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Promoting visible-light degradation of toluene over a simple constructed TiO_2/Pd nanocomposite as photocatalytic coating air purification filter

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Abstract

A series of air filters based on the TiO_2/Pd nanophotocatalyst were prepared using the colloid solution method in the presence of polyvinyl alcohol (PVA) as a surfactant. The morphology, surface area, and visible light adsorption properties, were investigated using FESEM, BET, and DRS analysis. As a result, the nitrogen adsorption-desorption isotherms of the catalysts showed that the N_2 adsorption ability of the resulted nanophotocatalysts (1.5 Wt.% of PVA) was better than the TiO_2 and other catalyst samples with more surface area. The photocatalytic activity of the nanophotcatalyst filter was investigated in the experiments of the photocatalytic degradation of toluene in a gaseous phase under visible light illumination. Among all the filters, the 10 Wt.% nanophotcaotalyst filter demonstrated the highest photocatalytic ability to decompose toluene. The photocatalytic efficiency of the nanophotocatalyst with the 1.5 Wt.% of PVA was remarkably enhanced by toluene removal. The LED lamp-60 W achieved the highest photocatalytic activity. The enhanced photodegradation of toluene is possibly due to the improved adsorption ability and the enhanced electron-hole pairs separation due to the presence of Pd nanoparticles on the surface of TiO_2 nanocatalyst and the electron transfer between the conduction band/defect level and Pd nanoparticles.

Keywords: Photocatalysis, Toluene removal, Gaseous phase, TiO_2/Pd nanophotocatalyst, Polyester nonwoven fabric

1. Introduction

Indoor air quality within buildings is a critical concern since many metropolitans generally spend more than 80% of their time in an indoor environment [1,2]. BTEX (benzene, toluene, ethylbenzene, and xylene) represents a group of volatile organic compounds that can cause adverse health impacts on occupants and constitute a serious threat to groundwater and indoor environments. Recently the degradation of them attracted substantial attention from researchers in all of the world [3,4]. Visible light active photocatalysis using TiO_2 semiconductors is one of the most attractive advanced oxidation processes based on the green chemistry principles [5, 6] and can be remarkably used as an efficient tool for the degradation of various environmental pollutants such as dye [7,8], drug [9], and so for. However, some literature has proved that the incorporation of Nobel metals as dopants into the TiO_2 matrix could promote the chemical or physical adsorption of organic substrates on the catalyst surface in an aqueous phase and also boost the impact of photocatalytic activity under visible light irradiation [10,11]. In a typical application for the TiO_2 photocatalysis system, TiO_2 has commonly been used in a powder and slurry form which is costly due to the post-recover process of separation (both time and money) [12,13]. Moreover, a pressure drop as the main drawback is also observed in packed columns for use in the gaseous phase test. For this reason and to solve these problems, TiO_2-immobilized onto solid material could overcome these limitations. Among supporting materials (polymer, glass, cement, red brick, and inorganic fibers), polyester nonwoven fabric is economical, flexible, corrosion-resistant, and easy to handle [14-16]. The support fabric can be used for many types of conservation and restoration work, e.g. to support fragile objects during conservation treatments, as a release material in wet processes (support while immersing objects in water where its structure allows instant drainage), and as an interleaving material.

However, there are fewer studies on the photocatalytic degradation of toluene in a gaseous phase using Pd-doped TiO_2 prepared by the colloid solution method. Thus, in this study, the results of characteristics of

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prepared TiO_2/Pd using various analytical techniques such as UV-DRS, FESEM, and BET were reported. The photodecomposition of toluene by the visible light responsive TiO_2/Pd under visible light in the gaseous phase was compared with commercial TiO_2. The study of the reaction factors including PVA concentration, catalyst loading in air filter, and LED lamp intensity were investigated to achieve the optimal conditions for the toluene photodecomposition reaction.

2. Experimental

2.1. Materials

Titanium(IV) oxide nanopowder (TiO_2) nanoparticles (21 nm), palladium chloride ($PdCl_2$), polyvinyl alcohol (PVA) as surfactant, sodium borohydride ($NaBH_4$) as reducing agent were purchased from Sigma-Aldrich.

