

Enhanced Degradation of Dyes present in Textile Effluent by Ultrasound Assisted Electrochemical Reactor

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Abstract

Textile industry being the backbone of any country plays a very essential part in the development of the country. The treatment of chemical dyes present in textile wastewater and its reuse for irrigational purposes has become a major concern for the researchers. The present study emphasis on proper degradation of commonly used reactive blue (RB) 19 dye present in textile effluents using ultrasound assisted electrochemical reactor technique and presenting the analysis of microparticles present in dyes and its quantitative composition before and after treatment by means of scanning electron microscopy (SEM) images at high magnification. The investigation was carried out using various parameters such as Concentration, pH and reaction rate. The testing setup also includes UV absorbance spectrophotometer, ultrasonic bath, DC power supply, weighing balance, suction apparatus, and thermometer. Our studies show that the Optimum dye degradation (i.e. 82.3 %) was achieved at time 120 minutes with pH of 3.22 for 50 ppm of solution and the maximum degradation (i.e. 85%) was achieved at 40 °C using acid (HCl) and Base (NaOH) in equal amounts after 120 minutes for solution of 30ppm. The work efficiency includes saving time, money and degrading the dyes from wastewater before toxic sludge formation.

Keywords: Dye Degradation; Ultrasonic Assistance; Electrochemical Process; Reactive Blue (RB) 19 Dye; Textile Effluent.

1. Introduction

The rapid Industrialization has polluted our Environment by releasing the toxic wastes and effluents. Because of dyeing activities, a very large amount of harmful industrial waste is being released by textile industries [1]. On a global scale discharge of 280,000 tons per year of textile effluents are estimated to be released significantly polluting the groundwater [2]. More than 50 million cubic meters of groundwater is being used by textiles and clothing industries in Tunisia, most of which are not renewable [3]. In this regard, many scientists and scholars are seeking measures to reduce the amount of water used in the dyeing process [4] and usage of eco-friendly dyeing materials [5]. Drinking water crisis due to environmental pollutions is a critical problem in any dry climate region. Textile dyeing operates using various classes of chemical dye and additional chemicals depending on the textile are used, which results in mixed wastewater [6] and their sludge is comprised of residual dyes such as auxiliary chemicals, surfactants, chlorinated compounds and salts [7]. Generally, textile dyes show a huge resistance to degradation, resulting in their removal from textile wastewater

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to be difficult. Therefore, the effluents do not diminish their color when they leave the plant [8]. Textile coloration is the main consumer of high-quality water, which at the end of the processing is carried into a wastewater treatment plant [9].

If an average consumption of 100 L of water per 1 kg textile material and overall consumption of 30 million tons textiles per year is assumed then the total water consumption can be estimated as 3000 million m³ of water per year. Textile dyeing practices have been acknowledged as a source that contributes considerably to the overall chemical load in the wastewater. In almost every case the residual dyes lead to intensively colored effluents [10]. Textile dyes hold a chemical anchor group in their molecular structure, which form a covalent chemical linkage to the cellulose fiber. In any textile dyeing process, the hydrolysis of the anchor group of dye molecules takes place in a competitive side reaction, ultimately releasing the end by-product of dyes as effluent or sludge [11]. Theoretically, it is believed that the recovery of other dyes e.g. direct dyes, vat dyes including indigo, sulfur dyes, is possible. Although the loss in functionality of the anchor group avoids any reprocess of consumed reactive dyes. Every time it very difficult to degrade the textile dye pollutants due to a very wide change in concentration and types. One of the main problem related to the use of reactive dyes is showing a low affinity for fabrics compared to high affinity for textile wastewaters. There is nothing wrong in saying that almost 15% of the dyes used in the textile dyeing get released to the effluents [12]. It is undesirable to release these effluents to the environment without any proper treatment and may prove to be toxic as well as mutagenic to different forms of aquatic life [13]. Textile dyeing process includes various operations such as pre-treatment, dyeing, printing, and washing of garments resulting in the generation of a large number of polluted effluents. The processing of one ton of textile produces about 230-270 m³ of wastewater and is needed to be treated prior to its release into the environment [14]. Dyes are the most unsafe chemical mixes found in textile effluents and should be treated properly as their occurrence in water bodies reduces light infiltration precluding the photosynthesis of aqueous flora [15, 16]. Problems associated with textile effluents include allergy, dermatitis, and skin irritation [17] and in a grave situation, it provokes cancer [18] and mutation in humans [19]. Generally, water gets polarized into H⁺ and OH⁻ radicals by using the ultrasonic procedure [20]. The manufacturing companies are widely using activated carbon for the removal of wastewater due to its high porosity and large surface area [21]. Adsorption technique is another cost effective and efficient technique for dye removal from the aqueous solution by treating effluents [22]. Among all other types of dyes, azo dyes are more frequently used dyes around the world with a maximum production rate of 70% [23]. A known semiconductor zinc oxide (ZnO) among various photo catalyst is widely used for the photolytic degradation of various organic pollutants due to its high photosensitivity, good stability and with low cost and non-toxicity [24]. It is also noted that the use of high-frequency ultrasound waves is effective in decolorizing the effluent of the dyeing process containing azo dyes, but ineffective in mineralizing them [25].

