Process Optimization of Color Removal From an Industrial Azo Dye

P. Kingston Stanley, Sanjeevi Gandhi. A, K. Suresh Kumar, S. Kalidass

Abstract: ***Pure and hygienic water is of great demand but most of the water sources are being contaminated by a variety of pollutants on a daily basis. Effluents from textile industries is one of the prominent causes of water pollution. They contain a mixture of various aromatic and azo dyes, which are made of complex structures and are highly recalcitrant. The dye color in the water bodies become a major aesthetic problem along with other ecological problems. A number of physical and chemical methods are available for dye removal apart from aerobic and anaerobic biological degradation. A majority of these methods have limitations and are less successful in the complete dye removal. Metal nanoparticles are recently being employed in the decolorization of the textile dyes and they show promising results in comparison to other conventional methods. The output of this paper is to design a high efficient process with complete automation. Instrumentation setup plays a major role in the automation of the process.***

Keywords: Color removal, Nano particle, Color optical sensor.

I. INTRODUCTION

In the recent times, water pollution has become an important concern because of its direct intervention in the human and animal lives. Due to rapid urbanization and industrialization, the level of toxic and hazardous chemicals released from various industries are causing a threat to the eco-system. Among the various industries that are causing pollution, the textile dyeing and finishing industry plays a prominent role [1]. More than 100,000 commercially available dyes are used in various processes of textile industry. Most of the synthetic dyes from the textile industry are composed of complex structures which are toxic, carcinogenic and are highly recalcitrant [2, 3]. Of all the industrial dyes, azo dyes are versatile because of their easy use and varied applications [4]. As a result of it accounts for the majority of all the textile effluent. These effluents, when released into water bodies, increase the turbidity and colloidal nature of the water, thereby affecting biochemical oxygen demand (BOD) and chemical oxygen demand (COD) and an alteration in the pH [5, 6].

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Hence, it becomes necessary to convert the effluents in to non-toxic products, before its release in to water sources. As the azo dyes are stable and impervious to degradation, the existing treatment procedures are unable to remove them completely. Although different treatment methods are currently in use, they have their own shortcomings [7, 8, 9, 10, 11]. So, it is highly essential for development of other efficient and promising methods. Nano remediation is one of the current method which is being used for the remediation of a variety of pollutants [12, 13, 14].

II. MATERIALS AND METHODS

Preparation of Dye samples

The azo dye Red RR was obtained from a textile dyeing industry at Tirupur. Dye solutions of required concentration were prepared by mixing the dye powder in distilled water and used for experiments.

Preparation of the Matrix

Encapsulation of bacteria in Alginate Beads

The experimental bacterial strain (Aneurinibacillus aneurinilyticus) was isolated from effluent sludge of a textile industry. The organism was immobilized in calcium alginate beads. For the immobilization process, bacterial cells were grown overnight in culture media and harvested by centrifugation at 7000 rpm for 10 minutes. The cells were washed with saline and suspended in 2.0% sodium alginate. This slurry was then extruded drop-wise into 4.0% Calcium chloride($CaCl_2$)solution. $CaCl_2$ acts as a cross- linking agent which stabilizes the beads. They were allowed to harden in the $CaCl_2$ solution for 10-12 hours at 4°C, after which they were washed with saline to remove excess amount of free cells. The diameter of the beads were measured was found to be in the range of 2mm to 5mm. The whole procedure was carried out under aseptic conditions. The beads with the bacteria were then used for decolorization experiments.

Immobilization of iron nanoparticles on the polymer substrate

Carrier polymers of different pore sizes were purchased from a local supplier and were cut into uniform sizes of 1x1x1 cm. The polymer was pretreated by soaking them in a dilute hydrochloric acid solution (4%) for a period of 8 – 12 hours. All the mandatory reagents required for nanoparticle synthesis viz., 1.0 M Ferric chloride ($FeCl_3$), 1.6 M Sodium borohydride ($NaBH_4$) and 0.1% Palladium acetate

[Pd(CH_3COO)2] were prepared using 40% ethanol. Iron nanoparticles were then synthesized onto the matrices by the sodium borohydride reduction method [15]. Palladium acetate was doped onto the synthesized nanoparticles to enhance the activity.

