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OXIDATION OF TEXTILE DYE REACTIVE YELLOW 84 IN AQUEOUS SOLUTION IN ORDER TO REUSE TREATED WATER

OXIDACIÓN DEL COLORANTE TEXTIL AMARILLO REACTIVO 84 EN SOLUCIÓN ACUOSA PARA EL REUSO DEL AGUA TRATADA

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Abstract

Reactive Yellow 84 (RY84) is one of the most widely applied textile dyes because of its outstanding tinctorial properties. However, it has a very low affinity and it makes necessary to use large amounts of salt when RY84 is applied on cellulosic substrates. In this study, RY84 was treated by ozone in model solutions using a semi-batch bubbling reactor in order to recover water and salt for a further utilization in dyeing processes. Ozonation processes were carried out on solutions prepared at concentrations of dye as a typical dyehouse wastewater. In a first approach, it was analyzed the influence of initial dye concentration, initial temperature without further heating, and pH, on the ozonation. Additionally, with the scope to study the effect of chemical auxiliaries on ozonation process and on the result of reusing of treated water, model solutions simulating real effluents were prepared and ozonated. It was demonstrated that reusing of ozonated water is feasible, despite of the presence of ozonation byproducts and its accumulation during successive reusing cycles.

Keywords: dyes, textile, water, ozone, reuse.

Resumen

El Amarillo Reactivo 84 (RY84) es uno de los colorantes textiles de uso más extendido debido a sus sobresalientes propiedades tintóreas. Sin embargo, posee una muy baja afinidad, lo cual hace necesario usar grandes cantidades de sal cuando es aplicado sobre sustratos celulósicos. En este estudio, se sometió a tratamiento con ozono al RY84 en soluciones modelo, utilizando un reactor semicontinuo de burbujeo, con la finalidad de recuperar el agua y la sal contenida en las soluciones para darles un uso ulterior en procesos de tintura. Los procesos de ozonación se llevaron a cabo sobre soluciones preparadas con concentraciones de colorante dentro del rango de aquellas halladas en un efluente de una instalación de teñido típica. En una primera etapa se estudió la influencia de factores como concentración inicial de colorante, temperatura sin calentamiento adicional, y pH sobre la ozonación. Adicionalmente, se prepararon y ozonizaron soluciones simulando un efluente real, para analizar el efecto de los auxiliares de tintura sobre el proceso de ozonación y sobre el resultado de la reutilización del agua tratada en procesos de tintura. Se comprobó que es factible la reutilización del agua ozonada, a pesar de la presencia y acumulación de subproductos de ozonación durante sucesivos ciclos de reuso.

Palabras clave: colorantes, textil, agua, ozono, reuso.

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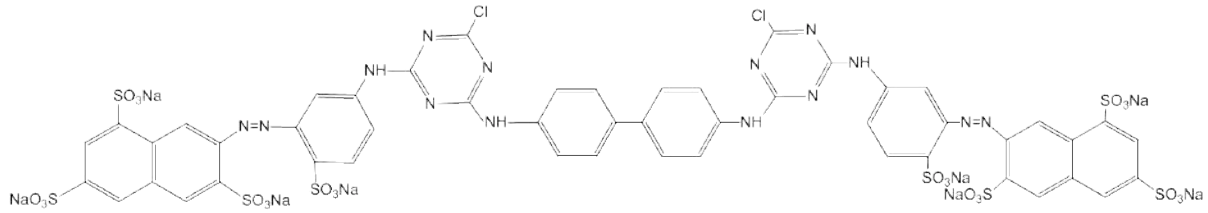

Figure 1: Chemical structure of Reactive Yellow 84.

1 Introduction

Worldwide, textile industry has an outstanding importance. But this industry is also a main environmental pollution source (Hauser, 2011). Textile industry releases to the environment wastewater containing dyes, salts and chemicals in high concentrations (Gomes *et al*., 2012). Textile wastewater contains also other pollutants such as metals, phenols, etc. Dyeing and printing operations are the main sources of those pollutants. In a typical dyeing process, up to 50 percent of applied dye is discarded in spent dyebath. Reactive dyes are one the most widely used tinctorial categories; but they have very low affinity, as such they require special attention (US-EPA, 1997). As a pretreatment, ozonation would be an option to improve color removal and biodegradation of reactive dyes contained in spent dyebaths. However, it depends on the ozonation time (Ulson De Souza *et al*., 2010). Ozone decolorizes wastewater breaking systems of double bounds that impart coloration to molecules; but complete degradation to carbon dioxide and water is difficult and expensive (Somensi *et al*., 2010; Tehrani-Bagha *et al*., 2010; Ruan *et al*., 2010; Sevimli *et al*., 2002; Mascolo *et al*., 2002).