There are three conventional methods used for the treatment of textile industry wastewater i.e. physical (which involves filtration, flocculation, and adsorption), oxidative (advanced and chemical treatment), and biological (through enzymes and microorganisms). In spite of the greater success stories by these conventional methods, key issues like regeneration/reusing ability of adsorbent, cost and lower sorption capacity in multiple pollutant systems are still the main concern that halts their use in the treatment process. This research aims at the conversion of toxic dyes of industrial wastewater into non-toxic end products with the ultrasound-assisted electrochemical process for the decomposition of dyes. Further aiming at improving water management to prevent industrial pollution. It also includes the disposal of treated wastewater and its reuse for irrigation purpose by saving the water and energy resources.

2. Testing Setup

2.1. Sampling

Following samples were used during the experiments.

Table 1. Details of samples

Serial Number	Sample description	Location	Coordinates
Sample No. 1	Reactive Blue Dye (RB) 19	Hattar industries, Khyber Pakhtunkhwa, Pakistan	33.9059° N, 72.8575° E
Sample No.2	Textile Industry wastewater	Hattar industries, Khyber Pakhtunkhwa, Pakistan	33.7662° N, 72.8262° E
Sample No.3	HCl, NaOH, Synthetic wastewater	Environmental chemistry lab, Environmental Engineering lab, UET, Taxila, Pakistan	33.7662° N, 72.8262° E

2.2. Instruments

Following instruments were used in the experiment.

2.2.1. pH Meter

A pH indicates acidity or alkalinity of the water-based solution by proper measuring their hydrogen ion activity in that specific solution. The difference in electric potential between a pH electrode and a reference electrode is measured using pH meter, and that's why pH meter is referred to as a "potentiometric pH meter". pH meter is shown in Figure 1.



Figure 1. pH meter

2.2.2. UV Absorbance Spectrophotometer

Absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region is referred by using Ultraviolet-visible spectrophotometry (UV/Vis). UV spectrophotometer is shown in Figure 2. In a visible range, the absorption or reflectance directly affects the associated color of the chemicals causing atoms and their molecules to undergo an electronic transition in the electromagnetic spectrum region. Unlike fluorescence spectroscopy, absorption spectroscopy deals with transitions from the ground state to the excited state. We had used this device for measuring the absorbance of effluent at a wavelength of 594 nm and 256 nm before degradation and then at the time interval of 30 minutes after starting the experiment.



Figure 2. UV absorbance spectrophotometer

2.2.3. DC power supply

A power supply converts one form of electrical energy to another by supplying electric energy to an electrical load and sometimes it is referred to as electric power converters. DC power supply is shown in Figure 3. 12-volt DC power supply was used for Direct Current used by an ultrasonic bath in with degradation of effluent was carried out. We had given the supply of 5 Volts for carrying out our experiment.



Figure 3. DC power supply

2.2.4. Suction Apparatus

Filtration using vacuum is one of the rapid filtration techniques used to isolate solids from the liquids. The air through a vacuum and the Buchner flask is drawn out by water flowing through the aspirator and by creating a pressure difference between the exterior and the interior of flasks respectively. Vacuum flask sucks the contents of the Buchner funnel while the filter at the bottom of the Buchner funnel separates the solids from the liquids. This machine has used at the end of degradation to filter the degraded matter from effluent. Suction apparatus is shown in Figure 4.



Figure 4. Suction apparatus

2.2.5. Ultrasonic bath

Ultrasonic cleaning is a process that uses ultrasound (usually from 20–400 kHz). We can use ultrasound with just water, but the using ultrasound with a solvent is suitable for cleaning the item. Also, the presence of soiling type enhances the effect. We had used this apparatus to place an electrolytic cell in it so that effluent placed in the electrolytic cell may degrade. We had set the frequency of 30 minutes so that after 30 minutes, bath get off automatically and analysis could be done every 30 minutes. Ultrasonic bath is shown in Figure 5.



Figure 5. Ultrasonic bath

2.2.6. Electrolytic Cell

An electrolytic cell made up of Perspex sheet was used to carry out the degradation experiments. Both the electrodes (working electrode and counter electrode) were placed in a vertical position and parallel to each other with an inter-electrode gap of 2.5cm in the cell. Stainless steel mesh and lead oxide were used as a counter electrode (cathode) and the working electrode (anode) with the dimensions of 14×15 cm² respectively. Electrolytic cell used in this research is shown in Figure 6.

2.2.7. Thermometer

It is a temperature sensor in which some physical change occurs with the temperature variation, and some means are used to convert this physical change into a numerical value (e.g. the visible scale that is marked on a mercury-in-glass thermometer). We have used a thermometer in our experiment frequently. After every 30 minutes, we had to measure the temperature of the solution in the electrolytic cell.

