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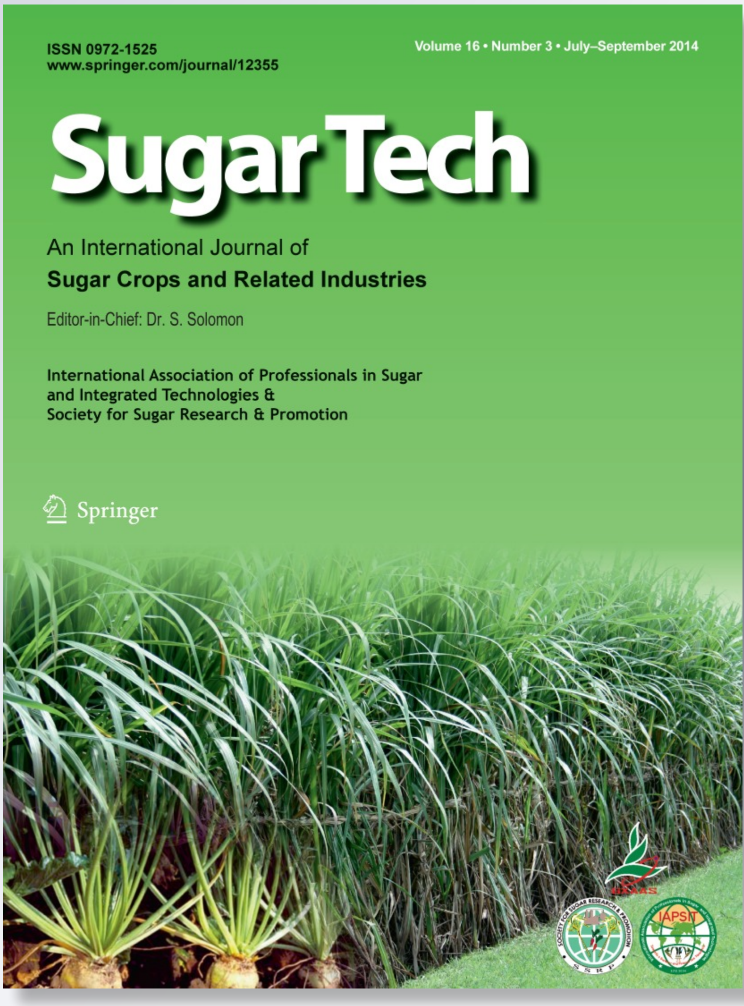

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RESEARCH ARTICLE

Performance Investigation of Electrochemical Treatment Process on Wastewater of Applicable Decolorization Resins in Sugar Factories

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Abstract The industrial waste water is considered as a serious threat to environment. Industries with high pollution load of chemical oxygen demand (COD) and biological oxygen demand such as sugar industries has a major role in environmental pollution. The aim of this study was to investigate the treatment possibility of this wastewater by using the electrochemical method. For this purpose quality parameters such as COD, color, turbidity and total dissolved solids (TDS) were investigated. The decrease of color, turbidity COD and TDS from effluent has been investigated at different voltages (10.8, 16.3 and 27.9 V) and various electrolysis times (10, 25 and 40 min) by using different electrodes (Al, Fe) at 4 cm middle distance. Experimental results demonstrated that the electrochemical process by using electrocoagulation and flocculation mechanisms can decrease color, turbidity and COD to 90.8, 98.9 and 50.5 %, respectively. The electrochemical process increased in pH with no considerable effect on the TDS of the effluent by water reclamation on the cathode side. Results showed that electrochemical process without additives could eliminate greatly various containments of wastewater. Therefore, an appropriate method for treating of wastewater can be designed and implemented.

