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Chapter

Bulk and Nanocatalysts Applications in Advanced Oxidation Processes

Luma Majeed Ahmed

Abstract

Advanced oxidation processes (AOPs) are considered to be vital methods for treating the contaminations produced mainly by the human activations. In present-day, UV light or solar light, bulk and nano-photocatalysts are often used to enhance this technology by creating the highly reactive species such as the hydroxyl radicals. Extreme hydroxyl radical is considered as a key to start the photoreaction. Photoreaction is widely used in treatment of Lab and industrial contaminations, preparation of compounds and produced the renewable energy, so it’s classified as green technique. In order to improve the efficiency of this reaction with fabrication the surface of the used photocatalyst such as metal doped, sensitized and produced a composite as bulk catalyst or nano catalyst.

Keywords: nanocatalysts, bulk catalyst, advanced oxidation processes, wastewater treatment, photocatalysis, Fenton reaction, photo-Fenton

1. Introduction

In this section, the advanced Oxidation Processes concepts will be related to use of the bulk and the nano- catalysts as vital materials for easily generating a highly oxidizing species and reactive oxygen species (ROSs) such as in aqueous or alcoholic solution [1]. ROSs are contains three primary kinds: superoxide anion (O$_2^•$), hydrogen peroxide (H$_2$O$_2$) and the hydroxyl radical (HO$^•$) [2], which produced from reaction of adsorbed oxygen molecule on catalyst’s surface with one electron in conductive band under illumination by light as UV, or visible or solar light, this mechanism is useful to reduce the recombination process and increased the life time of hole in valance band [3, 4]. As explained in Figure 1.

The ROSs are having the electron configurations as tabled in Table 1 [5–8].

2. Advance oxidation process applications

In the last few years, several researches have predominated in many universities and research centers on the scientific ventures to mainly treat the contaminations that produced by textile factories [9–11], reduced the degradation of food’s
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dye [12], decolorization of colored organometallic complexes [13], degradation of toxic cyclic compounds [14] and produced a hydrogen from alcohol as renewable energy [15]. The effective materials for all above mention research are generated the hydroxyl radical in aqueous solution with maximum oxidation power equals to 2.8 V [1]. Based on to the AOPs, the common sources for creation

Figure 1.
Essential mechanism for generating the ROSs under illumination of photo-catalyst particles [1].

Table 1.
Electronic configurations and chemical formulas for the ROSs types.

dye [12], decolorization of colored organometallic complexes [13], degradation of toxic cyclic compounds [14] and produced a hydrogen from alcohol as renewable energy [15]. The effective materials for all above mention research are generated the hydroxyl radical in aqueous solution with maximum oxidation power equals to 2.8 V [1]. Based on to the AOPs, the common sources for creation
of OH in AOPs are illustrated in Figure 2, which regards as power to start the dark or photo reactions [1, 16–19]. Fortunately, the benefits of AOPs are more than those of drawbacks. The benefits of AOPs are summarized up as [1, 20] follows to:

1. Create a large number of free radicals species.
2. Have the appropriate potential to depress the hazardous organic pollutants by complete their mineralization and producing CO₂ and H₂O.
3. Reduce the time of dark or photoreaction.
4. Have low economic cost.

Whereas, the drawbacks of AOPs [1, 21] are quenching the reaction rate with increasing the scavenger contains (mostly peroxide ion) and may be generated the undesirable hazardous products that prevented the complete of mineralization process, hence, the altered of pH or using further cost steps may be essentially to treat their problems.

3. Bulk and nano-catalysts

In general, the catalysts may be metal or alloy or semiconductor. Semiconductor is wide used as catalyst and can be element or compound as amorphous or crystalline or rock salt crystal. Because of semiconductors have intermediate properties between metal and insulator, which has given them rescannable electronic and structural properties, hence, semiconductor is classified as a better-known kinds, as mentioned in Figure 3 [22–24].

The usages of the bulk and nano catalysts are increment with increasing the development of life activations. The catalysts were known for the long time to increase the rate of reaction with decreasing the time of reaction and the activation energy in dark reaction or photoreaction. In order to use the catalyst in
photoreaction as photo catalyst, must have a band gap with raged about 1.1 eV to 5.0 eV \([1, 24]\). Referring to Figure 4, several band gap energy positions of some common photo catalysts can be displayed \([1, 25–27]\). The mainly problem in bulk and nano catalyst is recombination process, which results in diminishing the efficiency of used photocatalyst by returning the photoelectron from conductive band to valance band and reacting with photohole immediately. The recombination includes four kinds can be followed in Table 2 and Figure 5 \([1, 28–30]\).

