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Decolorization of Boron-Based Wood Preservatives by Ozonation for Recycling: Optimization of Process Parameters

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Abstract

Preservation of sawn timber from rubber trees with a water-soluble mixture of boric acid and borax generates a large quantity of colored wood preservative solutions and carries toxic metals, especially boron. To save costs, millers commonly reuse the colored mixtures in successive preservation cycles but regularly add boric acid and borax to maintain boron levels. However, this strategy affects both the color and the economic value of the treated wood. This paper presents a new strategy to treat the colored mixture of waterborne preservative solutions using ozonation for recycling purposes. At optimum conditions which are determined by response surface methodology, very high color removal (96 - 97 %) is achieved from ozone treatment of the wood preservatives with an initial COD concentration of 2,250 ± 40 mg/L and solution pH of 3.59 ± 0.02 for 65 min reaction time. The effluent can be reused in successive preservation cycles without degrading the wood quality in terms of the color of the processed timber and boron penetration in the end grain as well as the sides of the wood. Ozone treatment shows to be a promising color removal technology for sawn rubberwood industry, aimed at the reuse of waterborne preservatives, resulting in direct environmental and economic benefits.

Keywords: Waste reduction, Water reuse, recycling, Color removal technology, Ozone treatment, Statistical optimization, Wood preservatives, Clean technology

Introduction

Boron-based preservatives have been used for many years in the treatment of sawn rubberwood, which is very susceptible to biodegrading organisms. An inorganic aqueous solution of boric acid and borax has been recommended to treat rubberwood, in China and other Asian countries, because it is easy to handle and apply [1,2]. Furthermore, the mixture is considered more effective than copper- and zinc-based preservatives because of its rapid diffusion and broad-spectrum efficacy against a wide range of wood-destroying organisms, including termites, decay fungi, and wood-destroying beetles [3-6]. Moreover, wood after wood preservation using the boron-based mixture is odorless and colorless, which allows the treated wood to be subsequently painted or stained to the desired color in furniture making [3,7]. While several timber preservation methods can be used to treat rubberwood, vacuum pressure impregnation has been adopted by the industries that use borate compounds as a wood preservative. The pressure process forces wood preservatives into the outer layer of the timber, followed by natural diffusion of the chemical into the core during seasoning [5]. With the vacuum pressure impregnation, deeper and more uniform penetration of wood preservatives is obtained in a short time compared to dip diffusion processes [5,8,9]. Due to industrial demand for a cost-saving method in rubberwood treatment, it is a common practice to return the colored mixture of boric acid-borax to a storage tank and use it repeatedly in subsequent treatment cycles. A chemical treatment operator regularly adds boric acid, borax, and tap water to the storage tank to maintain the solution strength. While a freshly prepared aqueous solution of boric acid and borax is colorless, the color of the solution becomes dark brown and becomes darker with each reuse cycle. Reuse of darkening boric acid-borax solution reduces wood preservation costs as well as disposal costs for the industry. However, the reuse of darkening boric acid-borax solution affects the color of the treated wood and reduces the economic value of the wood, whereas natural wood color is often desired for furniture fabrication [10]. In addition, timber millers using boron-based solutions typically have no net generation of the spent borate solution, managing this waste at present relies on the industry for waste collection and treatment before discharge to the environment. Therefore, treatment of the colored boron solution and recycling of the effluent back into the preservative storage tank offers a viable method for pollution prevention by minimizing the use of, and, where applicable, reuse of the preservatives and water in the preservation process. It also reduces the responsibility of millers for waste collection.

