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NiO thin films for environmental photocatalytic applications: a review

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ABSTRACT/RESUME

Abstract: *Nickel oxide (NiO) thin film is metal oxide that has attracted much attention in recent years due to its environment friendliness. Like all metallic oxides semiconductors, thin films, NiO is gaining more and more attention as a promising photo-catalyst to replace powder catalysts, which are difficult in recycling. This paper summarized the photocatalytic activity of metallic oxides thin films. Furthermore, the results of several studies on the efficient photo-catalyst of NiO thin films and its components have also been shown. This review will be useful to researcher's investigate non-toxic materials, with low production cost and high efficiency in the field of environmental protection.*

I. Introduction

Pollution is considered one of the main problems facing the whole world, especially since it negatively affects our daily life. The examples of environmental pollution include, for example: Air pollution from oxides of sulfur and nitrogen, soil pollution due to agricultural chemicals such as chemical fertilizers, pesticides, and household and industrial waste, and Water pollution due to wastewater, oil spills, the pharmaceutical compound like antibiotics, and pollution by agricultural waste such as pesticides. In addition, using energy or burning waste to remove this type of pollution can lead to an increase in the percentage of carbon dioxide emissions into the atmosphere responsible for global warming. Therefore, it was necessary to seek an alternative to solve this environmental dilemma by finding a new environmentally friendly path. For this, researchers and scientists strongly recommend photocatalysis as an environmentally friendly technology to solve this problem [1]. This technique uses natural energy sources such as sunlight ،that are part of the environmental balance ،and a permanent and clean source of energy. Many studies concerned with the subject of environmental pollution showed the high effectiveness of this technology in removing many pollutants, especially organic ones. Numerous studies on the subject of environmental pollution have shown the high efficiency of photpcatalysts in eliminating many pollutants, in particular organic pollutants. Many scientific works have shown that almost all organic compounds can be completely oxidized on the surface of the catalyst. For this reason, the number of research studies interested in the development of this technique has increased in recent years (see figure 1). Several of these studies have focused on the use of photocatalysts to reduce organic pollution of water [2-4]. Others are interested in using photocatalysts to treat air pollution [5, 6].

The photocatalysis is the substance that causes certain reactions with light. Just like chlorophyll in photosynthesis. When exposed to light, it is active all day long as in the process of photosynthesis. The most powerful and cheapest known photocatalysis are metallic oxides (NiO, TiO2, CuO, ZnO...). Although it is very active with light, light cannot break it down. MOs (MOs) have a semiconducting behavior and low toxicity compared to metal sulfides, carbides etc [7]. Among different photocatalysts, MOs n-type semiconductors, such as titanium dioxide (TiO2), zinc oxide (ZnO), and

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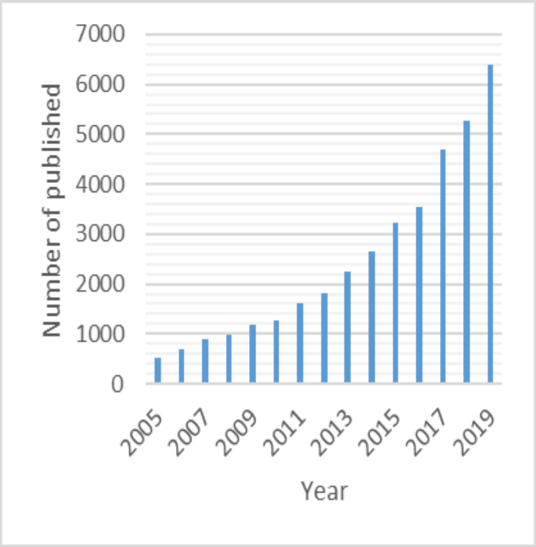

***Figure 1 .** Number of research papers per year containing keyword): "Photocatalysis" on Sience Direct*

tungsten trioxide (WO3) are popular and widely used because of their photosensitivity, nontoxicity, high oxidizing power and easy availability.