2.2. TiO_2/Pd nanophotocatalyst preparation

0.5 g of TiO_2 nanoparticle powder was dispersed into a clear solution of PVA surfactant/water with different concentrations including (0.5 Wt.%, 1 Wt.%, and 1.5 Wt.% of PVA) and then 0.08 mL of an acidic solution of $PdCl_2$ was added to the three TiO_2 nanocolloids. To prepare a stable TiO_2/Pd nanocolloids and Pd nanoparticles formation on the surface of TiO_2 nanoparticles, while stirring the nanocolloid for 30 min, 3 mL NaBH4 solution (0.1 M) was added to form a dark brown solution [19-21] and a UV lamp (20 W) was irradiated for about 45 minutes to dope Pd nanoparticles into TiO2 nanoparticles. Thereafter, the prepared nanocolloid was centrifuged for 10 min (2000 RPM). The obtained black precipitate (Pd nanoparticles supported on the TiO_2 nanoparticles) was washed with water and dried for 24 h at room temperature. Finally, TiO_2/Pd nanophotocatalyst was calcined at 300 °C for 4 h (45 °C/min) in an air atmosphere.

2.3. Air purification filter preparation

A stable nanocolloid based on the 2, 5, 10, and 15% by weight of the visible light active TiO_2/Pd nanophotocatalyst in water was obtained and stirred to prepare a visible light-activated photocatalytic coating composition. Further, the activated carbon was impregnated into a polyester nonwoven fabric, heat-sealed, air permeable, chemically inert, and with an extremely smooth surface finish having a thickness of 1.78 to prepare an activated carbon-containing porous substrate. The support fabric can be used for many types of conservation and restoration work, e.g. to support fragile objects during conservation treatments, as a release material in wet processes (support while immersing objects in water where its structure allows instant drainage), and as an interleaving material. The activated carbon-containing porous substrate contained 55 Wt.% of the activated carbon. Then, the activated carbon-containing porous substrate was immersed in the visible light-activated photocatalytic coating formulation and dried at room temperature to prepare an air purification filter.

2.4. Characterization

The commercial Titania nanoparticles and the prepared photocatalysts were characterized via various analysis techniques. The images of the all samples to determine the particle sizes, shape and morphology were obtained by TESCAN MIRA – High-resolution analytical FESEM. UV–Vis absorption spectra were recorded on UV–Vis spectrophotometer (S-4100, Sinco) with UV and visible light modes to confirm the role of Pd nanoparticles as dopant decorated on the surface of Titania. To calculate the specific surface area of each sample by using the BET method the (UPA-150, Mictotrac) was used.

2.5. Toluene removal study with the prepared photocatalytic air purification filter

The photocatalytic degradation activity and performance of each photocatalyst filter were evaluated using a designed photoreactor. The experimental setup of a gas-supply device, photodegradation reactor, and gas chromatograph is diagrammed in Fig. 1 schematically. The adjusted size of the catalyst filter used for each test was 10 cm 3 cm 1.78 mm g. The photocatalyst-coated filter was then placed in a quartz reactor, which was kept in the dark and maintained at room temperature.

During the evaluation of photocatalysis, one LED lamp was illuminated to vary light intensity (20, 40, and 60 W) to compare the photocatalytic reactivity under each condition. Solutions of toluene at concentrations of 15 ppm in air (79% N_2 and 21% O_2) were introduced into the quartz reactor at a flow rate of 20 mL/min. The photocatalyst was stabilized by gas flow for 120 min.

Toluene was allowed to reach adsorption equilibrium with the photocatalyst filters in the reactor before light irradiation. The concentration of toluene was measured over 280 min using a GC-FID (Agilent MODEL 8890 GC). The toluene removal rate was calculated using the following Eq. 1, by measuring a concentration of toluene before entering the chamber (C_0, here in after referred to as a first concentration) and a concentration of toluene in the air after passing through the chamber (C_t here in after referred to as a second concentration).