Ozonation is considered effective to reduce the color of the raw water that its color is caused by dissolved organic matter, e.g., humic and fulvic acids (biological decomposition products from vegetation) as well as high-colored wastewater of the textile industry [11-14]. Ozonation can simultaneously interact and break down low-biodegradability organic compounds in wastewater, increase biodegradability, and improve the subsequent biological treatment process [15-19]. Ozone is a strong oxidizing agent (E^o = 2.08 eV) compared with other agents, such as H_2O_2 (E^o = 1.78 eV) [14]. Ozone reacts with chromophore groups, generally organic compounds with conjugated double bonds, breaking the molecules down into smaller forms and decreasing the effluent color [14,20]. The reaction can proceed through 2 main routes based on pH: Direct molecular ozone oxidation and an indirect pathway involving ozone disintegration and formation of hydroxyl radicals (•OH). The latter is less selective and predominates under alkaline conditions, while direct oxidation is more selective and predominates under acidic conditions [16,21]. Success in the use of ozone has been reported, not only to treat dye wastewaters to meet effluent standards but also to focus on decolorization of textile industry wastewaters for reuse [11,12,22-25]. The application of ozone to treat the color of boric acid-borax solutions that are used as wood preservatives has never been reported. While The color of wood preservative solutions is caused by dissolved organic matter leached from the sawn timbers: Similarly, ozone decolorization of the repeatedly used boric acid-borax solutions from the pressure preservation process is potentially possible.

As has been previously reported in the literature, color removal efficiency by ozone is dependent on various operational parameters such as reaction time, ozone dosage, initial pH, initial pollutant concentration, and temperature of the solution [26-28]. To determine the effects of each operational variable, a traditional approach known as one-factor-at-a-time (OFAT) is widely practiced. Experiments are carried out by varying systematically only one studied parameter and keeping the others constant. Then, experiments are repeated to all the influencing parameters, resulting in unnecessarily large numbers of experimental runs, and considered to be a time-consuming method [28]. Moreover, the interaction between variables is not measurable, which can mislead the optimal conditions of the process. To determine the effect of operational parameters with the minimum number of experiments, response surface methodology (RSM) is a popular tool that has been employed to optimize the response of a process. In this case, it is decolorization or the color removal efficiency of ozone. Using RSM, it is possible to estimate linear, interaction, and quadratic effects of the studied variables and to provide a prediction model for the response [28]. The method has been used for modeling and optimization of the decolorization process by ozonation especially textile wastewater treatment [28-30]. However, no prior studies have examined the decolorization of the boron-based wood preservative solution by ozonation and the effect of the interaction of operational parameters. In this work, the feasibility of ozone technology for decolorization of the high-colored boric acid-borax solution, enabling recycling of the treated solution back into the wood treatment and preservation process, was investigated. The Box-Behnken design and RSM were applied to determine the mathematical model that best described the relationship between decolorization and process parameters for ozonation and to find optimum conditions for decolorization of the boron-based solution by ozonation. Those process parameters being investigated were ozonation time, initial pH of the solution, and initial COD concentration. Decolorization efficiency was monitored as the process response. Counter plots were drawn to predict the efficiency of the decolorization process under different values of the independent parameters.

Materials and methods

Boron-based solution: Source and characterization

An aqueous solution of boric acid and borax that is repeatedly used in the wood preservation process was collected from the wood preservative storage tank of a rubberwood timber mill in Thailand. There was a single supply, with a characteristic dark brown color. Before starting the ozonation experiment, pH was measured by pH meter (model no. 827, Metrohm, Switzerland). Color in ADMI units was measured by the Tristimulus Spectrophotometric Method (Methods 2120E) and Chemical Oxygen Demand (COD) was measured by Closed Reflux, Titrimetric Methd (Method 5220C) [31]. The boric acid concentration was analyzed by titration with a standardized NaOH solution. All measurements were in triplicate. Sample characteristics were as follows: pH, 7.95 ± 0.01; color, 1,013 ± 6 ADMI; COD, 2,822 ± 40 mg/L and Boric Acid Equivalent (BAE), 0.97 ± 0.003 % w/w.

Ozonation experiments

An acrylic tube reactor (diameter 60 mm×height 400 mm) was used and operated in batch mode. Ozone was generated from the air using a laboratory ASIA-TECH OZ-735 ozone generator that operates at a constant flow rate of 3.37 ± 0.13 L/min. The outlet stream from the ozone generator was passed through Teflon tubing connected to a porous diffuser that is laid at the bottom of the reactor for the generation of fine bubbles. The unreacted ozone in the off-gas from the top port was captured in 2 gas washing bottles, each of them containing 250 mL of 2 % potassium iodide (KI) solution. Mass of ozone trapped in KI was determined by titrating the solution from the gas washing bottle with 0.1 N $Na_2S_2O_3$ following Standard Method 2350E [31]. At the start of each run, the reactor was filled with 500 mL of the test solution with a predetermined influent COD concentration. The solution pH was adjusted to the target experimental values with sulfuric acid and sodium hydroxide. Ozone was then applied for a set time and then the color of the effluent was measured.

