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RESEARCH ARTICLE

PHOTOCATALYTIC DEGRADATION OF REACTIVE RED DYE AND OPTIMIZATION OF THEIR EXPERIMENTAL FACTORS

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ABSTRACT

Box-Behnken design of experiment for the optimization of the experimental parameters of the photocatalytic degradation of reactive red dye using dye concentration (mg/L), catalyst dosage (mg/L), pH and time of exposure as experimental factors was studied and reported. Stock solution of reactive red dye was prepared (0.00001M) and serial dilution was used to prepare twenty-seven (27) different concentration 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 was taken to obtain different percentage degradation for each experimental run. Analysis of these results was carried out using minitab 17 computer software. The result reveals the optimized conditions for the photodegredationas 10mg/L (dye concentration), 1mg/l (catalyst dosage), 5.85 pH and 90 minutes (time of exposure). This was confirmed experimentally after the optimization to prove that minitab17 software is highly effective for the optimization.

KEYWORDS

Photocatalytic, reactive red, Dye, Minitab17 and experimental.

1. INTRODUCTION

One of the techniques used for the treatment of effluents is photodegradation in the presence of a catalyst. This involves decomposition of a compound by radiant energy (Abrahart, 2018). It is the photochemical degradation of phenol derivatives which represents an alternative method for biological detoxification (Akpan, 2019). Photodegradation is the photochemical transformation of a molecule into a lower molecular weight fragment, usually in an oxidative process. This term is widely used in the destructive (oxidation) of pollutants by UV-based process (IUPAC, 1996). This is an effective method because the effluent is reduced into gases as much as possible (Afili, 2013).

Photodegradation takes place in the presence of suspended semiconductor particles which may be either by a direct process, by organic molecules adsorbed on the surface of the particles which interact with holes and hydroxyl radicals on the surface or they may be indirect, by interaction of the organic molecules with hydroxyl radicals in the bulk of the solution (Akpan, 2011). Photodegradation includes photodissociation, the breaking up of molecules into smaller pieces by photons. A common photodegradation reaction is oxidation. 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) (Baran et al., *2019*).

Design of experiment is a statistical based structural approach to process that will quickly yield significant increase in product quality. It is aimed at designing and developing process in shorter time to get maximum result. (Astrand et al., 2010). The basic principles of design of experiment (DOE) involves (1) randomization (2) replication and (3) blocking. It includes the steps for planning and analyzing an experiment. The practical steps needed for planning and conducting an experiment include recognizing the goal of the experiment, choice of factors, choice of response, choice of the design, analysis and then drawing conclusions. Simply, the purpose of design of experiment is the selection of the experimental points at which the response should be evaluated. Box-Behnken design is for three levels of variables that are evenly spaced.

The number of experiments required (N) is given by:

$$N = 2k\,(k - 1) + C_0 \qquad (1)$$

where k is the number of variables and C_0 is the centre points.

The main advantage of using Box-Behnken design is that the design avoids extreme conditions of experiments (Mall and Sharp, 1968). A group researchers discussed the use and application of Box-Behnken design in different fields of chemistry as an optimization procedure of analytical methods (Box et al., 2015; Madaren, 1983). After the selection of the appropriate experimental design, the nomination of levels of the variable is handled carefully based on the path of steepest ascent/descent, preliminary experiments, prior knowledge of the process attributes or literature review and certain instrumental limitations (Chen et al., 2007).

The usage of water is increasing day by day due to the increasing number of industries. The textile industries are one of the major water consuming industries, therefore great source of environmental pollution (Ren, 2000). In these industries, water is contaminated with different chemicals and auxiliaries, which are used for producing textile goods (Bird, 2015). Approximately, 10,000 different dyes and pigments are used in industrial production, representing the annual consumption of around 700,000 tones worldwide (Kabra et al, 2004). Environmental pollution on a global scale, as well as the lack of sufficient clean energy sources, have drawn much attention to the need for developing ecologically clean chemical technology materials and processes (Booker and Salker, 2006).

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It has been shown that the use of TiO_2, H_2O_2, and Fenton (a mixture of ferrous ion with H_2O_2) are more efficient in the photodegradation of organic pollutants (Chalcrabatii and Dutta, 2004). In comparison to that of direct photolysis. Among them, one of the common observations is that the enhancement of organic decomposition is due to the generation of powerful non-selective hydroxyl radical (OH) produced in the process of photodegradation (Davis et al., 2004). The efficiency of advanced oxidative processes for the degradation of recalcitrant compounds has been extensively studied (Zhange et al., 2009).