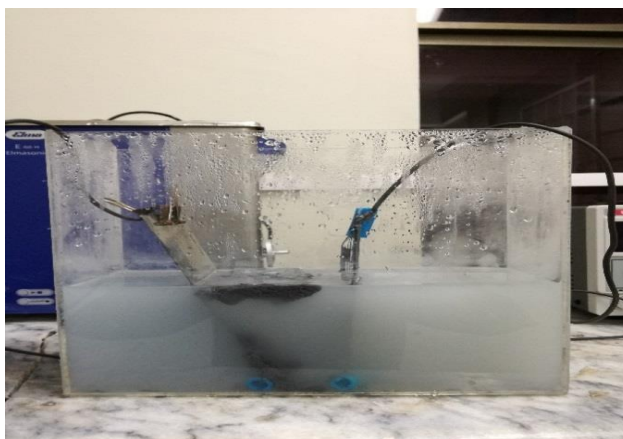


Figure 6. Electrolytic cell

3. Experimentation

An electrolytic cell made up of Perspex sheet was used to carry out the degradation experiments. Both the working electrode and counter electrode were positioned vertically and parallel to each other with an inter-electrode gap of 2.5 cm in the cell. Stainless steel mesh and lead oxide were used as a counter electrode (cathode) and the working electrode (anode) with the dimensions of 14, 15 cm² respectively. In all the experiments a current of 5 volts was provided by means of DC power supply. The designed electrolytic cell was immersed in the Digital Ultrasonic Bath as shown in Figure 8. Absorbance was measured at a wavelength of 594 nm and 256 nm with UV spectrophotometer. PH was measured with the help of pH meter and temperature with a thermometer after each 30 min time interval in all experiments. The test period for each experiment was 2.5 hours. Degradation experiments with different setups are explained below. A flow chart of the research methodology is shown in Figure 7.

- a) At 40°C (1 M HCl + 1 M NaOH)
- b) At 30°C (1 M HCl + 1 M NaOH)
- c) At 30°C (No acid, No Base)
- d) At 30°C (1 M HCl)
- e) At 30°C (1 M NaOH)

After the experimentation process, the collective data is used to plot a graph between time and dye removal percentage. The effluent of each 1 liter is taken in the electrolytic cell in which electrodes were adjusted with the help of a pipe. Five setups of experiments are done by keeping the temperature of ultrasonic bath 400°C and 300°C. Chemicals were added as 1 mL hydrochloric acid (HCl) and 1 mL sodium hydroxide (NaOH) to make a buffer solution and allowed it to degrade for 2.5 hours. Parameters like pH, temperature, and absorbance were measured at 30 minutes of the time interval.

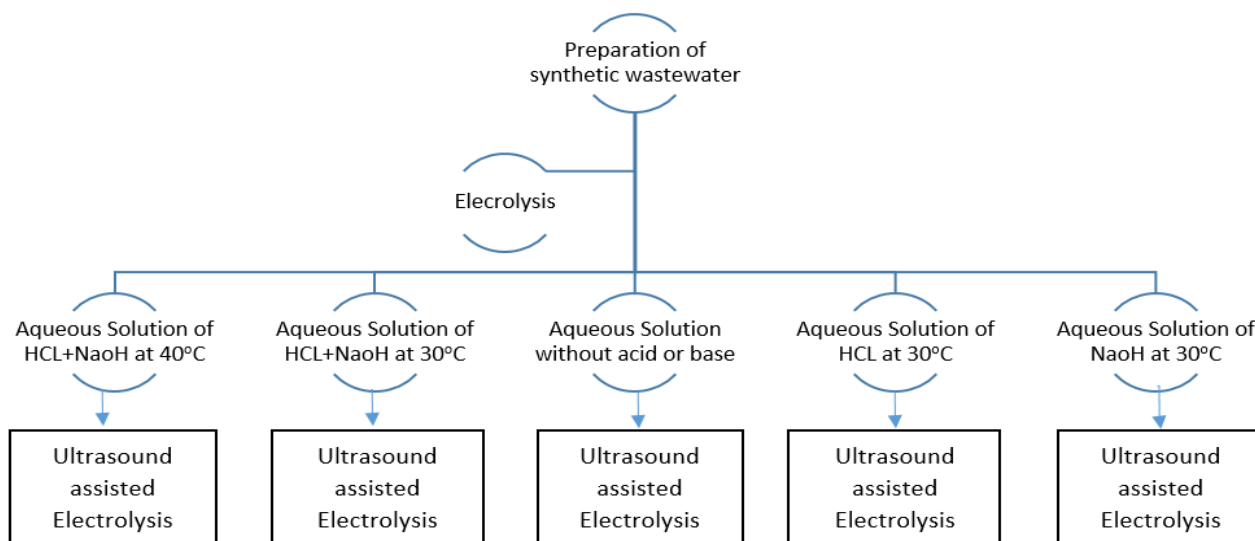


Figure 7. Flowchart of research methodology



Figure 8. Experimental setup

4. Results and Discussions

4.1. At 40 °C (1 M HCl + 1 M NaOH)

At 47 °C minimum absorbance occurs which represents maximum degradation of dye took place at 120 minutes. In the start of the experiment, absorbance was maximum and dye removal percentage was minimum. As temperature rises absorbance starts to decrease and reached the minimum value at 120 minutes. After this point absorbance starts to increase which shows that dye degradation process was stopped and the solution became secondary pollutant due to which absorbance increased after 120 minutes, ultimately shows the completion of reaction between the reactive dye and basic solution. Dye removal is 82 % at the point of minimum absorbance (i.e. 0.424 at 594 nm and 0.305 at 256 nm). Results are shown with the help of graphs in Figures 9 and 10.