Keywords Electrochemical process · Wastewater treatment · Decolorization resins · Sugar factories

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Introduction

By developing technology and increasing diversity of industries, the industrial waste water is considered as a serious threat to environment. Industries with high pollution load of chemical oxygen demand (COD) and biological oxygen demand (BOD) has a major role in environmental pollution. High COD and BOD will cause problems in sewage treatment and necessitates distinguishing between sewage treatments methods. A resin substrate can be used to separate unwanted ions from a solution which are passed through it or accumulate a valuable soluble mineral in water in other stages of recycling (Anand et al. 2001). Resins have different applications according to their types and performances such as water purification (Eccles and Greenwood 1992), juices and sugar extracts decolorization, (Application information 1997) purifying mixture of amino acids, and de-elementalization (Moreira and Ferreira 2005) and de-bitterness and repelling heavy metals from wastewater (Vera et al. 2003). The ion-exchange resins are used in sugar factories to decolorize and put off the ash of raw sugar after its solving in dissolvers to produce sugar head. An ion exchange resin, usually after 12–48 h use, depending on its application, lose its effectiveness and should be reduced. The reduction of resins use can be done by different solutions such as sodium hydroxide solution (5 %), sodium sianid (Bolto and Pawlowski 1983), potassium hydroxide solution (5 %) and potassium sianid (Gomes et al. 2001). In sugar factories the reclamation of decolorization resins is done by alkaline solution of 10–12 % sodium chloride (with 2 % concentration) and washing is done by a kind of effluent with a great amount of colorful mineral organic matters so it contains high amount of COD (Fig. 1). Industrial wastewaters such as effluents from decolorized reins contain very high levels of salt. Biological methods for wastewater treatment are not very satisfactory because of inhibition of salt in microbial growth. But saline wastewater due to the existence of anions and cations has high electrical conductivity and therefore electrochemical method is suitable for its treatment (Dalvand et al. 2009).

Over recent years some progressive ways which are used in industrial wastewater treatment and effluents containing organic pollutants, are electrochemical methods. One of methods which have been developed to overcome the drawbacks of conventional water and wastewater treatment technologies was electrocoagulation (EC). EC is an electrochemical process which uses direct current electricity in order to remove contaminant from solutions (Jiantuan et al. 2004). EC is a potentially effective method for treating different kinds of wastewater with high removal efficiency (Chen 2004). EC process provides a simple, reliable and cost-effective method for the treatment of wastewater without any demand to additional chemicals, and thus as a secondary pollution. It also reduces the amount of sludge which needs to be disposed. EC technique uses a direct current source between metal electrodes immersed in polluted water (Mollah et al. 2004). Electrochemical process is an alternative technology for wastewater treatment and valuable chemical recycling in wastes. In comparison with conventional process of coagulation and flocculation, they require fewer facilities. Some of advantage in this method included: it need low treatment time; it does not use any chemicals, it does not produce secondary pollution and less amount of sludge is produced (Dalvand et al. 2009).

According to the general principles of the method, the direct electrical current enters into liquid through anode and cathode. The main mechanisms in electrochemical process are electrocoagulation, electroflotation and electrooxidation (Sheng and Peng 1994). Bejankiwar et al. (2003) studied about the separation of color and organic compounds from waste of coffee industries by electrochemical methods and showed that steel anodes are very useful in color and COD removing (Bejankiwar et al. 2003). The most important parameters affecting the performance of electrochemical process are: potential differences between anodes and cathodes electrodes, electrolysis time and pH of treated solution (Aleboyeh et al. 2008).

The decolorized effluent by reduction resins in sugar factories due to reduction of resins by sodium chloride alkaline is salty and biological methods for this kind of sewage are not effective. On the other hand, effluent discharging into environment, in addition to high environmental pollution due to its high amount of COD, wastes a great amount of water which is used while reclaiming decolorization resins. Therefore this study aimed to investigate possibility of wastewater treatment derived from the reduction of decolorization resins in sugar factories by using electrochemical process and iron and aluminum electrodes.

Materials and Methods

This research is an experimental laboratory-scale study which is done on the effluent of decolorization resins of Khorasan Sugar Factory in a closed (Batch) system.