A substantial amount of dyestuff is lost during the dyeing process in the textile industry and decolourization of dye effluents has therefore acquired increasing attention (Esplugas et al., 2009). During the past two decades, photocatalytic process involving TiO_2 semiconductor particles under UV light illumination has been shown to be potentially advantageous and useful in the treatment of wastewater pollutants (Maradiya and Patel, 2002).

1.1 Statement of Problem

This research subjecting reactive red dye to photodegradation using TiO_2 as a catalyst in order to eliminate dye concentrations from industrial effluents and optimization of their photodegradation parameters.

1.2 Justification f the Study

Discharge of textile 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 (Chatterjee Mahata, 2002).

1.3 Aim and Objectives of The Research

The aim of the study is to carryout photocatalytic degradation on the reactive red dye using Box-Behnken design. The aim is achieved through the following objectives:

i. Characterizing the dye using UV-visible spectrophotometry, Fourier Transform infrared Spectrophotometry (FTIR) and Gas Chromatography Mass Spectrophotometry, GCMS.

ii. Carrying out degradation studies on the reactive red dye based on the following operational parameters; initial dye concentration, catalyst loading, time of exposure, and pH of solution using UV-Visible at the λ_{max} of each dye.

iii. Studying the inter-dependence of these variables and how they affect degradation of the dye using design of experiment (Box-Behnken)

iv. Determination of the optimized operational parameters.

1.4 Significance of Research

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

1.5 Scope of The Study

The research work is limited photodegradation studies of the reactive red dye using design of experiment and optimization of the experimental factors.

2. LITERATURE REVIEW

Dyes and dye intermediates with high degree of aromaticity and low biodegradability are introduced into the aquatic system resulting in increase of environmental risk. Effluents from textile industries are important sources of environmental contamination. Several studies of photocatalytic degradation of dyes have been reported (Chatterjee and Mahata, 2002; Herrera et al., 2000). 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 pollutant**s.** The hydroxyl radical is able to oxidize and mineralize almost every organic molecule, yielding CO_2 and inorganic ions as shown below:

$$^*OH + RH \longrightarrow R^* + H_2O \qquad (2)$$

$$R^* + O_2 \longrightarrow RO_2 \longrightarrow \text{Products and } CO_2 \qquad (3)$$

Azo dyes are characterized by the presence of one or more azo (-N = N -) groups in the dye molecule which forms bridges between organic residues of which at least one is usually an aromatic nucleus. The importance of azo dyes is due to the fact that they account for over 60% of the synthesized dyes structures are found in most dyestuff application class such as acid dyes, basic dyes, direct, azoic, reactive, mordant etc. The advantage of using azo links is to connect groups which are themselves not coloured such as benzene and naphthalene. Azo dyes are described as monoazo, disazo, trisazo, tetrakisazo and polyazo according to whether there are respectively one, two, three, four or more azo groups present in the dye molecule. TiO_2-based photocatalysis appears as the most emerging destructive technology. 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_2photocatalyst is largely available, inexpensive, and non-toxic and shows relatively high chemical stability.

Interest in the design of azo dyes containing heterocyclic moieties stems from high degree of brightness compared to azo dyes derived from aniline (Maclaren, 2000). The 2-aminothiophene based azo dyes are known as disperse dyes with excellent brightness of shade. This class of dyes was established as an alternative to more expensive anthraquinone dyes (Zhange, 2009). The thiophene containing azo dyes have many advantages including a colour deepening effect as an intrinsic property of the thiophene ring and small molecular structure leading to better dyeability. The heterocyclic nature of the thiophene ring has also allowed for excellent sublimation fastness on the dyed fibres (Davis et al., 2004). Increasing the electron-withdrawing strength of the substituent on the thiophene ring resulted in bathochromic shift (Afilli, 2013). Additionally, the sulphur atom plays a decisive role by acting as electron sink as explained by valence bond theory (Booker and Salker, 2006).

A one-pot procedure created an efficient route to the synthesis of thiophene based heterocyclic dyes (Akpan, 2019). A number of researchers studied aminothiophene derivatives as azo disperse dyes in the dyeing of syntheticfibres, blended polyester, wool and more recently in optical data storage devices. This class of compounds also showed semi-conducting properties.