It is desirable that textile industry reuse most of the water used in wet processes in order to minimize its environmental impact. Because of the high water consumption related to textile wet processes (Bes-Piá *et al*., 2010).

Reactive dyes are preferred in cellulosic fabrics dyeing because of they have outstanding properties of leveling, brightness and fastness. Particularly, they have outstanding wet fastness. However, the biological treatment of wastewater containing these compounds is very hard, because they have also good water solubility and poor biodegradability (Hauser, 2011). In addition, when reactive dyes are applied by batch dyeing, up to 50 percent of applied dyestuff is released to the effluent; it represents a great challenge in terms of minimizing color discharge (Hauser, 2011; Singh-Kalra *et al*., 2011).

This study reports decolorization and decomposition of the textile dye Reactive Yellow 84 (RY84) in model solution, under similar conditions to that found in a spent dyebath. The chemical structure of RY84 is showed in Figure 1.

Dyeing proofs using recycling water were carried out for 5 times, using a set of three direct dyes and three reactive dyes. Results of dyeing proofs were evaluated by AATCC Test Method 173-2005 widely applied in textile industry (AATCC, 2007).

There are several papers related to ozonation of aqueous model solutions of dyestuffs which scope is to study different aspects related to their kinetics and reaction mechanisms (Panda and Mathews, 2014; Guimaraes *et al*., 2012; Muthukumar *et al*., 2005). In the same way there are several studies on the use of ozone for the oxidation of recalcitrant organic compounds in combination with other advanced oxidation processes (Moctezuma *et al*., 2016).

2 Materials and methods

Reactive Yellow 84 has wide application in textile industry for high wet fastness dyeing of cotton fabrics. It is a monochlorotriazinyl compound and belongs to the high exhaustion class.

2.1 Influence of ozonation conditions on water treatment

The model solutions of RY84 were prepared simulating conditions found in dyehouse effluents. The sample of dyestuff used in this study was industrial grade. The experiments of ozonation were carried out in a semi-continuous reactor equipped with a porous diffuser at the bottom. The ozone-air mixture was produced from dry air in a discharge-type ozone generator (Peak Corporation) and it was introduced from the lower side through the diffuser in order to get fine bubbles. The concentration of ozone was 2 mg/liter and the flow of ozone-air mixture was 0.3 liter/min.

The decolorization and decomposition processes were monitored from absorbance using UV-Vis spectroscopy using a Genesys 10S UV-Vis spectrophotometer (Thermo Scientific), taking samples every 5-10 minutes. Measurements were made at 414 nm for the visible range and 226 for the UV range. Samples were taken through a teflon valve located on the lower side of the reactor. In a first approach, ozonation was carried out modifying process variables such as concentration dye, temperature and pH, taking in account that commonly exist variations of these parameters in real effluents.

The influence of initial concentration of dye was analyzed between 50 and 250 mg/L, meanwhile the effect of temperature and pH were analyzed ranging 20°C-80°C and 3-11, in each case.

2.2 *Influence of dyeing auxiliaries on ozonation process*

In other experiments series, model solutions were prepared adding separately salt and alkalis commonly used in reactive dyeing as chemical auxiliaries in large concentrations (Smith, 1986). The aim of these additions was to study their effect on ozonation, simulating spent dyebaths, initial dye concentration was maintained constant at 50 mg/liter and important parameters were varied. Concentration of salt was varied from 0 to 20 grams/liter (sodium sulphate, J.T. Baker ACS, 99.8%), concentration of anhydrous sodium carbonate (J.T. Baker ACS, 99.6%) was varied from 0 to 20 grams/liter and concentration of caustic soda (sodium hydroxide, J.T. Baker ACS, 98.9%) was varied from 0 to 2.5 grams/liter.

