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RESEARCH ARTICLE

COMPUTER SOFTWARE AIDED PHOTOCATALYTIC DEGRADATION OF VAT YELLOW DYE USED IN LEATHER AND TEXTILE INDUSTRIES

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ABSTRACT

This research investigated design of experiment for the optimization of the experimental parameters of the photocatalytic degradation of VAT yellow dye using dye concentration (mg/L), catalyst dosage (mg/L), pH and time of exposure (minates) as experimental factors. Stock solution of vat yellow dye was prepared (0.00001M) and serial dilution was used to prepare twenty seven (27) different concentrations of the dye solution which were subjected to photocatalytic degradation in a photo reactor using a ultraviolent light source under ambient environmental condition. The absorbances of these solutions before and after photo degradation were taken to obtain different percentage degradation for each experimental run. Analysis of these results was carried out using minitab18 computer software. The result revealed the best conditions for the photodegredation as (20mg/l) (dye concentration), 2(mg/L) (catalyst dosage), 3.39 pH and 45 minutes (time of exposure). This was confirmed experimentally after the optimization to prove that minitab18 software is highly effective for the optimization of photodegradation of tannery and textile effluent containing reactive red dye.

KEYWORDS

Photocatalytic, Parameter, Dye, Minitab18 and Optimization.

1. INTRODUCTION

Different techniques such as biological detoxification, pyrolysis, biosorption, complexation, precipitation, solvent extraction, physicochemical remediation, phytochemical remediation, microbial remediation, ion-exchange, membrane processes, adsorption using activated carbon, coagulation and others have been used for the treatment of effluents (Goncalves et al., 2019). These techniques are not capable of destroying the dye components of the effluents which may be carcinogenic and highly toxic if absorbed by human through consumption of food or water contaminated by the effluent (Halmann, 2002). Advanced oxidation processes (AOPs) have been developed and used to destroy the dye components of these effluents in a process known as photodegradation (Finar, 2001).

Effluents from textile and leather industries contain high level contaminants of unconsumed dyes, surfactants and trace metals. (Gilws, 2015). This constitutes a major source of environmental pollution which is indeed a National threat (IUPAC, 1996). Heterogeneous photocatalysis via combination of titanium (IV) oxide and hydrogen peroxide as photocatalysts and ultra-violet light is considered one of the most promising advanced oxidation processes for destruction of dye components in water soluble organic pollutants (Fujishima et al., 2019). In this research, design of experiment was used to optimize the photocatalytic degradation of vat yellow dye (Hallas, 2010). Optimization is a way of adjusting controllable variables in any process to find out the suitable factors level that gives the best possible outcome (response) (Griggin, 2015). Design of experiment is a statistical based structural approach to processes that will quickly yield significant increase in product quality. It is aimed at designing and developing process in shorter time and get maximum result (Fox and Dulay, 2020). The basic principles of design of experiment (DOE) involves: randomization, replication and blocking. It includes the steps for planning and analyzing an experiment.

1.1 Statement of Research Problem

This research is aimed at subjecting vat yellow dye used in leather and textile industries to photodegradation using TiO_2 and hydrogen peroxides as catalysts in order to eliminate dye concentrations from industrial effluents and optimization of their photodegradation parameters.

1.2 Justification of the Study

Discharge of textile and tannery untreated effluents to the environment and water bodies poses a threat to the environment and aquatic lives, hence the need for the degradation of these dyes using an economical and more environmentally friendly method.

1.3 Aim And Objectives

The aim of the study is to carry out photocatalytic degradation of VAT YELLOW dyes used in Textile and Leather industries, using design of experiment.

The aim was achieved by the following objectives:

(1) Characterizing the dyes using Thin.layer chromatography, UV-visible spectrophotometry, Fourier Transform infrared Spectrophotometry (FTIR) and Gas Chromatography Mass Spectrophotometry, GCMS.

(2) Carrying out degradation studies on the dyes based on the following operational parameters; dye concentration (mg/L), catalyst loading (mg/L), time of irradiation (min), and pH of the dye.

(3) Studying the inter-dependence of these variables and how they

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affect degradation of the dyes using design of experiment.

(4) Determination of the optimized operational parameters.

(5) Experimental confirmation of the optimized conditions.

1.4 Significance of Research

The significance of the research is that environment pollution is a Global issue and it requires serious attention.

