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SULFIDE REMOVAL BY CATALITIC OXIDATION FROM RAYON WASTE

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KEYWORDS
Sodium Hydrosulphide (NaHS); Potassium Permanganate Titration; Catalytic Oxidation; Hydrogen Peroxide (H_2O_2)

ABSTRACT

Catalytic oxidation of spent sulfidic caustic consist of SH^- ion or NaHS compound by H_2O_2 in neutral or acidic solution to elemental sulphur may provide a convenient and economical method for the control of sulphide wastes and their associated odors at pulp, paper and textile industry. Oxidation of sulfide in rayon waste by hydrogen peroxide was investigated in the presence of ferric sulfate catalyst. Kinetic equations and activation energies of H_2O_2 and SH^- ion to total sulphur and sulphate in rayon waste for catalytic oxidation reaction were calculated based on the experimental results. For the removal of sulfide from sulfide solution the most common process involves its catalytic oxidation to a more benign form sulfate. The rate of sulfidic catalytic oxidation was found higher at lower initial sulphide concentration and the rate of sulphide catalytic oxidation was found directly proportional to loading and hydrogen peroxide addition. Optimum total sulphide concentration was achieved when sulphide solutions in the presence of H_2O_2 in the ratios SH^-/H_2O_2 1:4.2. The potential user of H_2O_2 determine the optimal conditions for control of odor, corrosion and waste treatment cost due to sulfide consisting of sulphur ion, sulphate ion, etc. The catalytic oxidation of sulphides into sulphates by H_2O_2 may be applied directly to aqueous wastes containing these odorants

INTRODUCTION

The industrialization process also encourages the rayon industry to produce chemical waste containing a mixture of inorganic and organic components in large quantities which is burdening the environment. Waste reduction is driving the chemical industry towards clean technology as a secondary resource. Waste processing in the rayon industry is carried out appropriately to generate secondary waste resources while the mixing of several compounds in the waste stream provenience from liquid-liquid contact between NaOH and H_2S. Immediately, sodium hydroxide the majority react with H_2S becomes sodium hydrosulphide or sodium waste as spent sulfidic caustic (SSC)[2][3]

Sodium sulphide (Na_2S) and sodium hydrosulphide (NaHS) are reaction products of the scrubbing H_2S with NaOH solution by the Eq. (1), (2), and (3). Sodium hydrosulphide can occur as predominant sulphide species in waste, therefore the reaction product is influenced by the pH of the spent NaOH solution is required. Once, the pH of Eq 1 is generated at pH > 12, when the pH ease around 11.5, NaSH is formed. The following equations describe the scrubbing reaction of [3][4][5][6]H_2S and NaOH solution:

$H_2S + 2\,NaOH \rightarrow Na_2S + 2\,H_2O$ (1)

$H_2S + NaOH \rightarrow NaSH + H_2$ (2)

$H_2S + Na_2S \rightarrow 2\,NaSH$ (3)

Sulphur compounds as SH^- ion at spent sulfidic caustic can be removed through wet air catalytic oxidation, fenton's catalytic oxidation with precipitation using a mixture of ferrous and ferric salts, chemical catalytic oxidation with catalytic or non catalytic with hydrogen peroxide, catalytic oxidation of H_2S and Na_2S in aqueous solution by dissolved oxygen, incineration with auxiliary fuel, and biological catalytic oxidation of sulphide. However, these methods of treatment are expensive, using a mixture of ferrous and ferric salts is competitive process, and induce secondary pollution problems.[2], [6][13]

For the reduce of sulphide from waste and wastewater the most prevalent process involves its catalytic oxidation to a more benign form sulphate. In the chemical [14][24] catalytic oxidation method, ions SH^- are converted into sulphate (SO_4^{2-}) species , with the oxidants are hydrogen peroxide, sodium hypochlorite, potassium permanganate, chlorine and ferrate (VI) ions. The [6][25] catalytic oxidation of sulphide using hydrogen peroxide at low temperatures that the decomposition of sulphide was five times faster at 258 K than at room temperature.[26]

