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The effect of media ion and nitrogen gas dissolved in water on color removal with sonolysis, Fenton and sono-Fenton in the continuous flow ultrasonic reactor

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Abstract: Ultrasound has been researched as an alternative technique for color removal methods from water and wastewater. In this study the color removal from water using sonolysis, Fenton, and Sono-Fento was investigated and the effects of power intensity, media ions and nitrogen gas dissolved in water were investigated in model working solution with Basic Red 29 Dye in the continuous flow ultrasonic reactor. The experimentation was performed at 22 kHz ultrasonic frequency with different power intensity application in the ultrasonic system. The optimum color removal efficiency was achieved with 0.45 W/cm^2 power intensity. The color removal efficiency of Basic Red 29 was raised to two times with the coexistent effect of media ions while the media ions and dissolved gas increased to four times simultaneously. To conclude, the color removal efficiency followed from this order in sonolysis: N_2+ SO_4^{-2}+ HCO_3^-+NO_3^{-2} > HCO_3^-> SO_4^{-2}+ HCO_3^-+NO_3^{-2}> SO_4^{-2} >N_2> NO_3^{-2}. In Fenton process, the effects of Fe^{+2} and H_2O_2 concentration on system performance were examined obtaining 89% color removal efficiency by using 20 mg/L Fe^{+2} and 1000mg/L H_2O_2. With the hybrid Sono-Fenton process, the required Fe^{+2} amount decreased by 1/5, when the color removal efficiency increased to 98 % and there was no significant effects of media ions and dissolved gas on Fenton and Sono-Fenton treatment. As a result of toxicity studies, it was seen that the initial toxicity of wastewater decreased with treatment and the Sono-Fenton method was the most effective method on toxicity removal. It was determined that color removal using Sono-Fenton can be accomplished successfully, economically and environmentally friendly.

Keywords: sonolysis, Fenton, Sono-Fenton, color removal, media ions, nitrogen gas, toxicity

1. Introduction

Wastewater production of textile industry has recently gained great significance in environmental engineering in terms of synthetic dye content and toxic characteristic. The synthetic dyes are resistant to biological degradation (Tegli et al. 2014) and it remains a challenge to efficiently and accurately treat textile wastewater with conventinal metods due to its relatively high level of acidity, toxicity, and non-biodegredable organic load (Husain 2006). The textile industry wastewater is generally accepted as persistent, nonbiodegradable, photosynthesis limiting and toxic characteristics to the aquatic ecosystem (Weng et al. 2014).

The number of studies on alternative technologies aims to attain more effective color removal and reducing toxic effect of dyes has increased, although various conventional wastewater treatment methods have been used for color removal of these industries effluents to prevent their detrimental effects on aquatic ecosystem such as, adsorption (Khan et al. 2004), membrane processes (Sakoda et al. 1996) and ion exchange (Zhao et al. 2008), electrochemical treatment (Surme and Demirci 2014), advanced oxidation process (Lopez-Grimau et al. 2012), bio-sorption (Jin et al. 2014), nano-sized adsorption (Lin et al. 2015), and sonolysis (Zhang et al. 2013) can be listed among these alternative technologies.