Sensor Design and Calibration characteristics

Color sensor

The color sensor utilized in the proposed work is an IC - TCS3200. This IC consists of 8 x 8 array with a total of 64 photodiodes which the light-to-frequency. Out of this 64 photodiodes, 16 are used as blue(B) filter mode, another 16 photodiodes are used as green(G) filter mode, next 16 photodiodes are deployed as red(R) filter mode, and the remaining 16 photodiodes are used as white / no filter mode. The pins S2 and S3 are used to select the particular filter mode as per the tabulation made in table 1. To diminish the effect of variation in incident irradiance the four filter modes are combined together.

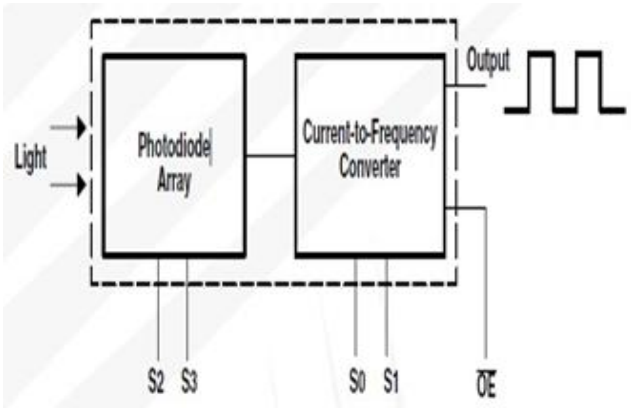

Fig. 1 Functional block of TCS3200

The pins S0 and S1 are used to select the scaling factor for frequency scaling as per the tabulation made in table 2. By selecting the switches S0 and S1 value high, 100% scaling can be achieved. For further reducing the scaling to 20% and 2%, the switches values can be 1 0 and 0 1 respectively.

Table. 1 Photodiode modes

S_2	S_3	PHOTODIODE TYPES
0	0	R FILTER MODE
0	1	B FILTER MODE
1	0	No FILTER MODE
1	1	G FILTER MODE

Table. 2 Output Frequency Scaling

S_0	S_1	FREQUENCY SCALING (fo)
0	0	Power Down
0	1	2%
1	0	20%
1	1	100%

Sensor Standardization

Calibration is the process of relating two unknown measurable quantities. Any sensor should be calibrated initially to meet the standards, provided by the manufacture in the datasheet. The colored solutions were prepared at five different known concentrations of Red dye & Blue dye solutions and measured with TCS3200 in all the four filter modes to obtain the output frequency of the known solution. And the above procedure repeated for many solutions and tabulated. Figure.2 &Figure.3 shows the different filter frequency results of various red & blue known concentrations. These results helped in choosing the right filter mode for designing the color removal process.

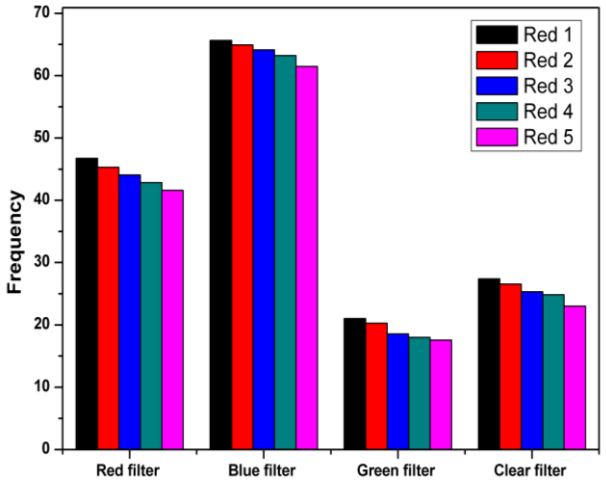

Fig. 2 Frequency vs Red Filter

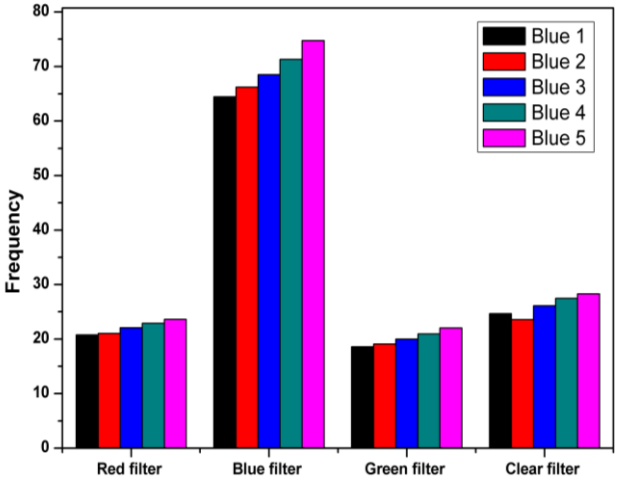

Fig. 3 Frequency vs Blue Filter

Experiments were carried out in a batch mode to find out the decolorization efficiency of the immobilized bacteria and immobilized nanoparticles. The same experiment was carried out in a closed and open loop format.

