



Journal home page: <http://ajarcde-safe-network.org> ISSN 2581-0405

Reaction Kinetics of Potassium Sulfate Formation from Bleaching Earth Liquid Waste and Potassium Hydroxide

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ARTICLE INFO

Article History:

Received: 26 May 2026

Final Revision: 21 June 2026

Accepted: 22 June 2029

Online Publication: 27 June 2026

KEYWORDS

Bleaching earth liquid waste, potassium sulfate, reaction kinetics, activation energy, industrial wastewater, conversion rate.

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ABSTRACT

This study examines the kinetics of potassium sulfate (K_2SO_4) formation from bleaching earth industrial liquid waste. The objective is to evaluate the effects of temperature and reaction time on the conversion rate of sulfuric acid, determine the reaction order, and calculate the activation energy. The process involves reacting the diluted acid-rich liquid waste with analytical-grade potassium hydroxide (KOH) under constant stirring at temperatures of 50–70°C for 20–40 minutes. The results show that conversion increases with higher temperature and longer reaction time, reaching an optimum conversion (X_A) of 96.22% at 70°C for 40 minutes. Kinetic analysis indicates that the reaction follows a second order model based on the highest coefficient of determination (R^2). The calculated activation energy (E_a) is 8580.048 J/mol with a frequency factor (k_0) of 367.0784, expressed in the rate constant equation $k = 367.0784 e^{\frac{-8580.048}{T}}$, adhering to the Arrhenius model ($R^2 = 0.844$) despite slight experimental deviations. These findings highlight the potential use of bleaching earth liquid waste as a raw material for potassium sulfate production and provide insight into its reaction kinetics.

Contribution to Sustainable Development Goals (SDGs):

SDG 2: Zero Hunger

SDG 9: Industry, Innovation, and Infrastructure

SDG 12: Responsible Consumption and Production

SDG 13: Climate Action

1. INTRODUCTION

1.1. Research Background

Industrial waste issues in Indonesia have become a major concern, particularly regarding the management of waste generated by the bleaching earth industry, which contains sulfuric acid with a pH of approximately 3. Bleaching earth (BE) is generally a type of bleaching clay derived from bentonite, with its main components consisting of SiO_2 , Al_2O_3 , bound water (H_2O), and ions such as Ca^{2+} , MgO , and FeO . The bleaching capability of bleaching earth is attributed to the presence of Al^{3+} on the particle surface, which enables adsorption of coloring

substances [1]. The liquid waste produced by this bleaching process contains hazardous substances, including chemical compounds and particulate matter, that can contaminate soil and water sources if not properly managed. In addition, improper waste disposal practices that fail to comply with environmental standards may lead to the accumulation of hazardous materials, negatively impacting public health and ecosystems. With the increasing demand for products processed with bleaching earth, it is essential to develop more effective and environmentally friendly waste-treatment technologies and practices [2].

The bleaching earth industry often generates liquid waste from washing processes or from activating bleaching earth with mineral acids such as sulfuric acid (H_2SO_4) or hydrochloric acid (HCl). This industrial waste contains hazardous compounds,



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particularly sulfuric acid (H_2SO_4), which, if not properly treated, can cause environmental pollution in water and soil. Several studies have developed research related to the formation of potassium sulfate through the utilization of various types of waste as potassium sources. A study conducted by Andini [3], specifically examined the reaction kinetics using oil palm empty fruit bunch ash extract and sulfuric acid (H_2SO_4). The extraction procedure was carried out with a composition of 100 grams of ash in 1 liter of distilled water (aquadest), which was allowed to stand for 7 days. The reaction was performed by mixing 150 ml of extract with 150 ml of 1N H_2SO_4 at temperatures of 50, 60, 70, 80, and 90°C, and times of 40, 50, 60, 70, and 80 minutes. The study concluded that the formation of potassium sulfate follows a second-order reaction model, with the best operating conditions achieved at 90°C for 80 minutes, yielding a conversion rate (XA) of 93.01% and a reaction rate constant. While research on the formation of potassium sulfate from agricultural wastes has been widely documented, investigation into the reaction kinetics using bleaching earth liquid waste is still scarce. Therefore, this study focuses on evaluating the kinetic parameters of potassium sulfate formation from bleaching earth liquid waste and potassium hydroxide across different operating temperatures and reaction times.