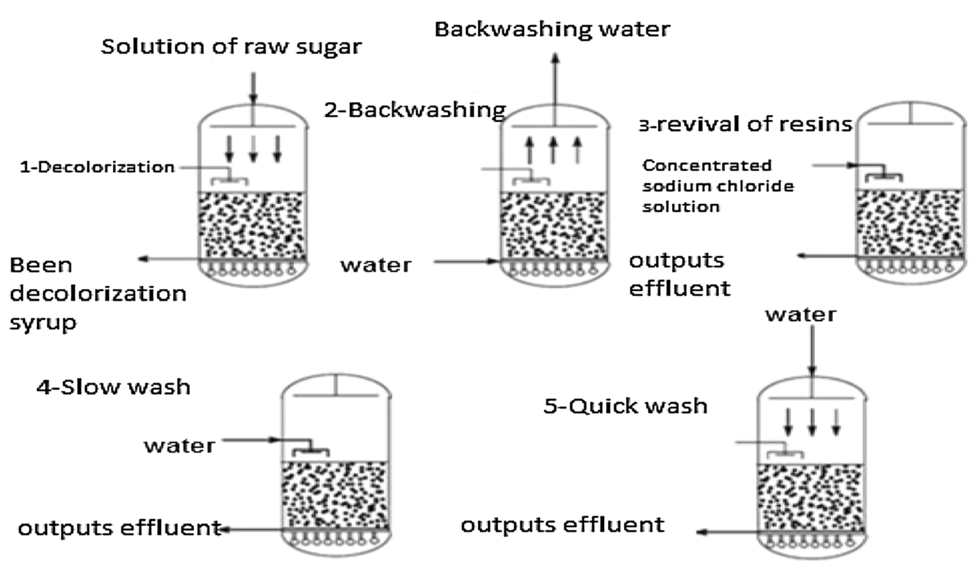

Fig. 1 Decolorizing of raw sugar and effluent formation

The laboratory apparatus which used for electrochemical treatment consist of an iron electrode and an aluminum electrode with 4 cm space between them. Each electrode had 5 parts and their effective area in the sample was 360 cm^2. The distance between parts on each electrode was fixed and 4 cm. The apparatus works with direct electrical current and voltage was changeable.

Color was measured by spectrophotometric method (Cambridge, England) at 420 nm wave length according to ICUMSA instruction in 2000, turbidity which is the symbol of suspended solid (SS) in the affluent was measured by turbidity meter (Model 6035, turbidimeter Jenway, Keison International Ltd, England) according to ICUMSA method, pH was measured by pH meter (Model 9812-5, HANNA instruments, USA), EC was measured by Conductivity meter (Model 380BA, UK) (ICUMSA 2000), COD by peptic device DRB 200, HACH, and spectrometer (DR 5000 HACH) at 620 nm wave length and Vials 0–15,000 under the American Standard method (AOAC 2002). After purification process, the filter papers (Whatman, 42, 15 cm) were used to filter the samples. The effluent sample was the sewage after discoloring by decolorizing resins which were sampled in Khorasan Sweet Sugar Factory. The characteristics have been given in Table 1 (Aleboyeh et al. 2008).

Procedure

In each test, at first, 10 l of decolorization resins effluents is poured into a plastic container with 20 l capacity and then electrochemical apparatus electrodes are fixed in the container and are connected to a direct current electricity supply by unipolar wires.

To establish a uniform mixture within the container and prevent deposition of decomposed organic materials on the electrode surface, stirring was done by a wooden rod and the place of iron and aluminum electrodes were changed during the process.

In each set up a constant voltage (27.9, 16. 3, 10.8 V) was established between electrodes and at different interval times (40, 25, 10 min), sampling was done from the plastic container.

Table 1 The properties of decolorizing resins effluent for electrochemical processes

Properties	Unit	Value
Color	ICUMSA (ICU)	9,780
COD	ppm	1,538
Turbidity	NTU	112.3
TDS	mg/li	7,450
Brix	%	2.6
EC	ms/cm	15.7
pH	–	7.2

The pH of samples was constant and equal to 7.2. Some researchers reported neutral pH as favorable condition for electrochemical process (Aleboyeh et al. 2008) and also the pH of effluent derived from decolorizatation resins columns was neutral, so the process was performed in neutral pH. Then samples were filtrated by paper filters and desired parameters were measured. After each test period, the electrodes were placed in dilute hydrochloric acid and then washed by plastic brush. Each experiment was done twice and the average was considered.