Dyes owe their colours to the characteristic property their molecules possess. Bright colours are as a result of strong selective absorption, the absorption bands being relatively narrow and rising steeply to high intensities. The absorption bands of dull colours are not sharp but extend across the entire spectrum (Bird, 2015).

Table 1: Absorbed and Complementary Colours

Wavelength, nm	Colour absorbed	Visible (complementary) colour
400-435	Violet	Yellow-green
435-480	Blue	Yellow
480-490	Green-blue	Orange
490-500	Blue-green	Red
500-560	Green	Purple
560-580	Yellow-green	Violet
580-595	Yellow	Blue
595-605	Orange	Green-blue
605-750	Red	Blue-green

3. MATERIALS AND METHOD

3.1 Materials

The materials and chemicals used in this work are sodium nitrite, sulphuric acid, acetic acid, propionic acid, sodium hydroxide, hydrochloric acid, Reactive red dye.

3.2 Equipment

The equipments used in this work are: 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).

$$\% \text{ Degradation} = \frac{A_i - A_f}{A_i} \times 100 \quad (4)$$

Where: A_i and A_f are the initial and final absorbance at a given time 't'.

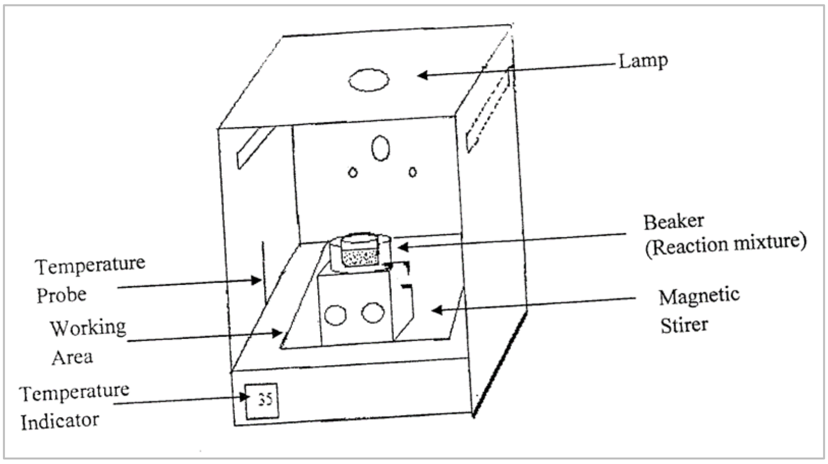

Figure 1: Schematic Diagram of a Photoreactor

Table 1: Result of the Photodegradation of the reactive red dye

Run Order	Dye Concentration (mg/L)	Catalyst loading (mg/L)	Ph rH	Time (Min)	Absorbance		Degradation %
					Initial	Final	
1	10	0	1.85	45	0.255	0.216	15.294
2	0	0	3.85	45	0.139	0.139	00.000
3	0	1	1.85	45	0.326	0.326	00.000
4	20	1	3.85	0	0.725	0.725	00.000
5	10	0	5.85	45	0.165	0.150	09.090
6	0	1	3.85	90	0.176	0.176	00.000
7	0	2	3.85	45	0.185	0.185	00.000
8	10	1	1.85	90	0.175	0.130	25.760
9	10	1	3.85	45	0.422	0.211	50.000
10	10	2	3.85	90	0.122	0.041	66.393
11	10	1	3.85	45	0.421	0.212	49.763
12	10	2	3.85	0	0.142	0.142	0.000
13	20	2	3.85	45	0.610	0.394	35.484
14	10	1	5.85	0	0.300	0.300	0.000
15	10	2	1.85	45	0.310	0.200	35.484
16	10	1	5.85	0	0.300	0.300	0.000
17	20	1	5.85	45	0.625	0.327	45.950
18	10	1	5.85	90	0.324	0.051	84.782
19	20	1	1.85	45	0.600	0.350	41.667
20	10	1	3.85	45	0.422	0.212	49.763
21	10	2	5.85	45	0.322	0.200	37.882
22	0	1	5.85	45	0.225	0.225	00.000
23	20	1	3.85	90	0.603	0.290	51.907
24	10	0	3.85	0	0.172	0.172	0.000
25	20	0	3.85	45	0.620	0.550	11.290
26	10	0	3.85	90	0.132	0.112	15.152
27	0	1	3.85	0	0.184	0.184	0.000