Sonolysis has been used for various dyes with their different natures in the literature. Ai et al. developed sonolysis to remove Rhodamine B azo dye in aqueous solution in addition to methyl orange, methylene blue and Reactive Brilliant red X3-B with high removal efficiency (Ai et al. 2010). Zhang et al. demonstrated that sonolysis enhanced the reduction efficiency of C.I. Acid Orange 7 in the zero valent iron processes (Zhang et al. 2005). Sonolysis and their hybrid applications combined with other methods like sono-Fenton have been noticed to attain more efficient decolorization and reduce chemical and energy requirement. Fenton reaction can be improved by combining UV (photo-Fenton, solar photo-Fenton), or ultrasound radiation (sono-Fenton), and electrical current (Electro-Fenton). The characteristics of the textile industry effluent is important for recent progress of the Fenton processes like sono-Fenton adressed as alternative to the treatment of textile effluents. (Ramos et al. 2021). Among advanced oxidation processes often used for treatment, sono-Fenton and photo-Fenton processes gain a lot of attention due to high pollutant degradation efficiency thanks to direct and synergistic effects of ultrasound and light which lead to the highest generation of reactive radicals such as •OH, •H and •OOH and thier catalytic role of ferrous ions (Moradi et al. 2020). Sayan and Edecan researched the removal of Reactive Blue 19 dye using sono-Fenton process. They reported that performing sonolysis with Fenton provided more efficient color removal because of accelerating synergetic effect of two systems (Sayan and Edecan 2008). The combination of the various advantages of these alternative methods such as, mass transfer accelerator, reaction rate acceletor, and catalyst effect the raise the removal efficiency, while the disadvantages such as, energy consumption, chemical requirement, and high cost are eliminated in the hybrid systems. Xu studied Reactive Brilliant red X3-B textile dye, which is persistent to UV radiation treatment, with different advanced oxidation process and he reached the highest decolorization efficiency in sono-photo-Fenton (US-Fe^{+2}–H_2O_2-UV) treatment (Xu 2001). Voncina and Majcen-Le-Marechal investigated six different reactive dyes with sono-oxidation (US-H_2O_2) treatment. They stated that advanced oxidation process related to sonolysis application combined with H_2O_2, UV, and ozone accelerated free radical formation efficiency. Since sonication improved mass transfer and chemical reaction speed (Voncina and Majcen-Le-Marechal 2003). Weng et al. studied with real textile wastewater and achieved successful decolorization efficiency using sono-Fenton process combined with 1 g/L zero valent iron dosage and 0.01 M hydrogen peroxide concentration applying 47 kHz sonic frequency while operating cost was estimated around 4.5 USD for 1 m^3 wastewater (Weng et al. 2014). Chu et al reported the results of the heterogeneous Fenton-like processes to remove organic dyes from water, and the synergistic effects of ultrasound (US) and Fenton with metalic biochar catalyst. The heterogeneous sono-Fenton-like triple (US–H_2O_2–catalyst) system exhibited a high dye-removal efficiency for methylene blue and orange (Chu et al. 2020). The main reason for using sonolysis together with other methods is that it requireshigh energy and cost to obtain effective removal efficiency. Zhang et al. reported that sonolysis did not enhance the colour removal of CI Reactive Black 8 combined application with Fenton process regarding to single Fenton treatment while improving COD removal (Zhang et al. 2007). The main disadvantage of other advanced oxidation methods (e.g., fenton) is the requirement of oxidizing chemicals and the possibility of forming toxic by-products as a result of treatment. An other study perfomed by same research team stated that ultrasound and Fenton process supported activated carbon system had 88% decolorization efficiency of Crystal Violet and acute toxicity of dye solution decreased after the treatment by Daphnia magna test (Zhang et al. 2013). The possibility of toxic by-products formation in Fenton treatment also depends on other ions being in the water. In addition, these ions can be effective in color removal by sonolysis eihter. The characteristics of the textile industry effluent is important for recent progress of the Fenton processes like sono-Fenton adressed as alternative to the treatment of textile effluents. There is no detailed study in which the effects of these characteristics on sonolysis, Fenton and hybrid sono-Fenton processes and efflux toxicity are examined in the literature.

The efficiency of such processes regarding the important characterization parameters (media ions, color, dissolved gas present in water) for model textile effluents was investigated in this survey.In this study the color removal from water using sonolysis, Fenton and sono-Fenton was researched in the continuous flow ultrasonic rector. The effect of media ions and dissolved nitrogen gases being in the water were investigated on the single and hybrid treatment processes by assessing toxicity.