In order to improve the activity of photocatalysts must depress the recombination with modify their surfaces with three main methods: surface sensitization, metalized photocatalyst surface and coupled for two or more photocatalysts as Composite. The details of these modification methods are mention in Table 3 and Figure 6 \([40]\).
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### Table 2.
The most common recombination types concepts.

| Kinds                        | Other name                          | Info                                                                                                                                                                                                 | Type of photocatalyst |
|------------------------------|-------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| Direct recombination         | Band-to-band recombination          | In this kind, the transition occurs as a radiative transition in direct band gap semiconductor. It is created when the Free photo electron in CB drops directly into free photo hole (an unoccupied state) in the VB and associated together. Note Figure 5(A). | ZnO have a direct band gap. |
| Volume recombination         | Centers recombination or Trap-assisted recombination | This case obtains, when defect of semiconductor by impurities that given a new levels (as traps of photoelectron and photohole). It leads to liberate heat as phonon in indirect band gap semiconductor. Note Figure 5(B). | Pure TiO$_2$ and defect of TiO$_2$, by metal, which had given an indirect band gap. |
| Surface recombination        | Recombination of an exciton         | This case occurs at low temperature, when the traps at or near the surface or interface of the semiconductor, capture the photo electron- hole as exciton. That attitude to dangling bonds caused by the sudden discontinuation of the semi-conductor crystal with energy just below the band gap value. Note Figure 5(C). | It happed in solar cells and light emitting diode (LED) containing shallow levels. |
| Auger recombination          | —                                   | This recombination involves three carriers: Free photo electron, free photo whole recombine, and the emitting the energy as heat or as a photon (non-radiative process). The transition of energy deals with as intra-band transitions, which resulting when either electron elevates in higher levels of conduction band or hole deeper push into the valence band. Note Figure 5(D). | This case can be obtained wit short lifetime when heavy doping defects (like Ag) in direct-gap semiconductors under present sunlight. |

Figure 5.
The schematic diagram of the most common recombination kinds.
| Kinds                | Info                                                                                                                                                                                                 | References |
|----------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------|
| Surface sensitization| This case favors for modified the wide band gap semiconductor by physical or chemical adsorption of colored materials mostly dye. The colored material will absorb the visible or solar light after irradiation, and excite it either singlet or triplet excited state. The excited colored material will inject its electron via the conductive band of semiconductor. | [31–34]   |
| Metallized photocatalyst surface | The metal deposits on the surface of semiconductor must choose with high work functions of metal compared to work function of the metal in semiconductor. The doped metal will act as sink of electron, with create a Schottky barrier. That will increase the lifetime of photo holes. Examples: Pt and Au doped on TiO$_2$, Ag doped on ZnO, Cr and Mn doped on ZnS. | [2, 4, 14, 35–37] |

**Coupled for two or more photocatalysts as composite**

When the energy of the irradiated light is not enough to promote electron from conductive band of the photocatalyst, that attitude to it has a big band gap, hence, can couple it with other semiconductor has a small band gap. This coupling process includes three kinds type (I), type(II) and type(III).

**Type I**

**Type II**

**Type III**

Examples: CdS-ZnS (Type I), WO$_3$-TiO$_2$ (Type II). And Mn$_2$O$_3$, ZnO, (Type III).