$$\text{Toluene removal rate (\%)} = (C_0 - C_t) / (C_0) \times 100 \qquad (1)$$

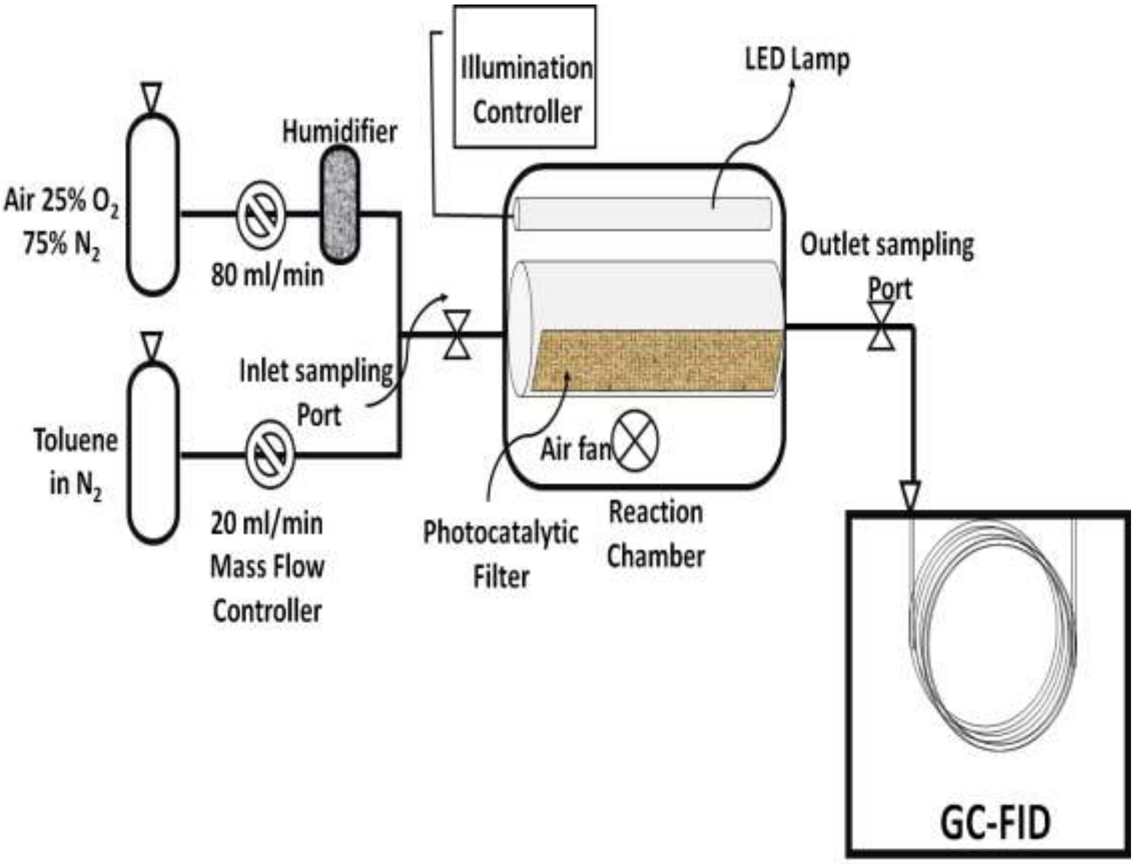

Fig. 1. Schematic diagram of the toluene removal experimental setup for the visible light activated photocatalytic reaction with TiO_2/Pd filter

3. Results and Discussion

3.1. UV-Vis-DRS analysis

The UV-DRS spectra of commercial TiO_2, synthesized TiO_2/Pd are illustrated in Fig. 2. DRS is an optical analytical technique used to quantify the amount of light scattered from a solid, powdered, or liquid sample over a range of wavelengths to study sensitization of TiO2 photocatalyst under visible light illumination. The absorption edge of the Pd doped TiO_2 nanophotocatalyst sample was shifted to the red band region compared to the P25 due to the surface plasmon resonance of Pd nanoparticles decorated on the surface of TiO_2 nanophotocatalyst. It also higher absorption band than pure TiO_2 in the visible region ($\lambda > 400$ nm) and lower band gap energy (E_g)by using the equation $Eg = 1240/\lambda$. The absorption edge red-shift as well as the increase in the visible region absorption of TiO_2 doped with transition metals could usually be explained by the difference in energy levels between the Pd nanoparticles dopant and the TiO_2 valence and conduction bands [22-24].

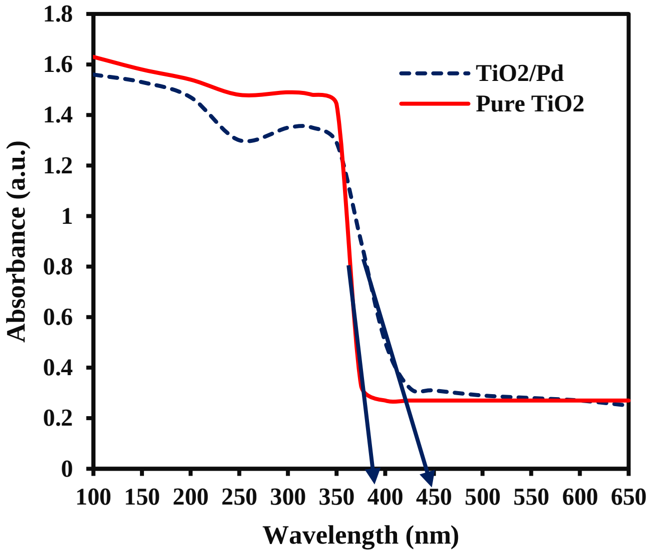

Fig. 2. The UV-DRS spectra of commercial TiO_2 (P25), and the synthesized TiO_2/Pd (1.5 Wt.% of PVA)