2.3 *Influence of ozonation byproducts on dyeing process*

The experiments related to recycling ozonated water for up to 5 times were conducted applying ozone treatment to RY84 solutions maintaining constant dye concentration (50 mg/liter), salt concentration (30 grams/liter) and carbonate concentration (10 grams/liter); concentrations commonly used in a real textile dyeing. Accumulation of byproducts was observed by UV-Vis spectroscopy.

The colorimetric results of dyeing using ozonated model solution were evaluated by reflectance spectroscopy on two series of cotton samples colorized with two sets of dyestuffs; one of them integrated for three reactive dyes and the other for three direct dyes, covering main SDC classifications and all of them azo compounds (AATCC, 2007): Reactive Black 5 (RB5), Reactive Red 141 (RR141), RY84, Direct Blue 80 (DB80), Direct Red 23 (DR23) and Direct Yellow 50 (DY50). The cotton samples were colorized without salt addition, which is necessary to reduce zeta potential between textile material and dye molecules (Aspland, 1997). Colorimetric differences were measured by a colorimeter ColorFlex EZ (Hunter Lab).

2.4 *Influence of ozonation on sodium concentration and FTIR spectra*

With the aim to verify that the sodium ion remains in the model solution during successive recycling, a solution of anhydrous sodium sulfate containing 50 mg/ liter of ion sodium was prepared and the presence of the sodium ion was detected using an atomic absorption spectrophotometer Perkin Elmer 3300.

Additionally, a 500 mg/L solution of RY84 was prepared and ozonated for up to 200 minutes. The decolorization and decomposition processes were monitored from the FTIR spectra variation in the 450 - 2000 cm^{-1} range using a Fourier transformation infrared spectrophotometer Serie 200 (Perkin-Elmer).

3 Results and discussion

According to published information, decolorization of textile dyes is accomplished by reaction of ozone with systems of conjugated double bonds responsible for the color (Perkins, 1999).

3.1 *Influence of ozonation conditions on water treatment*

Figure 2 shows the influence of the initial concentration of dye on the decolorization rate at each instant against initial coloration. The process becomes slower at higher concentrations because of the ozone concentration remains constant and its ratio to the dye is less.

Figure 3 illustrates the effect of initial temperature of exhausted dyebath on the decolorization rate of the dye solution. It is clear that high temperatures promote final degree of discoloration. However, it is interesting to note that during the first 15 minutes, the effect is negative. This most likely is because high temperatures favor decomposition of ozone.

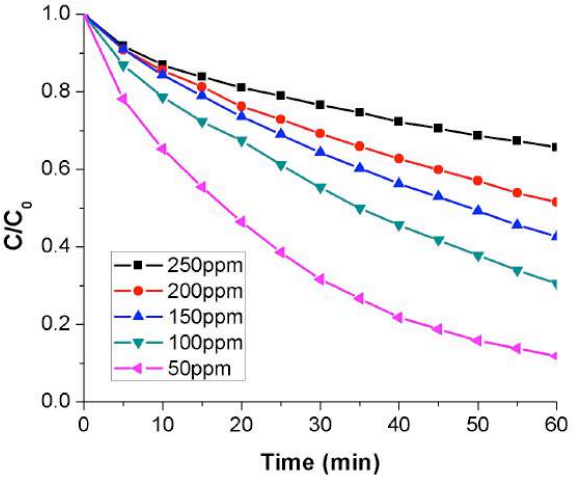

Fig. 2. Effect of concentration on ozonation of Reactive Yellow 84.

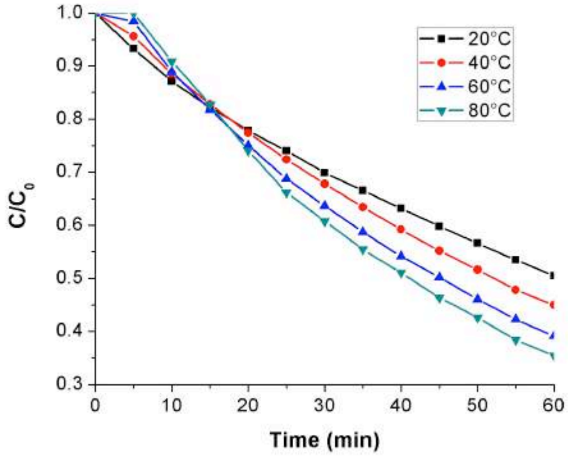

Fig. 3. Effect of temperature on ozonation of Reactive Yellow 84.