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*Table 3.*

The description of the methods for modifying photocatalysts [31–39].
| Application field | Type of used AOPs | Efficiency | References |
|-------------------|------------------|------------|------------|
| Textile dye       | O₂/UV-A(250 W)/ZnO/ H₂O₂ | 89.8% (Photodecolorization) (5 mmole/L of H₂O₂ (T = 25°C), (pH = 10) | [41] |
| Textile dye       | O₂/UV-A(250 W)/ZnO | 92.7% (Photodecolorization) (T = 35°C), (pH = 6.68) | [42] |
| Textile dye       | O₂/UV-A(250 W)/ZnO | 91.41% (Photodecolorization) (T = 38°C), (pH = 6.75) | [43] |
| Industrial dye    | O₂/UV-A/ZnO | 99.07% (Photodecolorization) (T = 15°C), (pH = 7.63) | [44] |
| Industrial dye    | O₂/UV-A(125 W)/ZnO | 74.4.5% (Photodecolorization) (T = 38°C), (pH = 8.6) | [32] |
|                   | O₂/UV-A(250 W)/ZnO | 98.5% (Photodecolorization) (T = 38°C), (pH = 8.6) | |
|                   | O₂/Solar/ZnO | 96.5% (Photodecolorization) (T = 42°C), (pH = 8.6) | |
| Textile dye       | O₂/UV-A(125 W)/ZnO | 94.40% (Photodecolorization) (T = 20°C), (pH = 7.7) | [10] |
|                   | O₂/UV-A(125 W)/ZnO/Fe²⁺ | 60.86% (Photodecolorization) (T = 20°C), (pH = 7.7) | |
|                   | O₂/UV-A(125 W)/ZnO/Fe²⁺+1% H₂O₂ | 16.44% (Photodecolorization) (5 x 10⁻⁴ mole/L of Fe²⁺ (T = 20°C), (pH = 7.7) | |
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| Application field | Type of used AOPs | Efficiency | References |
|-------------------|-------------------|------------|------------|
| Drug dye Cobalamin(Vit B12) | $\text{O}_2$/UV-A(250 W)/ZnO | 79.33% (Photodecolorization) (T = 30°C), (pH = 6.5) | [19] |
| | $\text{O}_2$/UV-A(250 W)/ZnO/ $\text{K}_2\text{S}_2\text{O}_8$ | 88.75% (Photodecolorization) (1 x 10^{-4} mole/L) of $\text{K}_2\text{S}_2\text{O}_8$ (T = 30°C), (pH = 6.5) | |
| | $\text{O}_2$/UV-A(250 W)/ZnO/ 0.025% $\text{H}_2\text{O}_2$ | 90.80% (Photodecolorization) (T = 30°C), (pH = 6.5) | |
| | $\text{O}_2$/UV-A(250 W)/ZnO/ $\text{K}_2\text{S}_2\text{O}_8$ + 0.025% $\text{H}_2\text{O}_2$ | 95.85% (Photodecolorization) (1 x 10^{-4} mole/L) of $\text{K}_2\text{S}_2\text{O}_8$ (T = 30°C), (pH = 6.5) | |
| Food dye Carmoisine (E122) dye | $\text{O}_2$/UV-A(250 W)/ZnO | 73.11% (Photodecolorization) (T = 18°C), (pH = 7.55) | [12] |
| | $\text{O}_2$/UV-A(250 W)/ZnO/ 0.1% $\text{H}_2\text{O}_2$ | 62.58% (Photodecolorization) (T = 18°C), (pH = 7.55) | |
| | $\text{O}_2$/UV-A(250 W)/ZnO/ $\text{Fe}^{2+}$ | 36.99% (Photodecolorization) (1 x 10^{-5} mole/L) of $\text{Fe}^{2+}$ (T = 18°C), (pH = 7.55) | |
| Lab materials Co(II) Complex of Schiff Base | $\text{O}_2$/UV-A(250 W)/ZnO | 99.11% (Photodecolorization) (T = 38°C), (pH = 7.55) | [13] |
| Industrial dye Methyl green dye | $\text{O}_2$/UV-A(400 W)/ ZnO NPs | 37% (Photodecolorization) (T = 25°C), (pH = 5.4) | [35] |
| | $\text{O}_2$/UV-A(400 W)/Ag(2%) ZnO NPs | 87.87% (Photodecolorization) (T = 25°C), (pH = 5.4) | |
| Liberated of hydrogen from Methanol as renewable energy | Ar/UV-B(1000 W)/ (0.5 Pt) $\text{TiO}_2$ NPS | 8.8% (Photo hydrogen production) (T = 25°C), (pH = 7.3) | [14] |
| | Ar/UV-B(1000 W)/ (0.5 Au) $\text{TiO}_2$ NPS | 4.5% (Photo hydrogen production) (T = 25°C), (pH = 7.3) | |
| Industrial dye Light Green SF Yellowish (Acid Green 5) Dye | $\text{O}_2$/UV-A(400 W)/ $\text{TiO}_2$ NPS | 90.2% (Photodecolorization) (T = 20°C), (pH = 7.3) | [45] |
| | $\text{O}_2$/UV-A(400 W)/ $\text{TiO}_2$ NPS | 88.1% (Photodecolorization) (T = 20°C), (pH = 7.3) | |
| Application field | Type of used AOPs | Efficiency | References |
|-------------------|------------------|------------|------------|
| **Industrial dye** | **Safranine O Dye** | O$_2$/UV-A(125 W)/TiO$_2$ NPS | 90.2% (Photodecolorization) (T = 30°C), (pH = 6) | [34] |
| | | O$_2$/UV-A(125 W)/TiO$_2$ NPS/Fe$^{2+}$ | 85.92% (Photodecolorization) (1 x 10$^{-3}$ mole/L) of Fe$^{2+}$ (T = 30°C), (pH = 6) | |
| | | O$_2$/UV-A(125 W)/TiO$_2$ NPS/Fe$^{2+}$/0.1% H$_2$O$_2$ | 92.73% (Photodecolorization) (T = 30°C), (pH = 6) | |
| | | O$_2$/UV-A(125 W)/TiO$_2$ NPS/0.1% H$_2$O$_2$+Fe$^{2+}$ | 98.83% (Photodecolorization) (1 x 10$^{-3}$ mole/L) of Fe$^{2+}$ (T = 30°C), (pH = 6) | |
| **Industrial dye** | **Acid Red 87 (Eosin Yellow) dye** | O$_2$/UV-A(250 W)/TiO$_2$ NPS | 63.58% (Photodecolorization) (T = 25°C), (pH = 6.09) | [16] |
| | | O$_2$/UV-A(250 W)/TiO$_2$ NPS/H$_2$O$_2$ | 50.44% (Photodecolorization) (1 x 10$^{-2}$ mmole/L) of H$_2$O$_2$ (T = 25°C), (pH = 6.09) | |
| | | O$_2$/UV-A(250 W)/WO$_3$ NPS | 27.84% (Photodecolorization) (T = 25°C), (pH = 6.09) | |
| | | O$_2$/UV-A(250 W)/WO$_3$ NPS/H$_2$O$_2$ | 21.54% (Photodecolorization) (1 x 10$^{-2}$ mmole/L) of H$_2$O$_2$ (T = 25°C), (pH = 6.09) | |
| | | O$_2$/UV-A(250 W)/WO$_3$/TiO$_2$ nanocomposite | 25.11% (Photodecolorization) (T = 25°C), (pH = 6.09) | |
| | | O$_2$/UV-A(250 W)/WO$_3$/TiO$_2$ nanocomposite+H$_2$O$_2$ | 73.88% (Photodecolorization) (1 x 10$^{-2}$ mmole/L) of H$_2$O$_2$ (T = 25°C), (pH = 6.09) | |
| **Industrial dye** | **Methyl green dye** | O$_2$/UV-A(250 W)/ZrO$_2$ | 92.31% (Photodecolorization) (T = 30°C), (pH = 5.4) | [46] |
| | | O$_2$/UV-A(250 W)/ZrO$_2$+Fe$^{2+}$ | 39.93% (Photodecolorization) (1 x 10$^{-3}$ mmole/L) of Fe$^{2+}$ (T = 30°C), (pH = 5.4) | |
| | | O$_2$/UV-A(250 W)/ZrO$_2$+1.5%H$_2$O$_2$ | 98.78% (Photodecolorization) (T = 30°C), (pH = 5.4) | |
| | | O$_2$/UV-A(250 W)/ZrO$_2$+K$_2$S$_2$O$_8$ | 74.62% (Photodecolorization) (1 x 10$^{-3}$ mmole/L) of K$_2$S$_2$O$_8$ (T = 30°C), (pH = 5.4) | |
| **Lab materials** | **Fe(II)-(4,5-DIAZAFLUOREN-9-ONE 11) COMPLEX** | O$_2$/UV-A(400 W)/MnO$_4$ | 22.64% (Photodecolorization) (T = 15°C), (pH = 4) | [47] |
| | | O$_2$/UV-A(400 W)/(1)MnO$_4$- (4) ZrO$_2$ nanocomposite | 40% (Photodecolorization) (T = 17°C), (pH = 4) | |
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4. Used of bulk or nano catalyst in AOPs