3.2. FESEM study

The different nanocatalysts, commercial TiO_2, TiO_2/Pd nanophotocatalyst (1.5 Wt.% of PVA) were imaged by FE-SEM (Fig. 3). The commercial TiO_2 consisted of spherical and regularly shaped particles with a wide size range (18–50 nm), as shown in Fig. 2 (as inset). The TiO_2/Pd nanophotocatalyst formed relatively uniform particles size (20–65 nm) with some nanoagglomerates, and had a grape cluster-type structure as shown in Fig. 2c. The prepared TiO_2/Pd nanophotocatalyst was less agglomerated due to the colloid solution and formed nanomicelles as nanoreactor [25,26], indicating that the addition of Pd nanoparticles could effectively suppress the crystal growth of TiO_2.

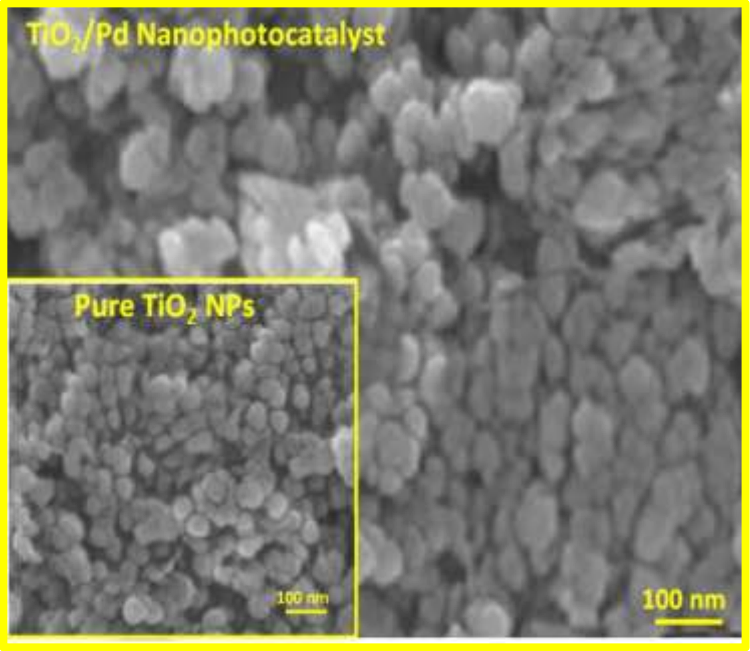

Fig. 3. FESEM images of the commercial TiO_2 nanoparticles (NPs), and TiO_2/Pd (1.5 Wt.% of PVA) nanophotocatalyst before impregnated into a polyester nonwoven fabric.

3.3. Brunauer-Emmett-Teller (BET) analysis

Fig. 4 presents the nitrogen adsorption-desorption isotherms of the resulting TiO_2/Pd nanophotocatalysts at different concentrations of PVA surfactant. The isotherms of the TiO_2/Pd nanophotocatalysts sample could be attributed to type IV, and the curves exhibited hysteresis loops at high relative pressures, indicating a mesoporous structure. Furthermore, the specific surface areas and average pore sizes of these samples as calculated by the BET method, TiO_2/Pd (1.5 Wt.% of PVA) resulted in BET surface areas of (113.425 m^2/g) and the average pore sizes of (10.1234 nm). This result may be because the average pore size is related to the crystallite size of a TiO_2/Pd nanophotocatalyst sample. As calculated by the BET method, TiO_2/Pd ((0.5 and 1.0 Wt.% of PVA) resulted in BET surface areas of 64.022 and 65.724 m^2/g and relatively large average pore sizes of 16.7723 and 17.2044 nm, respectively. Due to the high-concentration of PVA surfactant (1.5 Wt.%), the nanospheres of TiO_2 dispersed with high monodispersity and no growth and agglomeration, which would cause the high surface area of TiO_2 nanoparticles and create fine voids to decorate of Pd nanoparticles**.**