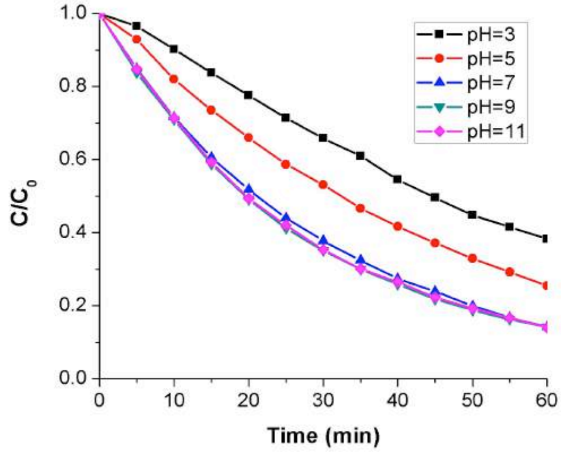

Fig. 4. Effect of pH on ozonation of Reactive Yellow 84.

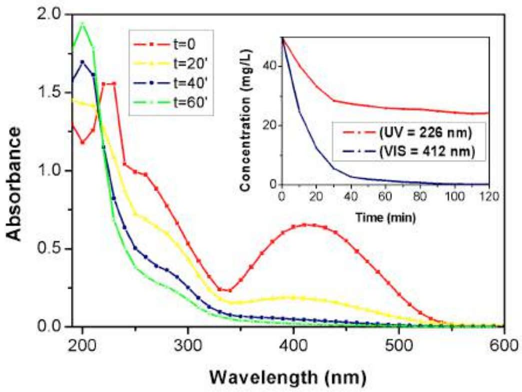

Fig. 5. UV-Vis spectra variation on RY84 ozonation processes. Inset: Decolorization and decomposition behavior of RY84 under ozonation.

Figure 4 shows the effect of pH on the rate of RY84 decolorization. It can be appreciated that the acid pH makes slower decolorization in a proportional way to the variation of pH. It is important to note that the pH in the acidic range was adjusted using sulfuric acid, in order to avoid using an ozonation byproduct, such as organic acids. The pH is one the most important and most studied variables in order to decide the use of a certain method of oxidation (López-Ojeda *et al*., 2015).

In the case of alkaline pH range, no significant variations are observed in the decolorization rate, even though according to the literature under alkaline pH the reaction mechanism is by radicals.

Under the conditions listed above, i.e. without adding large amounts of chemical aids, it takes an hour of ozonation to get the almost complete disappearance of color. Figure 5 presents the variation in the spectra of UV-Vis absorption for one hour ozonation on RY84. the chromophoric groups are destroyed within an hour of ozonation. However, the complete destruction of the compound takes longer. In fact, Figure 5 shows increasing values of absorbance around 210 nm in ozonation of RY84. These increases suggest formation of intermediates (Hassan and Hawkyard, 2002; Lin and Lai, 2000). In this sense, reduced absorption within the UV range is considered indicative of the destruction of intermediaries (Pines and Reckhow, 2003).

Figure 5 Inset A illustrates the variation of the absorbance maxima of RY84 in UV region at 226 nm and 412 nm for Vis region, transformed to concentrations of dye (mg/liter) by means of the corresponding calibration curves. It is clear from the figure that decolorization process takes place more quickly than total destruction of the compound.

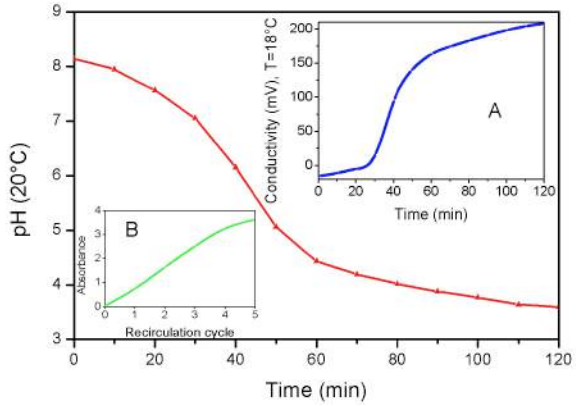

Fig. 6. pH variation on RY84 ozonation. Inset A: Conductivity variation on RY84 ozonation. Inset B: Accumulation of ozonation by-products along with RY84 ozonation-reuse cycles.