There are many common application of AOPs in environment fields by using the white photocatalyst or its modified such as ZnO, TiO$_2$, ZrO$_2$, ZnS, WO$_3$, CdS and Mn$_3$O$_4$. The efficiencies with used these photocatalysts are altered with using AOPs methods. The efficiency of the photoreaction depends mostly on the concentration of colored material, initial pH which affected on the surface of photocatalyst and the temperature. As shown in Table 4.

5. Conclusions

This chapter focuses on the source of hydroxyl radical which produces via the advance oxidation process. Indeed, this process interests in the forming the different species, which in the final step generates a hydroxyl radical. The photocatalyst enhances the generating of hydroxyl radicals (2.8 V) in aqueous solution under Uv-light or visible or solar. The photoexitation of photocatalyst leads to jump of electron to conductive band then return to valance band and liberates a hot this process called recombination. It is depressed the efficiency of photoreaction. However, some procedures used to modify the photocatalyst surface.

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| Application field | Type of used AOPs | Efficiency | References |
|-------------------|-------------------|------------|------------|
| Textile dye       | O$_2$/UV-A(400 W)/ ZnS NPs | 59% (Photodecolorization) (T = 15°C), (pH = 6.3) | [36] |
| Reactive blue 5 dye | O$_2$/UV-A(400 W)/ Cr-ZnS NPs | 94% (Photodecolorization) (T = 17°C), (pH = 4.1) | |
| Industrial dye    | O$_2$/UV-A(400 W)/ ZnS NPs | 95% (Photodecolorization) (T = 30°C), (pH = 75) | [39] |
| Congo red dye     | O$_2$/UV-A(400 W)/ CdS-ZnS nanocomposite | 98% (Photodecolorization) (T = 30°C), (pH = 75) | |

Table 4. Some applications of bulk and nano photocatalysts in AOPs, with environment chemistry and green chemistry.
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