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Kinetic Modeling of Humic Acid Production from Oxidized South Sumatera Lignite Toward Green and Sustainable Synthesis

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Abstract

This study investigates the reaction kinetics of hydrogen peroxide (H_2O_2) oxidation of low-rank coal (lignite) to produce humic acid. Utilizing H_2O_2 as a green oxidant enhances the environmental compatibility of the process, aligning with Sustainable Development Goals of responsible consumption and production. Scaling up laboratory processes requires practical insights into kinetic parameters that are essential for designing process equipment. The oxidation of coal with H_2O_2 is solid-liquid reaction, typically governed by three steps to control the process: diffusion through a thin film layer, diffusion through the solid state, and chemical reaction. Despite its potential, research on the kinetics of chemical oxidation in lignite remains relatively limited. In this study, coal particles (-60 +80 mesh) are oxidized using 10% H_2O_2. At specific time intervals, a certain volume of the solution is taken and measured through titration. The evaluation of kinetic models involves liquid film diffusion (sphere), Jander diffusion (three dimensional), ash diffusion, first order, second order, and surface reaction. The result shows that the oxidized coal demonstrates the addition of carboxyl groups. The kinetics model highlights the surface reaction as the controlling process. The estimated activation energy stands at 29.80 kJ/mol, with an Arrhenius constant of 121.10 min^{-1}. This study suggests the possibility of conducting the reaction at room temperature.

Keywords: Fulvic acid; humic acid; kinetic modeling; lignite; oxidation.

1. Introduction

Indonesia possesses a substantial total coal reserve of 39,891 million, but approximately 29.40% of this reserve is categorized as low-rank coal, including sub-bituminous and lignite coal, characterized by low economic value [1]. The utilization of this coal for direct industrial purposes is challenging due to its high-water content, low calorific value, and elevated volatile matter [2–4]. Notably, lignite, a type of low-rank coal, holds potential as a raw material for humic acid production. Research by Yan et al [5] has highlighted the ability to further oxidize lignite to synthesize humic acid. Humic acid, a beneficial chemical, finds applications in agriculture, such as enhancing water retention capacity in the soil, contributing to soil detoxification, and providing benefits to plant growth, including enzyme activity, root respiration, and resistance to abiotic stress [6].

According to previous studies [5,7–9], low-rank coal oxidation can be achieved through air and chemical oxidation. However, the application of air oxidation for humic acid synthesis was limited due to its high energy consumption. Recent studies have shown promising results in humic acid production through chemical oxidation using acid treatment [7]. The research group led by Fatima et al. successfully doubled the humic acid yield from low-rank coal (from 21.15% to 57.8%) by performing a nitric acid (HNO_3) oxidation pretreatment followed by extraction with KOH and NaOH [10]. HNO_3 is commonly utilized to alter the structure of coal in order to produce humic acid. However, the HNO_3 oxidation process reveal

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an issue for causing serious environmental problems [11]. Another study reported that introducing a hydrogen peroxide (H_2O_2) oxidation process increased the humic acid yield from 13.5% to 45.7%, a threefold improvement [12]. H_2O_2 oxidizes coal via reactive oxygen species (ROS), especially from hydroxyl species (•OH) [13]. Oxidation of carbon species in coal, introduces oxygen containing functional groups such as -COOH and -C=O. Skripkina et al. significantly increased the humic acid content from 20% to 70% through the oxidation of coal using sodium percarbonate ($Na_2CO_3 \cdot 1.5H_2O_2$) [14]. Humic acid derived from lignite is characterized by abundant aromatic functional groups, including carboxyl groups, phenolic acid, as well as methylene and ethylene connecting bonds [11,14,15].

Currently, designing green synthetic methodologies for oxidizing low-rank coal by H_2O_2 is essential to implement the sustainable and cleaner process to preserve the quality of environment [16]. As the demand for humic acid increases, there is a need to develop it into an industry. Consequently, laboratory processes must be scaled up, requiring practical information on kinetic parameters for the design of process equipment. In solid-liquid reactions, three steps control the process: diffusion through a thin film layer, diffusion through the solid state, and chemical reaction [17]. Among these steps, the slowest rate becomes the controlling factor. The exploration of the kinetics of chemical oxidation in low-rank coal is relatively scarce. To scrutinize the controlling step, the shrinking core model was employed. The models incorporated in this study encompass liquid film diffusion, first-order reaction, second-order reaction, surface reaction, Jander Diffusion, and ash diffusion.

