Tikrit Journal of Engineering Sciences

TJES Tikrit Journal of Engineering Sciences

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Enhanced Dye Removal Rate from Textile Wastewater Using Advanced Oxidation: A Case Study of Jeans and Wool Dying Factories in Hama City

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Abstract:

The study aimed to develop a laboratory reactor to treat textile wastewater from dyeing factories in Hama City using visible light and a catalyst comprising titanium dioxide, bismuth vanadate, and activated carbon. It explored varying catalyst concentrations (500-200 mg/L) and found 1500 mg/L to be optimal for the highest dye removal. The study also assessed the impact of pH on dye removal, noting pH 6 as optimal. Light wavelength (400nm to 660nm) significantly affected dye removal, with 400nm visible light showing the best results. Sodium persulfate at 1000 mg/L achieved 99% dye removal. Additionally, 500mg/L of ferrous sulfate was optimal for chemical precipitation. TiO_2/$BiVO_4$/AC photocatalyst exhibited the highest dye degradation efficiency among the variants, attributed to the BiO-TiO_2 p-n heterojunction. $BiVO_4$ enhanced photocatalytic activity by facilitating electron transfer, shifting the absorption spectrum to the visible light, reducing the TiO_2 band gap, and suppressing electron-hole recombination. Violet light proved most effective in dye degradation compared to other visible light sources. The study highlighted superior dye removal efficiency (99% within 180 min) when employing chemical precipitation, ion exchange, and photocatalytic degradation combined. Treated Textile wastewater can meet Syrian irrigation standards (Syrian standard specification No. 2580 of 2008).

Keywords:

$BiVO_4$; Dyes; Photocatalytic Degradation; Textile Wastewater; TiO_2.

Highlights:

- A case study of Jeans and wool dyeing factories in Hama city for textile wastewater treatment.
- The maximum dye degradation of 99% was achieved to repurpose the treated wastewater in irrigation.
- Preparing a photocatalyst to lowest the environmental damage of the spent catalyst (V2O5) in sulfuric acid industry.

Citation:

Alloush FS, Hillal MA. **Enhanced Dye Removal Rate from Textile Wastewater Using Advanced Oxidation: A Case Study of Jeans and Wool Dying Factories in Hama City**. *Tikrit Journal of Engineering Sciences* 2026; **33**(1): 1940.

Article History:

Received:	23	Dec.	2023
Received in revised form:	21	Jan.	2024
Accepted:	09	Feb.	2025
Final Proofreading:	19	Apr.	2026
Available online:	19	May	2026

https://doi.org/10.25130/tjes.33.1.27

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1.INTRODUCTION

Recent research has highlighted the development of effective nanomaterials for various applications across multiple fields, notably in photocatalysis and wastewater treatment. Photocatalysts have garnered significant attention for their utility in environmental applications. These catalysts, synthesized from heterogeneous metal oxide semiconductors, are often utilized for water pollution treatment. They exhibit efficiency in removing organic dye pollutants under light exposure. The catalyst's ability to generate electron-hole pairs is crucial for its photocatalytic activity, leading to the formation of free radicals that can trigger further reactions. [1] Among the metal-oxide nanoparticles utilized for this purpose, TiO_2 and $BiVO_4$ stand out for their exceptional photocatalytic properties. Nanostructured TiO_2, a well-known photocatalytic material, has a history of industrial applications. However, its effectiveness is traditionally limited to UV irradiation due to its wide optical band gap. To enhance its performance under visible light, strategies such as modifying its electronic structure through junctions with other metal-oxide nanomaterials have been employed. Doping TiO_2 with BiVo4 nanoparticles has been shown to improve its photocatalytic performance significantly. In this case, electrons and holes move from the TiO_2 conduction band to the $BiVO_4$ conduction band. Electrons and holes on TiO_2's surface help create more on bismuth vanadate's surface. These new electrons are essential for dye oxidation, assisted by free radicals formed during the process. [2] Moreover, the formation of a $NiO/Ag/TiO_2$ heterojunction by aligning the band positions of the two semiconductors at the metal-semiconductor interface can improve charge transfer efficiency, reduce electron-hole recombination, and enhance light absorption within the visible light spectrum. Research has also focused on synthesizing heterojunction nanocomposites to enhance their photocatalytic activity under visible light irradiation. By incorporating high-mobility charge carriers such as NiO and establishing effective p-n heterojunctions with TiO_2, [3] these nanocomposites exhibit enhanced visible light catalytic activity for pollutant degradation. This paper successfully developed a reliable textile heterojunction $TiO_2/BiVO_4/AC$ for dyeing factories, achieving a 99% removal efficiency, making the wastewater suitable for irrigation. In comparison, the reference study [3] used $NiO/Ag/TiO_2$, achieving a 90% removal rate. The presence of untreated dye in textile wastewater poses a significant environmental threat, impeding sunlight penetration and endangering aquatic life. Many dyes resist biodegradation and are carcinogenic, especially Azo dyes containing harmful breakdown byproducts. Governments are imposing strict regulations on the extraction of dyes from industrial waste, necessitating treatment before disposal into water sources. Traditional methods for treating textile wastewater have limitations; biological treatments face toxicity issues, physical treatments merely transfer dyes, and chemical processes lead to sludge formation. Photocatalytic degradation is increasingly favored due to the non- biodegradable nature of dyes. Advanced Oxidation Processes (AOPs) are economical and effective in breaking down dyes into harmless byproducts. A study is underway to develop a batch laboratory reactor for advanced oxidation of wastewater dyes from textile factories, focusing on Jeans and Wool dyeing facilities in Hama city. AOP disintegrates organic pollutants using visible or ultraviolet light with semiconductor catalysts like titanium oxide and bismuth vanadate. When the semiconductor, TiO_2, absorbs energy exceeding its band gap, electrons transition from the valence band (VB) to the conduction band (CB). This transition creates positive gaps (h+) in the valence band and electrons in the conduction band (e-). The electrons, holes, and free radicals generated by the Advanced Oxidation Process (AOP) contribute to the oxidation of dyes, producing CO2 and H2O]4 [. There is growing research interest in using advanced oxidation processes for the photocatalytic degradation of dyes, particularly those found in textile wastewaters. The utilization of AOP for the photocatalytic degradation of Wool and Jeans dyeing wastewaters to reuse them in irrigation has gained traction. A composite of two semiconductors, TiO_2 and $BiVO_4$, is employed as a co-catalyst to degrade dyes using visible light. $BiVO_4$ has attracted significant interest as a photocatalyst due to its narrow band gap and efficient degradation of organic and inorganic pollutants. $BiVO_4$ nanoparticles were synthesized for degradation of methylene blue. The catalysts were extensively characterized using SEM to evaluate their properties and performance. When samples were exposed to UV light at a neutral pH, the degradation rate reached 58.3% in 90 minutes. $BiVO_4$ demonstrated exceptional durability, maintaining catalytic activity even after five uses. This characteristic establishes $BiVO_4$ as a reliable nano-catalyst with economic and environmental benefits [5]. This paper successfully developed a reliable textile heterojunction $TiO_2/BiVO_4/AC$ for the photodegradation of dyes under visible light, achieving a 99% removal efficiency of the MB dye, which is considerers more effective than $BiVO_4$ under UV light [5]. Zawadzki et al. [6] discovered that the incorporation of persulfate ions in the presence of glucose and sunlight or

visible light enhances the efficiency of advanced oxidation. Ahmed et al. [7] employed AOP to remove organics from wastewater in a textile dyeing facility. The research findings indicated that AOP is more efficient at removing organic matter than biological treatment, particularly when the BOD/COD ratio is less than 0.3. Given that wastewater samples from Jeans and Wool dyeing exhibit a BOD/COD ratio below 0.3 (as shown in Table 4, the optimal treatment method AOP. To reduce the environmental impact of spent catalyst from the sulphuric acid industry(V2O5), the spent catalyst was used to prepare $BiVO_4$, which serves as a photocatalyst. This paper provides comprehensive details of the experimental procedures, reactor specifications, instrumentation used, and the obtained results. The investigation examined the effects of pH, catalyst concentration, and persulfate concentration on the efficiency of photocatalytic dye degradation, ultimately determining the optimal conditions for the process.