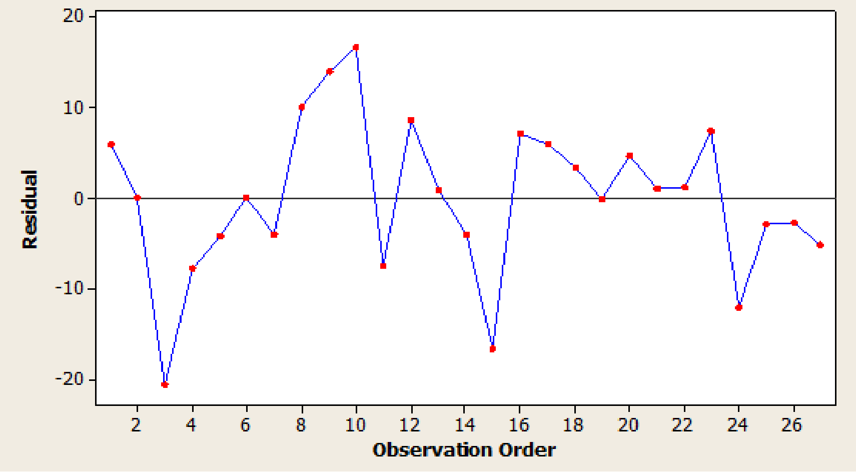

Figure 2: Standardization residual versus Observation Order

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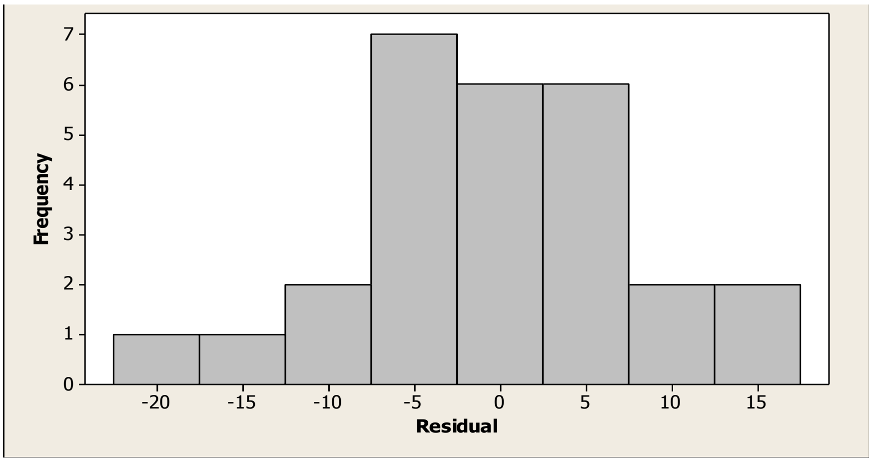