Complementing the above information, Figure 6 shows how the pH varies through the ozonation process, starting from the natural pH of a solution at 50 mg / liter concentration. This pH decreases from a slightly alkaline value to characteristic values of organic acids, which are known products of the ozonation of textile dyes (Perkins, 1999), so that reduction can be seen as a confirmation of the formation of such acids during RY84 degradation process.

The chemical identity of both intermediate and final products formed during ozonation of polyaromatic organic compounds depends on the chemical structure of the starting compound. It includes aromatic simple structure compounds, organic acids and ions such as sulfates, nitrates, etc. (Singh-Kalra *et al*., 2011; Perkins, 1999; Pines and Reckhow, 2003; Faria *et al*., 2009; Kuo and Huang, 1995; Hautaniemi *et al*., 1998). The conductivity increasing showed in Figure 6 Inset A is interpreted as confirmation of this formation of ionic species.

In this study formed intermediaries take up to 120 minutes completely destroyed. Figure 6 Inset B shows the estimated concentration variation of resulting ozonation by-products. The observed behavior, along with Figure 5 Inset A, confirms that more ozonation time is needed to remove those intermediaries.

3.2 *Influence of dyeing auxiliaries on ozonation process*

In textile dyeing processes large amounts of salt and alkali are added. In order to study the influence of these electrolytes, there were conducted two series of experiments by adding salt and alkali in similar amounts to those used in a common dyeing process.

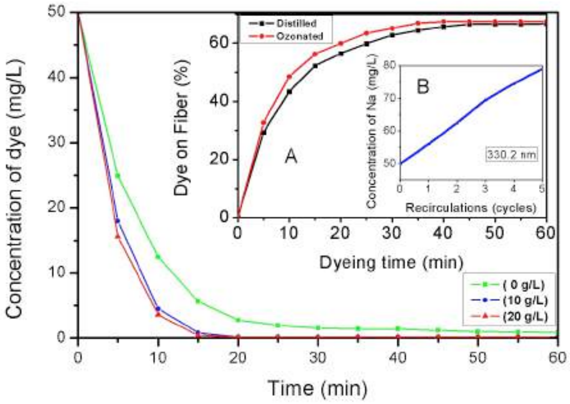

Fig. 7. Influence of sodium sulfate addition on RR141 ozonation process. Inset A: Influence of RY84 ozonation byproducts on kinetics of dyeing with DB80. Inset B: Sodium concentration through ozonation cycles.

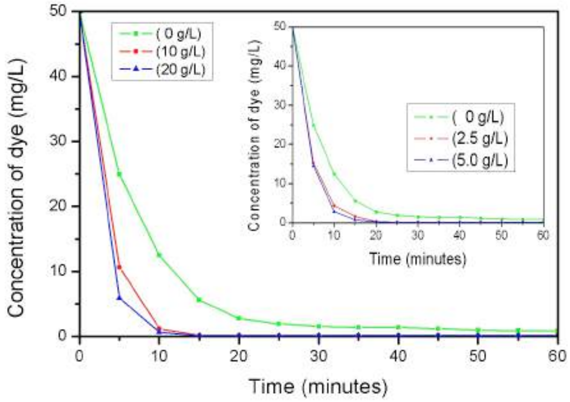

Fig. 8. Influence of sodium carbonate on RY84 ozonation process. Inset: Influence of sodium hydroxide on RY84 ozonation process.

Figure 7 shows the influence of the addition of sodium sulfate on RY84 ozonation. From the graph it is evident that the addition of 10 grams / liter has a marked influence on the rate of solution discoloration and major additions to this concentration have virtually no effect. It is important to highlight that salt concentrations used in dyeing with reactive dyes are much greater than 10 grams / liter.

Figure 8 shows the influence of the addition of sodium carbonate on the ozonation of RY84 and Figure 8 Inset shows the same for the addition of caustic soda. The presence of electrolyte has an important effect and this is independent of the concentration between studied limits.