2.APPARATUS AND MATERIALES

2.1.Materials

Isopropanol (70% vol), TiO_2 (white powder M=79.8 g/mole, density of 3780 g/L), H_2O_2 (30% vol.), HNO_3 0.1M, NH_4OH (28%w/w), $BiNO_3$ 1M (M=485 g/mole, density of 2900 g/L), NaOH 0.1M, HCl 0.1M, $FeSO_4$ (crystalline white powder, density=2840 g/L), $Na_2S_2O_8$ (white crystal, density = 2400g/L). All chemicals were purchased from Petrochemical Engineering Faculty laboratories, and used without any further purification.

2.2.Physio-Chemical Analysis of the Samples:

The values of colour, pH, conductivity, Turbidity, and TDS of water samples are measured by a colour Scale, a pH meter, a conductivity Scale (Milwaukee PH/mv/Ec/TDS/NaCl/ Temperature, Romania), and a Turbidity Scale (HACH, 2100 NTURBIDI METER 46500-02, USA). UV–vis absorption spectra of the prepared samples were acquired using a split-beam UV–vis spectrophotometer (YK Scientific, UV1810/UV1810S, China). All experiments were conducted in the laboratories of the petrochemical Engineering Faculty/Homs University, /Homs.

2.3.Catalyst Characterization

N2 sorption analysis was performed at 77 K using the Micrometrics ASAP2020 accelerated surface area and porosity system (Micrometrics, Norcross, GA, USA), with specific surface area and pore size distribution measured using Brunauer–Emmett–Teller (BET). Scanning electron microscopy (SEM) was performed. in the laboratories of the College of Science / Aleppo University.

2.4.Catalyst Synthesis

The textile $TiO_2/BiVO_4$/AC heterojunction nanocomposites were synthesized via co-precipitation. Typically, 1 M of TiO_2 (rutile) Nano powder was ultrasonically dispersed for 15 min in a 250 mL beaker in a mixed solution of 150 mL of ultrapure (UP) H_2O_2 and isopropanol in a volume ratio of 1:1. After aging for 12 hours at 22°C and drying at 60°C for 2 hours, the dried solution was calcinated at 400°C for four hours to further enhance stability. 1M of NH_4VO_3(recovered from V_2O_5 spent catalyst) and 1M of $BiNO_3$ in 20 mL of a mixed solution of UP water and 0.1N OF HNO_3 were separately mixed for 15 min, the pH was adjusted to six, and the mixture of NH_4VO_3 and $BiNO_3$ solution was cooled to room temperature, filtered, and the resulting precipitate $BiVO_4$ was dried at 300°C for three hours. $BiVO_4$ Nano powder was then added to the TiO_2 solution in ethanol and stirred for 15 min. The mixture was processed in an autoclave at 90°C for 1 hour, followed by drying at 180°C for 24 hours. Subsequently, it was washed with ethanol, and the resulting precipitate was dried for 6 hours at 60°C. TiO_2/ $BiVO_4$ was deposited on AC in a series of steps, including mixing, drying, hydrolyzing, and heating to achieve the desired composite structure. The precipitated nano powder was separated by centrifugation, then washed 3 times with UP water and absolute ethanol and dried at 60 °C for 24 h.

2.5. Activated Carbone Specification

Table 1 illustrates the specification of Activated Carbone.

2.6.Catalyst Characteristics

The surface area and porosity of the nanocomposite were measured quantitatively using nitrogen adsorption, the BET method. The samples were degassed at 150 °C for 48 h before recording N2 adsorption–desorption isotherms at 77 K (−196 °C). The BET surface area and porosity (pore size) of $TiO_2/BiVO_4$/AC and TiO_2 are listed in Table 2.

Table 1 Specification of Activated Carbone.

Variable	Moisture content	pH	Ash content	Organic content	Density
Value	8.5%	5.8	0.53%	78.7%	110 g/L

Table 2 Characteristics of the Composite ($TiO_2/BiVO_4$/AC).

Component	Weight percentage %	Specific surface m^2/g	Particle size (nm)
$BiVO_4$	10	120	29
Tio_2	10	100	40
AC	80	1500	43
The Composite	100	1000	

Figure 1 illustrates a scanning electron microscope (SEM) image of the catalyst (TiO_2 /$BiVO_4$/AC).

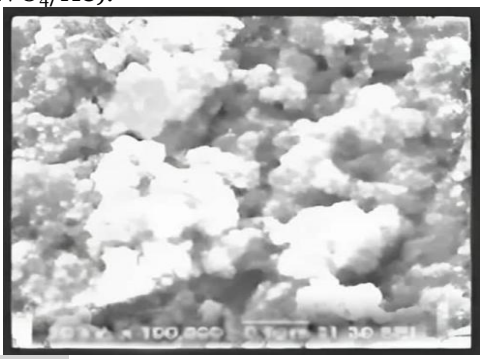

Fig. 1 The Scanning Electron Microscope (SEM) Image.

From Fig. 1, it can be deduced that the particles of each component of the composite (TiO_2, $BiVO_4$, and AC) are heterogeneous, with an average size of 43nm.

2.7.The Experimental Reactor Characteristics

The experimental reactor consists of the following, as illustrated in Fig. 2: an air pump (1), which is used to provide air bubbles at a flow rate of 3.5 L/min, while a lens (2) with a diameter of 0.13 m is strategically placed 0.12 m above the liquid surface to effectively focus and direct light onto the liquid. The reactor also includes a violet visible light lamp (3) that emits light with a wavelength of 400 nm and an irradiance of 18 Mw/m². To ensure turbulent flow, a variable speed mechanical mixer (4) is employed, capable of mixing at up to 300 rpm. The experimentation vessel is a closed rectangular glass container (5) with dimensions of 0.40* 0.15* 0.25 m, featuring inward bevelled corners designed to prevent liquid stagnation. Finally, the vessel is enclosed with a movable stainless-steel cover (6). The sketch of the experimental reactor is shown in Fig. 2. The reactor uses visible light, a catalyst, and additives to generate free radicals for oxidation. Oxygen supplied by the air pump supports the reaction. Photocatalytic oxidation occurs inside the reactor, degrading pollutants over time.

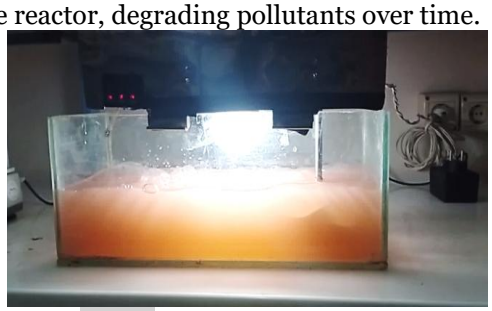

Fig. 2 Experimental Device.