1.5 Scope of The Study

The research work is limited to VAT YELLOW dye commonly used in textile and leather industries, Characterization of the dyes and photodegradation studies of the dye and optimization of their process parameters using design of experiment.

2. Literature Review

Advanced oxidation processes (AOP's), uniting together ozone and high output ultraviolet technologies, in conjunction with hydrogen peroxide and catalyst are successfully used to decompose many toxic and bio-resistant organic pollutants in aqueous solution to acceptable levels, without producing additional hazardous by-products or sludge which require further treatment. Advanced oxidation processes involve the generation of hydroxyl (OH*) radicals which oxidize the pollutants (Gordon and Gregory, 2018). The hydroxyl radical is able to oxidize and mineralize almost every organic molecule, yielding CO_2 and inorganic ions as shown below:

$*OH + RH \longrightarrow R^* + H_2O$

$R^* + O_2 \longrightarrow RO_2 \longrightarrow$ Products and CO_2

TiO_2-based photocatalysis appears as the most emerging destructive technology (Goncalves *et al*, 2019). The key advantage is that it can be carried out under ambient conditions (atmospheric oxygen is used as oxidant) and may lead to complete mineralization of organic carbon into CO_2. Moreover, TiO_2 photocatalyst is largely available, inexpensive, and non-toxic and shows relatively high chemical stability (Grifinth, 2012).

2.1 Degradation Studies

Chemical reaction involve kinetics which make use of traditional one-factor-at-a-time (OFAT) approach, examining the effect of parameters such as initial concentration of target compound, degradation time, catalyst dose and characteristics, pH, temperature, uv light source and intensity. If the factors involved in the process are independent (which is rarely the situation) the most common practice is OFAT while holding all others constant. However, the result of this univariate analysis shows inadequate optimization towards response(s) (Gewald, 2016).

2.2 Materials and Equipment

2.2.1 Materials

The materials and chemicals used for this research were sodium hydroxide, hydrochloric acid, tetraoxosulphate VI acid, sodium nitrite, titanium IV oxide. VAT YELLOW dye.

2.2.2 Equipment

The equipment used were: Mercury bulb (25W). B22, 220 – 240V, Jenway UV-Visible spectrophotometer model 6305, digital pH meter model Jenway 3505, 78HW – 1 magnetic stirrer, electronic digital weighing Balance, Shimadzu FTIR – 8400S Fourier transformed Infrared spectrophotometer, photoreactor, equipped with heat suction fan (model 361KL – 04W – B69, 12V DC, 0.56A, DC Brushless, Gas chromatography and Mass spectro-photometer, melting point apparatus (Gallenkamp).

3. Methodology

3.1 Characterization of The Dye

- Purification of the dyes
- Determination of Melting Point
- Percentage Yield

3.2 Ultra-violet-visible Spectroscopy

The wavelength of maximum absorption λ_{max} of the dyes produced who measured using JENWAY 6305 UV-visible spectrophotometer.

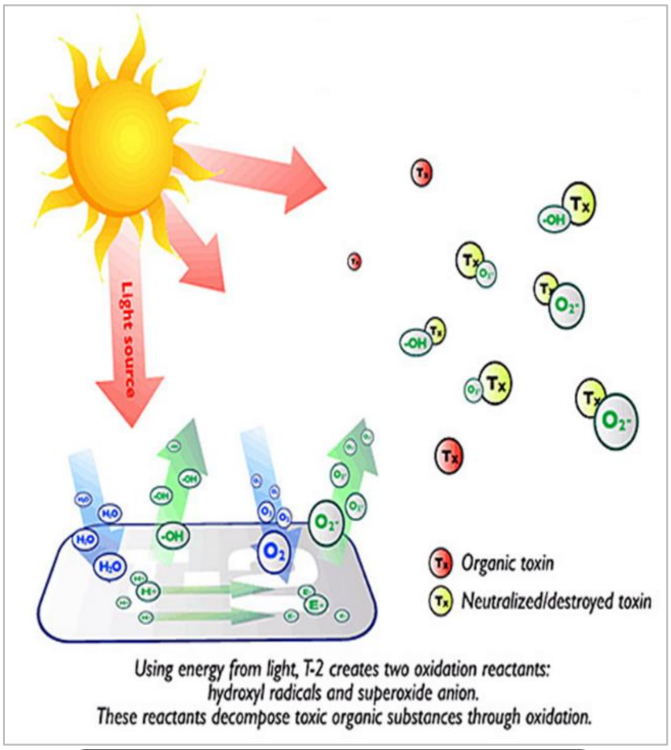
3.3 Fourier Transform Infra-red (FTIR) Measurement

The infrared peaks of the dye was determined using FTIR – 8400 series.