Experimental design and statistical analysis

The feasibility of applying ozone (O_3) to reduce the color content of borates solutions was investigated based on One-Factor-At-a-Time (OFAT) experiments, which vary one variable at a time while keeping others fixed. The effect of ozonation time (10 - 120 min), initial solution pH (3 - 9) and initial COD concentration (2,284 - 2,862 mg/L) on color removal efficiency (or decolorization) was measured. A Box-Behnken design (BBD) combined with Response Surface Methodology (RSM) was employed to study the main effects and interaction effects of 3 operational parameters: Ozonation time (x_1), solution pH (x_2), and initial COD concentration (x_3), as well as to seek the optimum conditions for decolorization of the colored boric acid-borax solutions by ozonation. High and low levels of the 3 process parameters being studied were selected based on the results from OFAT experiments, and those levels being studied are shown in **Table 1**. A total of 15 experimental runs were obtained from BBD using Minitab statistical software (version 17.0) as shown in **Table 2**. The response variables for the model (i.e., decolorization efficiency) were analyzed and the relationship between the input variables and the observed response variables was assumed to follow a 2nd-order polynomial model as shown in Eq. (1).

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i x_i + \sum_{i=1}^{3} \beta_{ii} x_i^2 + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} x_i x_j \quad (1)$$

where *Y* is the response (decolorization in %), x_i and x_j are the uncoded independent variables that influence the predicted response, β_o is a constant and β_i, β_{ii}, β_{ij} are the linear, quadratic and interaction effects.

Table 1 Process parameters and their levels used in the experimental design.

Process parameter	Range and levels		
	–1)Low(	**0)Middle(**	**1)High(**
Ozonation time)min.(, x_1	10	60	120
Solution pH, x_2	3	5	8
Initial COD concentration)mg/L(†, x_3	2,284	2,669	2,752

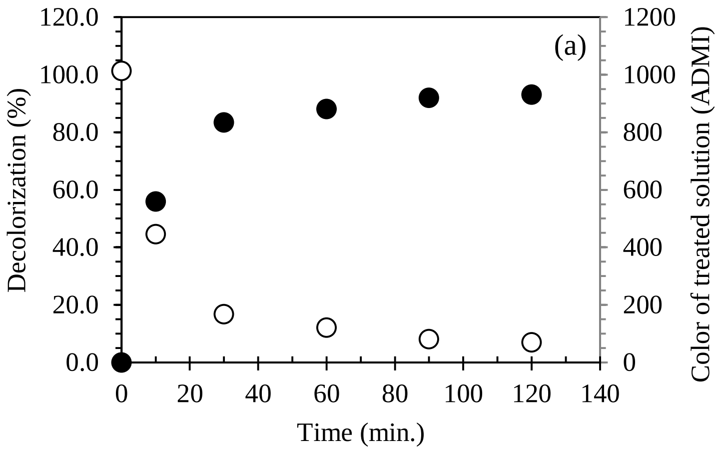
† samples prepared by dilution of the original aqueous solution of boric acid and borax with tap water to achieve 80 - 95 % of the original COD concentration.

Table 2 Box-Behnken design matrix and decolorization efficiency (%) using ozonation.

Experimental run	Coded			Uncoded			Decolorization (%)	
	x_1	x_2	x_3	Time. (min.)	pH	Influent COD (mg/L)	From experiment	From model
1	–1	0	+1	10	5.03	2,789	61.4	64.2
2	0	0	0	60	5.04	2,662	91.0	91.2
3	0	0	0	60	5.04	2,662	91.2	91.2
4	+1	0	+1	120	5.04	2,789	99.1	97.0
5	0	-1	+1	60	3.03	2,789	95.0	95.3
6	–1	+1	0	10	8.00	2,662	63.7	62.9
7	+1	0	–1	120	5.03	2,205	99.4	98.6
8	0	+1	–1	60	8.01	2,205	92.8	92.8
9	–1	-1	0	10	3.03	2,662	74.2	72.0
10	0	0	0	60	5.04	2,662	91.4	91.2
11	+1	–1	0	120	3.03	2,662	99.5	101.0
12	0	–1	–1	60	3.04	2,205	96.7	97.1
13	+1	+1	0	120	8.02	2,662	99.2	100.8
14	0	+1	+1	60	8.02	2,789	90.9	90.1
15	–1	0	–1	10	5.03	2,205	66.5	66.9