2. Materials and Method

2.1. Sonolysis

The ultrasonic decolorization studies were performed in continuous flow reactor with 22 kHz frequency, applying 26 W power. The ultrasonic reactor was used in this study (refer to Fig. 1). A model wastewater was used in the treatment studies including Basic Red 29 (refer to Fig. 2) dye (Sigma Aldrich) with 40 mg/L initial concentration in the 100 mL volume.

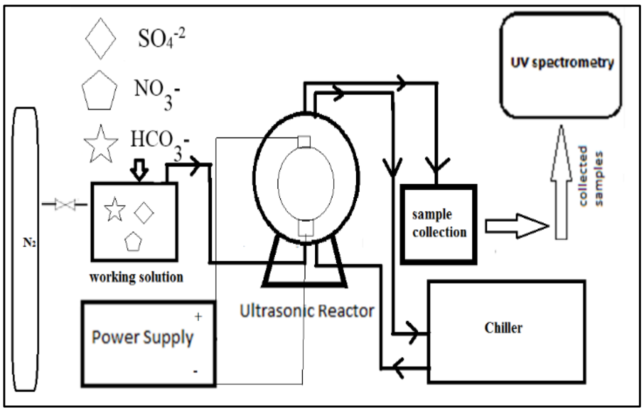


Fig. 1 Experimental setup

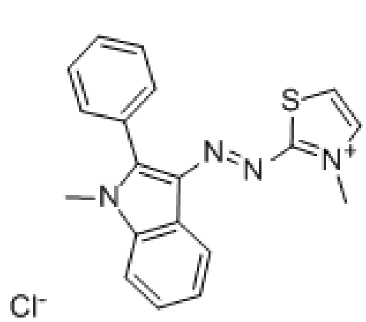


Fig. 2 Chemical formula of Basic Red 29

The reactor was performed at 5 mL/min flow rate to the ultrasonic reactor with 50 mL volume and the hydraulic retention time was determined as 10 minutes during decolorization treatment with sonolysis, Fenton, and Sono-Fenton processes. The absorbance of collected samples was obtained in UV-Vis spectrophotometer (Shimadzu UV-Vis 1600) at 506 nm and the decolorization efficiency calculated using Equation 1.

$$\text{Color removal efficiency } (\%) = \frac{C_i - C_e}{C_i} \times 100 \qquad \text{(Eqn. 1)}$$

Where,

C_i=influent Basic Red 29 concentration, mg/L

C_e =effluent Basic Red 29 concentration, mg/L

Each treatment was conducted with three independent experiments at the same conditions and the average results of all experiments was given in the result section.

2.2. Media ions effect on decolorization

In order to investigate effect of media ions present in water, SO_4^{-2}, HCO_3^-, and NO_3^{-2} ions were added to system in form of their water soluble sodium salt. Na_2SO_4 (Merck Millipore), $NaHCO_3$ (Merck Millipore), and $NaNO_3$ (Merck Millipore) were used to supply these ions to water with 0.1-0.2 M SO_4^{-2}, 4.1-8.2 mM HCO^{-3}, 4.0-8.0 mM NO_3^{-2} concentration added respectively to model wastewater considering their legally permissible concentrations in the surface waters as the quality parameters to examine their effect on decolorization.

2.3. Dissoved nitrogen gas effect on decolorization

Thedissolved nitrogen gas in water was determined in model wastewater solution in this study. The effect of dissolved nitrogen gas on decolorization was examined with 8 L/hour, 12 L/hour, and 16 L/hour nitrogen gases flowrates. The effect of three different nitrogen gases flowrate on ultrasonic color removal was determined during color removal from water using sonolysis and sono-Fenton.

2.4. **Fenton and Sono-Fenton**

Fenton (Fe^{+2}-H_2O_2) studies were conducted in a glass beaker. The $FeCl_2.4H_2O$ (Merck, 99% w/w) was used as Fe^{+2} source in Fenton treatment process. Firstly, The H_2O_2 concentrations were adjusted as 1000 mg/L in model solution and the effect of Fe^{+2} concentration was determined containing 2, 4, 6, 8, 10, 20, 40, 60, 80, and 100 mg/L Fe^{+2} using suitable primary $FeCl_2.4H_2O$ stock solutions.