The literature indicates that when the ozonation is carried out under acidic or neutral pH, is performed under direct mechanism, whereas when performed under alkaline pH is done under radical mechanism. However, such reaction mechanisms are not enough to support the behavior observed in Figures 7 and 8 on electrolytes addition (Hauser, 2011). Sodium carbonate shows greater influence on decolorization rate, despite of it is considered as a radical scavenger. It is important to mention here that neither expected effect was verified by analyzing the influence of pH directly. In Figure 4 it shows that this influence is minimal.

On the other hand, decolorization processes for RY84 are completed for 10-15 min. of ozonation in the presence of salt or alkali as we can see in Figures 7 and 8.

3.3 *Influence of ozonation byproducts on dyeing process*

In order to quantify the effect of the presence of ozonation byproducts on textile dyeing with reactive and direct dyes, salt and alkali were used along with the corresponding dye to prepare the model solutions simulating a spent hydrolyzed dyebath. Then, solutions were ozonated for 10 min. The model dyebaths containing salt and ozonation byproducts, were prepared. They were used for dyeing cotton samples prepared with a set of three reactive dyes and direct dyes three of the most common classes: Reactive Red 141 (RR141), Reactive Yellow 84 (RY84), Reactive Black 5 (RB5) Direct Red 23 (DR 23), Direct Yellow 50 (DY50) and Direct Blue 80 (DB80). Model spent dyebaths were prepared and recycled in cotton dyeing up to 5 times for each case using an textile standard method for evaluation of differences of color AATCC Test Method 173-2005 (AATCC, 2007). Figure 9 shows the results as color coefficient variation (ΔE).

From Figure 6 Inset B we can conclude that there is accumulation of ozonation byproducts during subsequent cycles of reuse of treated water. However, from Figure 9, we conclude that the influence of ozonation byproducts on the results of dyeing depends on the nature of the dyestuff used for dyeing cotton. It is higher for the case of reactive dyes and not as important in the case of direct dyes.

In Figure 7 Inset A and Figure 10 is shown the dyeing behavior of the three studied direct dyes during the first cycle of dyeing on cotton using ozonated water as compared with a dyeing carried out with distilled water as reference. As can be seen, the byproducts modify the dyeing kinetics, but not alter the concentrations at equilibrium, i.e. the percentage of exhaustion is the same for practical purposes.

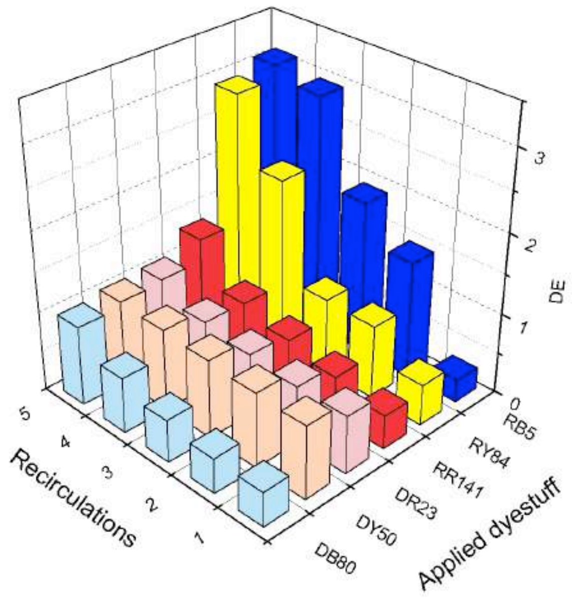

Fig. 9. Influence of RY84 ozonation byproducts on dyeing with direct and reactive dyes.

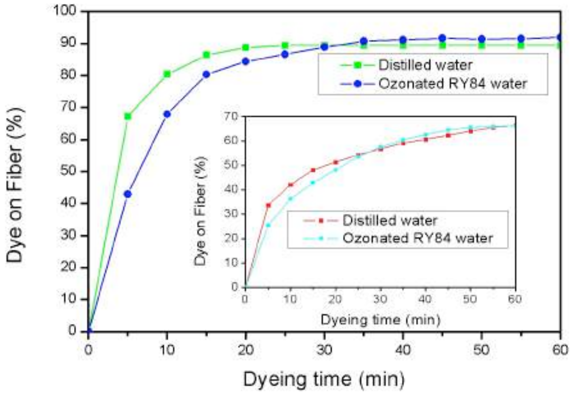

Fig. 10. Influence of RY84 ozonation byproducts on kinetics of dyeing with DR23. Inset: Influence of RY84 ozonation byproducts on kinetics of dyeing with DY50.