The aim of this study is to investigate the reaction kinetics of H_2O_2 oxidation of South Sumatera lignite for the production of humic acid. This work focuses on evaluating kinetic models that describe the oxidation behavior and identifying the rate-controlling steps involved in the solid-liquid reaction system.

2. Materials and Methods

2.1. Materials

The lignite sample was collected from South Sumatera, Indonesia. The coal had a calorific value of 4,469 cal/gram, ash content of 1.34%, water content of 26.74%, volatile matter of 36.76%, and fixed carbon of 35.17%. The H_2O_2 (50%) was obtained from PT. Samator Intiperoksida, Indonesia.

2.2. Oxidation of low-ranked coal

The coal was crushed and sieved. The fine particles (-60 +80 mesh) were collected to be used as raw material. The oxidation reactor is carried out in a three-necked flask assembled with a heating mantle, condenser, and stirrer motor. The oxidation was conducted by the addition of 5 grams of coal into 500 mL of 10% H_2O_2 in three different temperatures (40°C, 50°C, and 60°C). In a specific interval of time, a small amount of the solution was taken and measured using titration. The acidic content of oxidized coal (C_A, mgeq/mL) was calculated using Equation (1), with N_{KOH} was the normality of KOH, while n, V_{sample}, and V_{KOH} were 100 (the diluted sample is 100 times), the volume of the diluted sample (10 mL), and the volume of titrant (KOH).

$$C_A = \frac{n.N_{KOH}.V_{KOH}}{V_{sample}} \tag{1}$$

The total acid of coal that can be converted was calculated theoretically using Equation (2), with m_c was the initial mass of coal, MW_A was the average molecular weight of acid, and V_{H2O2} was the volume of H_2O_2.

$$C_{C0} = \frac{m_c}{MW_A.V_{H_2O_2}} \tag{2}$$

The conversion (x) was calculated using Equation (3)

$$x = \frac{C_{A,t} - C_{A0}}{C_{C0}} \tag{3}$$

For further analysis, the raw and oxidized coal were scanned using SHIMADZU IR Prestige-21 in the range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} and the KBr Pellets method.

2.3. Reactions kinetics model

The shrinking Core Model (SCM) involves several steps as illustrated in Fig. 1. The first step is the diffusion of the liquid reactant through the thin film, then it is followed by the diffusion of the reactant through the ash layer, and finally the reaction between the fluid reactant and solid reactant [18]. The equations used in this study are described in Table 1. The particle was assumed to be constant in size. The models involved liquid film diffusion (M1), Jander Diffusion (M2), ash film diffusion (M3), first-order reaction (M4), second-order reaction (M5), and surface reaction (M6).

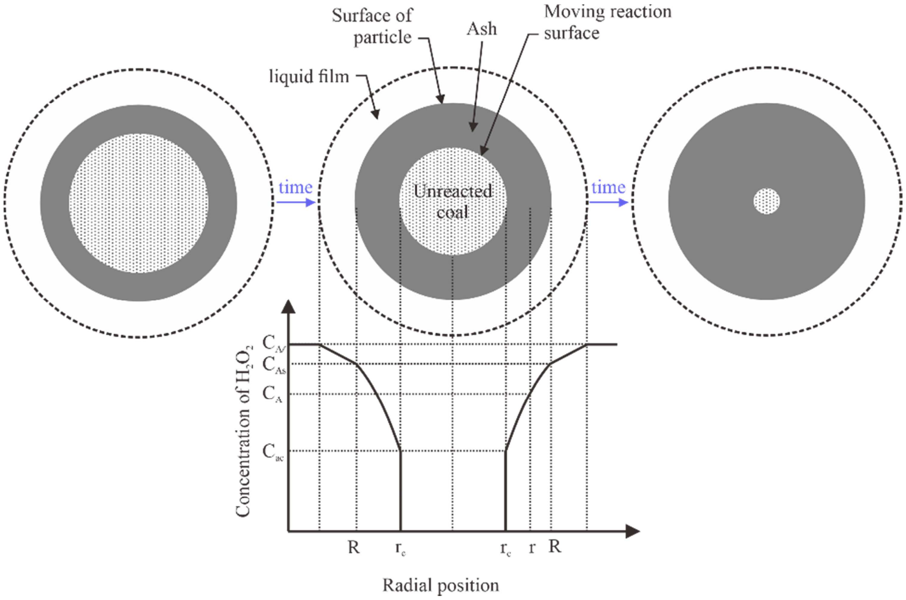

Fig. 1. Illustration of coal oxidation by H_2O_2 (solid-liquid reaction)