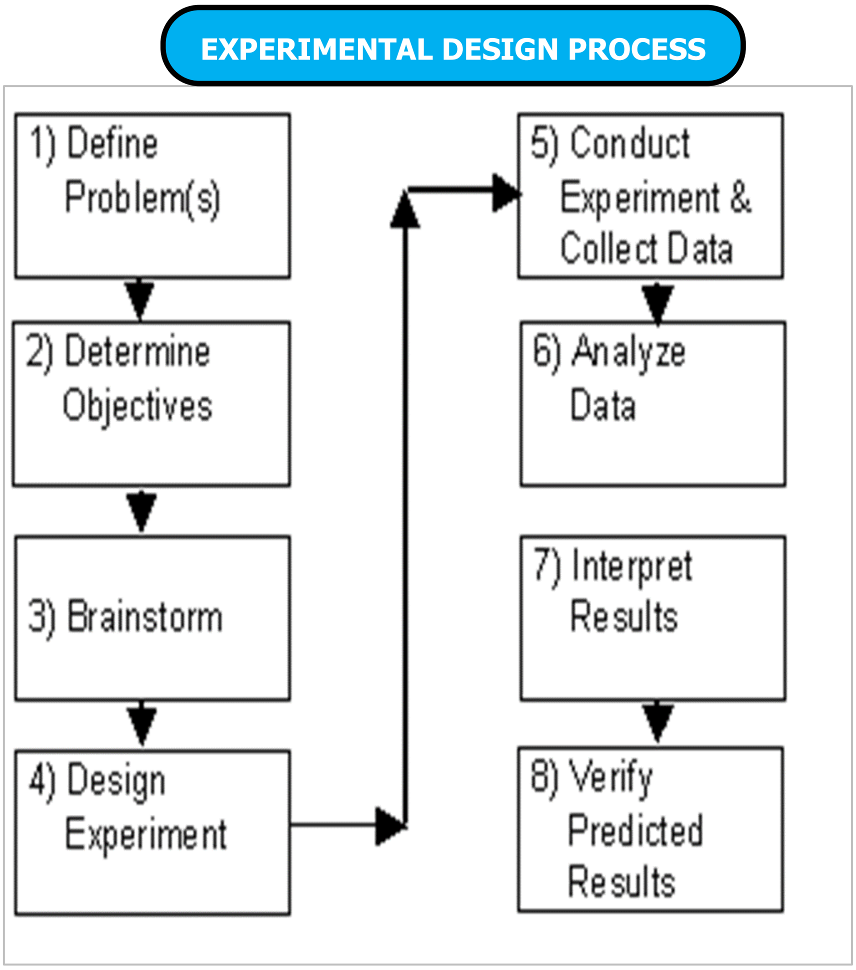
3.4 Gas-chromatography-mass Spectrophotometry Measurement

The structural elucidation of the dye was studied by the use of gas-chromatography – mass spectrophotometry.

3.5 Photodegradation



PHOTOCATALYTIC PROCESS



3.6 Design of Experiment Using Box Behnken Design

$N = 2k\ (k-1) + C_o$

Where

N = number of experimental runs,

k = number of factors,

C_o = centre point

For the ten dyes, the factors used were :

- Dye Concentration (mg/L)
- Catalysts loading (mg/L)
- Time of irradiation (min)
- pH

Number of experimental runs = $2k\ (k-1) + C_o$

$= 2(4)\ (4-1) + 3 = 27$

Exposure for dye concentration of 10mglL and a catalyst loading of 1mg/L..The plot indicates that greater than 70% degradation was obtained at the pH range of about 3.0 to 5.0 at the time of exposure of 60 to 80 minutes. This represents the optima conditions for the performances of the catalyst. when the active sites well fully utilized for photodegradation reaction.