In addition, the p-type semiconductors, such as nickel oxide (NiO) and copper oxide (CuO) have drawn researchers' attention to use as photocatalysts. These metal oxides are very important semiconductors, due to their wide applications in photovoltaic devices, which are the working principle of solar cells. Light is not only important for these cells generate electricity, but it is also necessary for photocatalysts in the degradation of organic pollutants. However, the nanoparticles of MOs can cause secondary pollutants in the mineralization of environmental contaminants. It comes to the problem of separating the catalyst from the solution after the reactions. That is why their commercial value is gradually diminishing. Because it is very difficult to remove these catalysts from the solution medium in order to carry out the necessary measurements after disintegration. Although it is tempted to be removed from the environment by filtration methods, it decreases their activities with the decrease in their quantity over time. The most practical way to overcome this problem was to use thin film photocatalysts. Scientists have agreed that thin films based photocatalytic process can be a new and alternative way to overcome this problem [8]. In a review study on the evaluation of environmental photocatalysis [9], Xin Li and his colleagues summarized the different pollutants degradable by two-phase photocatalysis, as shown in Figure 2.

Given the importance of protecting the environment for humanity and life around the world, many review papers have focused on many MOs photocatalysts and their applications. However, they have not interested in the state of catalysts: nanoparticles (powder) or thin films. This review article aims to show the importance of thin films of MOs as photocatalysts. We focus on the role of nickel oxide in the degradation of organic pollutants. NiO thins films are characterized by the combination of two important properties, namely high electrical conductivity and optical transparency, which has led to the increasing interest of researchers in this material.

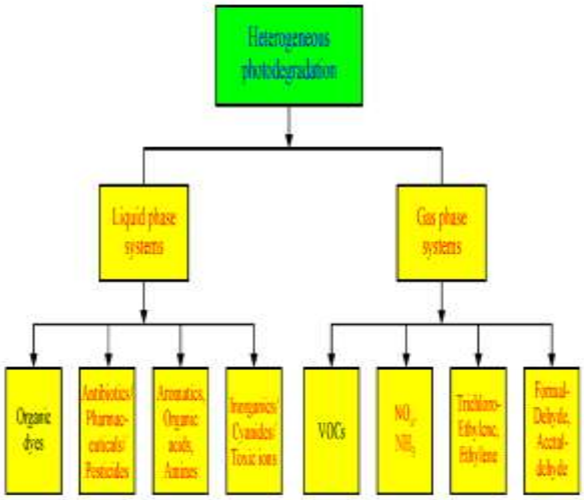

***Figure 2 .** Heterogeneous photo degradation systems for various pollutants [9]*

II. Photocatalysis

In 1972, Akira Fujishima and Kenichi Honda, while looking for new technologies to produce hydrogen, they described the process of water decomposition on the surface of titanium dioxide (TiO2) crystals under the influence of sunlight [10]. The process has been named "photocatalysis". From this moment, the development of a wide variety of photocatalysts and devices based on photocatalysis began.

Photocatalysis is a word made up of two parts: "photo" (light) and "catalysis" (decomposition, destruction). Therefore, it turns out the destructive light. The photocatalyst is a semiconductor material that creates a strong oxidizing environment on the surface under the effect of ultraviolet (UV) light. Photocatalysis offers a unique opportunity to completely oxidize organic compounds under mild conditions, since all processes take place at room temperature. This is what makes photocatalysis interesting for purifying large volumes of air and water.

When exposed to UV light, they transfer this energy to surrounding reagents to start the chemical reaction. It oxidizes certain harmful substances such as microbes, molds and bad smells, with which it is in contact, thanks to its strong oxidizing power, and transforms it into carbon dioxide, water and other small molecules [11]. The process of catalysis depends on a substance, which increases the rate of transformation of reactants without being affected or

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depleted. This substance is known as the catalyst. It increases the reaction rate by reducing the activation energy required. Therefore, the process of photosynthesis is a reaction in which light is used as a stimulant of a substance which will work to increase the rate of chemical reaction without having a role in the reaction itself (figure 3).