Sulfide oxidation is chemically believed to be a chain oxidation reaction so that the reaction becomes very sensitive to the reaction conditions. Chen and Morris found some correlation between the length of the induction period and the initial oxidation rate that the length of the induction period decreased with increasing the initial oxidation rate. Stoichiometry of chemical sulfide oxidation occurs very complex because various products and intermediates will be generated during the reaction process. The main intermediate products identified are sulfur elements (S^0), thiosulfate ($S_2O_3^{2-}$), sulfite (SO_3^{2-}) while sulfate (SO_4^{2-}) is the result of the reaction. The formation of intermediate products and formed products is described in the following reactions:[27]

$2HS^- + O_2 \rightarrow 2S^0 + 2\,OH^-$ (4)
$2HS^- + 2O_2 \rightarrow S_2O_3^{2-} + H_2O$ (5)
$2HS^- + 3O_2 \rightarrow 2\,SO_3^{2-} + 2H^+$ (6)
$2HS^- + 4O_2 \rightarrow 2\,SO_4^{2-} + 2H^+$ (7)

The sulphide concentration may not be estimated in a redox system between oxidants (H_2O_2) and reductants (S^{2-}) own time. The total sulphide be necessary analyzed to determine concentration of sulphate and reduction of sulphide in sulphide waste. The analysis was carried out by reacting the sulphide waste after being oxidized, with a solution of potassium permanganate as the reaction below:[28]

$5\,NaHS + 8\,KMnO_4 + 7\,H_2SO_4 \rightarrow 4\,K_2SO_4 + 8\,MnSO_4 + 7\,H_2O + 5\,NaOH$(8)

The initial concentration of H_2O_2 was determined by titration method using potassium permanganate solution. The H[31], [32] 2 O2 titration method using potassium permanganate solution with the following reaction equation :[29]

$5\,H_2O_2 + 2\,KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8\,H_2O + 5O_2$ (9)

The objective of this study is to investigate the effect of two experimental variables viz. the initial sulphide waste concentration and ratio $NaHS/H_2O_2$, which may affect the total sulphide concentration and considerable sulphide to sulphate during hydrogen peroxide catalytic oxidation of NaHS onto subsequent synthesis of NaSH. The effect of time, pH, temperature, total solid, and density were examined in order to use as indicators for specify of the reaction end point during industrial operation. Besides, the hydrogen peroxide and initial sulphide waste concentration was analyzed by potassium permanganate titration to examine the initial condition and the optimum catalytic oxidation condition.

RESEARCH METHOD

Materials and Reagents

The sulphide waste used in this study was collected from PT Rayon Utama Makmur, a synthetic textile and rayon industry located in Sukoharjo, Indonesia. It had a pH value of 11.75 (base), contained COD of 476,72 kg/m3. Another material used was hydrogen peroxide, sulphuric acid, distilled water, potassium permanganate and sodium oxalate, all of the material was provided by Merck.

Materials Standardization and Initial Condition

The chemicals for standardization and initial condition of waste used included potassium permanganate ($KMnO_4$) 0.25 N, sulphuric acid, sodium oxalate and distilled water. It was heated for 1 or several hours near the boiling point, then filter and standardize with sodium oxalate. The sodium oxalate 0.1 N was prepare in 100 mL. The sulphuric acid 1M making up to 500 mL and allowed to cool at room temperature. Titrate rapidly with the $KMnO_4$ solution to be standardized, while stirring, to a slight pink end-point color that persists for at least 1 min[31] .

Total Catalyst

Important parameters in determining the degree of sulfide oxidation are sulfide concentration, H_2O_2 concentration and catalyst addition. [32]Iron (III) sulfate ($Fe_2(SO_4)_3$) is taken as the starting material for catalysts for catalytic oxidation processes. The solids are then dried in the oven for 30 minutes at a temperature of 110 ± 5oC. Solids formed as a result of drying are then stored as catalysts for the oxidization process. The mass of the catalyst is calculated based on the mass of H_2O_2 used during the reaction which is 1% of the mass of H_2O_2.