Secondly, Sono-Fenton (US-Fe^{+2}-H_2O_2) studies were conducted in US reactor (refer to Fig. 1) using 1000 mg/L H_2O_2 (Merck, 35% w/w) concentrations and 20 mg/L Fe^{+2} concentrations. The effect of the media ions effect was determined with 0.2 M SO_4^{-2}, 8.2 mM $HCO_3^-$and 4.0 mM NO_3^{-2} concentration and the dissolved gas effect was investigated 16 L/hour nitrogen gas flowrates on Sono-Fenton processes.

2.5. **Toxicity Asesment**

Toxicity tests of effluent and influent samples were analyzed with Microtox Model 500 Analyzer (Azur Environmental, Carlsbad, CA) as cytotoxicity investigation. The lyophilized *Vibrio fischeri* bacterium (NRRL number B-11177, Microtox Acute reagent, Modern Water) with luminescence characteristics was used as a test microorganism. The *Vibrio fischeri* bacterium was incubated with reconstitution solution (nontoxic ultra-pure water, Modern Water) and osmatic adjuster solution (22% sodium chloride, Modern Water) at 15 °C during 15 min in glass cells. Four serial dilutions of the sample were prepared with dilution solution (2% sodium chloride, Modern Water). Toxic effects of the test result were recorded for each sample using Microtox calculation software (95% confidence) with light emission reduction percent value of 15 min exposure of *Vibrio fischeri to test sample.*

3. Results and Discussion

In this study the color removal from water using sonolysis, Fenton and Sono-Fenton technology was researched and the effects of media ions and dissolved nitrogen gas were determined in model synthetic Basic Red dye solution.

3.1. Sonolysis

The results of the experimentations process were given at 22 kHz frequency with different power intensity (W/cm^2) application to the system (refer to Fig. 3).

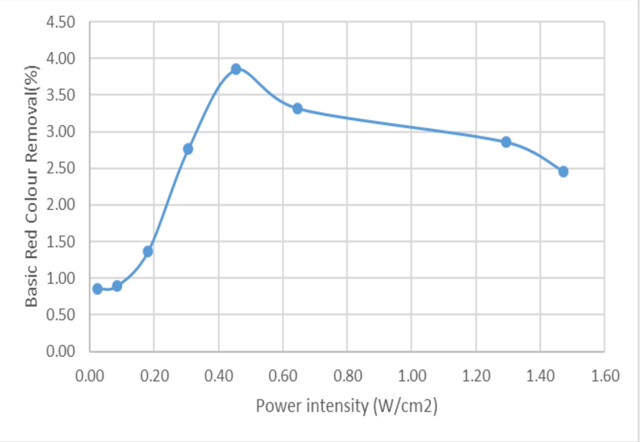


Fig. 3 Experimental setup

The optimum power intensity was determined as 0.45 W/cm^2, and raising the power intensity bigger than 0.45 W/cm^2 because the applied power is more than 0.45 W/cm^2 caused an increasement in heat or resonance of the ultrasonic system. The other sonolysis studies were performed this optimized power intensity with 0.45 W/cm^2. The results of the effect of sulphate ion on color removal were investigated for 0.1-0.2 M SO_4^{-2} (refer toFig. 4).