1.2. Literature Review

1.2.1. Bleaching Earth Production and Activation Processes

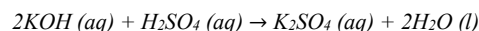
Bleaching earth (BE) is a type of bleaching clay derived from bentonite rock, with its primary composition consisting of SiO_2 , Al_2O_3 , bound water (H_2O), and ions such as Ca^{2+} , MgO , and FeO . The bleaching capability of bleaching earth is attributed to the presence of Al^{3+} on the particle surface, which enables adsorption and facilitates the removal of coloring substances. This capability also depends on the ratio of Al_2O_3 to SiO_2 in the bleaching earth [4]. The production stages of bleaching earth initiate with the processing of raw clay. Large lumps of raw clay are initially comminuted using a crusher to reduce particle size, thereby facilitating subsequent processing stages. These clay particles are then introduced into a dryer to minimize their moisture content, followed by conveyance to a pulverizer where they are finely ground to achieve a smaller and more uniform size distribution. Following this fine pulverization process, the material is transferred into a mixer for the blending phase. At this stage, water or a sulfuric acid (H_2SO_4) solution is uniformly sprayed across the entire surface of the clay particles, with the retention time adjusted based on the specific end-product specifications desired. Upon completion of the mixing process, the refined bleaching earth is directed to the final stage to be packaged in sacks (bagger) or stored in bulk storage units [5].

The spraying or activation process utilizing mineral acids (such as HCl or H_2SO_4) within the mixer aims to alter the mineral structure, thereby enhancing the bleaching efficiency and adsorption properties of the bleaching earth. Mechanistically, the acid solution dissolves and reacts with impurities such as tar, as well as calcium (Ca) and magnesium (Mg) salts that occlude the adsorbent pores [6]. This acid activation process initially triggers a cation exchange between the metal ions (Ca^{2+} and Mg^{2+}) located in the bentonite interlayer and the H^+ ions from the acid. This

phenomenon is subsequently followed by the dissolution of Al^{3+} ions and other metallic cations, such as Fe^{3+} from the bentonite lattice framework. The dissolution of ions imparts a net negative charge to the bentonite structure, which significantly enhances the adsorption capacity of the acid-activated clay. Furthermore, the multi-ion dissolution occurring during acid activation effectively opens up new pore networks and expands the specific surface area of the clay, thereby substantially optimizing the adsorption interactions [7].

1.2.2. Reaction Kinetic

Chemical reaction kinetics dictates the rate of reactant and product formation as a function of time, governed primarily by concentration, temperature, and pressure. In a homogeneous liquid-liquid system, these reaction rates are quantified through continuous variations in molar concentration. In this study, the kinetic behavior is evaluated through the reaction between a saturated potassium hydroxide solution and sulfuric acid-rich earth liquid waste. This liquid-phase reaction drives the neutralization of the acid and base components, leading directly to the chemical formation of potassium sulfate and water. The overall reaction is as follows [8] :



The reaction rate can be defined as [8]:

$$-r_A = kC_A^m C_B^n \quad (1)$$

The reaction rate is defined in Equation (1). To understand the reaction behavior, the experimental data were evaluated using integrated zero-order, first-order, and second-order kinetic models. The integrated rate equations used in the kinetic analysis are expressed as follows:

$$-r_A = k' C_A^m \quad (2)$$

Where, $k' = kC_B^n$

Therefore, Equation (2) can be expressed as follows:

$$-\frac{dC_A}{dt} = k' C_A^m \quad (3)$$

For a zero-order reaction where $m = 0$, the reaction rate remains constant regardless of the H_2SO_4 reactant concentration (provided the reactants are not depleted); thus, Equation (3) becomes:

$$C_A = C_{A0} - kt \quad (4)$$

If the reaction is first-order ($m = 1$), the equation is derived as:

$$-\ln(1 - X_A) = kt \quad (5)$$

For a second-order reaction ($m = 2$), the rate law equation is expressed as follows :

$$\frac{1}{C_A} = k't + \frac{1}{C_{A0}} \quad (6)$$

The activation energy is determined using the Arrhenius equation, which is expressed as [9] :

$$k = k_0 e^{-\frac{E_a}{RT}} \quad (7)$$

1.3. Research Objective

This study aims to evaluate the effects of reaction temperature and time variations on the conversion rate sulfuric acid in potassium sulfate production from bleaching earth wastewater. Furthermore, it seeks to determine the reaction kinetics model, establish the reaction order, and identify the optimum operating conditions that generate the highest conversion.