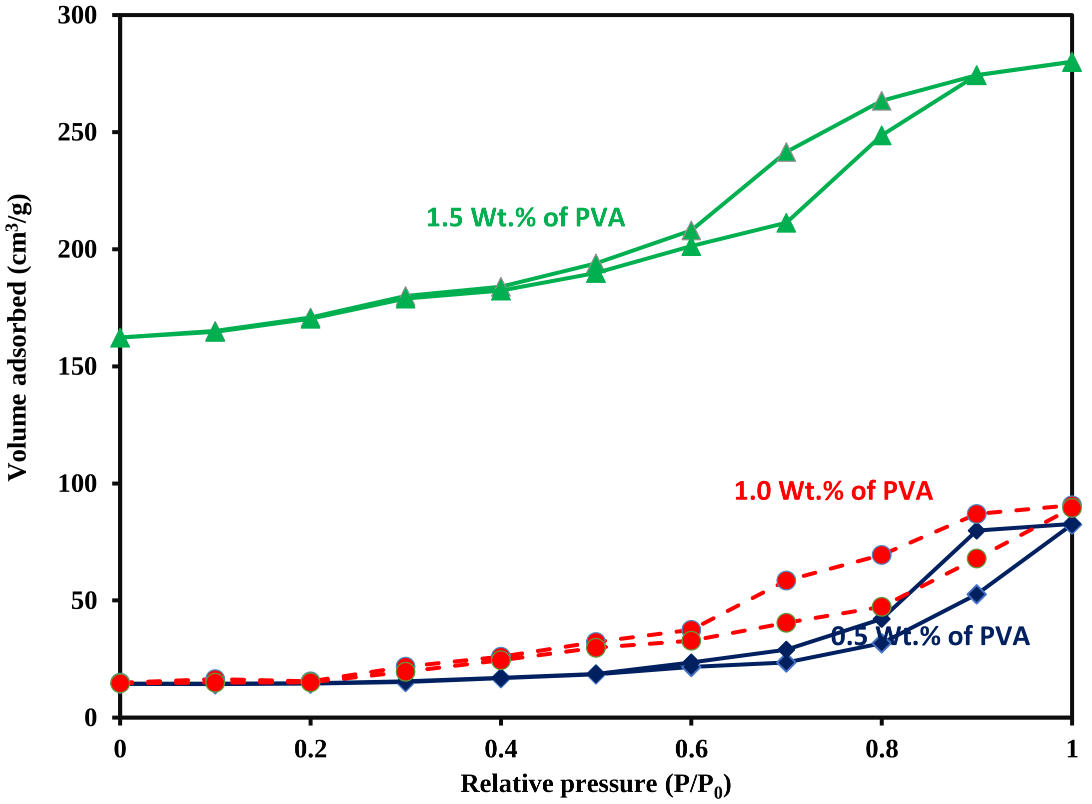

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Fig. 4. Nitrogen adsorption-desorption isotherms of the resulted TiO_2/Pd nanophotocatalysts at different concentration of PVA surfactant

3.4. The PVA surfactant concentration effect on the photocatalytic activity

The effect of different concentrations of PVA on the degradation of toluene by the TiO_2/Pd nanophotocatalyst was investigated to confirm the main active species and provide insight into the potential photocatalytic mechanism of TiO_2/Pd photocatalysts. Three different PVA concentrations were selected to prepare nanophotocatalyst. Fig. 5 shows that 1.5 Wt.% of PVA markedly increased the photodegradation rate of toluene, whereas the prepared nanophotocatalyst based on the 0.5 and 1.0 Wt.% of PVA barely inhibits the photocatalytic efficiency compared to pure TiO_2 nanoparticles. Thus, we conducted a set of comparative experiments. The prepared nanophotocatalyst with 1.5 Wt.% of PVA inhibited the degradation of toluene more strongly than both samples with low concentrations of PVA. Therefore, these results suggest that $^{\cdot}OH$ and $^{\cdot}O_2^{-}$ were the primary active species that governed the photocatalytic process, and no recombination of the prepared charge carriers (e^- and h^+) as the main limitation was not observed and more electron-hole pairs are produced [27-29].