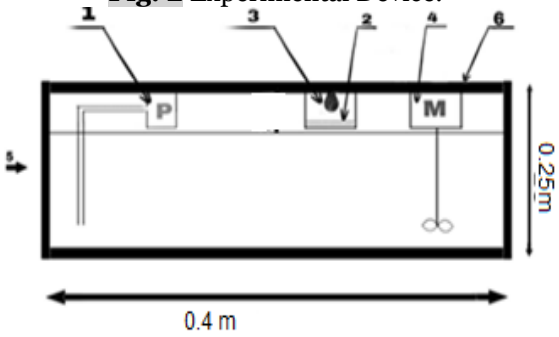

Fig. 3 Sketch of the Device, 1: Air Pump, 2: Lens,3: Light Lamp,4: Mixer,5: Glass Vesele,6: Cover.

2.8.Specifications of the Study Samples

Two types of water samples were used. The first type is artificial dye samples of both acidic dyes (Brilliant Blue, MO, Indigo, Reactive Red 73, Azo Carmine B, Tartrazine), and basic dyes (MB) each separately. These dyes are the most hazardous ones found in the wastewater of wool and jeans dyeing factories. Table 3 presents the characteristics of dyes provided by the laboratories of the Petrochemical Engineer Faculty/Textile Engineer Department. The second type of water samples is textile dye wastewater from jeans and wool dyeing factories in Hama city, specifically at the outlet of the bath dyeing process. The characteristics of these samples are listed in Table 4.

Table 3 Specification of Dyes Used in Preparing the Artificial Water Samples.

Name of dye	Type of dye	Chemical formula	ג (Wave length) nm	Dye uses
Azo carmine B	acidic	$C_{26}H_{15}N_4NaO_9S_2$	440 [8]	Wool dying
Brilliant blue	acidic	$C_{37}H_{34}N_2Na_2O_9S_3$	582 [9]	Wool dying
Indigo	acidic	$C_{16}H_{10}N_2O_2$	610 [10]	Jeans dying
MB	basic	$C_{16}H_{18}Cl\ N_3S$	664[11]	Wool dying
MO	acidic	$C_{14}H_{14}N_3NaO_3S$	466 [12]	Wool dying
Reactive Red 73	acidic	$C_{44}H_{24}Cl_2N_{14}O_{20}S_6Na_6$	525 [13]	Wool dying
Tartrazine	acidic	$C_{16}H_9N_4Na_3O_9S_2$	440 [14]	Wool dying

Table 4 The Characteristics of Jeans and Wool Dying Factories in Hama City.

Wool dying wastewater			Jeans dying waste water		
Variable	**Unit**	**Values**	**Variable**	**Unit**	**Values**
Turbidity	NTU	370	Turbidity	NTU	55
Colour	P.C.U	800	Colour	P.C.U	500
pH	-	8.7	pH	-	7.6
TSS	mg/L	3000	TSS	mg/L	200
TDS	mg/L	6000	TDS	mg/L	1400
Conductivity	µS/cm	6000	Conductivity	µS/cm	2140
COD	mg/L	8630	COD	mg/L	4500
BOD/COD	-	0.2	BOD/COD	-	0.1
Cr	mg/L	1.1	Cr	mg/L	0.94
Cu	mg/L	0.06	Cu	mg/L	0.05
Pb	mg/L	0.02	Pb	mg/L	0.01

2.9.Experimental Procedure

The experimental procedures are listed below:

1) Beakers of 1000 ml are filled with 500 ml of wastewater after characterizing it and adjusting the pH to the required value using 0.1 N HCl or 0.1 N NaOH.
2) The required $FeSO_4$ dose was mixed with the wastewater at a mixing speed of 100 rpm for two minutes, followed by a slow mixing of 60 rpm for ten minutes Finally, the mixing speed was decreased to 20 rpm for twenty minutes. Then, the mixers are turned off and the magnets are attached to the bottom of the beaker from the outside for 5.0 minutes.
3) Water samples from the supernatant were taken to characterize them.
4) The supernatant underwent ion exchange treatment to remove negative ions that could disrupt the oxidation process, and the treated samples were characterized.
5) The reactor is then filled with 6 L of the treated wastewater after adjusting the pH to the required value using 0.1 N HCl or 0.1 N NaOH.
6) The required catalyst dose was mixed with the wastewater at mixing speed of 250 rpm without irradiation for one hour.
7) The mixture was then exposed to violet visible light with a wavelength of 400 nm in presence of persulfate at the required dose to enhance AOP.
8) Regular samples (0.025 L) of wastewater were taken at half-hour intervals, filtered, and the dye absorbance value was measured using a spectrophotometer at a specific wavelength.

The catalyst's efficiency was evaluated using MB, MO, AZO carmine, Brilliant Blue, Indigo dyes in an aqueous solution. A tungsten lamp ($\lambda \geq 400$ nm, with $\lambda max = 800$ nm) with UV cut filter coating was used in all photodegradation experiments. The intensity of the light source was ~18 MW/m² measured by a light meter (EXTECH Instrument, Model 401027, Luximetro de Bolsillo) at 12 cm between the lamp and the cup using different doses of photocatalyst (500, 1000,1500, and2000 mg) suspended in 1 L of dye (200 mg/L) solution in a lab reactor. The solution was kept under continuous stirring in the dark for 60 min before irradiation to balance absorption and desorption between the produced photocatalyst and the dye solution. The maximum absorption wavelengths of the MB, MO, AZO carmine, Brilliant Blue, and Indigo dyes were measured using UV–visible spectrum at 664,466, 440,582, and 610 nm respectively. Absorbances were recorded at 30min intervals for 180 min to assess the dye's deterioration. In all tests, the blank sample consisted primarily of water. UV–vis spectrometry was used to measure the dye concentration after removing the catalyst from the liquid using a 0.45 µm PTFE syringe filter.

Dye removal percentage of the artificial water samples was calculated using Eq. (1):

$$\textbf{Dye removal percentage } \% = \frac{(C0-C)\times 100}{C0} \quad \textbf{(1)}$$

Where:

C_o (mg/L) is the initial dye concentration.

C (mg/L) is the dye concentration after a specific time.

COD removal percentage indicates dye removal percentage of wool and jeans wastewaters. The values of the BOD/COD ratio of the wastewater samples presented in Table 4 are less than 0.3. This clearly indicates the necessity of primary treatment to reduce COD,enhance AOP efficiency, and render the wastewater biodegradable. Three levels of ferrous sulfate (200, 500, and 700 mg/L) were utilized, with an initial pH of 5. Whatman filtration paper grade 1:11 nm was used.

3.RESULTS AND DISSCUSSION

3.1.Wastewater Effluent Characteristics after Chemical Precipitation

Tables 5 and 6 show characteristics of Wool and Jeans Dyeing wastewater after chemical precipitation with three levels of $FeSO_4$ concentration.

Table 5 Characteristics of Wool dyeing Wastewater after Chemical Precipitation.

Wool dying wastewater			**Wool dying waste water**			**Wool dying waste water**		
C $_{FeSO4}$ 200 mg/L			**C $_{FeSO4}$ 500** mg/L			**C $_{FeSO4}$ 700** mg/L		
Variable	**Unit**	**Values**	**Variable**	**Unit**	**Values**	**Variable**	**Unit**	**Values**
Turbidity	NTU	202	Turbidity	NTU	120	Turbidity	NTU	170
Colour	P.C.U	680	Colour	P.C.U	420	Colour	P.C.U	560
pH	-	8.1	pH	-	7.9	pH	-	7.9
TSS	mg/L	2700	TSS	mg/L	1420	TSS	mg/L	1600
TDS	mg/L	4900	TDS	mg/L	3050	TDS	mg/L	3070
Conductivity	µS/cm	5100	Conductivity	µS/cm	3840	Conductivity	µS/cm	3920
COD	mg/L	7237	COD	mg/L	5178	COD	mg/L	5060
BOD/COD	-	0.21	BOD/COD	-	0.24	BOD/COD	-	0.26
Cr	mg/L	0.96	Cr	mg/L	0.94	Cr	mg/L	0.92
Cu	mg/L	0.04	Cu	mg/L	0.03	Cu	mg/L	0.02
Pb	mg/L	0.014	Pb	mg/L	0.01	Pb	mg/L	0.09

Table 6 Characteristics of Jeans Dyeing Wastewater after Chemical Precipitation.