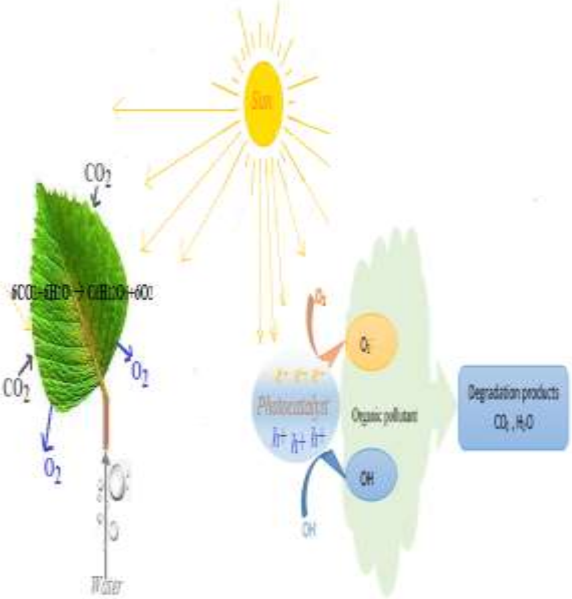

***Figure 4 .**Difference between photosynthesis and photosynthesis*

Semiconductors have been chosen to be a photocatalyst because these materials have a small energy gap between the valence and conduction beams. In order for the photocatalysis process to take place, the semiconductor material absorbs energy from the sun. The electron (e^-) travels to the conduction band, leaving a hole (positive space) (h^+) in the valence band and the positive hole transforms the water molecule into hydrogen and hydroxyl. The electron interacts with the oxygen molecule and gives a very strong oxidizing anion. This process continues as long as there is light available [12]. The most example that can be given as a photocatalyst is TiO_2 which energy gap is Eg=3.2eV. This energy is equivalent to the energy of a photon that has a wavelength equal to 388 nm and this photon is located in the ultraviolet range. Titanium dioxide has been widely used as a photocatalyst for several advantages, for example, titanium dioxide is inert, corrosion resistant and needs less processing and preparation than other semiconductors, and this makes it available at a low cost. It can also interact under normal conditions. In 1989, titanium dioxide-based paint was used to tint the ceiling and walls of an operating room in a hospital. The result was a decrease in amount of bacterial contamination in this room. Since that time, this substance has been used as an antibacterial material and to reduce pollution [3].

Zhong and Haghighat [5] claimed that TiO_2 is widely used in the new generation of building materials, including silicate, cement, glass, mortar and stone [5], [13]. These materials are photocatalytic, which can have several photochemical activities: air purification, self-cleaning and air sterilization (Figure 4).

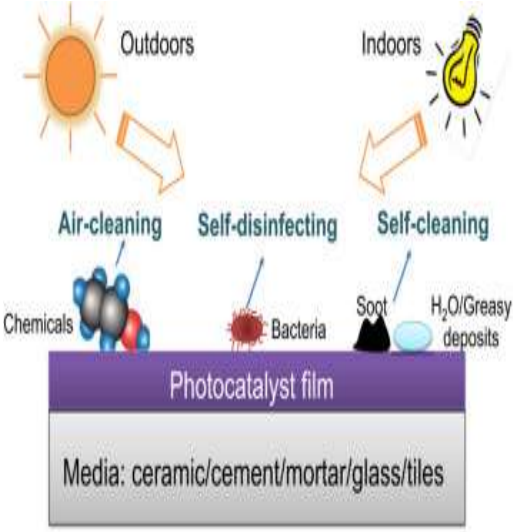

***Figure 3 .** Schematic diagram of multi-functional photocatalytic building materials [5].*

Using powdered titanium dioxide to purify water is an effective and used method, but it has its problems. According to Zhu and colleagues, the ecotoxicity of TiO_2 nanoparticles presents risks for aquatic ecosystems. The results of their study showed that for exposure to n TiO2 for 21 days, daphnia *magna* exhibited severe reproductive anomalies, growth retardation and a high mortality rate [14]. Moreover, nanoparticles (<100 nm) are suspected to have health risks [15]. Therefore, once the powder has done its job and the purification process is complete, removing the powder becomes a problem. The solution was to make a photocatalyst based on a film of titanium dioxide on a glass substrate. To remove an organic pollutant (as Methylene Blue (MB)) from aqueous solution, the film is placed in the aqueous solution under ultraviolet radiation and monitoring the degradation of the organic dye at over time.