Electrochemical Process Mechanism

When wastewater is treated by direct current electricity, a number of physicochemical processes happen in this process including oxidation, the dissolving of metal anodes, colloidal particles coagulation, and electrophoresis, and particles electro flotation, particles deposition of cathode metal ions, decomposition of salts, acids and alkali concentration and desalination of water (Yang and McGarrahan 2005).

Three main processes that occur during the electrochemical reaction included a) electrolytic reactions at the electrodes surface, b) formation of coagulants in aqueous phase, c) absorption of soluble or colloidal pollutants on coagulants and their remove by sedimentation or flotation was done according to Aleboyeh et al. (2008). The main mechanism for removing of colorful compounds and pollutants from solutions containing these compounds is clotting or coagulation process. In high cathode potentials, due to restoration of water and oxygen, hydroxyl ions can be formed:

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (1)$$

$$2H_2O + O_2 + 4e^- \rightarrow 4OH^- \quad (2)$$

When iron sheets are used as electrodes in reaction, Fe^{+2} and Fe^{+3} are formed in anode.

Anode reaction:

$$Fe \rightarrow Fe^{2+} + 2e^- \quad (3)$$

$$Fe \rightarrow Fe^{3+} + 3e^- \quad (4)$$

Cathode reaction:

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (5)$$

When aluminum sheets are used as electrodes in reaction, $A1^{3+}$ ions are formed in anode.

Anode reaction:

$$AL \rightarrow AL^{3+} + 3e^- \quad (6)$$

Cathode reaction:

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (7)$$

During the process due to production of hydroxyl ions in cathode and egression of hydrogen as a gas, increasing of pH is expected. As a result in high pH, gelatinous suspension of $A1(OH)_3$, $Fe(OH)_3$, $Fe\ (OH)_2$ are formed and remain in solution (Yang and McGarrahan 2005). Gelatinous suspension through surface complexion and electro static absorption mechanisms trapped the color compounds and other pollutants of thre solution and during sequestration of ions and aluminum hydroxide heavy clots, pollutants are removed by surface physical absorption mechanism (Phalakornkule et al. 2009).

Result and Discussion

Evaluation of the Electrolysis Time and Voltage Variation on the Color Removal Efficiency in the Electrochemical Treatment Process

Among electrochemical treatments, electrochemical oxidation and electrochemical coagulation are more effective than others in decolorizing effluents. Although some electrochemical processes involve the direct oxidation of pollutants at anode surfaces, most of the others involve production of active species to react with target pollutants. The rate of electrochemical oxidation depends on the type of electrolyte, electrolyte concentration, reaction temperature, and current density (Lorimer et al. 2001). Another promising electrochemical process is by coagulation (Daneshvar et al. 2004). Aluminum and iron sheets have been used as consumable anodes to generate coagulants that adsorb and remove organic dyes. An aluminum coagulant removes colorant by simple adsorption without involving chemical reactions while an iron-based coagulant provides ferrous ions (Fe^{2+}) to further degrade the dyes (Wilcock et al. 1992). Likewise the iron process seems more effective for removal of reactive dyes while the aluminum process is superior for removal of disperse dyes (Do et al. 1994).

The effect of electrochemical process on effluent color changes in different potentials and electrolysis time is shown in Fig. 2. As it can be seen at lower times (10 min) with increasing voltage from 10.8 to 27.9 V, the reduction of color percent was increased, but with passing times the increasing of voltage had an negative impact on samples color reduction. The reason of higher yield of colorful compound removing with increasing voltage probably was because of high potential, the concentration of iron and aluminum hydroxides which generated in electro chemical cell increased and as a result more hydroxide deposits and clots were produced to remove the pollutant. The reverse is also true, viz., at lower voltages (10.8–16.3 V) by increasing reaction time, the efficiency of electrochemical process for coagulating and removing organic compounds which caused color has increased, so the maximum color reduction percentage was 90.8 % in 10.8 V for 40 min. In higher voltages (27.9 V) increasing time from 10 to 40 min has a negative impact on process efficiency. Results that reported by Aleboyeh et al. (2008) also confirmed the point that increasing in current density and electrolysis time up to an optimal level, cause reduction in dyed materials of wastewater. Rising of these factors to more than an optimal level has no effect on color decreasing and can only increase energy consumption (Aleboyeh et al. 2008). In addition, the presence of salt is one of the factors that affect the wastewater treatment through the electrochemical process. With increasing of added salt, the electrical conductivity solution becomes higher, which result to give more electrical energy to existent organic matters in sewage and highly increase the electrochemical decomposition. The electrolysis of sodium chloride produces free chloride which is highly oxidizing and helps to break down the organic compounds (Phalakornkule et al. 2009). Sodium chloride in solution significantly reduces the energy consumption, and accelerates the clot formation by preventing iron and aluminum coating of solution in electrochemical system (Yang and McGarrahan 2005).