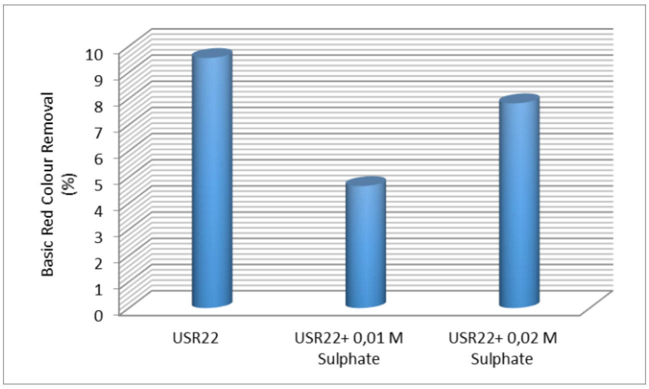


Fig. 4 The effect of sulphate ion on color removal

Another parameter considered in the color removal with sonolysis is the presence of media ions such as sulphate, bicarbonate, and nitrate presented in the liquid. According to Fig. 4, it was determined that suphate ion had negative effect on color removal efficiency of sonolysis. Dissolved ions accompanying in water can lead to the formation of ultrasonic cavitations, but they can have a negative effect as they can also cause an additional reactions such as conversion of the sulphate to sulfite or persulphate (Gayathri et al. 2010).

H_2O + ultrasonication→ H• + OH• (Eqn. 2)

SO_4^{-2} + H• + OH•→ $SO_4^{\bullet -}$ + H_2O (Eqn. 3)

$SO_4^{-2} + SO_4^{\bullet -} \rightarrow S_2O_8^{-2}$ (Eqn. 4)

Sonolyses catalyzes the production of persulphate, by activating sulphate with formation of hydroxyl radical by ultrasonication. If persulfate reaction in Eqn.4 has faster kinetics than Eqn. 3, the degradation rate of Basic Red 29 was enhanced by the US activated persulphate. Thus, its negative effect on color removal in the case of sulphate ion addition (refer to Fig. 4) could be explained by Eqn 2-4. The results of the effect of nitrate ion on color removal were investigated for 4.0-8.0 mM NO_3^{-2} (refer to Fig. 5).

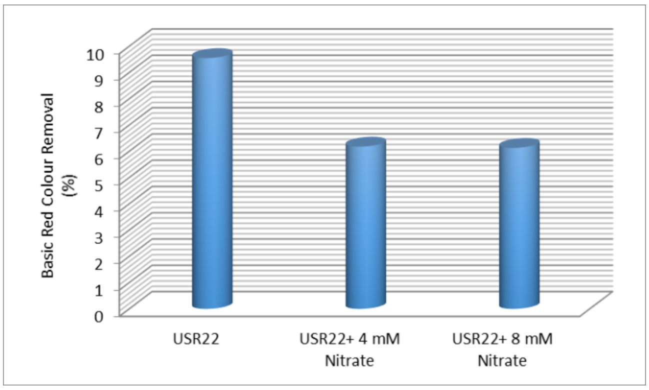
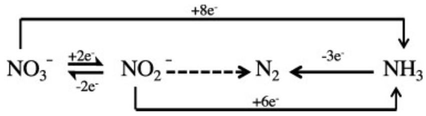


Fig. 5 The effect of nitrate ion on color removal

The nitrate ion addition affected the color removal adversely in parallel with the sulphate ion results. Similarly, it causes an additional reactions such as conversion of the nitrate to nitrogen and ammonia (Koparal and Ogutveren, 2002). The ultrasonical reduction of nitrate ions to nitrogen and ammonia could be explained as following reactions (Govindan et al. 2015).



(Eqn. 5)

The formation of additional reaction of nitrite, nitrogen gas, and ammonia could be decreased the ultrasonic color removal efficiency. The results of the effect of bicarbonate ion on color removal were investigated for 4.1-8.2 mM HCO_3^- (refer to Fig. 6).