Wood treatability study

Dipping, which consists of immersing wood in a preservative solution for a specific time, is applied to demonstrate the possibility of recycling boric acid-borax solutions that were treated with ozone as a wood preservative solution. Sets of sawn rubberwood billets with dimensions of 100×250×20 mm^3 were submerged in a bath of the ozonated boric acid-borax solution and allowed to soak for 3 days under ambient conditions (30 - 35 °C) in the laboratory. Control experiments were conducted using (a) a freshly prepared boric acid-borax solution (1:1 ratio) and (b) the original solution received from the factory as wood preservatives. All tested solutions had a Boric Acid Equivalent (BAE) of ~0.9 % (w/w). After the specified time, billets from each bath were air-dried. Photos of the end grains and sides of the wood were taken to compare colors. %BAE was tested for acceptable wood preservative levels by the curcumin/salicylic acid color test defined in Thai Industrial Standard (TIS 2423-2552) [32].



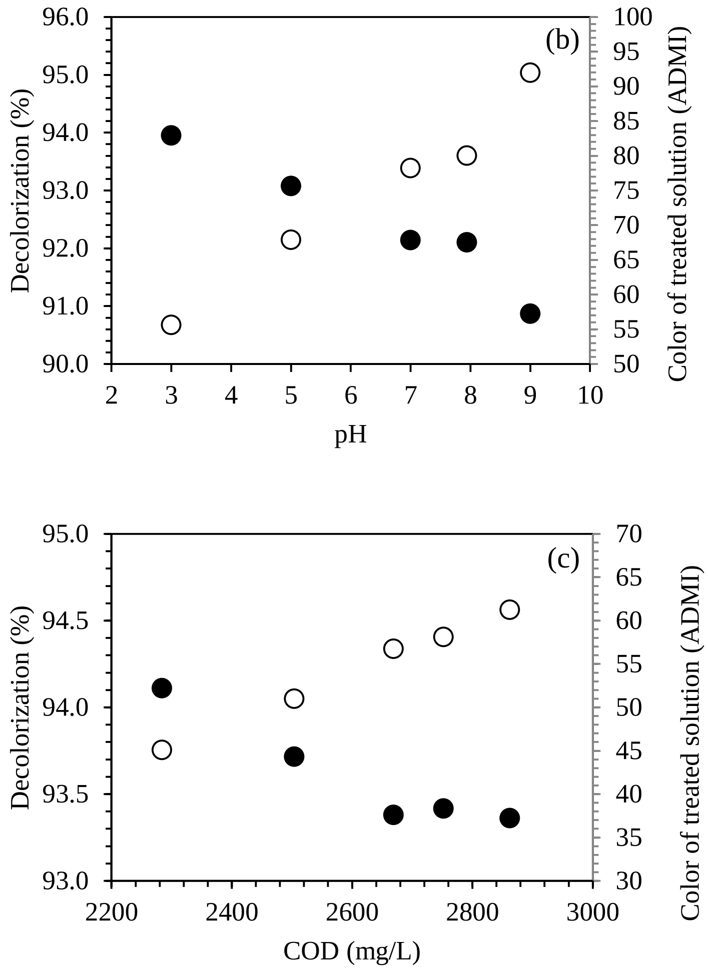


Figure 1 Effects of (a) ozonation time, (b) pH, and (c) initial COD concentration on color removal efficiency (●) and color values in ADMI (○) of the ozonated boric acid-borax solution.

Results and discussion

Decolorization of the used boric acid-borax solution by ozonation

Effect of ozonation time

Figure 1(a) shows the effect of ozonation time on color removal efficiency and color characteristics of the treated solutions. The color removal efficiency increased with the increase in the ozonation time applied to the solution, which subsequently led to an increase in ozone concentration in the liquid phase. It should be noted that more than 50 % of the color had been removed within 10 min. After 90 min, the color did not significantly reduce further, and by 120 min around 92 to 93 % of the color had disappeared. Color of the treated solution reduced from 1,013 ± 6 ADMI to 69 ± 3 ADMI after 120 min, suggesting that ozonation is a promising process for decolorization of boric acid-borax solution that was repeatedly used.