This graph illustrates the contour plot of degradation versus pH and catalyst loading for the dye concentrates of 10mglL and time of exposure of 45 minutes. Greater than 70% degradation was obtained the pH of about 3.5 to 4.0 and catalyst loading of 1.4 to 1.5g/L.

This graph illustrates the contour plot of percentage degradation against pH and dye concentration for catalyst loading of 1mg/L at time of exposure of 45 minutes. Greater than 70% degradation was obtained at pH of 3.5 to 4.5 at dye concentration of about 15mglL.

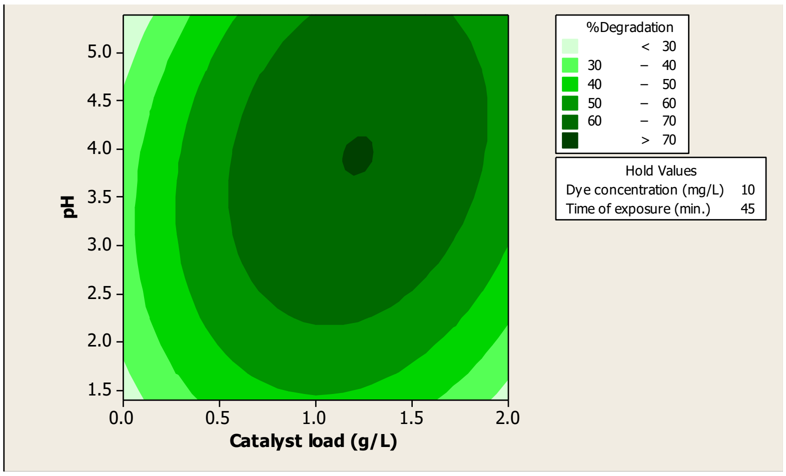


Figure 1: Contour Plot of % Degradation versus pH, Catalyst loading

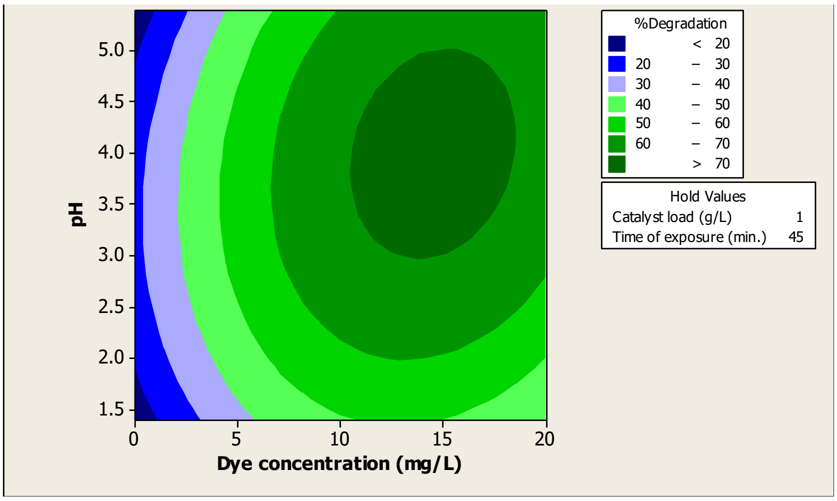


Figure 2: Contour Plot of % Degradation versus pH, Dye concentration

Table 1: Box-Bechken Design of Experiment for the VAT YELLOW Dye

Run Order	Dye Concentration	Catalyst loading	pH	Time	Absorbance		Degradation
	(mg/L)	(mg/L)		(min)	Initial	final	(%)
1	20	2	3.39	45	0.720	0.200	72.222
2	10	2	3.39	0	0.410	0.410	0.000
3	0	1	3.39	0	0.301	0.301	0.000
4	20	2	3.21	45	0.700	0.176	74.879
5	10	2	1.39	45	0.304	0.200	34.211
6	10	0	3.39	45	0.201	0.201	00.000
7	10	0	3.39	90	0.259	0.129	50.193
8	10	0	1.39	45	0.375	0.280	13.846
9	10	0	3.39	0	0.189	0.189	0.000
10	10	1	3.39	45	0.428	0.135	68.458
11	10	2	3.39	90	0.188	0.108	42.553
12	0	1	3.39	90	0.306	0.306	00.000
13	0	1	5.39	45	0.300	0.300	00.000
14	20	1	3.21	90	0.800	0288	64.000
15	10	1	1.39	0	0.220	0.220	0.000
16	20	1	3.39	0	0.750	0.750	0.000
17	0	2	3.39	45	0.102	0.102	00.000
18	10	0	5.39	45	0.325	0.298	08.308
19	10	1	5.39	0	0.282	0.282	0.000
20	10	1	1.39	90	0.605	0.385	36.364
21	10	2	5.39	45	0.507	0.201	60.355
22	10	1	3.39	45	0.428	0.134	68.692
23	10	1	3.39	45	0.429	0.136	68.685
24	20	1	1.39	45	0.701	0.320	54.380
25	0	1	1.39	45	0.201	0.201	00.000
26	10	1	5.39	90	0.346	0.118	65.896
27	20	0	3.39	45	0.701	0.610	12.981