Figure 3: Histogram of frequency versus Standardized residual

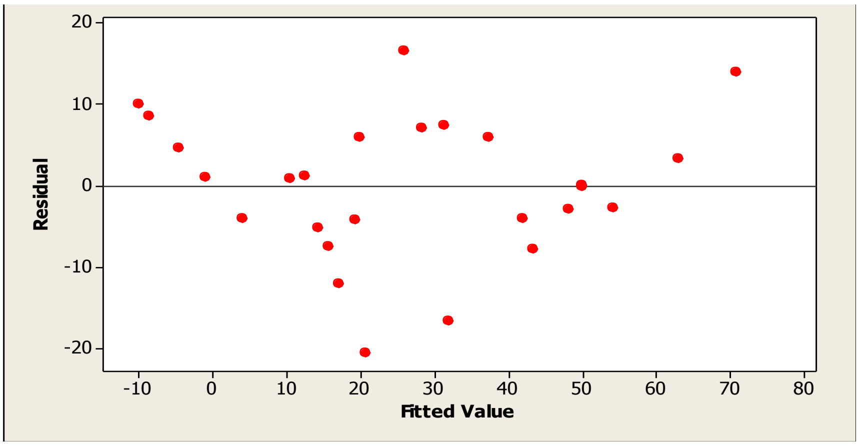

Figure 4: Standardized residual versus fitted values

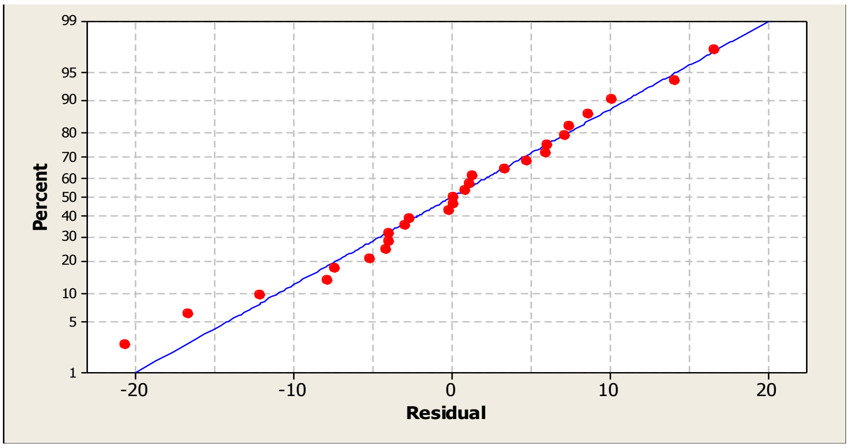

Figure 5: Normal Probability Plot

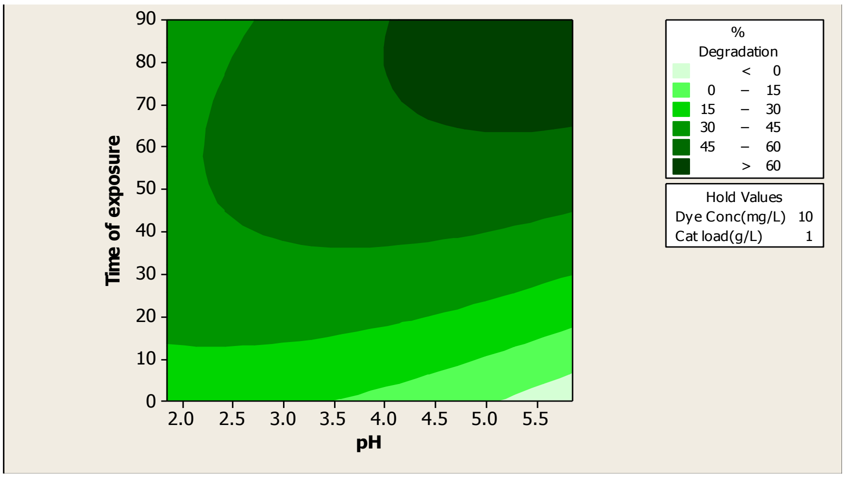

Figure 6: Contour plot of % Degradation versus Time, pH

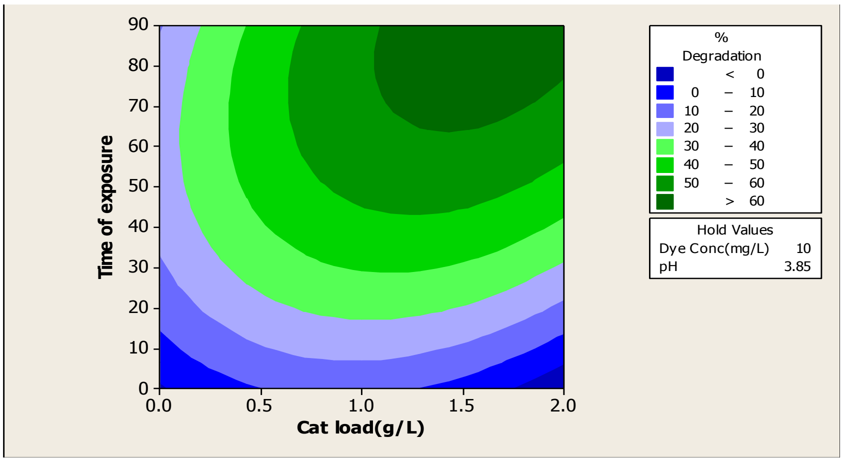

Figure 7: Contour Plot of % Degradation versus Time, Catalyst load

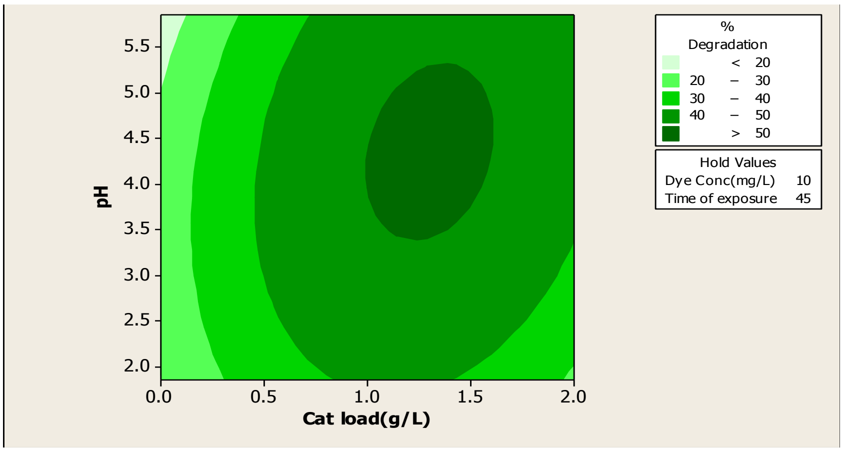

Figure 8: Contour Plot of % Degradation versus pH, Catalyst loading

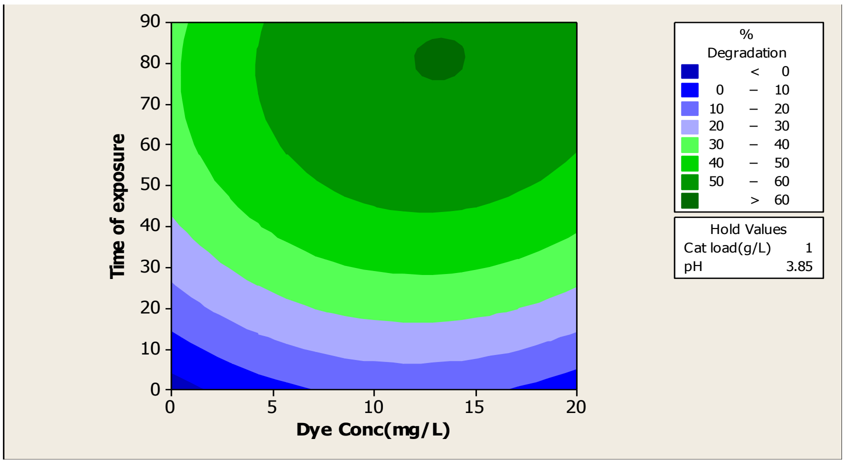