Effect of pH

The ozonation of the repeatedly used boric acid-borax solutions at various initial pH values (3 - 9) was examined. The selected pH values represent acid, neutral, and alkaline regions which influence the mechanism of the reaction and the types of created products as reported in the work of Sumegová *et al.* [21]. **Figure 1(b)** shows that ozonation in acidic conditions is slightly more efficient than in neutral and alkaline conditions with respect to decolorization efficiency. For example, after 90 min of ozone treatment, 94.0 % color removal was achieved at pH 3, while about 92.1 and 90.9 % color removal was obtained at pH 7 and pH 9, respectively. Thus, low initial pH enhanced color removal with ozonation.

Effect of initial COD concentration of the used boric acid-borax solution

The effect of the initial COD concentration of the used boric acid-borax solution on color removal efficiency with ozonation for 90 min at pH 3 was investigated. A decreasing trend in decolorization efficiency with an increase in initial COD concentration as shown in **Figure 1(c)** was observed. Since a constant ozone dose was applied in each batch experiment, a higher initial COD concentration resulted in a decrease in the ratio of ozone to organic molecules in solution leading to insufficient ozone to oxidize the organic molecules. Hafeez *et al.* [33] reported that higher ozone flow enhanced color removal, but our experiments were limited to the ozone generator that operates at a fixed flow rate of air (3.37 ± 0.13 L/min).

Optimization of process variables by response surface methodology (RSM)

A mathematical model to describe the relationship between process variables and the response, as well as optimum conditions for ozonation was investigated and determined by Response Surface Methodology (RSM). Box Behnken design (BBD) is selected to generate experimental plans for 3 levels (–1, 0, and +1) as shown in **Table 2**. Three operational variables including ozonation time (x_1, 10 - 120 min.), solution pH (x_2, 3 - 8), and initial COD concentration (x_3, 2,284 - 2,752 mg/L) were independent parameters of decolorization efficiency. Results from the decolorization of the colored boric acid-borax solutions by ozonation at various experimental conditions (15 runs) are shown in **Table 2**.

Model determination and significance of RSM model

A model suitability analysis was performed to explain the change in the decolorization efficiency resulting from ozonation at different lengths of time (min), solution pH, and initial COD concentration. The relationship between 3 independent variables and the response was analyzed using 4 models: (1) Linear model; (2) Linear + square model; (3) Linear + interaction model; and (4) Quadratic model as shown in **Table 3**. The relationship between color removal efficiency by ozonation (Y, %) and the input variables is best described by a full quadratic model because the model has the lowest standard error of the estimate (S), and the highest values of the coefficient of determination (R^2), as well as adjusted-R^2 as shown in **Table 3**.

Table 3 Standard error of estimate (*S*), determination coefficient (R^2), and adjusted determination coefficient (Adj-R^2).

Model	***S***	***R^2(%)***	**Adjusted-R^2 (%)**
Linear	7.12	78.92	73.17
Linear + square	2.37	98.30	97.02
Linear + interaction	8.17	79.78	64.61
Full quadratic	2.19	99.08	97.43

Using the experimental data as shown in **Table 2**, the following 2nd-order polynomial model was generated as shown in Eq. (2):

$$Y = 30 + 0.645x_1 - 6.77x_2 + 0.047x_3 + 0.01621x_{12} \\ +0.000019x_{13} - 0.00029x_{23} - 0.003699x_1^2 + 0.504x_2^2 - 0.00001x_3^2 \quad (2)$$

This full quadratic model yields an R^2 value of 0.9908, which indicated that only 1 % of the total variation can't explain by the model. Moreover, the contribution of each variable to the response is demonstrated in Eq. (2). Ozonation time (x_1) has a positive linear contribution for decolorization, whereas the pH of the solution (x_2) exhibits a strong negative contribution for color removal efficiency in this study. A positive contribution is also shown by initial COD in influent (x_3) and the interaction of x_{12}, as well as x_{13}, but with low intensity. The quadratic effect of pH (x_2^2) shows a high positive in comparison to x_1^2 and x_3^2 which have a low negative contribution to the response.