Total Sulphide Analysis

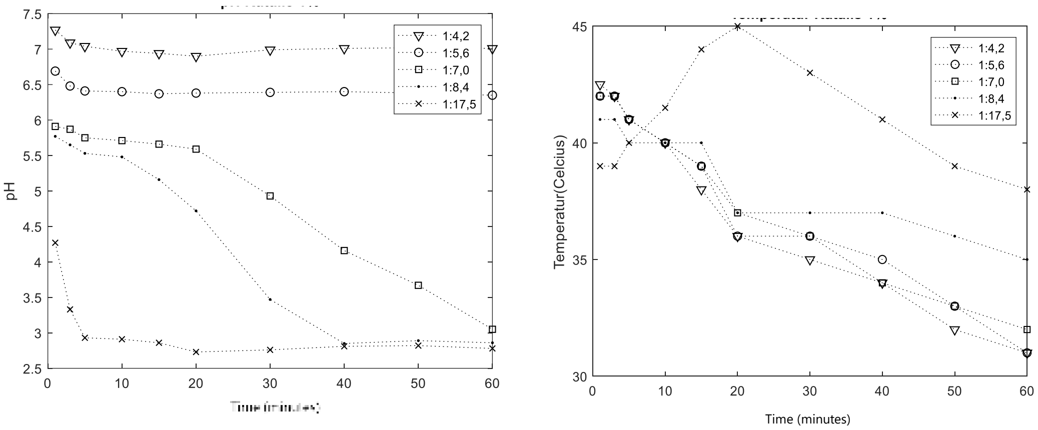
The procedure of total sulphide analysis was put 4 mL of working solution and 16 mL H_2SO_4 1 M into a 100-mL erlenmeyer flask and heat rapidly below 90°C. Titrate rapidly with the $KMnO_4$ solution to analyzed, while stirring, to a slight pink end-point color that persists for at least 1 min.

RESULT AND DISCUSSION

pH and Temperature Profile

The catalytic oxidation reaction is a pH and temperature suspended process in that pH appears an important role in the decomposition of sulphide to sulphate generation in the catalytic oxidation reaction. The effect of catalytic oxidation pH value of sulphide waste on the total sulphide removal by catalytic oxidation was studied in the pH range of 4 to 7.5 by adding H_2O_2 as proceeds catalytic oxidation process. The experiment was conducted to investigate the affect of pH and temperature on the degradation sulphide concentration process efficiency and find the trend of temperature and optimum pH catalytic oxidation. The results obtained are presented in Figure 1 showing the profile of time and pH during catalytic oxidation of sulphide, and Figure 1 showing that time and temperature at catalytic oxidation. As shown in Figure 1, the experiment at ratio 1:4.2 have pH 7.01 to 7.27 with initial ratio 1:4.2 has the best stable pH value. For the catalytic oxidation reaction, a decrease in pH occurs with an increase in the ratio of H_2O_2/NaHS and an increase in reaction time.

The pH value of 7.27 gives the highest result of all variations for the addition of 1% catalyst, namely in the 1st minute the ratio is 1:4.2 while the lowest pH value is known to be 2.73 obtained in the 20th minute the ratio is 1:17.5. The ratio 1: 4.2 have best trend temprature degradation from 42 to 31oC, which runways reaction has been controlled.