Jeans dying wastewater			Jeans dying waste water			Jeans dying waste water		
$FeSO_4$ 200 mg/L			$FeSO_4$ 500 mg/L			$FeSO_4$ 700 mg/L		
Variable	Unit	Values	Variable	Unit	Values	Variable	Unit	Values
Turbidity	NTU	40	Turbidity	NTU	24	Turbidity	NTU	29
Colour	P.C.U	380	Colour	P.C.U	200	Colour	P.C.U	310
pH	-	7.6	pH	-	7.6	pH	-	7.8
TSS	mg/L	170	TSS	mg/L	36	TSS	mg/L	40
TDS	mg/L	1250	TDS	mg/L	924	TDS	mg/L	930
Conductivity	µS/cm	1808	Conductivity	µS/cm	1415	Conductivity	µS/cm	1397
COD	mg/L	3940	COD	mg/L	2790	COD	mg/L	2690
BOD/COD	-	0.14	BOD/COD	-	0.19	BOD/COD	-	0.21
Cr	mg/L	0.91	Cr	mg/L	0.67	Cr	mg/L	0.51
Cu	mg/L	0.03	Cu	mg/L	0.01	Cu	mg/L	0.008
Pb	mg/L	0.012	Pb	mg/L	0.01	Pb	mg/L	0.007

Tables 5 and 6 demonstrate that the utilization of $FeSO_4$ at a concentration of 700 mg/L results in a decrease in COD and conductivity values compared to concentrations of 500 mg/L and 200 mg/L. Conversely, at a concentration of 700 mg/L, the use of $FeSO_4$ result in higher turbidity and colour values due to excessive deposition of iron salts. Consequently, the optimal concentration of $FeSO_4$ is determined to be 500 mg/L.

3.2.Effect of PH on Dye's Removal with AOP

pH is key parameter in photocatalytic dye degradation, as it can influence the rat of dye removal. It can influence dye adsorption onto the semiconductor surface because the catalyst surface charge depends on solution pH. pH effect is related to the surface-charge properties of the photocatalysts; and could be explained based on the point of zero charge (PZC). The point of zero charge for TiO_2 particles is pHpzc = 6.8. At pH values below the pHpzc (pH < 6.8) or in acidic solution, the catalyst surface becomes positively charged and anion-attracting/cation-repelling. Conversely, above PZC the surface is negatively charged and the surface becomes cation attracting/anion repelling [15]. A neutral or slightly acidic pH is often considered optimal for photocatalytic reactions because it favours the generation of reactive oxygen species, while minimizing side reactions. However, the specific impact of pH can vary depending on the photocatalyst type, and the target pollutants being treated. TO enhance photocatalytic efficiency across various water samples, the effect of pH was optimized across four experimental sets. The first set consisted of artificial acidic dye samples (Indigo, MO, Tartrazine, Brilliant Blue, and Azo Carmine B), each tested separately, at an initial concentration of 200 mg/L. The second set was an artificial basic dye sample (Methyl Blue) with an initial concentration of 200 mg/L. The third set was an artificial reactive dye sample (Reactive Red) with an initial concentration of 200 mg/L. The fourth set was samples of dyeing wastewaters from jeans and wool. Each experiment was conducted under violet visible light, using 1500 mg/L of the catalyst at various pH values ranging from 5 to 12, tested individually. Graphical representations of the results for acidic, alkaline, reactive, and wastewater samples are shown in Figs. 4-7. Figure 4 illustrates the effect of pH on Acidic Dyes removal percentage, Fig. 5 displays the effect of pH on Alkaline Dye removal percentage, and Fig. 6 illustrates the effect of pH on Reactive Red Dye removal percentage.

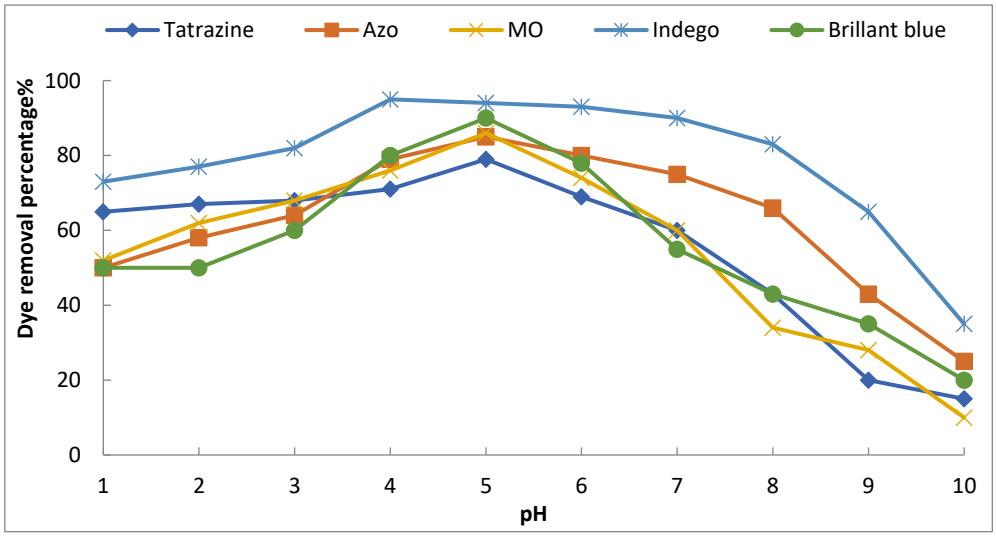

Fig. 4 Effect of pH on Acidic Dyes Removal Percentage. C $_{catalyst}$ 1500 mg/L,C $_{dyes}$=200 mg/L, Violet Visible Light,ƛ=400nm,p=18Mw/m².

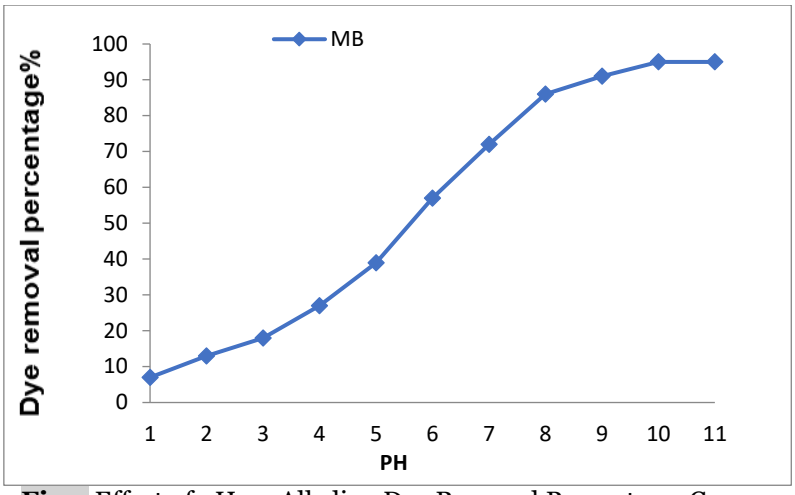

Fig.5 Effect of pH on Alkaline Dye Removal Percentage. C $_{catalyst}$ 1500 mg/L, C $_{dyes}$=200 mg/L, Violet Visible Light,ƛ=400nm,p=18Mw/m².