The model-predicted values of decolorization were calculated under the tested experimental conditions, and the results are shown in **Table 2**. The experimental values and the model-predicted values of decolorization are plotted in **Figure 2**. The correlation coefficient, R^2, has a value of 0.987 explaining the goodness of fit of the model. Joglekar and May [34] suggest that R^2 should be at least 0.8 for a good fit. Hence the model-predicted values of color removal efficiency by ozonation are in close agreement with the experimentally observed values.

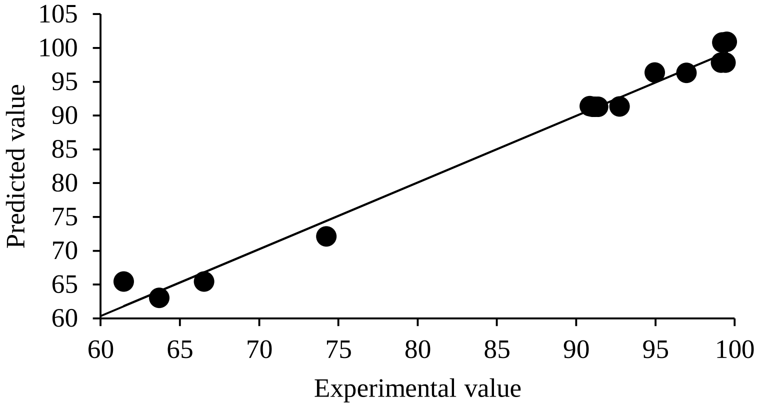


Figure 2 A plot of the measured and the model-predicted values of decolorization (%).

The significance of the RSM model is evaluated from ANOVA results as shown in **Table 4**. The Model *F*-value of 60.82 along with a low probability value ($p < 0.001$) implies that the model is statistically significant. In addition, the significance of each regression coefficient is also demonstrated in **Table 4**. In general, larger *F*-values and smaller *p*-values indicate more significant coefficient terms [35]. Based on the ANOVA results in **Table 4**, ozonation time and solution pH are statistically significant both in the linear term (x_1, x_2) and the square term (x_1^2, x_2^2). In addition, the interaction between ozonation time and solution pH (x_{12}) appears to have a small effect on the decolorization efficiency. Whereas both the linear term and the square term of initial COD in influent (x_3, x_3^2) as well as the other interaction terms (x_{13}, x_{23}) had no significant effect, with *p*-values higher than 0.05.

Table 4 ANOVA results for the acquired model.

Parameters	Sum of squares	DF	Mean square	F	*p*-value
Model	2,620.28	9	291.14	60.50	<0.001
x_1: ozonation time	1885.56	1	1,885.56	391.84	<0.001
x_2 solution pH	35.63	1	35.36	7.35	0.042
x_3: initial COD	10.51	1	10.51	2.18	0.200
x_{12}	20.15	1	20.15	4.19	0.096
x_{13}	0.44	1	0.44	0.09	0.774
x_{23}	0.09	1	0.09	0.02	0.898
x_1^2	455.25	1	455.25	94.61	<0.001
x_2^2	33.85	1	33.85	7.04	0.045
x_3^2	1.03	1	1.03	0.21	0.663
Residual error	24.06	5	4.81		

R^2 : 0.9909, Adj-R^2 : 0.9745

Individual and interactive effect of modeled parameters on decolorization efficiency

Using Minitab software, regression model-based contour plots were constructed for different pairs of variables holding the remaining variables at the central level (60 min, pH 5.0, and initial COD concentration of 2,662 mg/L). It is evident from **Figures 3(a)** and **3(b)** that initial COD concentrations between 2,205 mg/L and 2,789 mg/L have the least effect on the color removal efficiency compared to ozonation time and pH**.** Moreover, all 3 process parameters have little interaction with each other since the contours in **Figure 3** were almost parallel to the y-axis. Ozonation time has a positive effect on decolorization as can be seen from an increase in decolorization efficiency from 70 to 95 % with an increase from 10 to 120 min (**Figure 3(a)**). Meanwhile, pH had a negative effect on decolorization efficiency (**Figure 3(b)**). Those observations agree with the prediction of RSM as demonstrated in Eq. (2). However, maximum decolorization efficiency can be obtained at higher pH when longer ozonation times are used (**Figure 3(c)**).