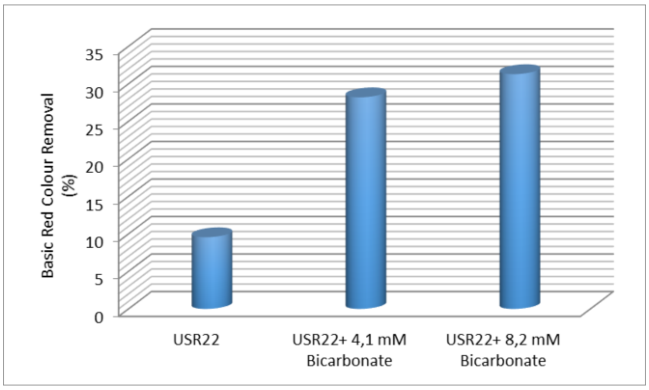


Fig. 6 The effect of bicarbonate ion on color removal

The addition of bicarbonate ion raised the color removal efficiency of Basic Red 29 three times unlike sulphate and nitrate ions. Bicarbonate ions could be identified as accelerator agents in the sonoysis because the rate constants of bicarbonate ions with sulfate and hydroxyl radicals are relatively high. Hence the presence of bicarbonate ions can eventuate in a competition for reaction with free radicals (Feizi et al. 2019). The results of the effect of nitrogen gas on color removal were investigated for 8, 12 and 16 L/h gas flowrate (refer to Fig. 7).

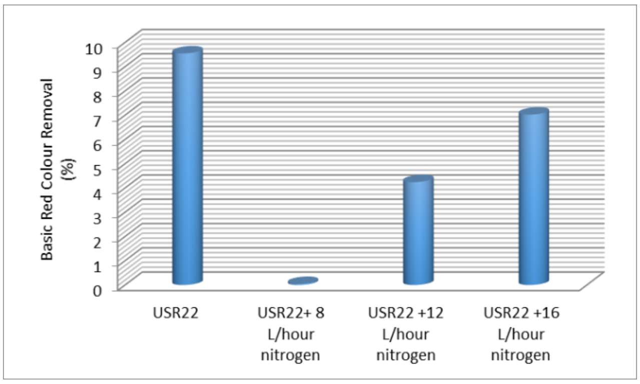


Fig. 7 The effect of nitrogen gas on color removal

The gas dissolved in the liquid affects the physical and chemical properties of acoustic cavitation bubbles and variation of the amount of dissolved gas in the liquid alters the production of collapse. The change of the type of dissolved gas modifies adiabatic rate, thermal transmission, the surface tension of the liquid, and the hot spot temperature of micro-bubbles. For these reasons the effect of dissolved gas on color removal with ultrasound must be investigated. After the nitrogen gas studies, it was observed that the addition of nitrogen gas did not have a significant effect on sonolysis. The simultaneous effect of media ions (0.2 M SO_4^{-2}, 8.2 mM HCO^{-3} and 4.0 mM NO_3^{-2} and dissolved nitrogen gas (16 L/hour) on color removal in sonolysis is given in Fig.8.

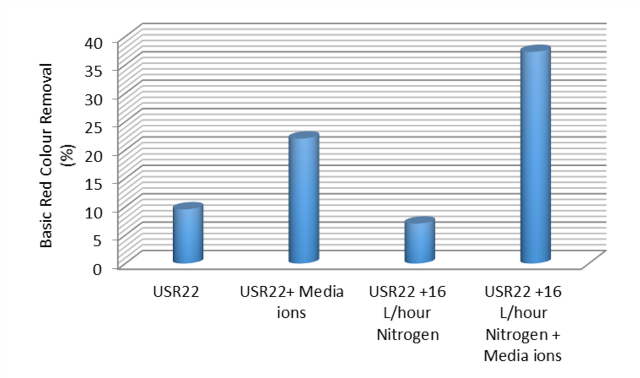


Fig. 8 The simultaneous effect of media ions (0.2 M SO_4^{-2}, 8.2 mM HCO_3^- and 4.0 mM NO_3^{-2} and dissolved nitrogen gas (16 L/hour) on color removal

The color removal efficiency of Basic Red 29 was raised to two times with the coexistent effect of media ions while the media ions and dissolved gas increased four times simultaneously. To conclude, the color removal efficiency followed from this order in sonolysis: N_2+ SO_4^{-2}+ HCO^-_3+NO_3^{-2}> HCO_3^-> SO_4^{-2}+ HCO_3^-+NO_3^{-2}> SO_4^{-2} >N_2> NO_3^{-2}.