Derived effluent from decolorization resin columns also contains a large amount of salt so its electrical conductivity is high and at low voltages by less energy consumption a desirable result can be achieved. The equation to calculate the color removal efficiency in this study is as follows (Aleboyeh et al. 2008):

$$\text{Color removal efficiency} = \frac{C_0 - C}{C_0} \times 100 \qquad (8)$$

C_0 and C are the wastewater color concentration (based on ICUMSA unit) before and after of electrochemical treatment, respectively.

Evaluation of the Effect of Electrolysis Time and Voltage Changes on Electrochemical Process Efficiency in Removal of COD

COD is defined as MEq (molar equivalent) of consumed oxygen in organic compounds oxidation by using a powerful oxidizer such as dichromate and permanganate under acidic conditions. Determination of the amount of COD is an important factor because it is a key indicator in determining the amount of organic pollution in aqueous systems. The high amounts of COD and BOD showed the poor quality of water. Electrochemical method is based on reduction of contaminant organic material, so COD number can be calculated by the electrical current that uses in electrochemical decomposition of organic pollutants (Yang et al. 2011). Figure 3 shows the dependence of electrochemical treatment efficiency to electrolysis time and

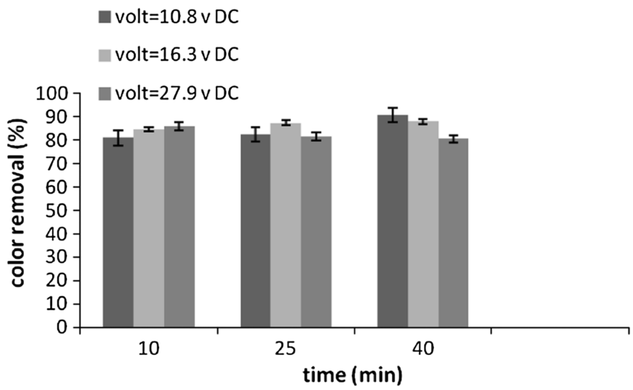

Fig. 2 The correlation between color removal and electrolysis time shifts and voltage

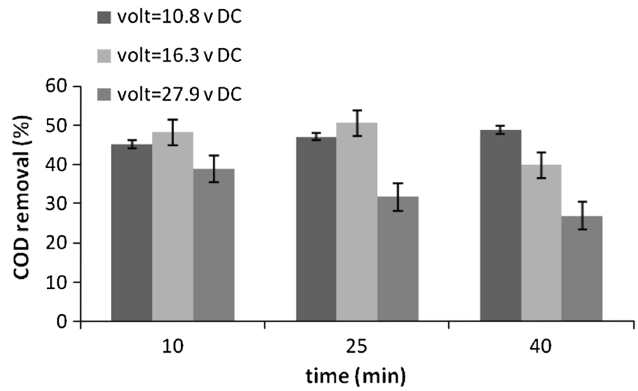

Fig. 3 COD removal efficiency with changes of electrolysis time and voltage

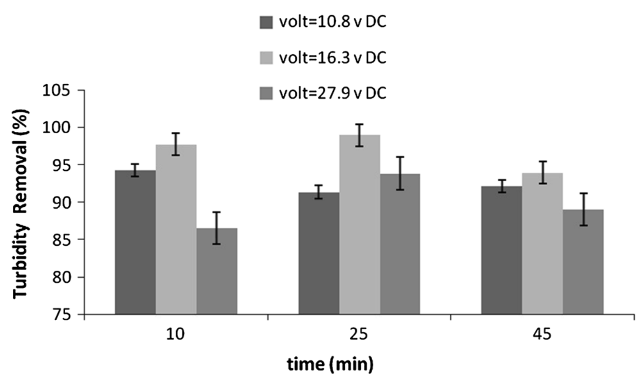

Fig. 4 Turbidity removal efficiency of waste water with voltage and electrolysis time changes