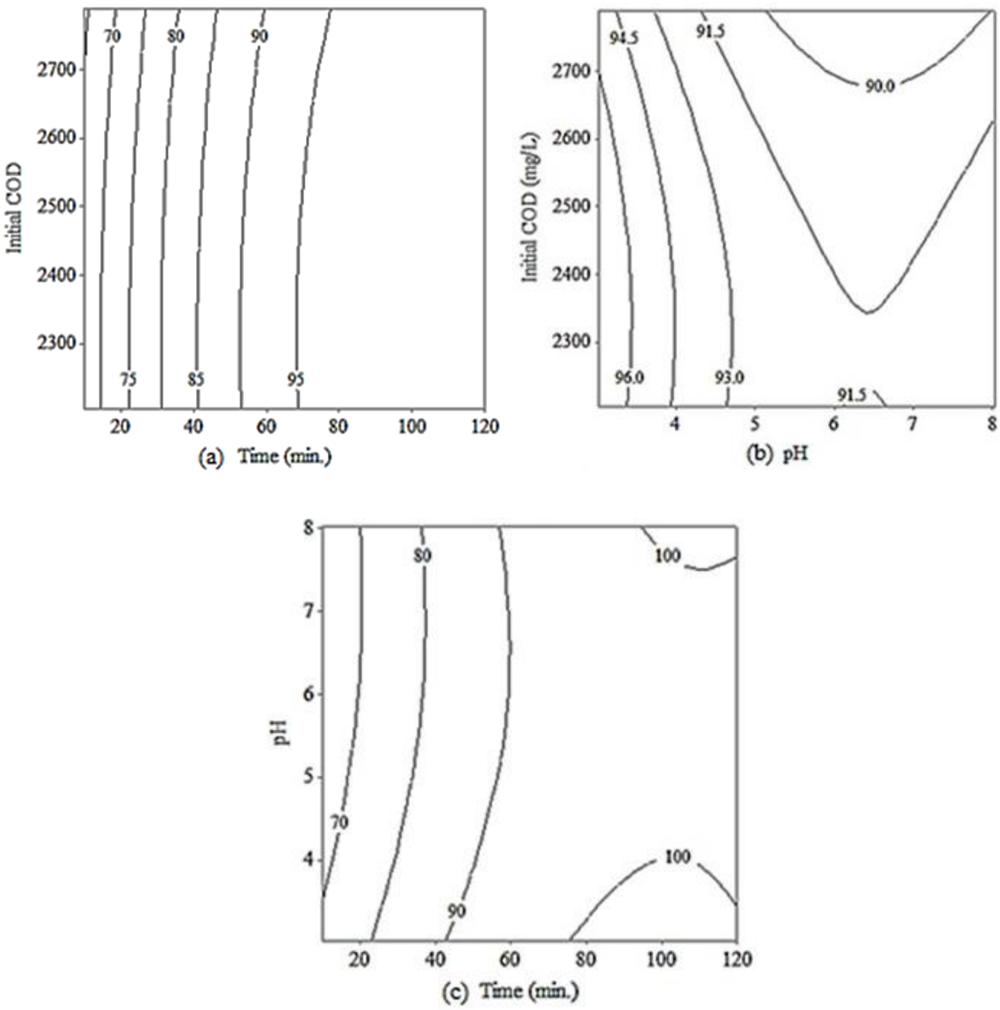


Figure 3 Contour Plots on decolorization (%) with respect to interaction effects of operational parameters: (a) initial COD and ozonation time, (b) initial COD and pH, (c) pH and ozonation time.

Condition optimization and confirmation test

The response optimizer function in the response surface designs of Minitab software was used to find the optimal conditions for decolorization of the used boric acid-borax solution by ozonation. The optimal values were found to be ozonation time 65 min, solution pH 3.59 ± 0.02 with initial COD 2,250 ± 40 mg/L, with a predicted decolorization efficiency of 97 %. Verification experiments showed a decolorization efficiency of 96.5 ± 0.6 % (average of 3 runs) indicating that the full quadratic model, set out in Eq. (2), was accurate and reliable for predicting the decolorization efficiency of the repeatedly used boric acid and borax solution by ozonation. Therefore, the proposed model can be properly used to predict the decolorization efficiency for various process parameters at any point in the design space.