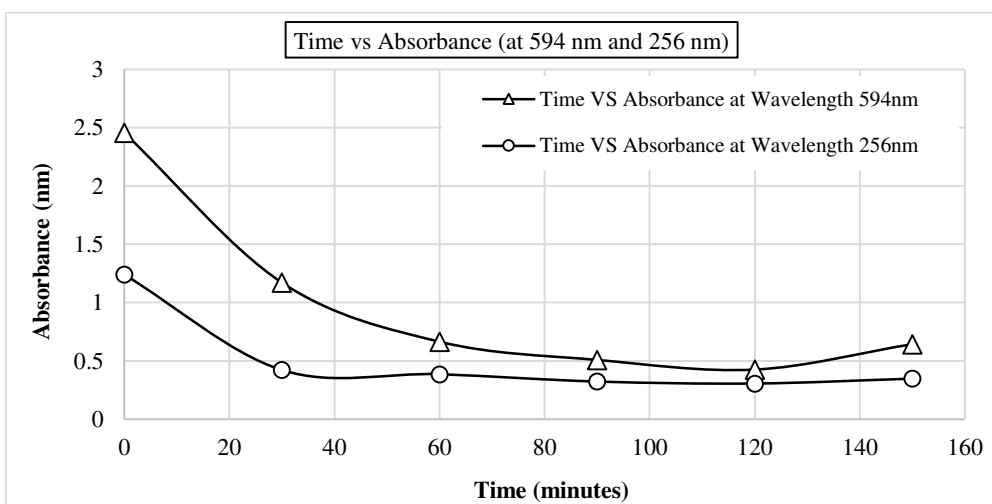


Figure 9. Time vs absorbance graph using both acidic and basic solution at 40 °C

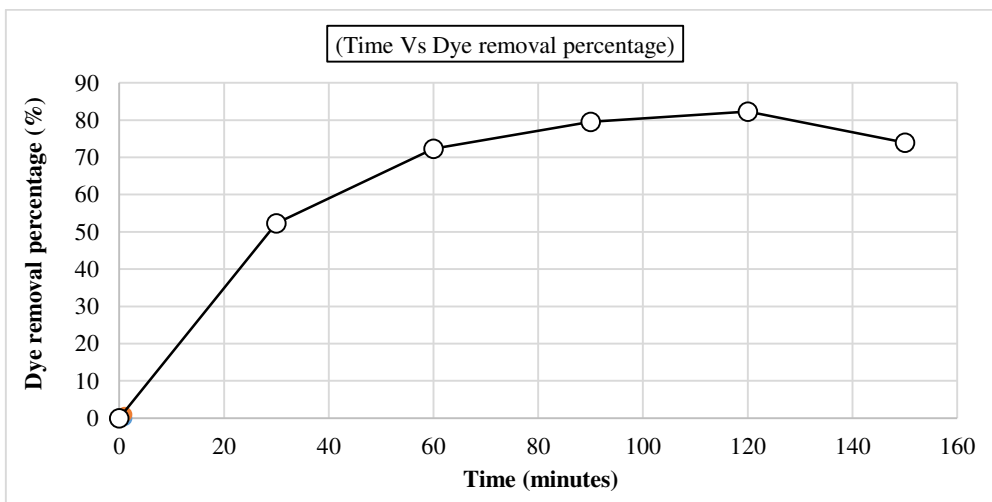


Figure 10. Time vs dye removal percentages using both acidic and basic solution at 40 °C

The absorbance vs time graph in Figure 9 shows two curves (at wavelength 594 nm and 256 nm). In the start of the experiment, the absorbance value was maximum showing minimum degradation. The absorbance curve shows decrease in values with respect to time due to an increase in the degradation of dyes. After a certain time, the absorbance value increases due to the presence of secondary pollutants which are resolved by using the filtration process. An increase in absorbance value causes the dye degradation process to stop.

4.2. At 30 °C (1 M HCl + 1 M NaOH)

The minimum absorbance was achieved at 90 mints and have absorbance 0.682. After that absorbance again starts to increase which means that the dye degradation process stops converting into secondary pollutants. The dye removal at 90 mint was maximum which 72.38% is. The comparison with 40 C shows that maximum dyes removal obtained earlier but the removal efficiency less. Like the previous experiment, the solution becomes secondary pollutant after maximum removal efficiency due to which absorbance increases after 90 minutes. Results are shown with the help of graphs in Figures 11 and 12.