An ideal photocatalyst should have the following properties:

- fully stable physical and chemical structure,

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- high photocatalytic activity,
- it should be easily synthesized and easy to obtain,
- be able to become active with visible light or near ultraviolet rays,
- harmless,
- non-toxic,
- it should be cheap.

III. Metallic oxides thin films

Metallic oxides (MOs) in general are bodies made up of metal atoms and oxygen atoms. They are, therefore, either simple or complex. Most of these oxides are n or p type semiconductors. p-types (hole conduction) are recognized to be relatively unstable because of their tendency to exchange oxygen from their network easily with air. The second family grouping together the n-types (electron conduction) are more stable and have properties more favorable to chemisorption [16]. MOs are classified into two broad categories: simple MOs are composed of only of a metal such as: two or more metals such as form NiO, SnO2, TiO2, SiO2…, and mixed MOs: BaTiO3, CaTiO3, Mg2SiO4.

Each semiconductor is characterized by an energy band structure. It is represented by a band diagram, in which the valence band groups together the energy levels occupied by the holes and the conduction band groups together the levels occupied by free electrons. The two bands are not completely filled which favors the displacement of electrons in the conduction band and displacement of holes in the prevalence band. When an electromagnetic wave interacts with a semiconductor, it will be completely absorbed by it if its energy is able to transfer electrons from the valence band to the conduction band. That is to say, if this energy is at least equal to that of the width of the forbidden band (the gap Eg). Bandgap is an intrinsic characteristic of semiconductors and plays an important role in various technological applications and modern devices, including photocatalysts [17, 18]. The gap is very sensitive to preparation methods and faults, as shown in Table 1. It is for this reason that several treatments and elaboration techniques have been developed in order to modify the value of the gap of MOs thin films and to improve their optical, electrical and structural properties. MOs thin films stand out as one of the most versatile materials, owing to their diverse properties and functionalities.

IV. The photocatalytic activity of NiO thin films

The interest of researchers in MOs is increasingly noticed. This is justified, because they have many properties. The main characteristics of MOs thin films are high optical absorption (as NiO) and nontoxic, low cost fabrication, high chemical and thermal stability and good photo and electrochemical stability [19, 20]. Some of them have a good ability to resist atmospheric corrosion (ZnO and TiO). Moreover, MOs have the advantage of being able to be synthesized into a large variety of nanostructures such as nanowires, nanohelix, nanotubes, nanorods, nanodendrites, nanoflakes, nanocombes and nanoflowers [21].

Metallic oxides thin films are material which many applications include solar cells, super capacitors, photocatalysis, light-emitting diodes (LEDs), UV emitters and photodetectors, field effect transistors and gas sensors [2, 3]. Certain polluting gases such as Formaldehyde (sources: paper, varnish, combustion appliances, pressed wood products, etc.), H2 gas and ammonia have dangerous consequences on the environment and on health (carcinogenic gas).

The molecules of these gases adsorbed on the surface of the NiO or thin film of the grain boundaries thereof, can capture a free electron. This therefore results in a reduction in electrical conductivity. Thus, NiO nanostructures can be used as materials for solid-state gas sensors to detect gas especially in closed environments [31]. Nevertheless, the most important is in the field of environmental protection, due to their photocatalytic activities. However, its photocatalytic performance in the degradation of pollutants is strongly affected by the methods and conditions of thin film preparation. The quality of the catalyst strongly depends on the morphology of the surface, the size of the particles, the specific surface, the crystalline quality, the transmission and the absorption of electromagnetic waves and the gap value. The nature of the substrate can also have an effect on the quality of the thin films. Scanning electron microscopy images of our NiO thin films produced by sputtering on different substrates clearly show the difference in surface morphology and therefore structural properties (figure 5).