2. MATERIALS AND METHODS

2.1. Material and Tools

This study utilized liquid waste from the bleaching earth activation process, obtained from PT Madu Lingga Raharja, as the primary raw material. Based on the laboratory analysis conducted by the Center for Standardization and Industrial Services (*Balai Standardisasi dan Pelayanan Jasa Industri - BSPJI*), the liquid waste is presented in Table 1. Additionally, solid potassium hydroxide (KOH) of analytical grade, which was purchased from UD. Nirwana Abadi, was employed as a supporting reagent in this research.

Table 1. Chemical composition of the bleaching earth activation liquid waste

No	Parameter	Unit	Result test
1	SO ₄	mg/L	177709.69
2	Fe	mg/L	9460.83
3	Mg	mg/L	735.04
4	Al	mg/L	0.4

The apparatus required for this experiment comprised hot plate, thermometer, analytical balance, magnetic stirrer, beaker glasses, and filter paper.

2.2. Research Procedure

2.2.1. Prepararton of Raw Material

The treatment of the liquid bleaching earth waste was carried out by preparing a liquid waste sample. A 10 mL sample of the waste was taken and placed into a volumetric flask, then diluted with distilled water to a volume of 100 mL. Next, the potassium hydroxide (KOH) solution was prepared. Based on stoichiometric calculations, 2.07 grams of KOH crystals were weighed using an analytical balance. The KOH crystals were then dissolved with aquadest in a volumetric flask to a final volume of 100 mL.

2.2.2. Reaction Process of Potassium Sulfate Formation

This process was conducted by mixing the diluted waste solution with the potassium hydroxide (KOH) solution inside a glass beaker. The mixture was stirred using a magnetic stirrer at a constant speed of 300 rpm to maintain reaction homogeneity. The reaction temperature was maintained at predetermined variations of 50°C, 55°C, 60°C, 65°C, and 70°C. The reaction time was counted after the waste sample reached the target temperature, with variations of 20, 25, 30, 35, and 40 minutes. After the conditions for each temperature and time variation were reached, the pH of each sample was measured. The resulting solution was then cooled to room temperature and filtered through filter paper to separate the residue from the filtrate.

2.2.3. Analysis of Residual Acid Concentration

The concentration of residual sulfuric acid was analyzed using the acid-base titration method on the filtrate obtained from the reaction between potassium hydroxide and sulfuric acid. The acid-base titration was performed using 0.1 N sodium hydroxide as the titrant and phenolphthalein as the indicator. A 10 mL volume of the filtrate was measured using a volumetric pipette and transferred into an Erlenmeyer flask as the analyte. Next, 2 to

3 drops of phenolphthalein (PP) indicator were added to the analyte. The titration was conducted by slowly adding the 0.1 N sodium hydroxide (NaOH) titrant from a burette into the Erlenmeyer flask until the equivalence point was reached. This equivalent point was indicated by a constant color change to faint pink. Once this point was reached, the volume of NaOH used was recorded accurately to calculate the concentration and the number of moles of residual sulfuric acid in the filtrate. The residual mole data obtained were then used as the primary basis for calculating the sulfuric acid conversion, reaction rate, and kinetic constants at each time and temperature.

3. RESULT AND DISCUSSION

3.1. Effect of Temperature and Reaction Time on the Conversion

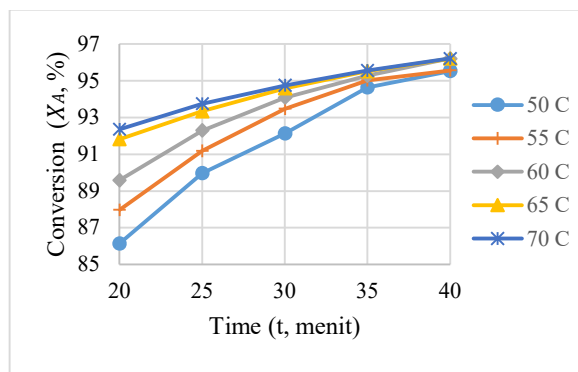


Fig.1 Effect of Temperature and Reaction Time on the Conversion of Potassium Sulfate Formation

Based on Figure 1, the conversion of sulfuric acid (X_A , %) exhibits a progressive increase that is directly proportional to the extension of the reaction time (t , minutes). This correlation indicates that a longer reaction duration consistently leads to a higher conversion value. In the nascent stage of the reaction, the conversion efficiency remains relatively low because the frequency of effective collisions between the reactant molecules has not yet extensively occurred. However, as the reaction time progresses, the frequency of molecular collisions increases substantially, prompting more reactants to undergo transformation. Consequently, the conversion values gradually increase until they stabilise.