Table 1: Mathematical model involved in this study

Equation		Type	Denoted
Linear Form	Derivative Form		
$k_1 t = x$	$\frac{dx}{dt} = k_1$	Liquid film diffusion (sphere)	M1
$k_2 t = \left[1 - (1-x)^{\frac{1}{3}}\right]^2$	$\frac{dx}{dt} = \frac{3\,k_2\,(1-x)^{2/3}}{2\,(1-(1-x)^{1/3})}$	Jander diffusion (three dimensional)	M2
$k_3 t = 1 - 3(1-x)^{\frac{2}{3}} + 2(1-x)$	$\frac{dx}{dt} = \frac{k_3\,(1-x)^{1/3}}{2\,(1-(1-x)^{1/3})}$	Ash diffusion	M3
$k_4 t = -\ln(1-x)$	$\frac{dx}{dt} = k_4(1-x)$	First order	M4
$k_5 t = (1-x)^{-1} - 1$	$\frac{dx}{dt} = k_5\,(1-x)^2$	Second order	M5
$k_6 t = 1 - (1-x)^{\frac{1}{3}}$	$\frac{dx}{dt} = 3k_6\,(1-x)^{2/3}$	Surface reaction	M6

3. Results and Discussion

3.1. Effect of oxidation process on functionalities of coal

The FTIR scanning results of raw coal and oxidized coal are presented in Fig. 2. A broad peak appeared around 3422.69 cm^{-1} for both samples corresponding to the hydrogen bond of coal, associated with -OH or -NH stretching. The symmetric and asymmetric stretching of C-H bonds in the saturated hydrocarbon groups $–CH_3$ and $–CH_2$ occur at 2921.75 cm^{-1} and 2850.87 cm^{-1} respectively. The aromatic C=C bond in this study appears as sharp peaks at 1618.05 cm^{-1} compared to another study which appears at 1600 cm^{-1} [19]. According to the result, the intensity of $–CH_3$ for oxidized coal is higher than the raw coal, it may be attributed because the oxidation breaks the $CH_2–CH_2$ long-chain macromolecular into smaller molecules [20]. The result is corresponding to other researchers [20,21].

Introducing a new peak at 2360.54 cm^{-1} indicates the existence of CO_2 [22]. In another study, Li and Yuan explain that the oxidation process of low-rank coal using H_2O_2 produces CO_2 as the by-product [23]. The gas might be entrapped in the solid state when the reaction takes place. The peak at 1706.77 cm^{-1} is attributed to the introduction of the C=O bond of the carboxylic group or ester carbonyl group [8]. The existence of a C–O–C bridge is shown in the oxidized coal at wavenumber 1119.04 cm^{-1}.

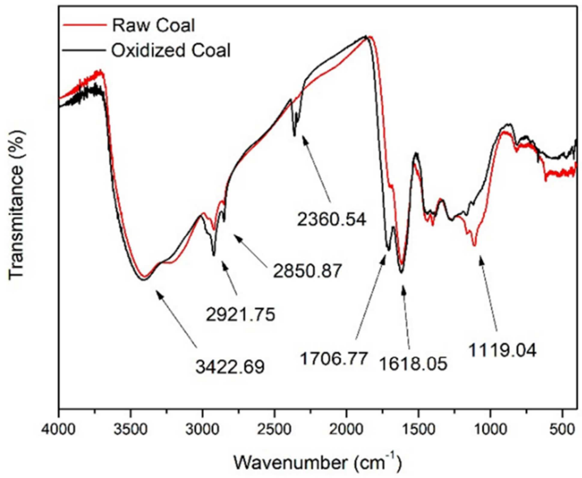

Fig. 2. FTIR spectra of raw coal and oxidized coal

3.2. Evaluation of kinetics model

The evaluation of kinetics models in this research is conducted using a temperature of 60°C. The kinetics constant is calculated by minimizing the sum of squares of errors on the time vs conversion and time vs first derivative, simultaneously. The constant results from each model are presented in Table 2 and plotted in Fig. 2. This study assumes that the amount of acid generated during the oxidation process is in proportionate with the reacted coal [23]. Therefore, the conversion is calculated using Equation 3. The shrinking core model (SCM) is also used to evaluate the controlling steps during the reaction. Previous study mentioned that SCM is usually used to evaluate solid-liquid reaction in many cases [17,18,24–26].