Table 1 . Band Gap (Eg) value for certain MOs materials

MOs Thin films	Elaboration methods	Annealing temperature	Eg (ev)	substrate	Reference
SnO	Pulsed laser deposition (PLD)	575°C	2.8	zirconia	[22]
TiO_2	RF reactive sputtering	300	3.54	Glass	[23]
TiO_2:Zn (4,87 at% Zn)	RF reactive sputtering	300	3,12	Glass	[20]
CuO	Spin coating	550°C	1,89	Quartz	[24]
CuO	Ionic layer adsorption (ILA)	/	1.92	Glass	[19]
NiO	CBD	200	3,65	glass	[25]
NiO	CBD	100	3,80	glass	[25]
NiO: Cu (3at.% Cu)	Electrodeposition	160	3,2	ITO	[26]
NiO	Spin coating	350	3,94	Glass	[27]
NiO : Li (5 at.% Li)	Spin coating	350	3,85	Glass	[25]
WO_3	Thermal evaporation	/	3,30	tantalum	[28]
Ga_2O_3	RF megnetron sputtering	1000	2.05	silica	[17]
Bi_2ZnTiO_6	RF magnetron sputtering	550	1.48	Si(100°	[29]
Co_3O_4	Chemical bath deposited (CBD)	200	3,6	Glass	[25]
Co_3O4	CBD	150	2,9	Glass	
ZnO	CBD	673	3.03	glass	[21]
ZnO:Cu (1at.% Cu)	CBD	673	2,96	glass	[21]
ZnO	Sol-gel spin coating	400	3,24	quartz	[30]

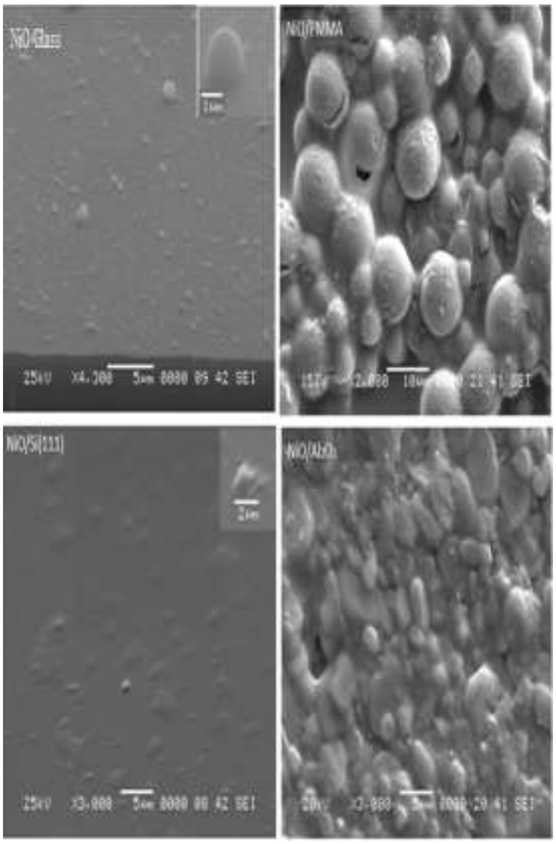

Figure 5 . *Surface morphology of NiO films on different substrates.*

Nickel oxide is an ionic chemical compound comprising in principle a “Ni” cation and an “O” anion, of formula NiO. It exists in nature as Bunsenite (a rare mineral) but it is easily synthesized from nickel compounds. It comes as a greenish gray powder. NiO crystallizes in the NaCl (rock salt) type structure. Six oxygen atoms bound each nickel atom and the same is true for the oxygen atom, which has six Ni atoms surrounding it. This face-centered cubic structure (C.F.C) has a parameter of a = 4.1769Å at 26 °C, the space group Fm3m and a rhombohedral unit cell with α = 60 °. Nickel oxide is an Electrochromic with a refractive index of 2.33 and an optical transmittance greater than 70% in the range 300nm - 900nm. NiO changes from transparent to black rapidly and reversibly following electron extraction (oxidation) and vice versa [32-34]. NiO has strong absorption in violet and red. In case of excess oxygen, i.e. Ni is oxidized directly to oxygen or doped with Li +, NiO appears black [10]. When an electromagnetic wave interacts with a semiconductor as NiO, it will be completely absorbed by it if its energy is at least equal to that of the gap (Eg). NiO thin films have a wide gap which changes value with the deposition technique (between 3.0 and 4.3 eV) preventing them from absorbing photons having an energy lower than that of the gap, and making them transparent to visible light [18, 35].