Reuse of boric acid-borax solutions after ozonation

The color of the end grain sawn rubberwood billet after treatment with the ozone-treated borates solution was quite like that of the block immersed in the freshly prepared 1:1 ratio of boric-borax solution (**Figure 4(a)**). In contrast, the color of the sawn rubberwood billet treated with the repeatedly used boric acid-borax solution was darker than the others. Similar results are observed when comparing color from the side view of sawn rubberwood billets (**Figure 4(b)**). Treatment with ozone does not affect the penetration of the boron-based preservative solution as shown in **Figure 5**. Regardless of the types of the boric acid-borax solution: The color on the side-view of sawn rubberwood samples is red after testing with the curcumin/ salicylic acid method for boron determination [32] implying successful penetration of the preservative solution to the grain of wood samples. Wood quality, in terms of color appearance, was not affected by reuse of the boric acid-borax solution treated with ozonation.

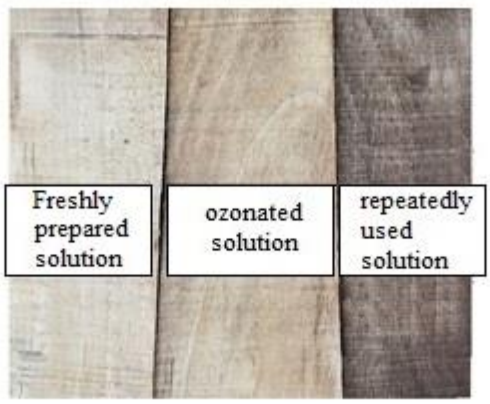


Figure 4 Color characteristic of (a) the end grains and (b) the side-view of sawn rubberwood billets after submersion in a bath of 3 different types of boric-borax solution.

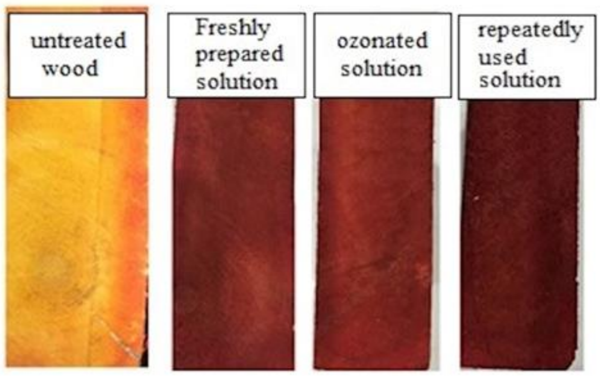


Figure 5 Wood color after qualitative determination of boron by the curcumin/salicylic acid color test.

Conclusions

Ozonation was shown to be effective to remove the color of boric acid-borax solutions allowing the solution to be recycled as wood preservatives without degrading the economic value of the preserved timber. A Box-Behnken design with the Response Surface Methodology was successfully applied to find the optimum conditions for decolorization of the boric acid-borax solution by ozonation. The proposed full quadratic model is accurate and reliable to describe the relationship between decolorization efficiency and operational parameters including ozonation time, initial pH of the solution, and initial COD concentration, as well as to predict the decolorization efficiency for various process parameters at any point in the design space. The optimal conditions for decolorization of the repeatedly used boric acid-borax solution were found to be as follows: 65 min ozonation and solution pH 3.59 ± 0.02 with initial COD of 2,250 ± 40 mg/L. Color removal of 96.5 ± 0.6 % was obtained under the optimal conditions. Results from the wood preservation process showed that the color of the end grains and the side-view of wood samples being treated with the ozonated boric-borax solution were close to wood samples that are treated using a freshly prepared boric-borax solution. Moreover, successfully boron penetration into the wood that was treated with the ozonated solution was observed. To our knowledge, this is the 1st study using ozonation to treat the colored borax/boric acid mixture for reuse in the preservation of sawn rubberwood: Ozonation not only maintains wood quality but also reduces the volume of wastewater that must be processed before releasing to the environment.

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