Fabrication of a pilot setup

The Bio-Process setup was prepared by packing the synthesized PUF in a glass column as depicted as shown in Figure.4. The prepared color dye solutions collected in the tank and pumped to the column with the flow rate of 20ml/min. To allow this minimum flow rate a bypass (T junction) valve also were used. A similar set was made for the bacteria also replacing the polymer column with sodium alginate beads which had the bacteria immobilized in them.

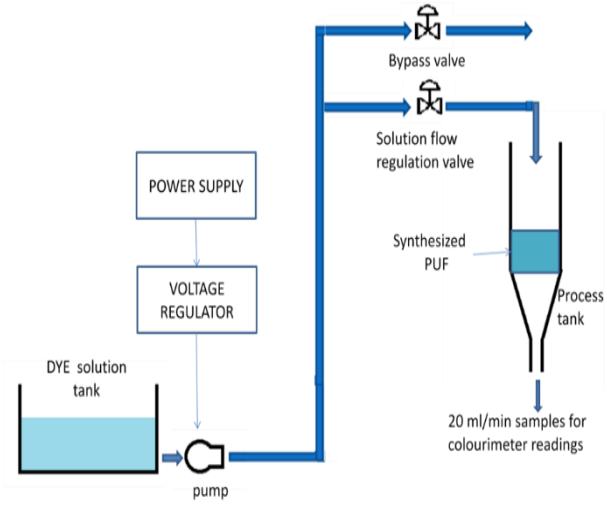

Fig. 4 Bio-Process setup

III. RESULT AND DISCUSSION

Flow rate optimization

The automated process of decolorization requires an optimum flow rate for the colour removal process. Hence a closed loop system with a TCS3200 as a colour sensor, a pump as the actuator and the PWM signal as the control for operating the pump. The controller was designed for the above the process to remove the colour effectively. The pump was tested with various input voltages to obtain the flow rate and tabulated. Figure 5 shows the various regulated output flow rate for different input voltages. For the efficient decolourization, a flow rate of 20ml/min was fixed and regulated by regulator under open loop test. For the closed loop, PWM signals were generated by the designed controller for the effective decolorization.

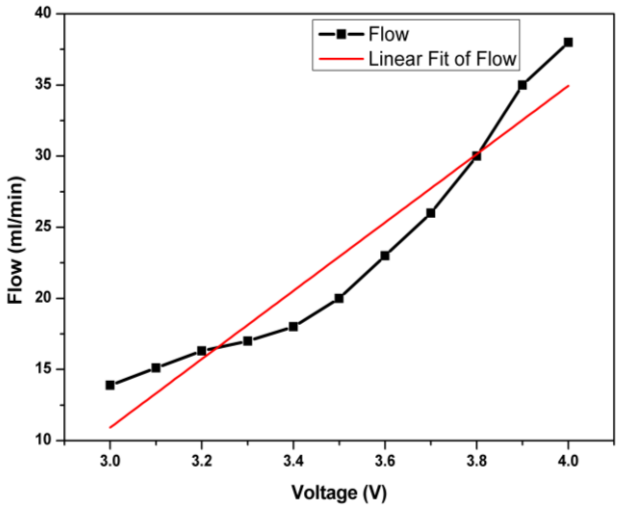

Fig. 5 Actuator Voltage vs regulated flow

Open Loop Test

The system ran in open loop test and the decolorizing efficiency were calculated. Under open loop a constant flow rate of dye solutions were pumped to column. From the Figure.6, it is evident that the decolorizing efficiency goes down after few rounds of decolorization for PUF matrix. The shelf life of PUF matrix in decolorizing the dye is estimated in this experiment.