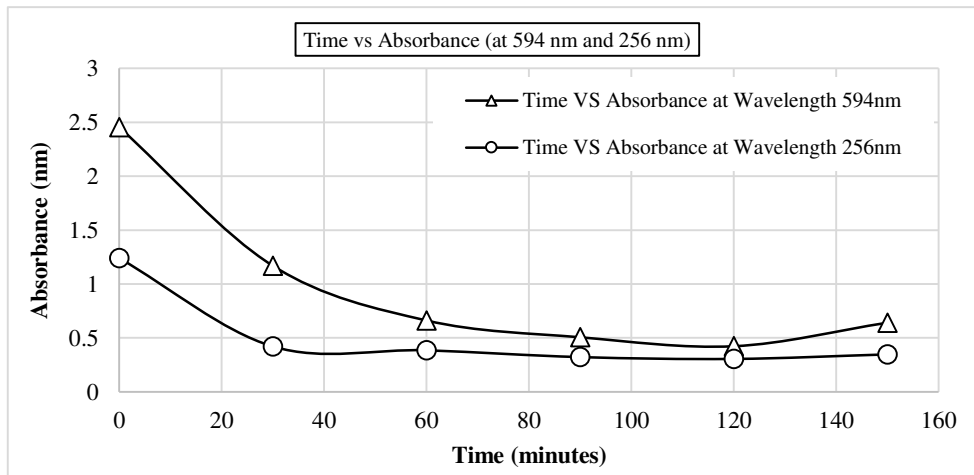


Figure 11. Time vs absorbance using both acidic and basic solution at 30 °C

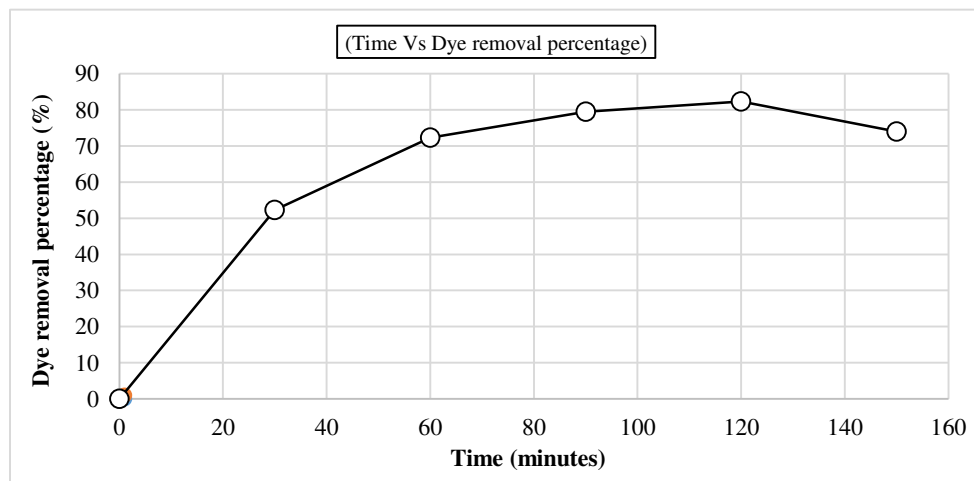


Figure 12. Time vs dye removal percentage using both acidic and basic solution at 30 °C

It is noted that the graphical trend of section 4.1 and section 4.2 is almost similar, as in both experiments the dye removal percentage starts to decrease after the formation of secondary pollutants, ultimately causing degradation process to stop.

4.3. At 30 °C (No acid, No Base)

In this experiment, the process of degradation of dye was very less. The data from the table 1 shows that pH and temperature increased continuously with the passage of time till the end of experiment which shows that without acid and base; the process of the degradation process is very low and also dye removal efficiency was very less. The maximum removal efficiency achieved was only 28.57 % at 150 minutes. The results are shown with the help of both Table 2 and Figure 13. It is noted that the dye degradation process shows a uniform behavior. Also, in this experiment, there was no formation of secondary pollutants and dye removal percentages values are very small as compared to other experiments in section 4.

4.4. At 30 °C (1 M HCl)

This experiment was done only by adding acid in the solution. PH decreases continuously as reaction proceeds but increases at the final interval attaining less absorbance and maximum dye removal with 57.12%. Although this experiment was considered time-consuming without adding both acid and base but was better than previous neutral reaction (discussed in case 4.3). The results are shown with the help of Table 3 and Figure 14.

Table 2. Dye removal percentages with varying temperature, pH, and time

Time (min)	Temperature	pH	Absorbance at (594 nm)	Absorbance at (256 nm)	Dye removal percentage
0	19	5.2	2.846	0.396	0
30	40	5.56	2.358	0.401	17.15
60	47	5.91	2.295	0.439	19.36
90	51	6.2	2.198	0.404	22.77
120	54	6.23	2.171	0.4	23.72
150	56	6.7	2.033	0.407	28.57