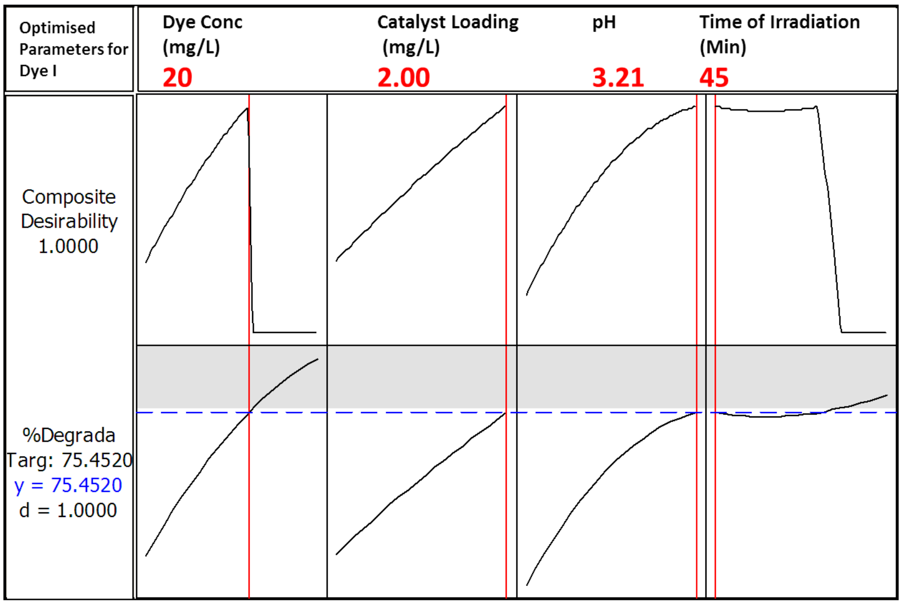


Figure 3: Response Optimization for Dye

Table 2: Experimental Confirmation of Each of The Optimized Results

Dye	Dye Conc. (mg/L)	Catalyst Loading (mg/L)	pH	Time (min)	Initial Absorbance	Final Absorbance	% Degradation
A	20	2.0	3.21	45	0.911	0.009	99.012

4. Discussions

Figures 1, 2, and 3 showed plots of standardization residual against observation order. It helped to show whether the fit is uniformly good or different for lower or higher values of the residual. Residual plots are the best single check for violation of assumptions, such as:

i. Variance not being constant across the explanatory variables.

ii. Fitted relationships being non-linear.

iii. Random variation not having a normal distribution

Residuals are random variables and arc Junctions of data. Examining residuals is a key part of all statistical modeling including Design of Experiments (DOE's). Residual tell us how well or otherwise the model fits the data. One problem with using residuals is that their values depend on the scale and units used. Since the residuals are in units of the dependent variable Y there are no cut-off points for defining what a "large" residual is. The problem is overcome by using standardized residuals. They are calculated by residual divided by standard error of the residual. The standard error of each residual is different, and using standardized residuals helps one to get round the problem.

5. Conclusion

The photocatalytic degradation of vat yellow dye using UV light as radiation source and TiO_2 as photocatalyst had been achieved using minitab 18 software to carry out the experimental design.

Summary and Recommendation

This work revealed the optimization of process parameters namely dye concentration (mg/L), catalyst loading (mg/L), time of irradiation (mins) and pH of VAT YELLOW dye.. Ultraviolet light was used to investigate the photodegradation using Titanium oxide TiO_2 as photocatalyst.

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