The alginate beads is packed together to form a sodium alginate filter. The construction of this experimental setup is entirely different from the PUF setup, where in the outlet flow rate is maintained constant and there is no inlet continuously. The dye solution is passed in to the column and the eluates (20ml) were collected at regular time intervals (every hour). Percentage of degradation values were calculated. The efficiency of the alginate beads is proportional to the retention time of the dye solutions in the presence of the bacteria. From the Figure.7, it is evident that the decolorizing efficiency goes down after few rounds of decolorization for alginate beads.

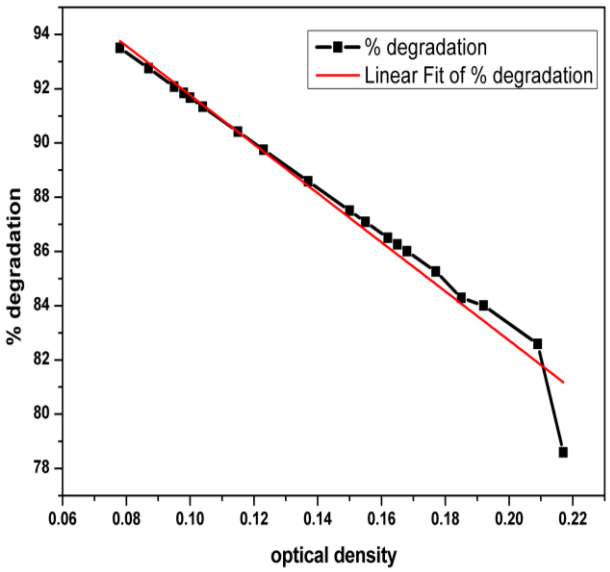

Fig. 6 % of color degradation vs optical density from PUF experiment

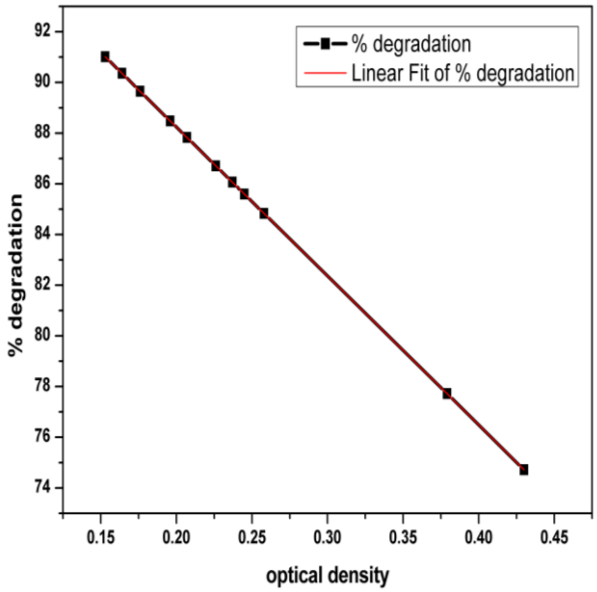

Fig. 7 % of color degradation vs optical density from SA experiment

Closed Loop Test

The entire setup is run in a closed loop with a suitable controller. The experimental setup discussed in section 2.4 is run as closed loop system with a suitable control algorithm implemented in LabVIEW. In the closed loop system the output of the process tank which is expected be decolorized solution is monitored using color sensors to check for any amount of dye present in the solution. The two sensors are arranged in such a way that it measures the presence/absence of the color present in the dye solution. The two sensor are placed before and after the process tank and the datas are collected and compared to conclude on how much of color is being removed from the dye solution. Based on this data the PID controller algorithm is implemented in LabVIEW and does the operation of the pump is controlled. The output of the process tank is collected in the settling tank if the dye is completely decolorized else the dye solution is fed back into the process tank for the further decolorization. Figure. 8 (Closed loop setup), Figure. 9, is the closed loop experimental setup for the decolorization process. So the feedback as well as feed forward control scheme implemented in the closed loop process ensures maximum decolorizing efficiency.