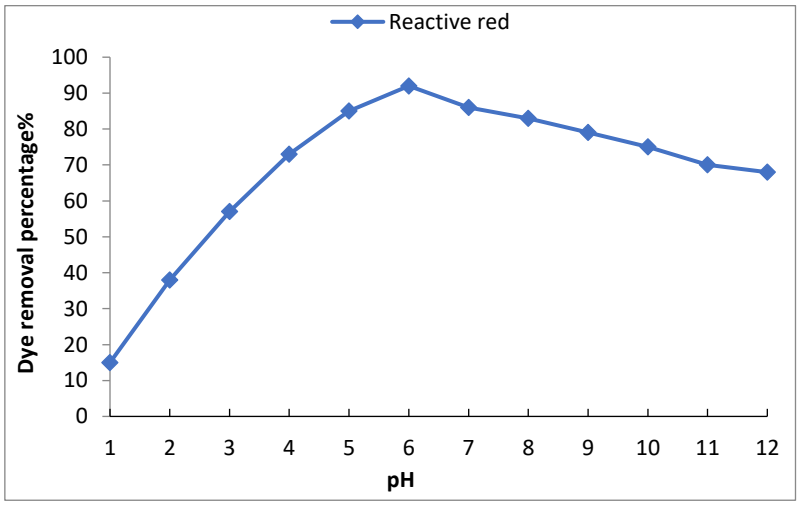

Fig.6 Effect of pH on Reactive Red Dye Removal Percentage. C $_{catalyst}$ 1500 mg/L,C $_{dyes}$=200 mg/L, Violet Visible Light,ƛ=400nm,p=18Mw/m².

It is evident from Figs. 4-6 that the optimal pH range for acid dyes lies between 4 and 5. Increasing pH from 6 to 10 decreased dyes removal percentage from approximately 85% to approximately 40% after 180 min. The optimal pH range for alkaline dye is 9 to 10, since increasing pH from 1 to 9 increases the dye removal percentage from 7% to 91%. The optimum pH for value for Reactive Red dye is 6, as at this pH the dye removal percentage is the highest. Figure 7 shows the effect of pH on the percentage of dye removal from the wastewater Sample. Figure 7 shows that wastewater samples with an initial pH of 6 had the highest dye removal percentage for both jeans and wool dye wastewater samples. pH values are taken in considered in the subsequent experiments.

3.3.Effect of Catalyst Dose on Dyes Removal Percentage

To study the influence of catalyst dosage on dye removal percentage from Textile wastewater, samples of 6L were taken from wool and jeans dyeing factories, as described in Tables 6 and 7. The initial pH of the samples was adjusted to 6. Three different catalyst concentrations (1000, 1500, and 2000 mg/L) were used. Regular samples of the treated wastewater were taken; the dye removal percentage was calculated. The results were documented and illustrated in Fig. 8. From Fig. 8, the optimal catalyst concentration to achieve the highest dye removal percentage is 1.5 g/L, yielding a COD removal of 95%. However, raising the catalyst concentration beyond 1500 mg/L diminishes the dye removal rate. This can be attributed to the obstruction of active centers caused by the overlapping of catalyst grains, consequently reducing the dye removal rate.

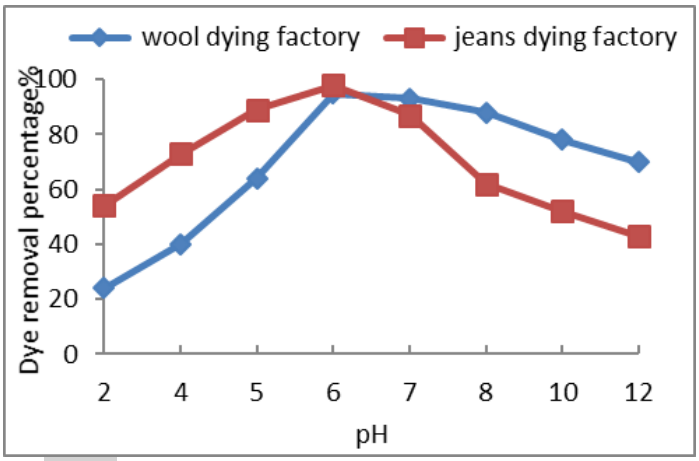

Fig. 7 Effect of pH on Dye Removal Percentage from Textile Wastewater Sample. $C_{catalyst}$ 1500 mg/L, C_{dyes}=200 mg/L, Violet Visible Light,ɹ=400nm,p=18Mw/m².

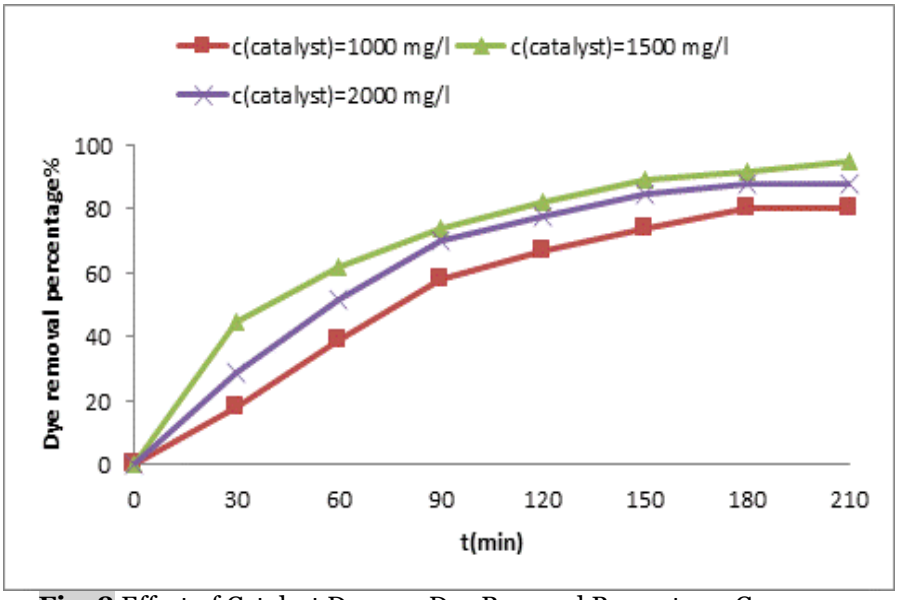

Fig. 8 Effect of Catalyst Dose on Dye Removal Percentage. C_{dyes}=200 mg/L, Violet Visible Light,ɹ=400nm.

3.4.Effect of Dye Dose on Dyes Removal Percentage

The rate of catalyst loading with dyes is determined by Eq. (2). Loading ratio based on the concentrations of dyes (C_{dyes} mg/L), and the used catalyst ($C_{catalyst}$ mg/L) [16].

$$\textbf{Loading ratio of catalyst with dyes \%} = \frac{\textbf{C dye} \times \textbf{100}}{\textbf{C catalyst}} \quad \textbf{(2)}$$

During these experiments, artificial water samples were prepared with different concentrations (200 - 900 mg/L) of Methyl Blue, Brilliant Blue, Tartrazine, Methyl Orange, and Azo dyes. A consistent catalyst concentration of 1500 mg/L was used. The pH was set at 4 for acidic dyes and 9 for basic dyes. Samples at consistent intervals were filtered, and the catalyst-loading ratio was computed; subsequently, the dye removal percentage was calculated. The outcomes are detailed in Fig. 9. It appears from Fig. 9 that the optimal catalyst-loading ratios for effective removal of Brilliant Blue, Methyl Orange, and Methyl Blue are 1.5, while for Azo and Tartrazine dyes are 2.5.