applied voltage changes for COD reduction of effluent from reduction of decolourization resins. Increasing reaction time in many purification methods can lead to more contact between pollutants and purifier and will increase the final efficiency. However, usually after a specified time (which depends on the type of treatment process) it will not cause significant increase in treatment efficiency. As it was proven in this study at low voltage (10.8 V) by increasing the time, the removing of COD by electrochemical process has increased, and the maximum removal efficiency is obtained after 40 min. Similar results have been reported by Dalvand et al. (2009) which found by increasing electrolysis time the efficiency of COD removing will increase. According to their results, one of the main reasons for the increasing of COD removing by passing time is the increase of the production of iron and aluminum hydroxide clots during the reaction time. COD removing mechanism during the electrochemical process is very complicated and can include oxidation-reduction of electrochemical restoration, electrostatic absorption and physically trapping of contaminant particles.

At voltage of 16.3, with rising of time to 25 min, the treatment efficiency has increased and additional time has a negative impact on removal of pollutants. Increasing time at the voltage of 27.9 has a negative effect on purification yield and by time enhancing from 10 to 40 min, the COD removal efficiency reached the minimum (Fig. 3).

According to the survey results the highest COD removal efficiency was obtained at voltage of 16.3 V and time of 25 min. According to Figs. 2, 3 and 4 when the amount of removed color combinations and turbidity factors were high, the maximum rate of COD removing has been observed. The less COD removing compared to color materials is probably due to decomposition of color factors and their conversion to other organic composition through electrochemical process (Dalvand et al. 2009).

Some researchers also examined the electrochemical treatment performance in sewage removal; Vigo et al. (1983) concluded a decrease of 97 % in sewage by using electrochemical oxidation in treatment of olive-oil factories waste water (Vigo et al. 1983). Difference in this survey results with mentioned studies can be attributed to the differences in sample and reaction condition. Waste water restored from decolorization resins due to high sodium chloride concentration has high electrical conductivity so in performed processes in this study at higher voltages the solution boiled and frothy layer of dark color was formed which indicated the active mechanism of electro flotation. Also evaporation of a part of solution water leaded to a condensed sample and raises the solid concentration in the effluent and decrease the process efficiency by depositing on the electrode surface.

The Effect of Voltage and Electrolysis Time Changes on Turbidity Removal Efficiency of Waste Water by Electrochemical Process

Turbidity is the amount of light absorption or scattering by suspended solids (SS) in the water. Since light absorption and scattering depends on the size and surface properties of SS, turbidity is not a quantitative value for SS. One of the main goals of waste water treatment is to remove floating and SS from it so study electrochemical treatment really fulfilled it. Figure 4 represents the electrochemical process impact on turbidity removal efficiency which shows the existent SS in the effluent from reduction of decolorization resins in different times and voltages. As can be seen in the Fig. 4, at 10.8 V with arises the time (from 10 to 40 min), there was not a significant change in turbidity of refined sample. Although at 16.3 V with increasing time to 25 min removal efficiency has an increasing trend but shifts are not considerable. The maximum efficiency was equal to 98.9 % at 25 min and 16.3 V and then declined with a greater slope to 40 min. By sufficient energy supply, organic compound are reduced in cathode and oxidation was occurred in anode and smaller molecules are formed.

The main mechanism to removing of colored compounds from aqueous solution is coagulation or clotting process. According to the researches, hydrolysis products including AL^{+3}, Fe^{+3}, Cl^{-1} are involved in flock formation and removing processes. Due to iron and aluminum hydroxides formation, the absorption of COD production factors, color and turbidity in created flocks, is possible. Figure 5 shows the Fe $(OH)_2$ formation and colloidal particles such as compounds causing color or turbidity through electrochemical process.