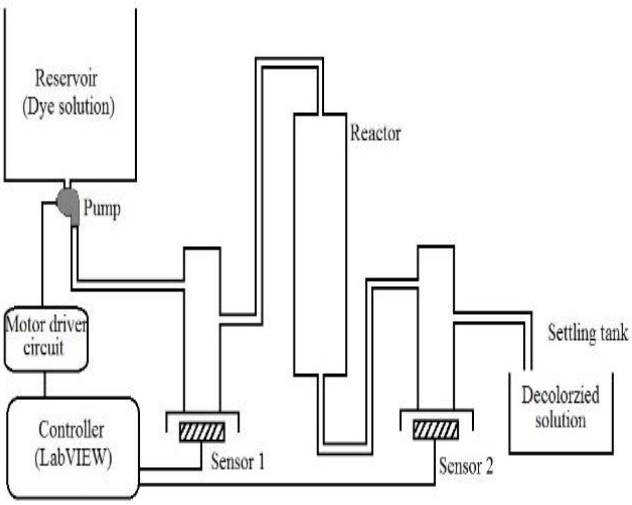

Fig. 8 Closed loop setup

Fig. 9 Experimental setup

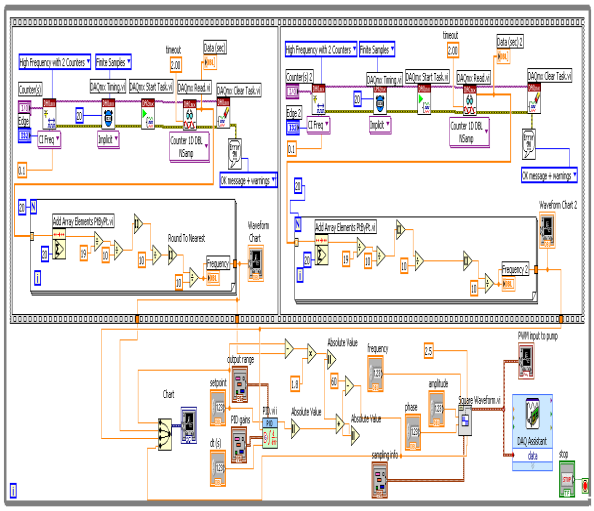

Fig. 10 Frequecny counter labview program (block diagram)

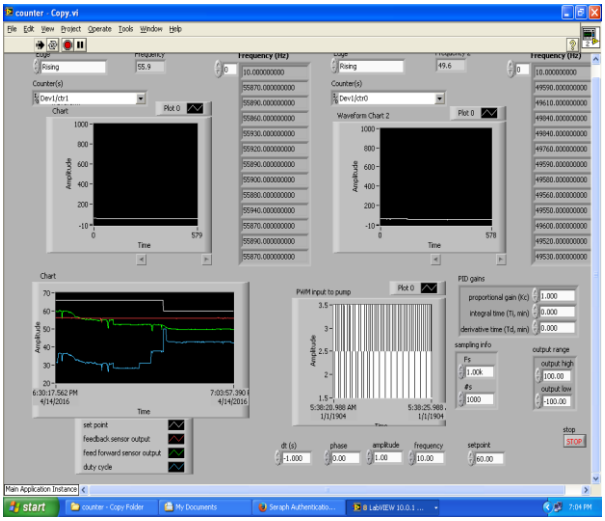

Fig. 11 Frequecny counter labview program (front panel)

Unlike the nanoparticles immobilized in PUF, alginate immobilized bacteria take more time to decolorize because the microbes require a doubling time. It is during this phase that the microbial cells double and grow rapidly and release enzymes which subsequently metabolize the dyes. Closed loop color removal (Figure.9) in which the feedback as well as the feed forward sensors are interfaced to the real-time system. The flow rate is regulated by the pump which is driven by PWM input. The setup was made to run as a temporary system in order to obtain the gain values for the controller. Figure.10 and Figure.11 are the Block Diagram and the Front Panel design respectively in LabVIEW Software.

IV. CONCLUSION

The sensor data is acquired via a NI-DAQ in LabVIEW and the PID control algorithm is implemented to adjust the flow rate by PWM circuit.

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The main objective of the proposed work to attain the maximum color removal for the given solution is successfully achieved. The effectiveness of the proposed methodology is evaluated by using similar test offline. The comparison study of PUF and SA test were carried out successfully. Nano particle immobilized on the polymer gives better decolorization when compared to the sodium alginate immobilized microbes. In the above experiments, only a standard volume (1 liter) of the dye solution was tested against the immobilized bacteria and nanoparticles. Further analysis with higher volumes of dye solutions should be done in order to determine the saturation point for the individual columns. Technologies are emerging towards greater heights as such this conceptual model is made out to give an efficient way in the decolorization aspect.

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