Figure 9: Contour Plot of % Degradation versus Time, Dye Concentration

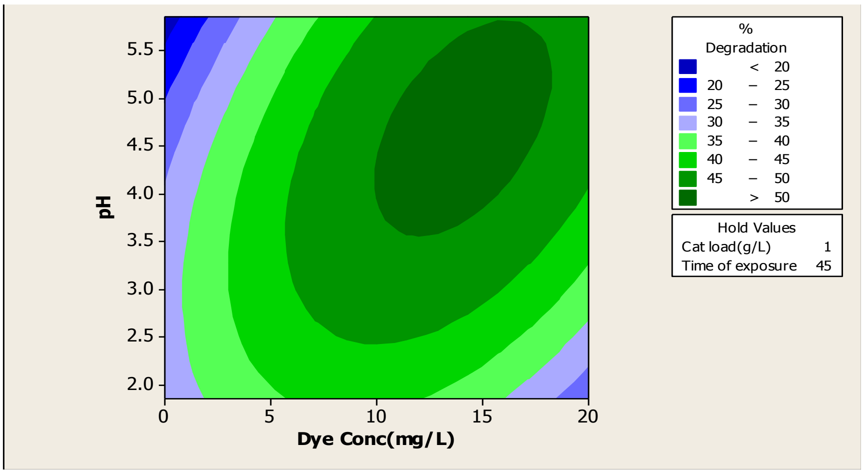

Figure 10: Contour Plot of % Degradation versus pH, Dye concentration

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4. DISCUSSIONS

Figures 1, 2, 3, 4, 5, 6, 7, 8, and 9 showed different 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 standardised residuals. They are calculated by residual divided by standard error of the residual. The standard error of each residual is different, and using standardised residuals helps one to get round the problem.