3.2. Testing of Research Data on Reaction Kinetics Model

The kinetic model of potassium sulfate formation from bleaching earth and potassium hydroxide wastewater was determined by testing experimental data against three basic models: zero-, first-, and second-order. Experimental data were plotted into the linear equation of each model to highlight the effect of reactant concentration on the reaction rate [10]. The best model was determined based on the coefficient of determination (R^2) value closest to one, which indicates the highest readiness in predicting the behavior and performance dynamics in the system.

3.2.1. Analysis of Zero Order Kinetics Model

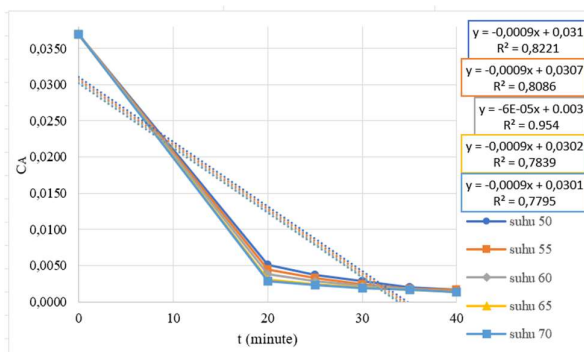


Fig. 2 Zero-Order Kinetic Model of Potassium Sulfate Formation

For zero-order kinetics, the relationship between reactant concentration (CA) and time (t), as illustrated in Figure 2, decreases over the reaction period. Based on the plot, the coefficient of determination (R^2) for the zero-order model ranges between 0.7795 and 0.954. Initially, the reactant concentration is relatively high as the majority of the reactant remains unreacted. As the reaction progresses, more reactants are consumed, leading to a decline in CA within the system. This continuous decrease indicates that the reaction proceeds as long as sufficient contact between the reactants is maintained. However, a characteristic of zero-order kinetics is that the reaction rate is independent of reactant concentration, which appears less consistent with the observed reaction.

3.2.2. Analysis of First-Order Kinetics Model

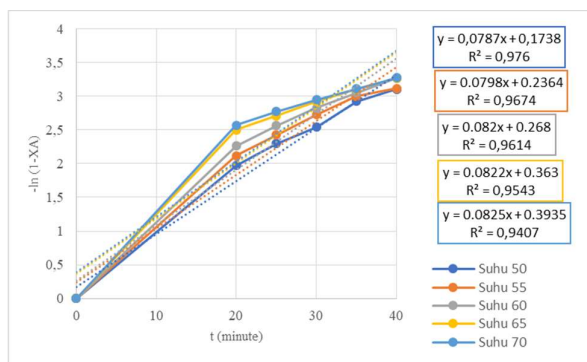


Fig. 3 First-Order Kinetic Model of Potassium Sulfate Formation

In first-order kinetics, the relationship between reactant concentration, represented by $-\ln(1-X_A)$, and time (t), as shown in Figure 3, indicates an upward trend as the reaction progresses. Based on the figure above, the R^2 values for the first-order model range from 0.9407 to 0.9543. During the initial stage of the reaction, the $-\ln(1-X_A)$ value remains relatively low due to the minor conversion achieved. However, as the reaction time extends, the amount of reactants reacted increases, leading to higher conversion and a gradual rise in the $-\ln(1-X_A)$ value. This suggests that the reaction rate increases with prolonged contact time between reactants. Although the first-order model demonstrates relatively high linearity, its coefficient of determination is generally lower than that of the second-order model. Analysis of Second Order Kinetics Model

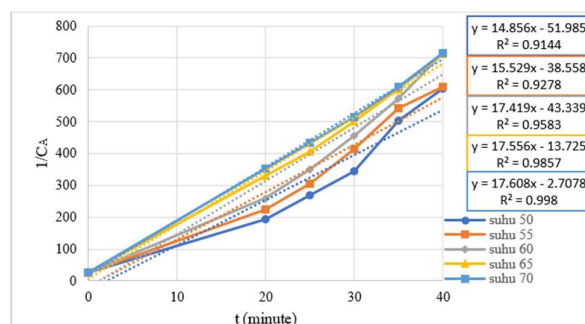


Fig.4 Second-Order Kinetic Model of Potassium Sulfate Formation

In second-order kinetics, the relationship between reactant concentration ($1/CA$) and time (t), as shown in Figure 4, indicates an upward trend as the reaction progresses. Based on the figure, the R^2 values for the second-order model range from 0.9144 to 0.998, which are generally higher than those obtained for the first-order model (0.9407–0.9543). This suggests that the second-order kinetic model provides a better mathematical representation of potassium sulfate formation under the investigated conditions.