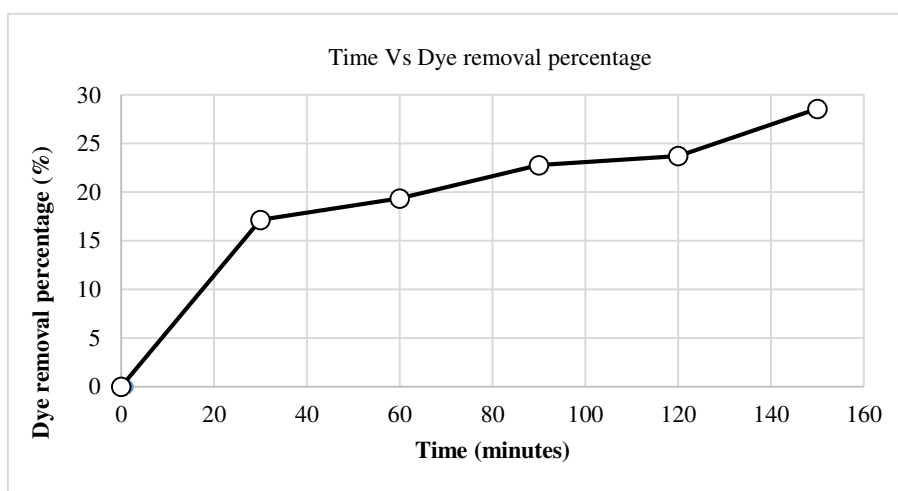


Figure 13. Time vs dye removal percentage using aqueous solution at 30 °C

Table 3. Dye removal percentages with varying temperature, pH, and time

Time (min)	Temperature	pH	Absorbance at (594 nm)	Absorbance at (256 nm)	Dye removal percentage
0	24	2.25	2.456	0.387	0
30	37	2.2	1.965	0.335	20
60	40	2.09	1.536	0.335	37.46
90	51	2.07	1.289	0.443	47.52
120	56	2.05	1.256	0.45	48.86
150	49	2.25	1.053	0.387	57.12

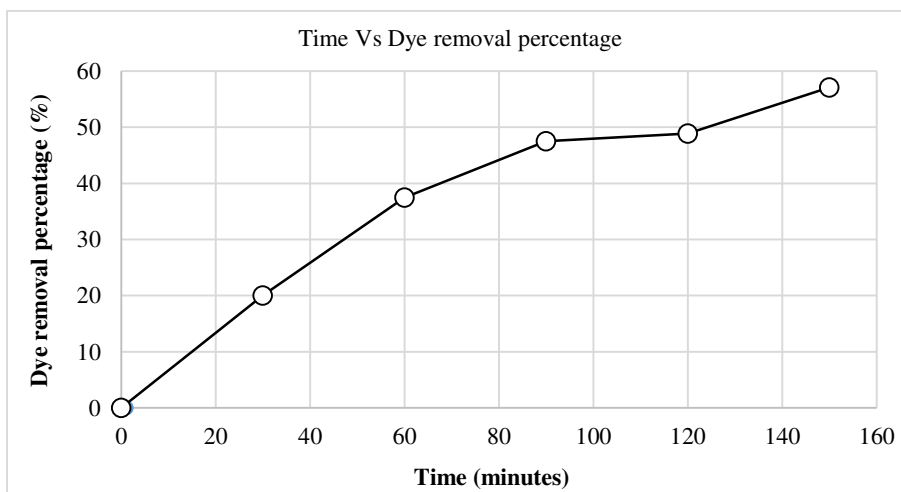


Figure 14. Time vs dye removal percentage using HCL at 30 °C

4.5. At 30 °C (1 M NaOH)

In this experiment pH decreased with the passage of time and temperature increased continually and at the same time absorbance decreased with a minimum value at 150 minutes which shows the point of maximum dye removal percentage. The results are shown with the help of both Table 4 and Figure 15.

Table 4. Dye removal percentages with varying temperature, pH, and time

Time (min)	Temperature (°C)	pH	Absorbance at (594 nm)	Absorbance at (256 nm)	Dye removal percentage
0	25	10.33	2.33	0.364	0
30	41	9.5	2.24	0.437	3.86
60	43	8.5	2.125	0.439	8.8
90	43	7.37	1.938	0.466	16.82
120	45	6.13	1.625	0.512	30.26
150	48	5.89	1.072	0.52	54

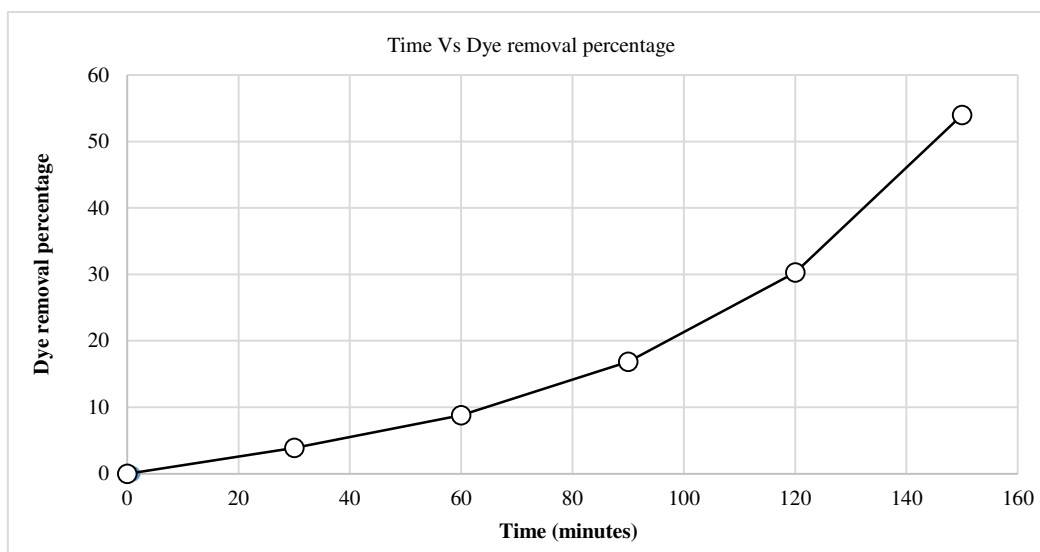


Figure 15. Time vs dye removal percentage using NaoH at 30°C

The graphical representation between time VS percentage dye removal is shown in Figure 16. It is a collective percentage dye removal graph obtained in all five experiments.