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The optical properties of NiO allow it to be used in many potential applications in different technologies. Due to the good electrochemical stability and the low cost of its thin films, many researchers have been interested in improving its photocatalyst activity. Moreover, the researchers concentrated on researching other form of catalyst other than powder because, powder have drawbacks relating to its recovery and agglomeration of particle during experimentation [36]. Noua et al. [37] investigated Photocatalytic activity of the pure NiO thin films for the Methylene blue degradation under visible light irradiation. This was achieved by sol-gel dip-coating method onto a glass substrate, by dissolving nickel acetate tetrahydrate in methanol and annealed the films in air for 2 hours at 550°C. The study revealed high photocatalytic activity in the order of 89% for 4.5h of irradiation exposure. For Wang et al. [38], the polycrystalline NiO nanowires prepared by pulsed laser deposition technic, showed a photocatalytic activity for the degradation of the acid scarlet dye much better than in the case of powdered NiO. Moreover, a very efficient electrochemical degradation of Imidaclopride s was carried out by thin films of nickel oxide (NiO) deposited on Ni substrates by a sol-gel method using nickel dichloride hexahydrate ($NiCl2$ $6H2O$) dissolved in n-butyl-alcohol ethanol. Liu et al. [39], reaching removal yields of 66.3% have, successfully carried out the electroreduction degradation of Imidacloprid, in alkaline aqueous solution. To improve the photocatalictic quality of NiO, Boulila et al. [38] have developed barium-doped nickel oxide nanofilms deposited on glass substrates using a spray pyrolysis method. The photocatalytic properties of thin films were investigated by measuring the degradation rate of methylene blue under ultraviolet irradiation. The result of their study showed that between 70–80% of MB was consumed after 300 min ultraviolet irradiation with the addition NiO with Ba concentrations in the range 0–3%.

Now coupling NiO with another semiconductor having suitable band gap energy can greatly enhance the catalytic activities. The Scientists thought about the separation of the photogenerated electron hole. This is achieved by coupling a suitable p-type and n-type semiconductor. Light excitation on the p-n heterojunction results in an integrated electric field, which diverts the photogenerated electron to the conduction band of the n-type material and from the holes to the valence band of the p-type material (Figure 6). Therefore, Due to the enhanced charge separation, the positive holes, which are prerequisite for photocatalytic decomposition, can approach toward the surface to participate in the chemical reactions without recombination with electrons. This mechanism necessarily delays the rate of recombination between pairs of electrons and photogenerated holes [36, 40, 41]. Figure 7 shows the band edge position at the p–n junction.

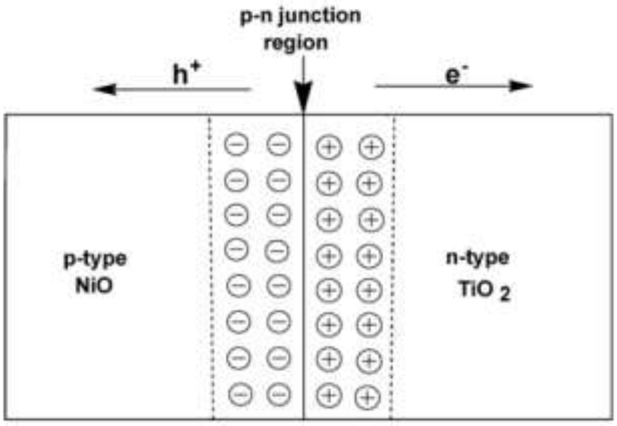

Figure 6 . *Schematic diagram of the electron-hole separation process in a p-n junction [43].*