A greater change in the $1/CA$ value over time indicates an increase in the reaction rate. The stronger linear relationship between $1/CA$ and time (t) observed under various temperature conditions demonstrates that the second-order model more effectively describes the kinetic behavior of the system. Furthermore, the slope of each linear trendline directly represents the reaction rate constant (k). As observed from the graphs, the slope increases with increasing temperature, indicating an increase in reaction rate. This phenomenon is attributed to the increase in molecular kinetic energy at higher temperatures, which significantly enhances the frequency of effective collisions between reactant molecules, thereby accelerating the overall reaction rate [11]. The high linearity observed in the second-order model indicates that the reaction rate is strongly influenced by changes in reactant concentration throughout the reaction process. Therefore, potassium sulfate formation under the investigated operating conditions tends to follow a second-order kinetic model.

3.3. The Effect of Temperature on Activation

To investigate the influence of temperature on the reaction kinetics, the reaction rate constants determined at each operating temperature were converted into Arrhenius parameters. The corresponding values are summarized in Table 2.

Table 2. The Effect of Temperature on Collisions and Activation Energy (E_a)

Temperature		k	ln k	1/T
°C	K			
50	323,15	14,856	2,6984	0,0031
55	328,15	15,529	2,7427	0,0030
60	333,15	17,419	2,8576	0,0030
65	338,15	17,556	2,8654	0,0030
70	343,15	17,608	2,8684	0,0029

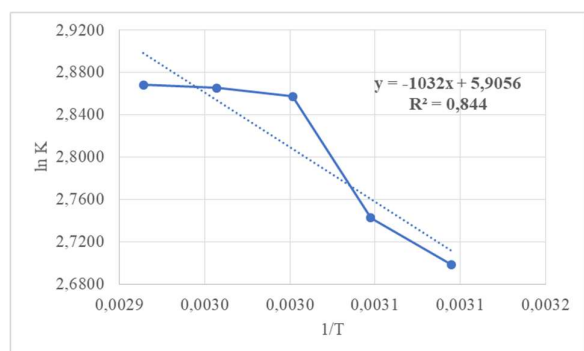


Fig. 5 Correlation Between 1/T and ln K

Figure 5 shows the plot of $\ln k$ versus $1/T$. Although a linear trend is observable, the data points do not strictly form a straight line. According to the Arrhenius equation, the linear relationship is expressed as $\ln k = \ln A - \frac{E_a}{R} \left(\frac{1}{T} \right)$ where a perfect fit yields a straight line with a slope of $-\frac{E_a}{R}$. The slight deviation observed in the experimental plot, indicated by the scattering of data points around the trendline, still maintains a representative correlation with the Arrhenius model ($R^2 = 0.844$). The linear regression derived from this plot is :

$$y = -1032X + 5.9056$$

$$\ln K = -1032 \left(\frac{1}{T} \right) + 5.9056$$

The activation energy (E_a) calculated from the slope using the ideal gas constant ($R = 8.314 \text{ J/mol.K}$) was 8580.048 J/mol . Meanwhile, the frequency factor (k_0), determined from the intercept ($\ln k_0 = c$), is 367.0874 . Consequently, the final Arrhenius rate constant equation is expressed as $k = 367,0874 e^{\frac{-8580,048}{T}}$.

4. CONCLUSION

Higher reaction temperatures and longer reaction times significantly increase the conversion of potassium sulfate. In this study, the optimum operating condition for potassium sulfate production from bleaching earth wastewater was achieved at 70°C for a 40-minute reaction time, resulting in the highest conversion X_A of 0.9622 or 96.22%. Furthermore, the reaction kinetics analysis established that the formation of potassium sulfate from bleaching earth wastewater and potassium hydroxide was better represented by a second-order kinetic model. The temperature dependence of the reaction rate is described by the Arrhenius equation $k = 367,0874 e^{\frac{-8580,048}{T}}$ with a calculated activation energy (E_a) of $8580,048 \text{ J/mol}$.

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