5. CONCLUSION

The photocatalytic degradation of reactive red dye using UV light as radiation source and TiO_2 as photocatalyst has been achieved using minitab 17 software to carry out the experimental design. The observations in these investigations clearly demonstrate the importance of choosing the optimum degradation parameters to obtain a high degradation rate, which is essential for any practical application of photocatalytic oxidation processes. The experiments showed that higher photodegradation efficiency can be achieved under the optimized conditions. Thus, this can be used as an efficient technology for photocatalytic degradation of the coloured wastewater discharged from the textile and leather industries. The results obtained also showed that degradation rate can be influenced by operational parameters such as pH, irradiation time, catalyst loading and dye concentration apart from the presence of electron acceptors and other additives that can enhance the process.

1. Photocatalyst in the presence of TiO_2 suspension and UV light brings about effective destruction of azo dye of different chemical functional groups.

2. The rate of photodegradation is influenced by operational parameters namely photocatalyst, pH, dye concentration and time of exposure.

3. Optimization of the operational parameters to achieve optimum percentage degradation is achievable using minitab.17 software.

SUMMARY

This work revealed the optimization of process parameters namely dye concentration (mg/L),catalyst loading (mg/L), pH and time of exposure (minutes) of reactive red dye. .Ultraviolet light was used to investigate the photodegradation using Titanium oxide T1O2as photocatalyst in slurry form. In this research work, the photocatalytic degradation of the dyes was carried out successfully using TiO2-P25 (Fluka) as photocatalyst in the presence of ultra violet light. The rates of degradation of the dye solution were determined on the basis of absorbance measurement using the JENWAY 6305 UV-V is Spectrophotometer. The effects of operational parameters such as dyes concentration, catalyst loading, irradiation time and pi I have been investigated. The influence of pH on the rate of degradation cannot be over emphasized as changes in pH influenced the adsorption of dyes molecules onto the TiO2 surfaces, an important step for the photocatalytic oxidation to take place. The hydroxyl radicals are considered as the predominant species at neutral or high pH levels. In alkaline solution, •OH are easier to be generated by oxidation of more hydroxide ions available on TiO2 surface, thus enhancing the efficiency of the process.

The dye concentration was also found to be another important factor in the photocatalytic reaction. The rate of degradation relates to the probability of •OH radicals formation on the catalyst surface and to the probability of •OH radicals reacting with dye molecules. As the initial concentration of the dye increased, the probability of reaction between pigment molecules and oxidizing species also increased, leading to an enhancement in the decolorization rate. On the contrary, the degradation efficiency of the dye decreased as the dye concentration increased beyond an optimum value. The presumed reason is that at high dyes concentration the generation of •OH radicals on the surface of the photocatalyst are reduced since the active sites are covered by dye ions. Another possible cause for such results is the UV-screening effect of the dye itself. At a high dye concentration, a significant amount of UV may be absorbed by the dye molecules rather than the TiO2 particles and this reduces the efficiency of the catalytic reaction.

Dye degradation is also influenced by the amount of photocatalyst used. The dye degradation increased with increasing photocatalyst concentration which is a characteristic of heterogenous photocatalysis. The reason generally advanced for this is that increase in the amount of photocatalyst increases the number of active sites on the photocatalyst surface, which in turn increases the number of hydroxyl and superoxide radicals. Again, when the concentration of the photocatalyst increased above the optimum value, the degradation rate decreased due to the interception of the light by the suspension. Furthermore, the increase of photocatalyst concentration beyond the optimum may result in the agglomeration of catalyst particles, hence the part of the catalyst surface becomes unavailable for photon absorption, and degradation rate decreases. The irradiation time also played a role in the degradation of the dyes. It was evident that the percentage of decolorization and photodegradation increased with increase in irradiation time. The optimized operational parameters for Dye were 10mg/L for dye concentration, catalyst loading of 1.0mg/L , pH of 5.85 and time of exposure of 90 minutes.

RECOMMENDATIONS

In view of the above findings, the following recommendations are therefore made.

1. Since the use of titanium dioxide has proved to be effective in the degradation of these dyes, the technology could be extended to treatment of textile effluents containing other dyes and pigments of varying chemicals structures.

2. Future research is needed for the development of alternative and efficient catalysts for harnessing solar energy in the Photocatalytic oxidation process.

3. Highly efficient photoreactors and solar pannels should be designed for photocatalytic treatment of textile waste effluents for both small and large, scale textile industries.

4. Other dyes can be subjected to photodegradation reaction based on their chemical structures.

5. Other photocatalyst can be used to investigate their efficiency on the reactions.

6. Other sources of ultra violet light can be used to compare the rate of photodegradation.

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