(a) (b)

Figure 1 Relationship between (a) time and pH and (b) time and temperature during catalytic oxidation of sulphide

Effect of Sulfide Removal Efficiency on Oxidation Reaction Rate

The sulfide concentration measured through the ratio of H 2O2/NaHS ratio was used as the inlet concentration of sulfide in the reaction. The measured sulfide concentration through the outlet of the batch reactor is used as the outlet concentration or total sulfide concentration. Sulfide removal efficiency in this study is defined as the conversion of reactions that take place between sulfide waste and hydrogen peroxide where the removal efficiency is a reduction of the initial amount of sulfide in the reaction with the final amount of sulfide at the time the reaction has been running for one hour. The efficiency of sulfide removal can be calculated through the following equation:

$$\%\, \boldsymbol{removal\ sulfide} = \frac{\boldsymbol{C_{SH^-\,in} - C_{SH^-\,out}}}{\boldsymbol{C_{SH^-\,in}}} \times \mathbf{100\%} \qquad \textbf{[1]}$$

Figure 2 Reaction removal efficiency profile every time for 1% addition of catalyst

Reaction with the addition of a catalyst reaction speed reaches the optimum point at the 10th minute by 79% for a ratio of 1: 4.2 and 80% for a ratio of 1: 8.4. Based on the exposure for each concentration and temperature shows the range of magnitude of the resulting reaction time with the maximum conditions for the removal percentage plot to be in minutes 15th. The tendency of the flattening curve characterized by the reaction time range is what will provide an outline of the clues to the location of the optimum point of the sulfide oxidation reaction time.

Table 1 Reaction order profiles and reaction speed constants and reaction removal efficiency during catalytic oxidation processes

H_2O_2/NaHS ratio	Temperature		Ph		p_1	p_2	k*	Removal Efficiency		SSE
	1'	60'	1'	60'				1'	60'	
4,2	43	31	7.27	7.01	2,99	1.9 3	35. 282	0.7 7	0.8 1	0.04
5,6	42	31	6.69	6.35				0.77	0.81	0,10
7,0	42	32	5.91	3.05				0.7 8	0.82	0,13
8,4	41	35	5.77	2.86				0.78	0.83	0,14
17,5	39	38	4.27	2.78				0.79	0.84	0,18

*k = (mol/L)1-n $^{\text{minute-1}}$ **with** n = $p_1 + p_2$

The obtained SSE value is displayed in the Table with a value less than 1 so that the SSE value is assumed to be close to zero or has reached the minimum value with a range of SSE values from 0.04 - 0.1 8. The calculated SSE deviation value is a relative error value in the simulation carried out as a summation of the deviation of the research data conversion and the conversion of the fitting results carried out . Based on the table of the smallest SSE values, namely at a ratio of 1: 4.2 with an SSE value of 0.04, this can be seen from the profile of the research data and fitting results in the Figure that is not much different although in other ratios there is no visible deviation that has an effect.

Total Sulphate Concentration Profile

In the operation phase the amount of sulfide eliminated cannot be released from the batch reactor in a short reaction time because it can lead to further oxidation of the sulfide to sulfate. Therefore, the sulfate concentration is relatively high during the initial reaction operation phase and increases gradually with an increase in reaction time.

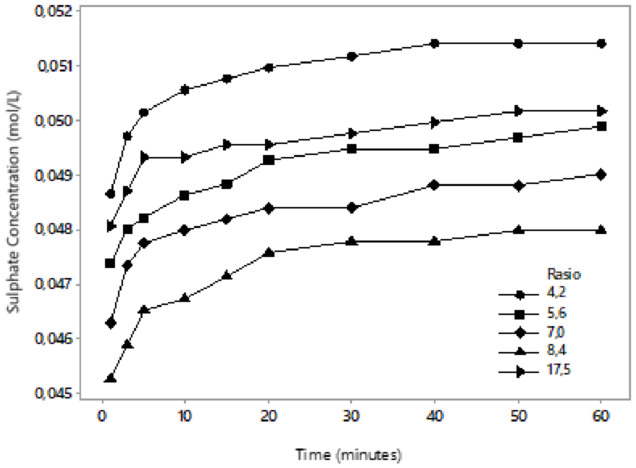


Figure 3 Profile of total sulfate resulting from catalytic oxidation reaction to reaction time

Based on Figure 3, it is known that the total sulfate profile resulting from the catalytic oxidation reaction shows a curve that increases with the addition of the reaction time and the addition of the amount of H 2 O2 to the solution. The graph of the sulfate concentration against the reaction time at concentrations of 1:4.2 and 1:5.6 shows the slope result close to zero so that the curve tends to be parallel to the addition of the reaction time. The addition of hydrogen peroxide concentration does not provide fluctuating increase in sulfate formation. The graph of the total sulfate fitting increases during the catalytic oxidation process.

