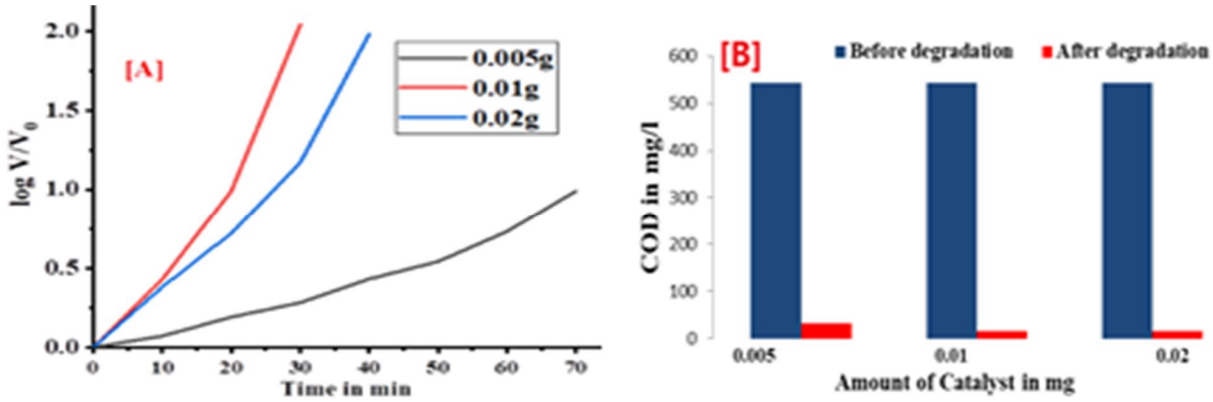

Figure 8. Effect of catalyst loading on the rate of degradation and COD values of Acetic acid under UV light

3) *Effect of Temperature:* Temperature is one of the essential factors which effects the rate of photodegradation. It is clear that the increase in temperature increases the acid degradation, and observed that the rate of appearance of colour is not very significant at low temperature. However, the reaction is more significantly influenced at high temperature since the diffusion rate increased with temperature an increase of temperature could bring about an increase in the degradation rate. The rate constant for different temperature is given table 3 and figure 9.The thermodynamic parameters were calculated for photodegradation of acetic acid it is in table 4.

Table3. Effect of temperature on the photodegradation of Acetic acid under UV light
Acetic acid = 0.001N
Nickel oxide Nanoparticle = 0.01g

Temp in k	Rate constant 10^3k Sec^{-1}	COD Values in mg/l	
		Before degradation	After degradation
308	2.94	544	16
318	3.28	544	48
328	5.05	544	16

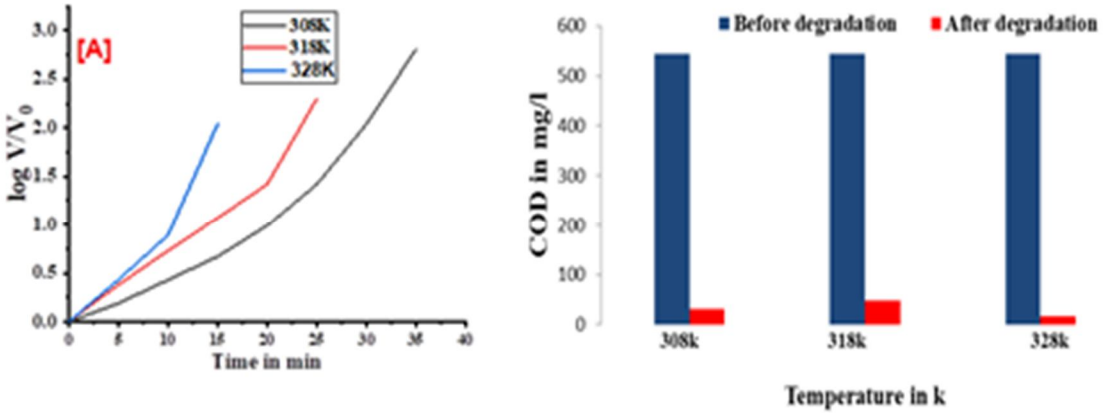

Figure 9. Effect of temperature on the rate of degradation and COD values of Acetic acid under UV light

Table 4: Thermodynamic parameter for Acetic Acid

Temperature in k	$\Delta H^{\#}$ kJmol^{-1}	$\Delta S^{\#}$ Jk^{-1} mol^{-1}	$\Delta G^{\#}$ kJmol^{-1}	Ea
308	41.83	-160.95	93.96	10.61x10^{3} calories
318	41.74	-160.12	95.31	
328	41.66	-160.28	96.96	