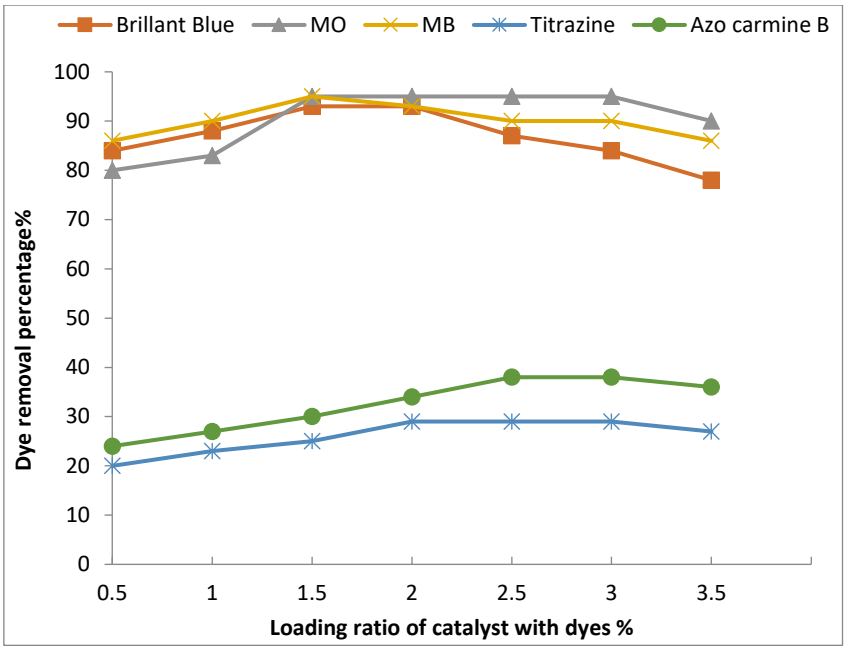

Fig. 9 Effect of Catalyst-Loading Ratio with dyes on Dye Removal Rate. C $_{catalyst}$ 1500 mg/L,C $_{dyes}$=200 mg/L, Violet Visible Light,λ=400nm,p=18Mw/m²

3.5.Effect of Visible Light Weave Length

Advanced oxidation was carried out on Textile wastewater utilizing the tested catalyst at a concentration of 1500 mg/L and an initial pH of 6. Various wavelengths of visible light were employed, including visible red at 660 nm, visible yellow at 585 nm, visible blue at 470 nm, and visible violet at 400 nm all at once. Samples of the treated water were taken, and the dye removal percentage was calculated. The findings were documented and presented in Fig. 10. From Fig. 10, it seems that violate visible light yields the highest dye removal rate. It seems that AOP is more effective for dyeing wastewater from jeans than from wool.

3.6.Effect of Sodium Persulfate on Dye Removal Percentage

For discussing the impact of sodium persulfate on the dye removal percentage from jeans and wool dyeing wastewater samples. The Advanced Oxidation Process (AOP) was carried out at a catalyst concentration of 1500 mg/L, a sodium persulfate concentration of 1000 mg/L, and a glucose concentration of 1800 mg/L, under ultrasound waves (to promote mixing and violet visible light (wavelength 400 nm). The experiments were repeated without persulfate. Samples were analyzed periodically to determine the percentage of dye removal in the presence and absence of persulfate. The results were presented in Fig. 11. Sodium persulfate can be activated by light to generate sulfate radicals, which react with organic compounds to break them down.

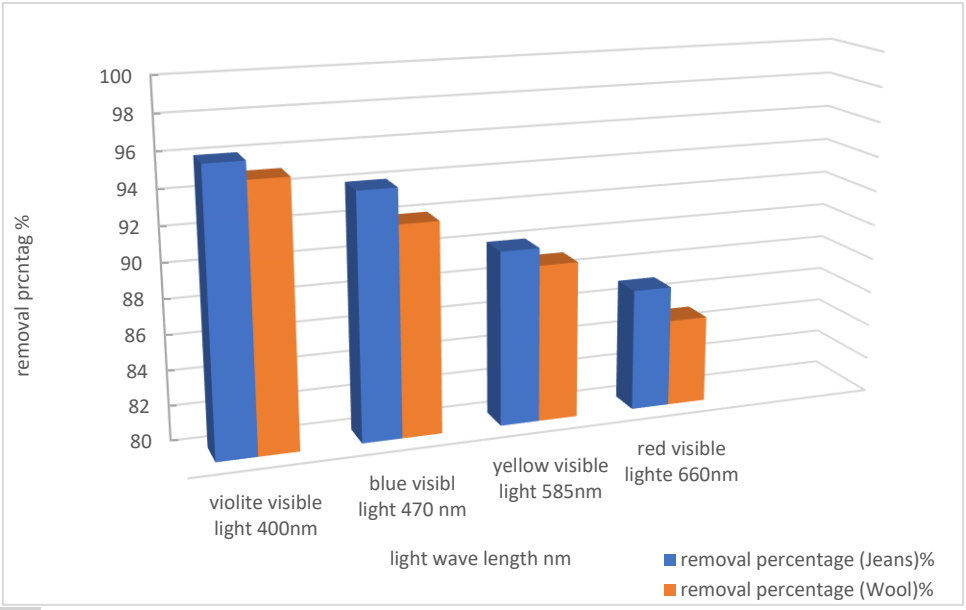

Fig. 10 Effect of Weave Length of Visible Light used. C $_{catalyst}$ 1500 mg/L, C $_{dyes}$=200 mg/L, p=18Mw/m².

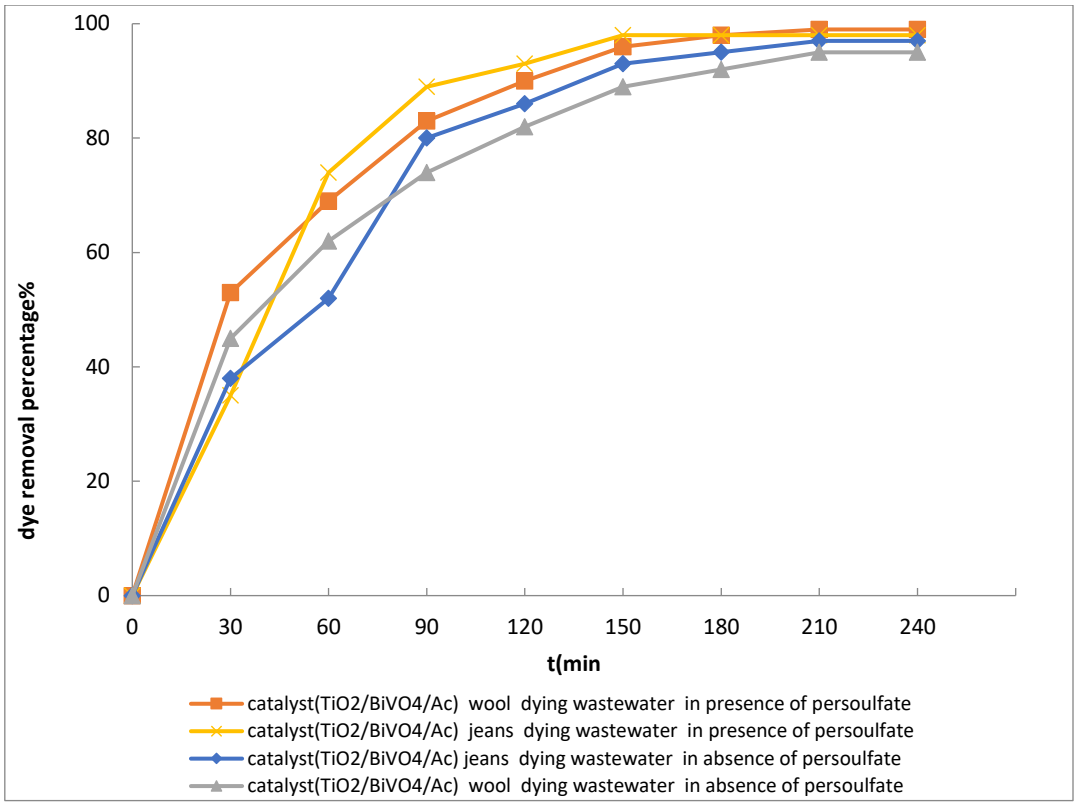

Fig. 11 Effect of Persulfate in Oxidizing Dyes in Textile Wastewater. C Catalyst 1500 mg/L, C Dyes=200 mg/L, Violet Visible Light,ɹ=400nm,p=18Mw/m².