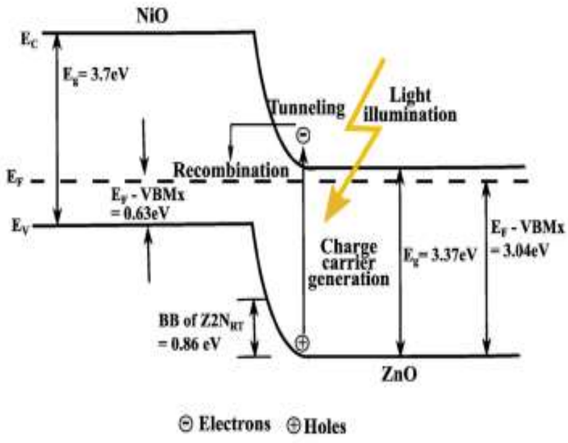

Figure 7 . *A energy band structure showing the formation of the p–n junction [39]*

n-NiO(111)/p-NiO(111) heterojunction was created by laser annealing using KrF excimer pulsed laser [42]. This p-n junction showed enhanced photocatalytic activity by increasing oxygen vacancies and Ni. Its Photocatalytic efficacy was assessed by measuring the decomposition rate of 4-chlorophenol under UV light. The photocatalytic reaction rate constants were determined to be 0.0059 and 0.0092 min-^{1} for the as deposited and the laser-treated samples, respectively [42].

The group of Chen Shifu [43] has studied the photocatalytic quality of the NiO/TiO_2 p-n junction photocatalyst. The result of their study showed that the photocatalytic activity of the p-n NiO/TiO_2 junction is much greater than that of TiO2 on the photocatalytic reduction of $Cr_2O_7^{2-}$.

Recently, Farh et al. [44] prepared n-ZnO/p-NiO heterostructures by sol-gel dip-coating technique. They used the degradation test of methylene blue (MB) aqueous solution under solar light irradiation for the photocatalytic characterization of the films. They found the degradation rate of methylene blue equal to 98.67% for 4.5h of irradiation.

In addition, a kind of three-dimensional (3D) reticulated ZnO/CNF/NiO heteroarchitectured composite was prepared by chemical vapor deposition (CVD) method [45]. This composite an excellent photocatalytic performance 2.5 times higher than that of regular ZnO / NiO composite. The bridge effect of carbon nanofibers (CNF) facilitated the separation efficiency of electrons and holes.

To improve photocatalytic efficiency, many nanocomposites thin films have been tested. Mossad and Najmy [46] prepared thin films of NiO/SiO2 by spin coating sol-gel technique on Silicon substrates. They evaluated the photocatalyst films by the photodegradation of methylene blue dye irradiated with UV light. Their results revealed a correlation between the photon yields and the annealing temperature of NiO/SiO2 films. Maximum photon efficiency is observed at 600 ◦C. Jana, Mondalb and Ghosh from India [36] have succeeded in fabricating electrodes with heterojunction nanostructures by coupling NiO and Fe_2O_3. NiO/Fe_2O_3 thin films were electrodeposited on FTO glass substrate and air annealed at 600 ° C for an hour. This heterostructure is versatile and shows high catalytic performance towards electro-oxidation methanol in alkaline media even at very low concentration. Further, it shows enhanced photocatalytic activity towards decomposition of phenol and commercial dyes e.g., Congo red (CR), Rhodamine B (RhB) of phenol, commercial dyes and photo reduction of toxic Cr (VI).

V. Conclusion

This review has introduced the properties and applications of NiO thin film. It is an excellent metallic oxide material for photocatalyst applications, efficient, low cost, durable and with a wide band gap of 3.25–4.0 eV. This gap has a critical role in photocatalysis process, which is an efficient and economic method to decompose organic pollutants. The coupling of different semiconductors with NiO to form the p-n heterojunction provides a more effective charge and acts as photo-degradation for the polluting dye and organic pollutants. Doping of metal onto NiO thin films are also in favor of increasing photocatalyst activity than undoped NiO. Further studies are encouraged in developing novel composites of NiO thin films with excellent photocatalyst activity.

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