Total Sulphide Concentration Profile

The reaction order and reaction speed constant resulting from the fitting as well as the reaction removal efficiency during the oxidation process for catalytic reactions are carried out at various H 2 O2/NaHS ratios. In redox systems oxidants in the form of H 2 O2 and SH− reductors will be present together in solution so as to estimate the overall sulfide concentration cannot be separated over time. Therefore, in this study determined the kinetic constant of the reaction rate, the concentration of the initial sulfide, hydrogen peroxide and catalyst was studied and interconnected with each other.

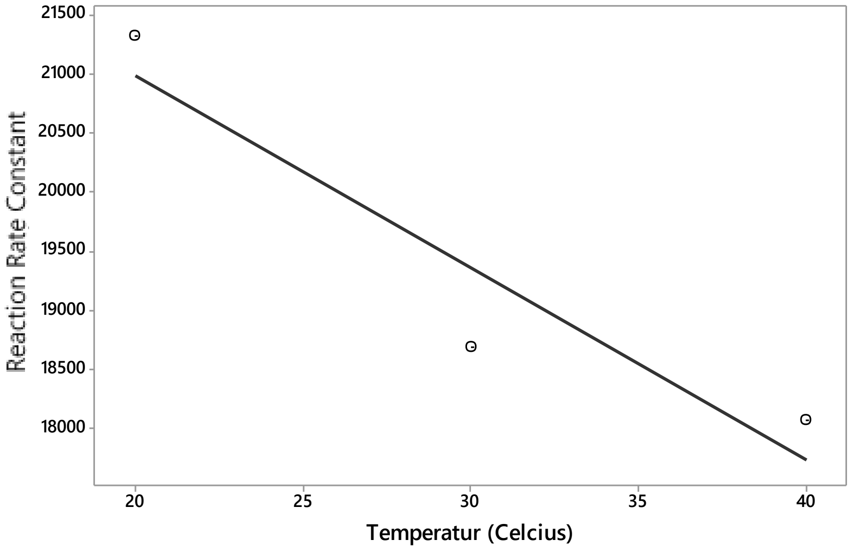


Figure 4 Profile of reaction speed constant to initial sulfide concentration for non-catalytic isothermal reactions

It is generally found that the oxidation of chemical sulfides proceeds very slowly from the order of the 15th minute until the end of the reaction. Based on the research conducted by Chen and Morris it was found that the overall reaction order is 1.9 with orders of 1.34 and 0.56 respectively for total sulfide and oxygen. According to studies conducted by O'Brien and Birkner it is known that the reaction order for sulfides in total is 1.02 and for oxygen 0.80. Based on the exposure of Cline and Richards who simulated data with kinetic rate equations obtained the order of reaction for each reactant is 1. In this study, it was reported that the reaction order for sulfides was 1.93 – 2.99.[33][34][35]

CONCLUSION

The sulphide concentration removal in an aqueous solution was accomplished by chemical catalytic oxidation process. From the present work following conclusions can be drawn: (1) rate of sulfide catalytic oxidation was found higher at considerable of ratio NaHS/H 2 O2 sulfide concentration; (2) rate of sulfide catalytic oxidation was immediately compatible to catalyst loading reaction and moles ratio of hydrogen peroxide addition; (3) at uncontrol temperature rate of sulfide catalytic oxidation by hydrogen peroxide rise to a big extent; (4) Sulfide reduction in rayon waste is also influenced by reaction and catalyst temperatures where the temperatures applied in this study are 20 o C , 30 o C and 40o C and the mass of the catalyst used is 1% of the amount of H 2 O 2 ranging from 0.21 – 0.89 gr Fe2(SO4)3.

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