It appears from Fig. 11 that the use of sodium persulfate as an irreversible electron acceptor resulted in a reduction of oxidation time from 180 minutes (AOP without persulfate) to 130 minutes with persulfate. Additionally, an increase in dye removal percentage from 95% without sodium persulfate to 99% with sodium persulfate. This enhanced oxidation rate aligns with the criteria for wastewater usability in irrigation, as per Syrian Standard 2008 NO. 2580 [17].

3.7.Effect of Sunlight on Advanced Oxidation of Textile Wastewater:

Moving on to the effect of sunlight on the advanced oxidation of Textile wastewater, experiments were conducted using samples from jeans and wool dyeing factories. The lab reactor was exposed to sunlight and concentrated the wastewater using a lens. These experiments took place from July 25th, 2023, to August 16th, 2023, for six hours daily between 10:30 a.m. and 2:30 p.m. Wastewater samples were treated with the specified catalyst, sodium persulfate, and glucose. Regular samples were taken, filtered, and the dye removal percentage was determined, with the results presented in Fig. 12. It seems from Fig. 12 that exposure to sunlight for 6 hours leads to a substantial 99% removal of dye. The removal percentage is the same as that obtained by using violet visible light. The combination of sunlight, sodium persulfate, and glucose has proven effective in degrading dyes from both wool and jeans dyeing factories, highlighting sunlight as a viable option for Advanced Oxidation Processes (AOP). Figure 13 shows a comparison of dye removal efficiency from textile wastewater after using TiO_2/AV, $BiVO_4$/AC, and TiO_2/$BiVO_4$/AC. Fig. 13 shows that composite TiO2/$BiVO_4$/AC provides the highest dye removal rate. Figure.14 illustrates a comparison of dye removal efficiency when treating wastewater using the advanced oxidation process only, chemical precipitation and the advanced oxidation process, and chemical precipitation, ion exchange, and advanced oxidation process. Fig.14 shows that superior dye removal efficiency was achieved chemical precipitation, ion exchange, and photocatalytic degradation were combined. The wastewater characteristics are detailed in Table 7 after the AOP treatment.

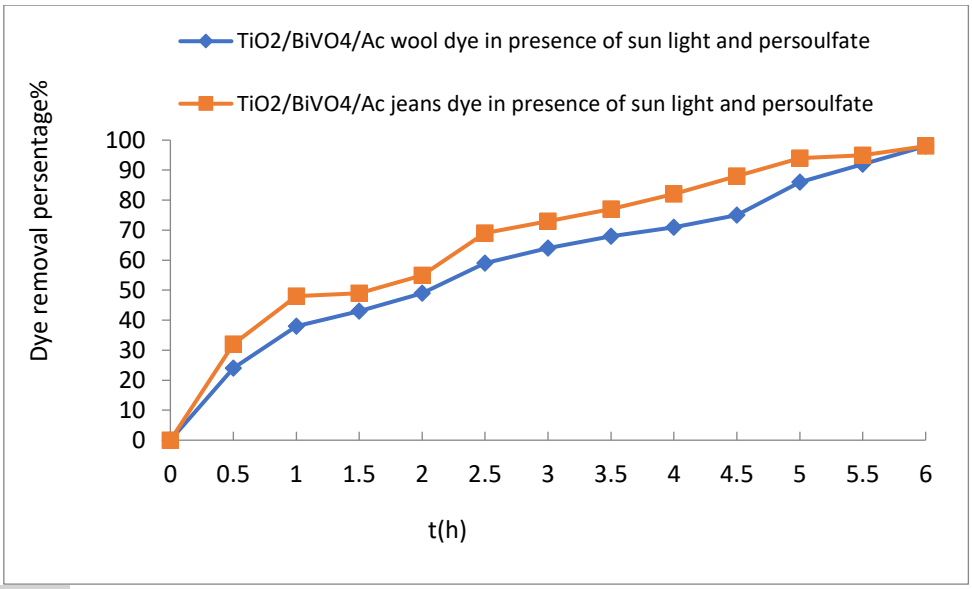

Fig. 12 Effect of Sunlight on Oxidizing Wastewater of Jeans Dyeing and Wool Dyeing. $C_{catalyst}$ 1500 mg/L,C_{dyes}=200 mg/L, Violet Visible Light,ɔ=400nm,p=18Mw/m².

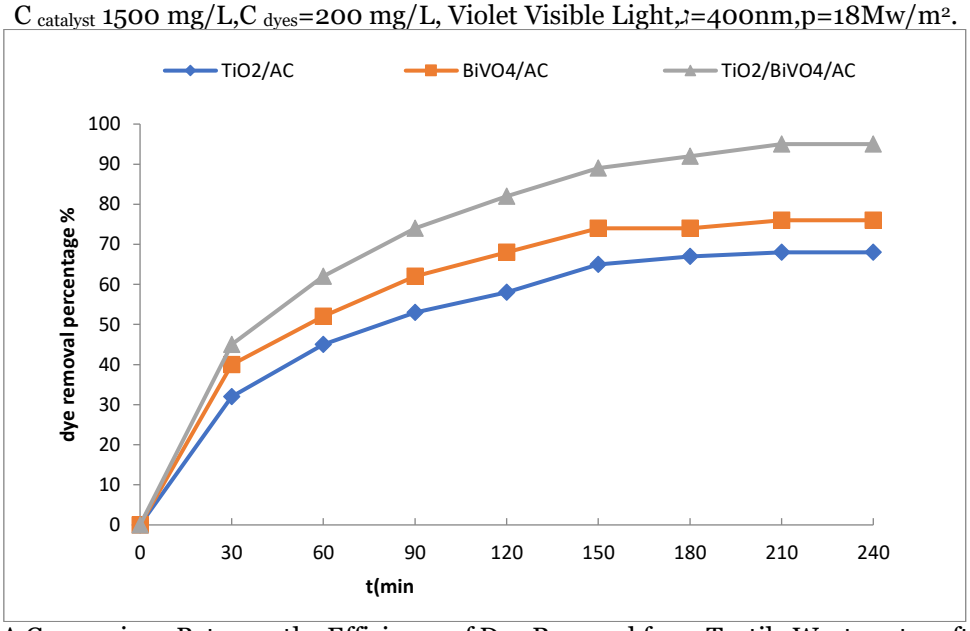

Fig. 13 A Comparison Between the Efficiency of Dye Removal from Textile Wastewater after using Each of TiO2/AV, $BiVO_4$/AC, and TiO2/$BiVO_4$/AC.