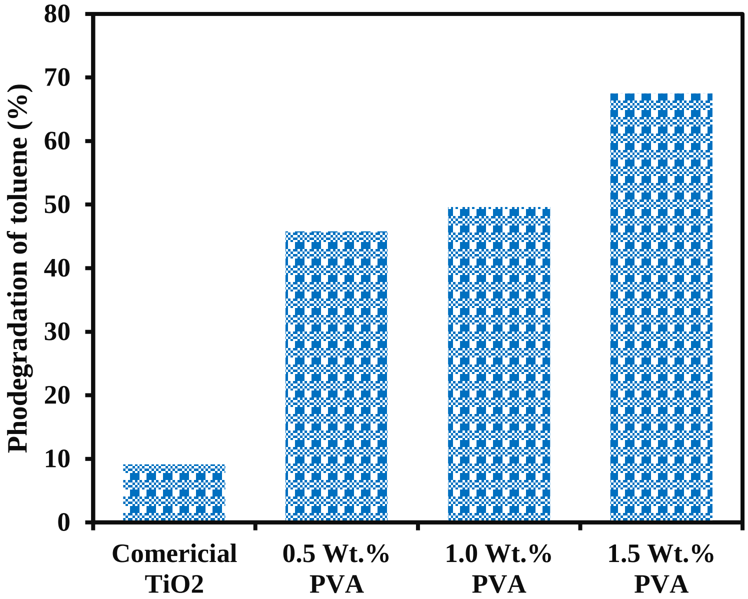

Fig. 5. The PVA surfactant concentration effect on the photocatalytic activity of toluene degradation under visible light irradiation

3.5. The effect of TiO_2/Pd nanophotocatalyst loading

The photocatalytic performance of the synthesized TiO_2/Pd nanophotocatalyst which was prepared with 1.5 Wt.% of PVA surfactant as the best sample was assayed with different loading of nanocatalyst composition in the obtained filter. Using one to four filter, the TiO_2/Pd nanophotocatalyst catalyst's activities toward the photodegradation of toluene were measured under visible light irradiation with LED lamp (20 W) (Fig. 6). The results clearly illustrate that photocatalytic activity increased with increasing concentration of the TiO_2/Pd nanophotocatalyst until 10 (Wt.% of TiO_2/Pd nanophotocatalyst) and the maximum photodegradation efficiencies toward toluene was 67.5 %, while those under filters with more concentration of TiO_2/Pd nanophotocatalyst have a slight decrease in photocatalytic activity. So, this filter with 10 (Wt.% of TiO_2/Pd nanophotocatalyst) was chosen as optimum for all further experiments.

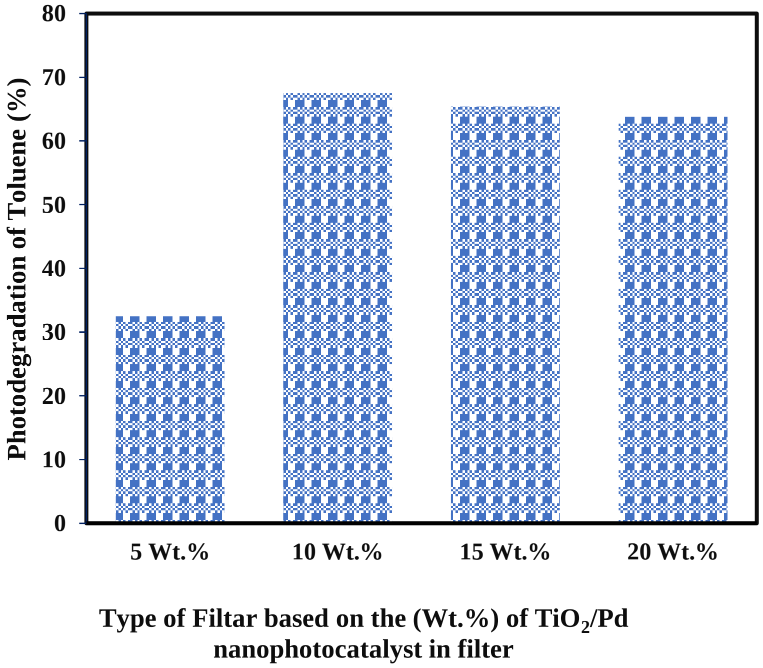

Fig. 6. TiO_2/Pd (1.5 Wt.% of PVA) nanophotocatalyst loading in the prepared filter for toluene degradation under visible light irradiation

3.6. The effect of light intensity

Light irradiation is the driving force for photocatalytic reactions; therefore, the intensity of irradiation directly affects the photocatalytic reaction rate. As the intensity of light increases, more electron–hole pairs are produced. The photodegradation of toluene in TiO_2/Pd TiO_2/Pd (1.5 Wt.% of PVA) nanophotocatalyst at different visible light intensities was tested by illuminating 3 different LED lamps including 20, 40, and 60 W (Fig. 7) at same condition. There was a distinct enhancement in photodegradation with increased visible light energy (Fig. 8a and c). Under visible light, the efficiency of toluene photodegradation increased remarkably by LED-60 W lamp, as shown in Fig. 7. The high photocatalytic activities can be attributed to the large specific surface area and strong absorption in the visible light region to produce more electron–hole pairs [30, 31].