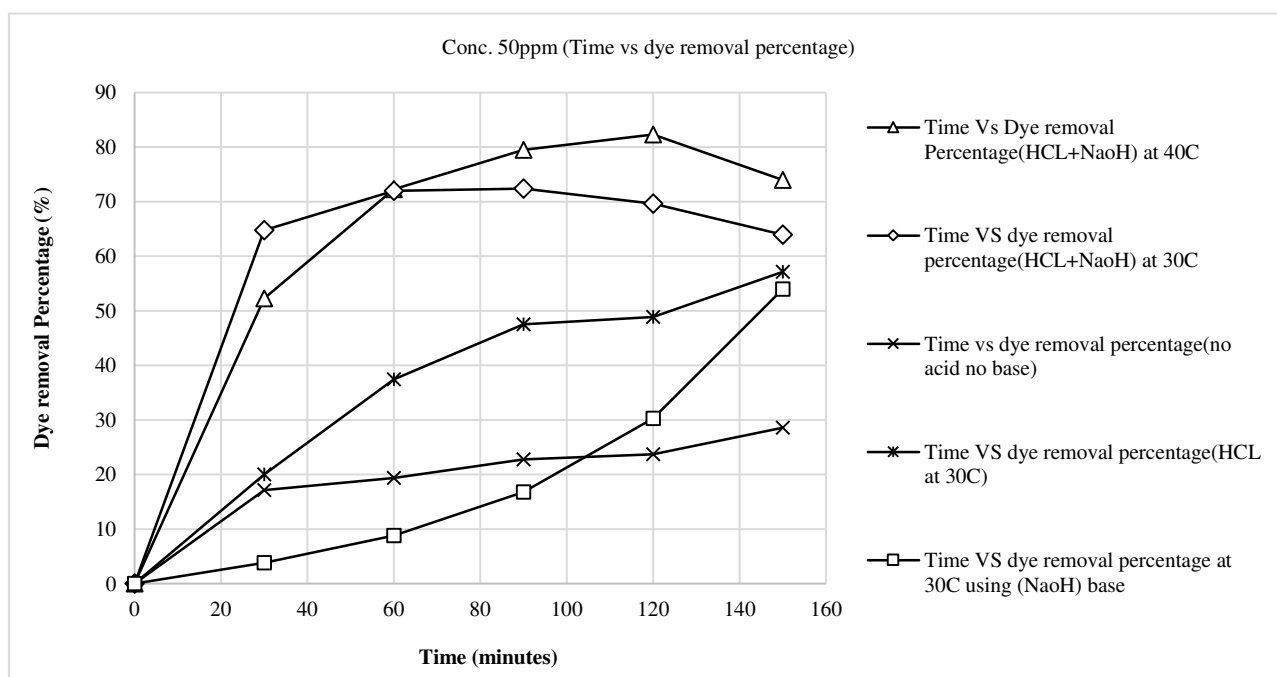


Figure 16. Comparison of dye removal percentages using different experiments

It is observed that the degradation of Rhodamine B dye using Hydrodynamic cavitation in the presence of intensifying additive is 99.9% with 55% TOC reduction [26] on another hand the degradation of Reactive Blue (RB) 19 dye (i.e. 82.3 %) at 120 minutes with pH value of 3.22 for the solution of 50ppm using the process of ultrasound-assisted electrochemical reactor and for the solution of 30ppm, we achieved maximum degradation of 85% at 40°C after 120 minutes using acid (HCl) and Base (NaOH) in equal amount.

4.6. Scanning Electronic Microscopy (SEM)

After performing experiments on reactive blue dyes, the aqueous solution was analyzed using scanning electron microscopy (SEM). The sample before treatment and after completing the experiments was analyzed and their results are shown in Figures 17 and 18. The results show the deposition of lead Oxide and stainless-steel mesh microelements with a waste aqueous solution. Their composition is shown in their respective spectrum images.