On the other hand, when the reuse of spent dyebath for dyeing cotton is made using reactive dyes, there are noticeable differences in the obtained results of quality coloration. From the second cycle of recirculation the quality of coloration is out of the limit specified for the used test method.

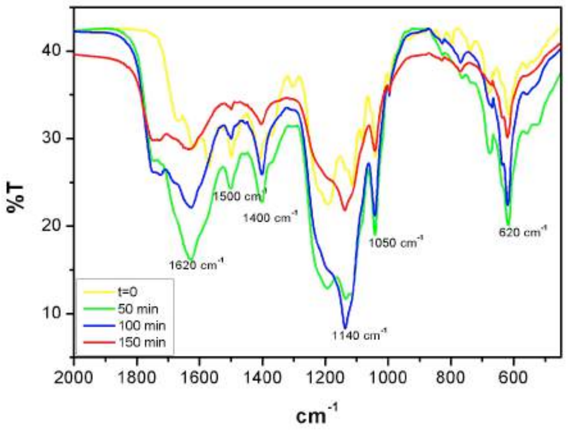

Fig. 11. FTIR spectra variation on the RY84 ozonation.

3.4 *Influence of ozonation on sodium concentration and FTIR spectra*

Figure 7 Inset B shows the analysis of the presence of sodium ion during subsequent cycles of ozonation and reuse by atomic absorption spectroscopy. A slight increase is observed in the sodium concentration even though no salt additions were made to the dyeing process. This is because the dyes have sodium in their structures and also contain varying amounts of salt due to its manufacturing process. Dyes are extracted from the reaction mixture by a method known as "salting out". In addition, manufacturers add salt to the final product to standardize.

FTIR spectrum variation for 200 minutes of ozonation on 500 mg/liter solution of RY84 in the region between 600-2000 cm^{-1}, RY84 presents several important absorption bands (Figure 11). One of them, at 620 cm^{-1}, corresponds to SO_3^{2-} bending; along with another at 1140 cm^{-1}, from stretching of S-O bond, confirms desulfonation of RY84 during ozonation process. In the same way, one band at 1050 cm^{-1} corresponding to C-O bond stretching and another band at 1400 cm^{-1} from COO^- ion stretching confirms the formation of organic acids. Additional bands situated at 1500 cm^{-1} and 1620 cm^{-1}, correspond to NO_2 formation and secondary amines, respectively.

Conclusions

The initial concentration of dye on the decolorization rate has a marked negative influence when the ozone concentration remains constant at a low range as used in this study. It is important to note that in the case of dyebaths from dyeing intense colors.

The initial temperature of ozonation also has a strong positive influence on the rate of discoloration. This is important if we consider that the exhausted dyebaths have a high temperature. However, during the opening minutes the influence is negative because increase the decomposition of ozone to molecular oxygen.

The pH in the acidic range shows a negative influence on the decolorization rate. However, this is not the case for exhausted dyebaths containing reactive dyes, which always have high pH because of large amounts of alkali used during their application. In the case of alkaline pH, the observed influence is not as marked as one would expect from the change of reaction mechanism.

When chemical auxiliaries are present within the minimum range used in a dyeing process, the ozonation of RY84 in aqueous solutions for relatively short treatment times, 10-15 min, is enough for enabling the exhausted dyebath for reuse. The effect of chemical auxiliaries on the reaction rate does not depend on the reaction mechanism.

The accumulation of ozonation byproducts during cycles of reuse of water has a negative influence on the results obtained by using this water in dyeing processes. However, this influence depends on the type of dye that is used to reuse water, being very marked when dyeing with reactive dyes and much less when dyeing with direct dyes. Additional variations existing within a single dye class must be considered.

The ozonation byproducts of RY84 affect dyeing kinetics when dyeing is made using direct dyes, but not alter the concentrations at equilibrium. Therefore, ozonation byproducts do not affect results of cotton dyeing with direct dyes up to for 5 times.

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