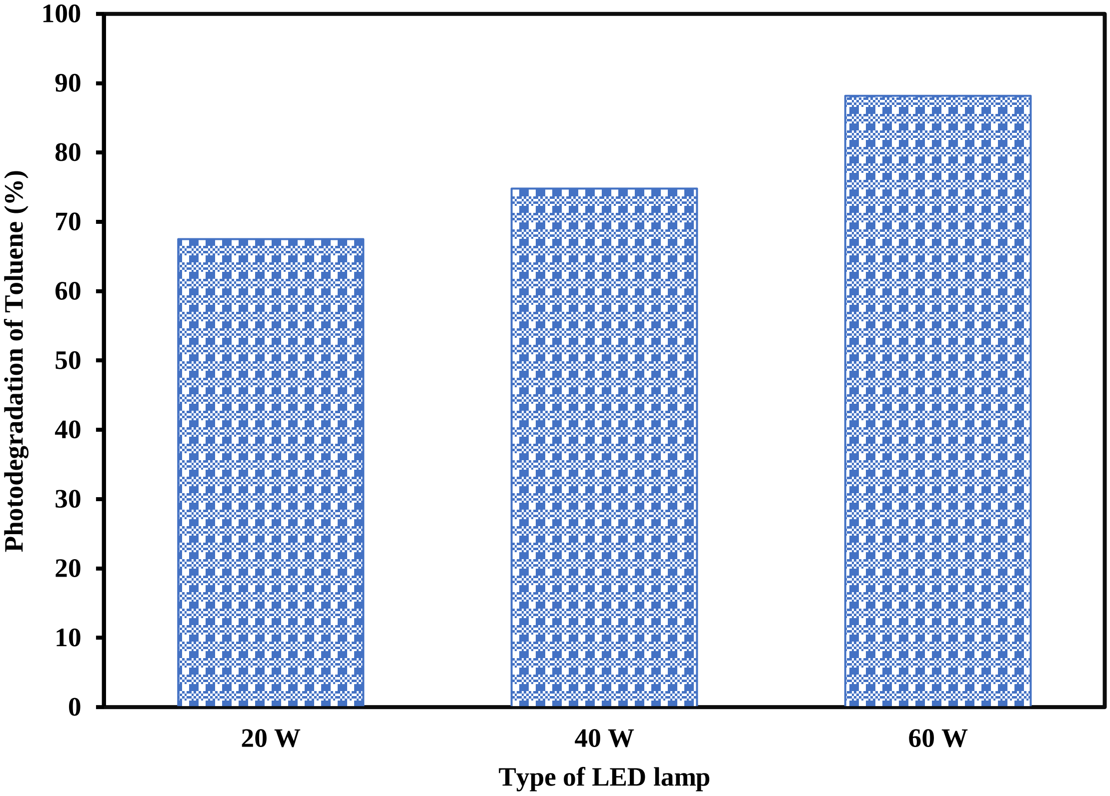

Fig. 7. The effect of LED-lamp intensity on the photodegradation of toluene

3.7. Proposed mechanism

The common mechanism of photodegradation is probably as follow: that toluene on the surface of TiO_2/Pd immobilized of polyester nonwoven fabric would be oxidized to H_2O, CO_2, or any inorganic harmless substances with $^{\bullet}OH$ or superoxide($^{\bullet}O_2^-$) radicals, which are generated under visible light irradiation [32]. When TiO_2/Pd immobilized of polyester nonwoven fabric sencitized and activated by visible light illumination, due to the transfer of an electron from the valence band (VB) to the conduction band (CB) without recombination of charge cariers (e^- and h^+) as a main limitation in photocatalysis systems, a series of highly reactive oxygen species such as $^{\bullet}OH$, $^{\bullet}O_2^-$, $^{\bullet}O_2H$, and H_2O_2 are generated and these are then capable of decomposing the toluene molecules (Fig. 8).