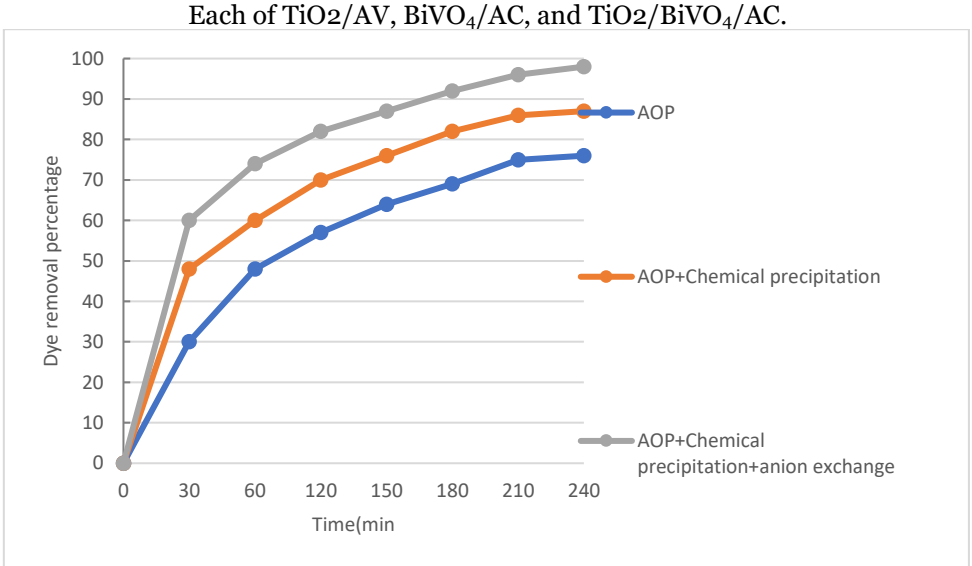

Fig. 14 A comparison between the Removal Efficiency of Dye when Treating Wastewater Using only AOP, Using Chemical Precipitation and AOP, and Using Chemical Precipitation and Ion Exchange and AOP.

Table 7 Characteristics of Textile Wastewater after AOP.

Wool dying wastewater			Jeans dying waste water		
Variable	**Unit**	**Values**	**Variable**	**Unit**	**Values**
Turbidity	NTU	20	Turbidity	NTU	4
Colour	P.C.U	44	Colour	P.C.U	0.5
pH	-	7.2	pH	-	7.2
TSS	mg/L	120	TSS	mg/L	13
TDS	mg/L	200	TDS	mg/L	37
Conductivity	µS/cm	500	Conductivity	µS/cm	150
COD	mg/L	51.78	COD	mg/L	100
BOD/COD	-	0.34	BOD/COD	-	0.24
Cr	mg/L	0.9	Cr	mg/L	0.63
Cu	mg/L	0.02	Cu	mg/L	0.08
Pb	mg/L	0.09	Pb	mg/L	0.09

Comparing the specifications of wool dyeing wastewater after chemical precipitation in Table 5 with its specifications after oxidation in Table 7, it is evident that during AOP, turbidity was reduced by 83.3%, colour by 89.5%, electrical conductivity by 89.4%, and COD by 95%. Additionally, comparing the specifications of jeans dyeing wastewater after chemical precipitation listed in Table 6 with its specifications after oxidation in Table 7, it illustrates that during the Advanced Oxidation Process, turbidity was reduced by 83.3%, colour by 97.5%, electrical conductivity by 89.4%, and COD by 94.6%. To remove the anions generated by advanced oxidation and reduce wastewater conductivity, the treated wastewater as subjected to negative ion exchange (A21) under specific dimensions and flow rate. The treated wastewater was then analyzed, and the results are presented in Table 8.

Table 8 Characteristics of Tested Wastewater after Treating it with Ion Exchanger.

Wool dying wastewater			Jeans dying waste water		
Variable	**Unit**	**Values**	**Variable**	**Unit**	**Values**
Turbidity	NTU	5	Turbidity	NTU	2
Colour	P.C.U	8	Colour	P.C.U	2
pH	-	7.2	pH	-	7.2
TSS	mg/L	10	TSS	mg/L	8
TDS	mg/L	30	TDS	mg/L	30
Conductivity	µS/cm	120	Conductivity	µS/cm	100
COD	mg/L	36	COD	mg/L	50
BOD/COD	-	0.34	BOD/COD	-	0.28
Cr	mg/L	0.9	Cr	mg/L	0.62
Cu	mg/L	0.06	Cu	mg/L	0.008
Pb	mg/L	0.02	Pb	mg/L	0.009

Comparing the specifications of wool dyeing wastewater after ion exchange in Table 8 with its specifications after oxidation in Table 7, it is apparent that during the ion exchange process, turbidity was reduced by 83.3%, color by 89.5%, electrical conductivity by 89.4%, and COD by 95%. Moreover, comparing the specifications of jeans dyeing wastewater after ion exchange in Table 8 with its specifications after oxidation in Table 7, during the ion exchange process, turbidity was reduced by 50%, color by 60%, electrical conductivity by 50%, and COD by 64.3%. When comparing the specifications of raw wool dyeing wastewater in Table 4 with its specifications after ion exchange in Table 8, it is clear that during the Advanced Oxidation Process, chemical precipitation, and ion exchange, Turbidity was reduced by 98.6%, color by 99.5%, electrical conductivity by 98%, and COD by 99.8%.Similarly, comparing the specifications of raw jeans dyeing wastewater in Table 4 with its specifications after ion exchange in Table 8, it is evident that during the Advanced Oxidation Process, chemical precipitation, and ion exchange, Turbidity was reduced by 96.4%, color by 99.6%, electrical conductivity by 95.3%, and COD by 98.9%.It is clear that the treated wastewater, as characterized in Table 8, can be used for irrigation according to the Syrian standard specification 2008 number 2580[17].

4.CONCLUSIONS

The study's key findings can be summarized as follows:

- The $TiO_2/BiVO_4$/AC textile heterojunction nanocomposite was successfully synthesized, with TiO_2 nanoparticles uniformly deposited in spherical shapes on $BiVO_4$ surfaces via a co-precipitation method. Enhanced porosity and reduced band gap contributed to superior dye removal compared with other composites ($BiVO_4$/AC, TiO_2/ AC), thereby showcasing exceptional photocatalytic performance. $TiO_2/BiVO_4$/AC exhibited remarkable efficiency in degrading organic compounds, such as anionic and cationic dyes (Indigo, Azo carmine, MB), with high stability and reusability. This research demonstrates the catalyst's high efficiency in degrading dyes effectively.
- AOP with sodium persulfate outperforms AOP without sodium persulfate, particularly

in oxidizing Tartrazine, Azo, and reactive Red Dye using violet visible light.

- Sunlight combined with persulfate and glucose is equally effective as violet visible light in breaking down dyes from wool and denim dyeing factories.
- Treated textile wastewater from the combined treatment meets the irrigation standards outlined in the Syrian standard specification 2008 number 2580.
- Further research is recommended on utilizing advanced oxidation processes to treat effluents from refineries and fertilizer plants to eliminate phenols and nitrates.
- Advanced oxidation processes are suggested for treating industrial effluents from dye factories in Hama city.
- Recycling sludge from chemical precipitation treatment into energy-efficient bricks by mixing it with clay is a potential method. Textile sludge blended with clay at a 30% ratio can be fired at 1200 degrees Celsius to create these bricks.

NOMENCLATURE

AC	Activated carbon
AOP	Advanced oxidation process
$BiVO_4$	Bismuth vanadate
BC	Conduction band
BOD	Biochemical demanded
BV	Valence band
CO_2	Carbone dioxide gas
COD	Chemical oxygen demanded
Cr	Chrome
Cu	Copper
$FeSO_4$	Ferrous sulfate
H2O	water
Hcl	Hydrochloric acid
MB	Methyl blue dye
MO	Methyl orange dye
NH_4OH	Ammonium hydroxide
Pb	Lead
SEM	Scanning Electron Microscopy
TDS	Total dissolved solide
TiO_2	Titanium dioxide
TSS	Total suspended solide
Greek symbols	
ƛ	Wave length

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