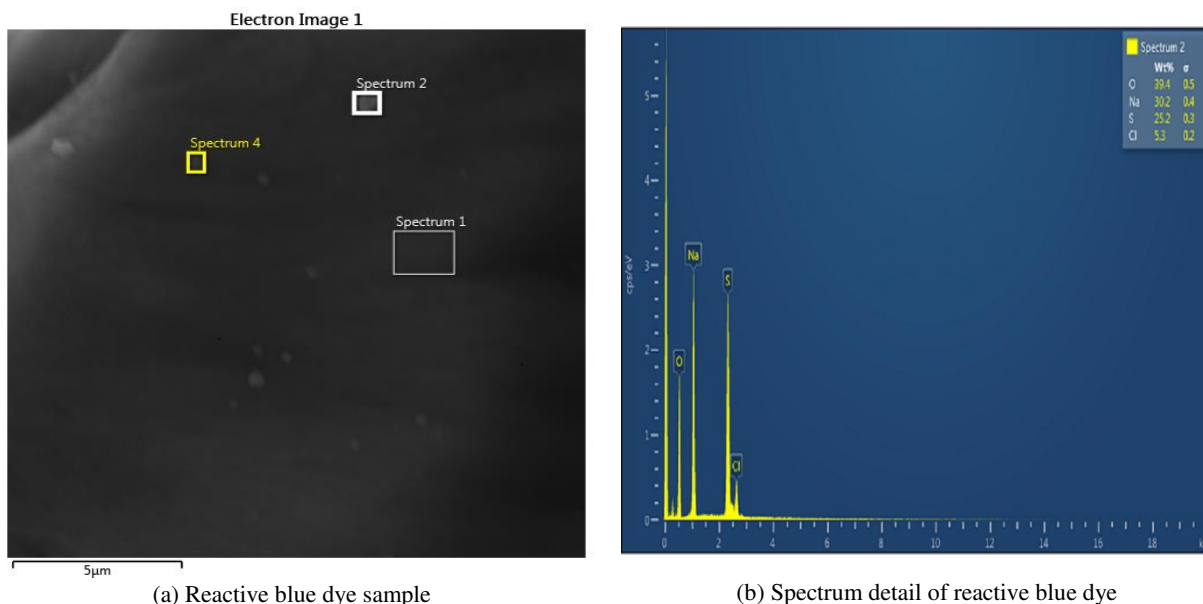


Figure 17. SEM of blue dye sample

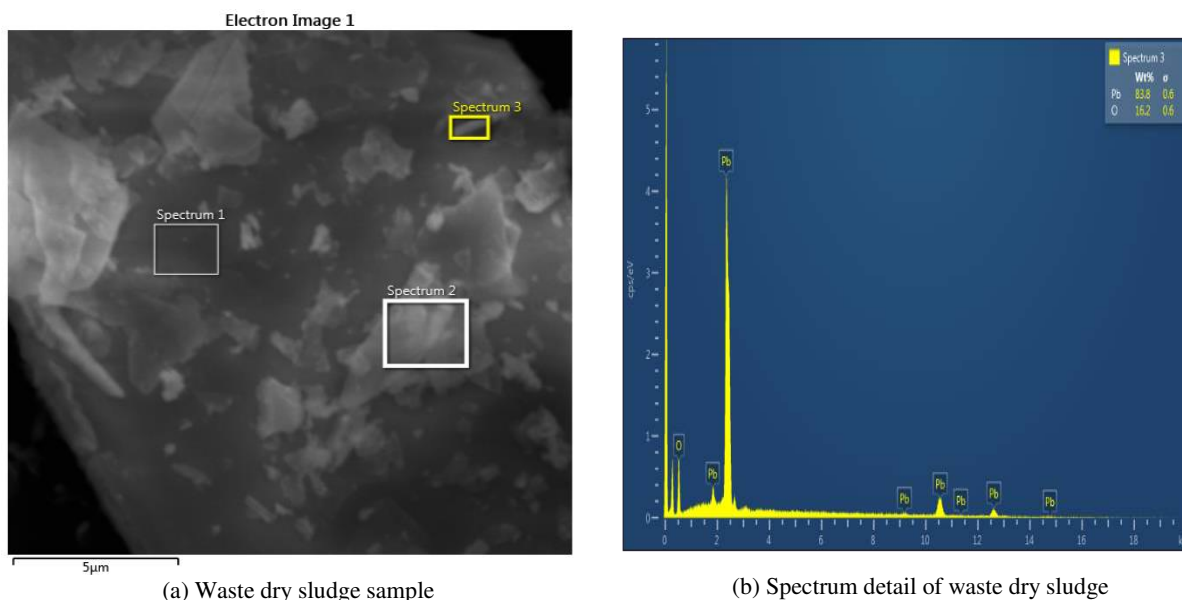


Figure 18. SEM of wastewater sludge

5. Conclusion

After performing a series of experiment on dye solution it was easy to conclude that the efficiency of degradation of dyes can be enhanced by changing the parameters like pH, temperature, and types of chemicals used. This work concludes that maximum degradation of dyes occurs at 40 °C using acid (HCl) and base (NaOH) in equal amount. Maximum degradation of dye (i.e. 82.3 %) at 120 minutes at pH of 3.22 for the solution of 50ppm and for the solution

of 30ppm, we achieved maximum degradation of 85% at 40°C after 120 minutes. After that time absorbance started to increase showing that degraded dye converting in secondary pollutant making the solution turbid. The combined graphical representation of time and dye removal percentage is a more appropriate way in comprehending the concept of dye removal using ultrasound assisted electrochemical process.

6. Conflicts of Interest

The authors declare no conflict of interest.

7. References

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