4) *Effect of Light Intensity:* The photodegradation rate constant in UV light is compared with sunlight. It is perceived that the photodegradation rate constant is increased in UV light compared to sunlight for prepared Nickel oxide nanomaterials. The reason beyond that is when a photon occurrence on a semiconductor(NiO) energy that overtake the band gap energy of the semiconductor .An electron is jump up from the valence band to the conduction band leaving a hole in the valence band .The excited state conduction band electrons and valence band hole can recombined and dissipate energy in the form of heat and get trapped into the metastable surface states, respectively with electrons acceptors and donors that happened to be adsorbed on the semiconductor surface .The stored energy is dissipated within a few nanoseconds by recombination in the absence of suitable e^{-} /h^{+} scavengers .If a suitable scavenger is available to trap the electron recombination is prevented i.e. subsequent redox reaction may occur. Therefore, the Nickel oxide nanomaterials act as a very good photocatalyst and is active under UV light compared to sunlight. The rate constant for degradation in sunlight are given in table.5

Table 5: Effect of rate of degradation in sunlight and UV light

Catalyst 0.01g	Concentration of Acetic acid In [N]	Sunlight 10^{3}k Sec^{-1}	Time taken for 95% Degradation in min	UV light 10^{3}k Sec^{-1}	Time taken for 95% Degradation in min
Nickel oxide Nanoparticles	1x10^{-3}	0.59	130	2.94	35

5) *Reuse of Catalyst:* The reuse of photocatalyst was examined to see the photodegradation efficiency of the acetic acid solution. After the degradation of acid, the acid sample was kept outside without expose the UV-light for 9hrs and supernatant liquid sample was decanted. The catalyst was thoroughly washed with double distilled water and reuse for the photodegradation by taking new acetic acid solution. The reuse of photocatalyst shown almost same degradation efficiency compared to the fresh sample. This can be recommended the photocatalyst can be regenerated and reused.

V. BIOLOGICAL ACTIVIY

The in-vitro antimicrobial activity of Nickel oxide Nanoparticles was evaluated against the gram-positive Staphylococcus aureus (MTCC-7443) and gram-negative Escherichia coli (MTCC-40) by disc diffusion method in Mueller Hinton Agar Plate [27-31]. 100 µL of the pathogenic bacteria cultures were transferred onto plate for 24 hours cultures of test microorganisms in broth were used for the seeding and poured to the Petri plates and allowed to cool to room temperature, in laminar air flow. Nickel oxide Nanoparticles were loaded into 6mm sterile discs and placed on the culture plates and incubated at 37^0C for 24 hours. The comparative stability of discs containing Gentamycin was made. By measuring the diameter of the ZOI formed around the disc, the antibacterial activity of Nickel oxide Nanoparticles was determined. The diameters of the zone of inhibitions (in mm) were measured after completion of the incubation. The antimicrobial activity of Nickel oxide Nanoparticles was investigated by zone of inhibition by Kirby-Bauer disc diffusion method. Disposable plates inoculated with the Gram-positive and Gram-negative bacteria, such as Staphylococcus aureus and Escherichia coli

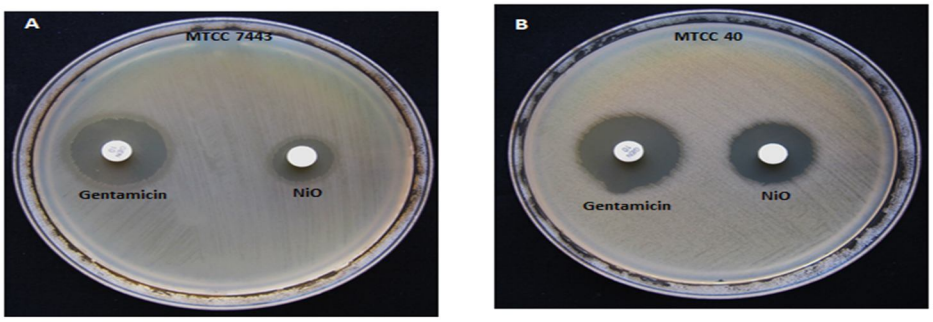

Figure 10.Appearances of inhibitory zones with (A) Staphylococcus aureus and (B) Escherichia coli bacteria.

Figure.10. shows plates to which a bacterial suspension (approximately 10^6 CFU/ml) was applied the bacteria grew to form a confluent lawn; the growth inhibition could be measured as the expansion of the clear zones surrounding the disc on the Petri dish. Nickel oxide Nanoparticles inhibited bacterial growth by the clear inhibition zone (a concentration of 10 µg/ml). The diameter of inhibition zones (in millimeters) around the Nickel oxide Nanoparticles against test strain are shown in Table 6.