According to Fig. 3 and Table 2, the fittest model is obtained from the M6 kinetics model. It provides the lowest sum square of error (SSE) of both evaluations (*f(x)* and *df(x)*). It means that surface reaction becomes the controlling rate process. The diffusion of liquid through film and ash can be neglected as the particle size is small enough [19,23,27]. The reaction equation of coal and H_2O_2 can be expressed by Equation (4):

$$coal + H_2O_2 \rightarrow CO_2 + aromatic\ rings - COOH + small\ molecular\ acid \qquad (4)$$

In order to study the influence of reaction temperature on the oxidation of coal from South Sumatera, this study involved 3 different temperatures. The result shows that the conversion increased as the temperature is raised (Fig. 3) which is similar to Wang et al [19]. Kinetic energy increases as the temperature is raised, which leads to an increase in the probability of collision between reactant molecules. Therefore, the reaction is faster when the temperature increases. However, in this study, the reaction temperature is limited to 60°C. When the temperature exceeds 60°C, the reaction produces a significant amount of foam due to an excessive amount of carbon dioxide (CO_2). The maximum conversion was obtained at 60°C and 160 min with a conversion of 75.38%.

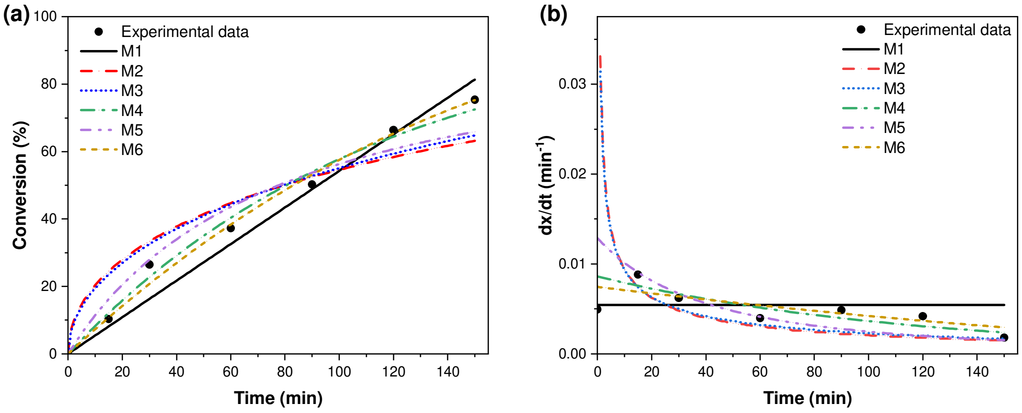

Fig. 3. Comparison of experimental data with kinetic model predictions of coal oxidation using H_2O_2: (a) conversion and (b) derivative of conversion over the time (symbol: experimental data; line: model)

Table 2: Calculated kinetics constant

Kinetics Model	Constant (min^{-1})	SSE	
		f(x)	*df(x)*
M1	5.43×10^{-3}	1.70×10^{-2}	2.96×10^{-5}
M2	5.36×10^{-4}	5.22×10^{-2}	4.22×10^{-5}
M3	1.39×10^{-3}	4.32×10^{-2}	3.93×10^{-5}
M4	8.61×10^{-3}	5.23×10^{-3}	1.91×10^{-5}
M5	1.29×10^{-2}	2.11×10^{-2}	7.27×10^{-5}
M6	2.48×10^{-3}	4.41×10^{-3}	1.38×10^{-5}

According to the previous section, the surface reaction (M6) is the controlling step. Therefore, the M6 model is fitted into the data. The data fitting of the M6 model at different temperatures is presented in Fig. 4. The calculated constants for 40°C, 50°C, and 60°C are 1.25×10^{-3}, 1.96×10^{-3}, and 2.48×10^{-3}, respectively. These results can be used to predict the reaction time needed for each temperature.

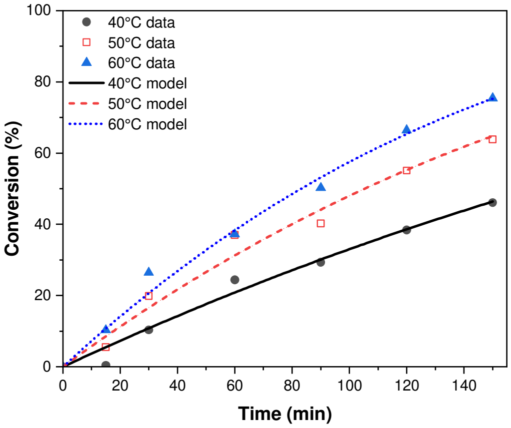

Fig. 4. Effect of temperature on the oxidation kinetics of coal using H_2O_2 (symbol: experimental data; line: model)