The Effect of Electrochemical Process on Effluent pH Changes

As it can be seen in Fig. 6 in all refined samples by electrochemical method, depending on the voltage intensity and sample contact time with electrodes in compare with control samples increasing of pH was observed. As it shown in this figure at 10.8 V by increasing the time (from 10 to 40 min) pH has a rising trend too. At 40 min with increasing voltages from 10.8 to 27.9 V the amount of effluent pH increases. Most reactions happen at 16.3 V and 25 min in anode and cathode so the highest pH value was achieved and it was equal to 10.8.

The increase in pH of waste water with rising current density is probably due to higher energy consumption during the passing time at high voltages. As a result of this increasing of energy consumption, anodic and cathodic activities and water molecules reduction in cathodes will significantly increase and lead to raising the amount of hydroxyl ions in the environment (Eq. 9)

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2 \quad (9)$$

On the other hand, the rate of hydrogen ions ejection from the environment as H_2 gas will increase, thereby by passing the time effluent pH grows up gradually (Dalvand et al. 2009). Better efficiency of electrochemical processes in removing SS and COD at 16.3 V and 25 min is probably due to this pH rising.

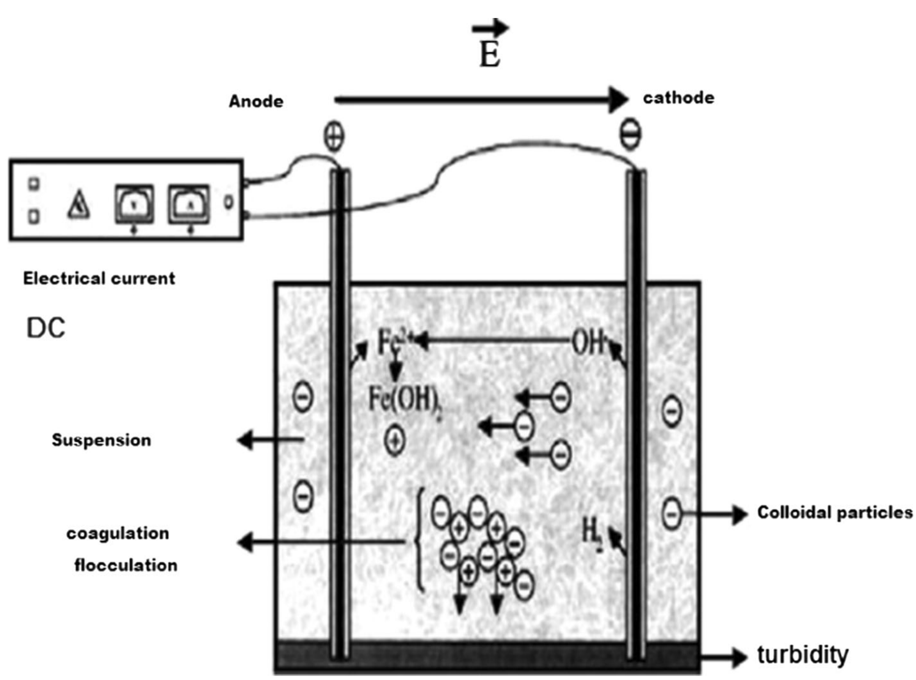

Fig. 5 Iron hydroxide formation and effluent colloidal particles deposit

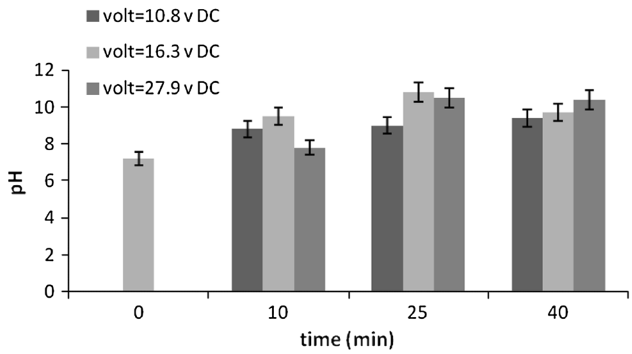

Fig. 6 pH changes trend with electrolysis time and voltage within electrochemical process