Table 6: Antimicrobial effect of Nickel oxide Nanoparticles by Zone of Inhibition (mm) against test strains

Test Bacteria	Nickel oxide Nanoparticles	Positive control *Gentamycin* (10 mcg)
Staphylococcus aureus MTCC 7443	15.01 ± 0.21	32.01 ± 0.14
Escherichia coli MTCC 40	22.01 ± 0.84	30.31 ± 0.08

VI. CONCULSION

In the present manuscript, the Nickel oxide nanomaterials are synthesized by electrochemical method. The electrochemically synthesized Nickel oxide nanomaterials were characterized by SEM, EDAX, XRD, IR and UV analysis. The photodegradation by this semiconductor offers a green technology for removal of organic dyes present in waste water and industrial effluents. The photocatalytic study for the synthesized nickel oxide nanomaterials was investigated by the kinetics of photodegradation of acetic acid by volumetric method against NAOH solution.The kinetics of photodegradation of Acetic acid recommended that the dematerialize of acid follows 1^{st} order kinetics. The photodegradation rate in UV light is high compared to sunlight hence the synthesized Nickel oxide nanomaterials acts as a very good photocatalyst and is active under UV light. The completeneness of degradation was confirmed by COD measurement. The COD values revealed that -96% of the acid had been degraded. The synthesized nanomaterials show appreciably good inactivation of different strains of bacteria.

VII. ACKNOWLEDGEMENT

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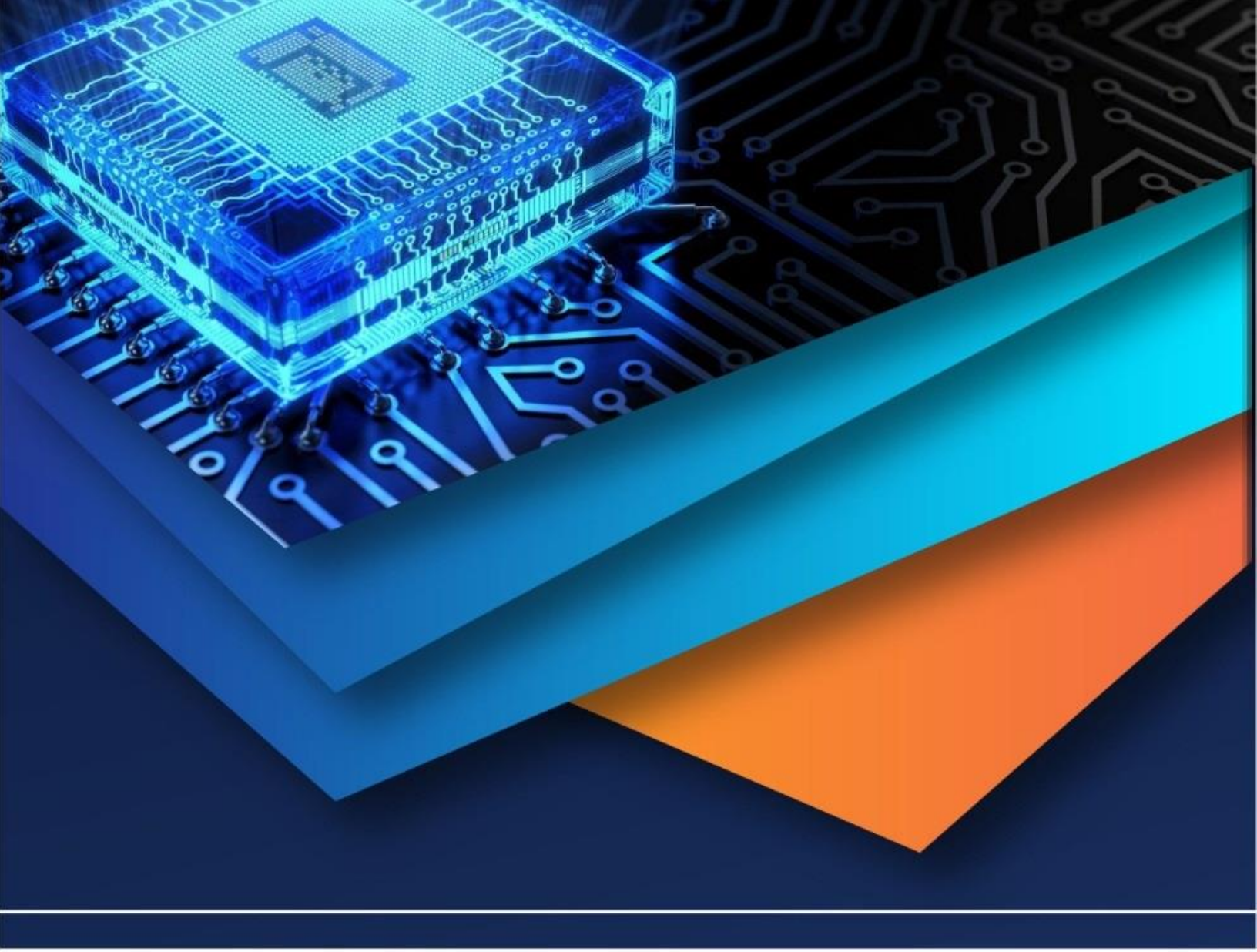

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