3.2. Fenton and Sono-Fenton

The effects of different Fe^{+2} concentrations on color removal were given in Fig. 9 in Fenton (Fe^{+2}-H_2O_2) studies to show the single effect of Fenton on Basic Red 29 removal.

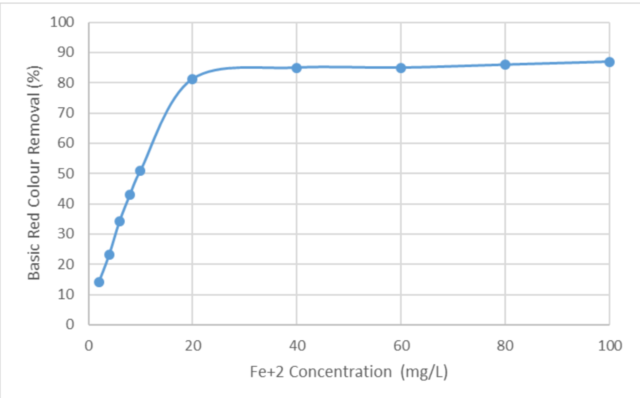


Fig. 9 The effect of Fe^{+2} concentration on Fenton (H_2O_2 concentration is 1000 mg/L and pH:3.96)

When the H_2O_2 concentration was set as 1000mg/L, the colorremoval efficiency raised to 12.0 % from 89 % as the concentration of Fe^{+2} was raised from 2 to 100 mg/L. It was determined that the applied Fe^{+2} concentrations showed same color removal trend between 20 and 100 mg/L Fe^{+2} concentration. Hybrid Sono-Fenton studies were carried out in an ultrasonic reactor by setting Fe^{+2} concentration to 20 mg/L and H_2O_2 concentration to 1000 mg/L. As in sonolysis studies, the effects of media ions and nitrogen gas on the Sono-Fenton were also investigated. The comparative graph of these studies is given in Fig.10.

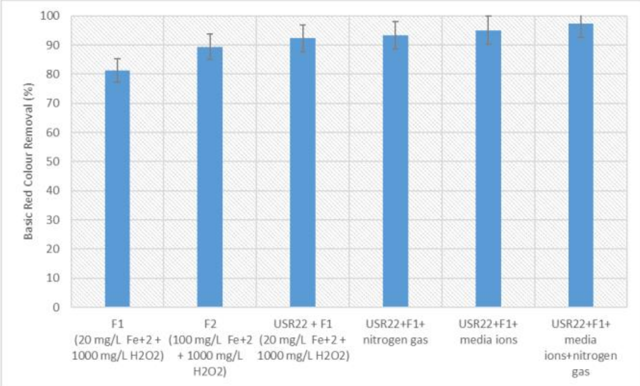


Fig. 10 The simultaneous effect of media ions

Comparing these results with literature, Weng et al. examined the effect of their anions by adding NaCl, Na_2SO_4, Na_2HPO_4, $NaNO_3$, and $NaClO_4$ salts while examining the removal of RS5 dye with Sono-Fenton at the end of the study, it was seen that Cl-, SO_4^{-2}, NO_3^- and ClO_4^- anions did not havesignificant effect, but $H_2PO_4^-$ anion suppressed the system (Weng et al. 2013). In another study, the effect of Cl^- and SO_4^{-2} ions was investigated. Accordingly, Cl^- ion was negative for the destruction of Blue2B (B54) and Red12B (R31) dyes; It has been observed that SO_4^{-2} ion does not have effect on destruction (Malik and Saha, 2004). After the nitrogen gas studies, it was observed that the addition of nitrogen gas did not have a significant effect on Fenton and US/Fenton reactions, as in the study of Saravanan and Sivasankar, 2015. They reported that the maximum decolourization occurs at acidic pH of 3.0, Argon gas bubbling, 8.82 mmol/L hydrogen peroxide, 10 g/L NaCl addition and the colour removal are the highest for Fenton's reagent-treated Reactive Black 5 dye solution which followed pseudo-first-order rate kinetics.