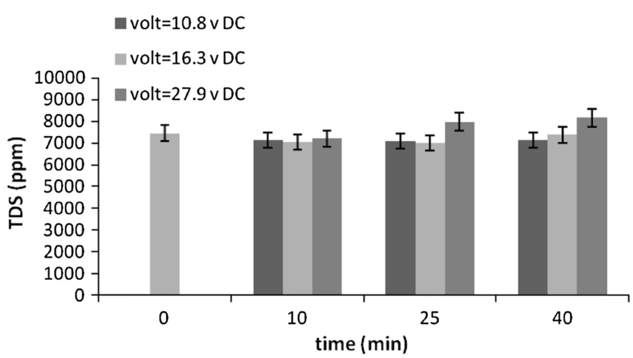

Fig. 7 Effect of electrochemical processes on changes in wastewater TDS

As mentioned earlier, at high pH, gelatinous suspension such as Al $(OH)_3$, Fe $(OH)_3$ and $Fe(OH)_2$ are formed in solution which during depositing, remove colored compounds and SS through complex mechanisms of surface and electrostatic absorption.

Result of the research that conducted by Dalvand et al. (2009) to remove the dye (198 Red) reactive from colored wastewater and Hsing et al. (2007) to remove orange acidic dye confirmed that the ultimate wastewater pH has increased in higher voltage and in high voltages, effluent pH has increased in shorter time. At a fix voltage, after a specific time pH is reached to a stable value (pH 10) and after that it does not have any considerable changes (Dalvand et al. 2009; Hsing et al. 2007).

Effect of Electrochemical Processes on Dissolved Solids (TDS) Effluent

One of the important substances found in water are solids that in two forms of total dissolved solids (TDS) and SS are seen in the water. TDS mainly contain various minerals in the water but does not include gas and colloidal material. The presence of solid particles is effective in electrical conductivity and is directly associated with specific conductivity, but this relationship is not linear. Thus TDS can be measured faster by using the conductmetery devices, evaporation and distillation techniques, electro gravimetery, ion exchange and reverse osmosis methods can be used for removing particles from solution. Modern methods are based on electronic, and are chosen by consideration of economic efficiencies (ICUMSA 2000). In this study the effect of the electrochemical process on removal of particles from the water soluble of effluent of reduced resin were investigated. As it can be seen in Fig. 7, this process had no significant effect on the removed of TDS. The maximum reduction was about 6 % which is related to the time of 25 min and voltage of 16.3 V. In the voltage of 27.9 V, as previously mentioned, because the effluent samples boil, some water evaporated from effluent samples, thus by increasing the time (from 10 to 40 min), the concentration of dissolved solids is increased too. In the Fig. 7 the zero time was related to control sample.

Conclusion

In this study, the electrochemical process by using iron and aluminum electrodes, for treating of wastewater of reducing decolorization resins of sugar factories were done. According to findings of this study electrochemical process by using EC and flocculation mechanisms can decrease the color of effluent about 90.8 %, COD to 50.5 % and the turbidity to 98.9 %. By reduction of water in the cathode, effluent pH increased 3.5 U and as a result it cause to better absorption and removing of colored substances and suspended and colloidal matters. It also has been shown that it had no significant effect on removing of dissolved solids of effluent from the column resin (only 6 %). According to the results, the efficiency of removing of COD, color and turbidity increased by increasing voltage and response times to a certain extent, but no direct relationship and more increasing of the voltage and time, have a negative effect on removal efficiency. The difference between the results of this study by the previous studies can be attributed to differences in the samples under investigation and reaction conditions. Decolorization resins wastewater contains high concentration of sodium chloride and high electrical conductivity therefore at higher voltages or times due to evaporation and concentration of the effluent water and deposition of material on the electrodes, the absorption potential was reduced and follow this mass transfer to the electrode surface was decreased and this lead to decrease in efficiency of the removal pollutants by electrochemical process. According to results optimum condition for decolorisation resin wastewater treatment by electrochemical process is the voltage of 16.3 for 25 min.

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