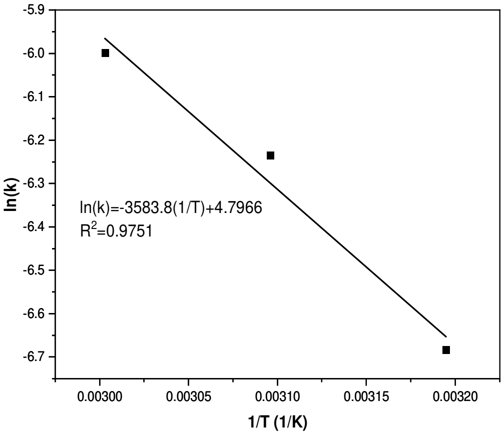

Fig. 5. Plot of Arrhenius kinetic parameter (M6) as a function of temperature

The obtained kinetic parameter (k) is then plotted as a function of temperature using the Arrhenius equation to evaluate the Arrhenius constant (A) and activation energy (E_a) (Fig. 5). The linear form of the Arrhenius equation is presented in Equation (5), where R is the gas constant (8.314 $J.mol^{-1}.K^{-1}$). According to the evaluation, the estimated value of Ea and A are 29.80 $kJ.mol^{-1}$ and 121.10 min^{-1}, respectively. This result reveals the activation energy is lower than 40 $kJ.mol^{-1}$. Therefore, it can be suggested that for the oxidation of coal using H_2O_2 can be reacted at room temperature [28].

$$ln(k) = -\frac{E_a}{R}\left(\frac{1}{T}\right) + ln(A) \tag{5}$$

The study on the kinetic oxidation of coal using H_2O_2 is still limited, however energy activation can be compared by others organic compound oxidation using H_2O_2. Table 3 provides a comparison of the activation energy of H_2O_2 oxidation from several organic compounds. According to Table 3, this work has higher activation energy compared to others. It is because of the complex chemical structure of the coal.

Table 3: Comparison study of the activation energy for the oxidation reaction using H_2O_2

Chemical Compound	Ea, $kJ.mol^{-1}$	Reference
Cresol	12.90 – 16.25	[29]
Methyl diethanolamine	14.66	[30]
Butane-2,3-diol	10 ± 2	[31]
Hexane-1,2-diol	14 ± 5	[31]
Low-rank coal	29.80	This work

3.3. Future and opportunities

The development of coal oxidation contributes significantly to the humic acid industry, which is growing in applications across agriculture, environmental remediation, and other sectors. Additionally, scaling up from laboratory to industrial production is necessary to meet the increasing demand. This process requires a deeper understanding, particularly of the reaction kinetics involved in the chemical oxidation of low-rank coal to produce humic acid. Key parameters for reactor design and equipment must account for the solid-liquid reactions, which involve diffusion through a thin film layer, bulk solid, and the chemical reaction itself.

This study aims to identify the controlling step in the reaction between H_2O_2 and low-rank coal, offering practical insights for process optimization. The results reveal that the surface reaction is the determining step. Given the limited research on lignite oxidation, these findings provide valuable guidance for optimizing industrial systems. The study also highlights that coal oxidation leads to the formation of carboxyl groups, which aligns with the demand for humic acid and enhances process yield.

Moreover, the findings indicate that the oxidation of low-rank coal can occur at moderate temperatures (40°C to 60°C), significantly reducing energy consumption and improving cost-effectiveness. The availability of lignite as a raw material, particularly from South Sumatra, presents an opportunity for localized production. However, adopting green processes and minimizing environmental impact are essential for ensuring the long-term sustainability of the industry.

4. Conclusion

The evaluation of the kinetic model on the oxidation of low-rank coal using H_2O_2 has been conducted in this study. The study of the kinetics of chemical oxidation in low-rank coal remains relatively limited. Hydrogen peroxide (10%) is utilized to oxidize the coal (-60 +80 mesh). At specific intervals, small portions of the solution are extracted and subjected to measurement via titration. The oxidized coal presents an introduction of the carboxyl group. While the kinetics model reveals that the controlling process is the surface reaction. The estimated activation energy is 29.80 $kJ.mol^{-1}$, while the Arrhenius constant is 121.10 min^{-1}. The present study suggests that the reaction can be conducted at room temperature. Therefore, the oxidation of lignite using H_2O_2 is a breakthrough process as it requires low energy and uses safer chemicals. This study contributes to Sustainable Development Goals by promoting environmentally friendly utilization of lignite and contributing responsible and production to the development of the humic acid industry.

5. Conflicts of interest

There are no conflicts to declare

6. Formatting of funding source

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