Mohamed and Awad [32] present a case study on the effect of a produced air purification filter in the removal of toluene with concentration of 26 mg/L. The principal targets of their investigation were to synthesize Ag nanoparticles/TiO_2 filter for the first time via the deposition of silver nanoparticles on a commercial immobilized TiO_2 tissue sheet by impregnation methodology and to study the activity of this fabricated Ag/TiO_2 tissue-based filter system for toluene photodegradation under UV light from indoor air. The results illustrated that under the experimental conditions, Ag/TiO_2-based filter was able to removal polluted air by two different stages; the first one is a slight adsorption phase in dark with approximately 15% of toluene removal within 60 min. The second stage is a photooxidation phase under UV irradiation in which the toluene removal efficiency was significantly enhanced with extension of the operational time and reached 97% during this stage.

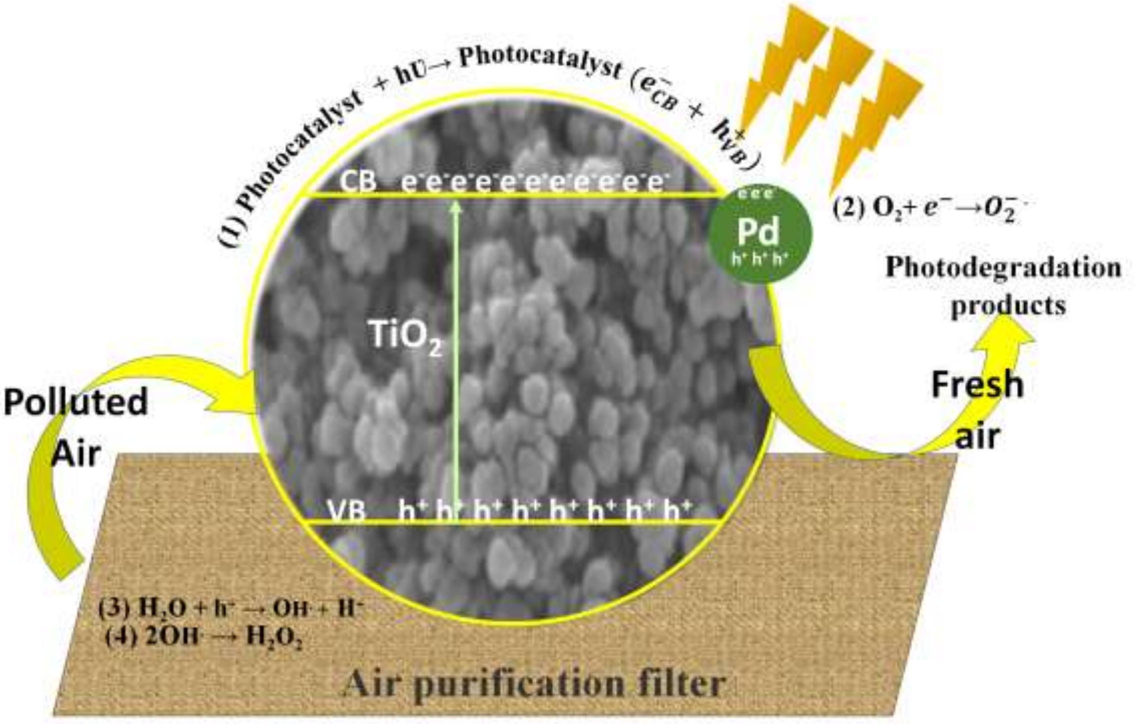

Fig. 8. The proposed mechanism for visible light photodegradation of toluene on the surface of air purification filter

4. Conclusion

The TiO_2/Pd nanophotocatalyst prepared by colloid solution method and immobilized on polyester nonwoven fabric, exhibited significantly high efficiency on photocatalytic activity comparatively with the commercial TiO_2 for photodegradation of in gaseous phase under visible light irradiation. The characteristics of the prepared TiO_2/Pd by means of various analytical techniques such as UV-DRS, FESEM, and BET were reported. The study of the reaction factors including PVA concentration, catalyst loading in air filter, and LED lamp intensity were investigated to achieve the optimal conditions for the toluene photodecomposition reaction. Toluene removal reaction increased with increasing of nanophotocatalyst loading due to more active sites of catalysts and more visible light adsorption. Then removal of toluene tended to a mild decrease with increasing of initial concentration of nanophotocatalyst in the prepared filter. In addition, the toluene removal had the highest degradation activity, about 80.0 %, when the LED lamp-60 W was used for the test study.

Conflicts of Interest

The author declares no conflict of interest.

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