3.3. Toxicity assessment

The toxicity reduction of Basic Red 29 containing synthetic wastewater was illustrated in Fig. 11.

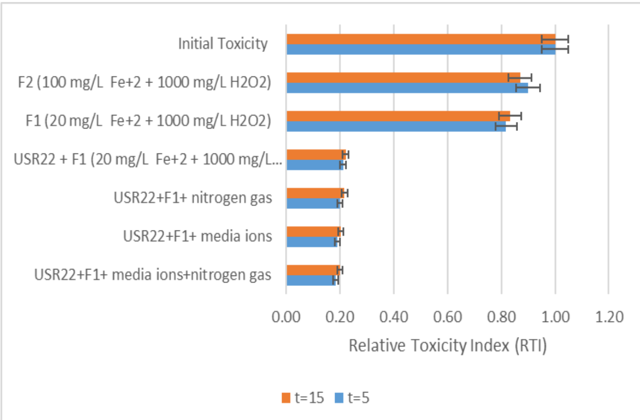


Fig. 11 The simultaneous effect of media ions

Toxicity assessment showed that 40 mg/L Basic Red 29 containing wastewater had a high toxic effect on *Vibrio fischeri* bacteria. The phenol solution was used as control according to Microtox Test Procedure with 50 mg/L concentration. The LC50 concentration of untreated wastewater was calculated as 20 mg/L (2%). In this paper, even if the sonolysis, Fenton, and Sono-Fenton treatment slightly reduced the toxicity of untreated wastewater in parallel with color removal, the toxicity of wastewater was significantly decreased with Sono-Fenton (US-Fe^{+2}-H_2O_2) treatment. However, the wastewater treated with Sono-Fenton (US-Fe^{+2}-H_2O_2) still showed toxic characteristic after 99% Basic Red 29 removal.

4. Conclusion

In this study the color removal from water using ultrasound technology was researched and the effect of applied power intensity, media ions, and nitrogen gas in water was determined with ultrasonic color removal of Basic Red 29 dye in the continuous flow reactor. The optimum color removal efficiency was achieved with 0.45 W/cm^2 power intensity. As a result of the study, it was determined that color removal from water using ultrasound technology can be accomplished successfully. The color removal efficiency of Basic Red 29 was raised to two times with the coexistent effect of media ions while the media ions and dissolved gas increased four times simultaneously. To conclude, the color removal efficiency followed from this order in sonolysis: N_2+ SO_4^{-2}+ HCO_3^-+NO_3^{-2} > HCO_3^-> SO_4^{-2}+ HCO_3^-+NO_3^{-2}> SO_4^{-2} >N_2> NO_3^{-2}. In Fenton process, the effects of Fe^{+2} and H_2O_2 concentration on system performance were examined obtaining 89% color removal efficiency by using 20 mg/L Fe^{+2} and 1000 mg/L H_2O_2. With the hybrid Sono-Fenton process, the required Fe^{+2} amount decreased by 1/5, when the color removal efficiency increased to 98 %. As a result of toxicity studies, it was seen that the initial toxicity of wastewater decreased with treatment and the Sono-Fenton method was the most effective method for toxicity removal.

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Authors' contributions:

FKÖ: Conceptualization, Methodology, Software, Data Curation, Validation, Formal Analysis, Writing- Original Draft Preparation, Visualization, Investigation.

Conflict of interest disclosure:

The author declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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