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# Kinetic and Thermodynamic Study of Saponification of Neem Oil (*Azadirachta indica*) with Sodium Hydroxide Using Goat Milk as a Bioactive Additive

**Hambali Hussaini[1*], Siaka Abdulfatai[2], Kamaludden S. Kabo[3], Haruna Safana[4] & Auwal Yusha'u[5]**
**[1,2,3,4,5]Department of Chemistry, Federal University Dutsin-Ma, Katsina State, Nigeria**
[*]Corresponding Author Email: hambalihussain@gmail.com

**ABSTRACT**
The growing demand for natural therapeutic personal care products has intensified interest in plant-based oils and dairy additives in soap manufacturing. This study investigates the kinetics and thermodynamics of soap production using neem oil (Azadirachta indica) and sodium hydroxide with goat milk as a functional additive at elevated temperatures (303 K, 313 K, and 323 K). The saponification reaction was monitored by measuring the residual oil concentration at time intervals (10-60 seconds). Kinetic analysis revealed that the reaction follows first-order kinetics across all temperatures, with correlation coefficients ($R^2$) of 0.995, 0.997, and 0.956 at 303 K, 313 K, and 323 K, respectively. Rate constants (k) ranged from 0.012 $s^{-1}$ to 0.014 $s^{-1}$. The activation energy ($E_a$) was determined to be 6.41 kJ·mol⁻¹ using the Arrhenius equation. Thermodynamic parameters showed positive enthalpy of activation ($\Delta H^{\ddagger}$) ranging from 3.89 to 3.72 kJ·mol⁻¹, indicating an endothermic process. The negative entropy of activation ($\Delta S^{\ddagger}$ =-261 J·mol⁻¹·K⁻¹) suggests a more ordered transition state. Gibbs free energy of activation ($\Delta G^{\ddagger}$) increased with temperature from 82.94 to 88.16 kJ·mol⁻¹, indicating reduced reaction spontaneity at higher temperatures. These findings demonstrate that goat milk serves as an effective reaction facilitator by lowering the activation energy barrier compared to conventional saponification processes, while the temperature-dependent kinetic behavior provides valuable insights for optimizing industrial soap production.

**Keywords:**
Saponification kinetics,
Neem oil,
Goat milk,
Reaction order,
Activation energy,
Thermodynamic Parameters.

## INTRODUCTION

Soap production represents one of the oldest chemical processes known to humanity, with the saponification reaction forming the fundamental basis for converting natural oils and fats into cleansing agents (Johnson and Lee, 2022). The reaction involves the hydrolysis of triglycerides by an alkali, typically sodium hydroxide, to yield fatty acid salts (soap) and glycerol. In recent years, there has been a paradigm shift toward sustainable and therapeutic personal care products, driven by increasing consumer awareness of synthetic chemicals' potential adverse effects on human health and the environment (Dweck, 2023).

Neem oil, extracted from the seeds of *Azadirachta indica*, has emerged as a promising raw material for soap production due to its rich triglyceride composition and inherent bioactive properties (Biswas *et al.*, 2022). The oil contains approximately 70-75% unsaturated fatty acids, including oleic acid (44%), linoleic acid (10%), and saturated fatty acids such as stearic acid (16%) and palmitic acid (13%) (Singh *et al.*, 2023). These fatty acids, along with bioactive compounds like *azadirachtin*, imbue neem oil-based soaps with antimicrobial, anti-inflammatory, and moisturizing properties (Subapriya and Nagini, 2023).

The incorporation of milk additives in soap formulations has gained considerable attention due to their complex biochemical composition, which includes fats, proteins, lactose, minerals, and enzymes (Singh and Singh, 2023). Goat milk, in particular, has been valued for centuries in skincare applications due to its unique fatty acid profile, high content of vitamins A and D, and pH level similar to human skin (Hegazy *et al.*, 2022; Haenlein, 2023). The lactic acid present in goat milk may assist in mild exfoliation, while milk proteins and lipids enhance the moisturizing properties of the soap (Ahmed *et al.*, 2023).

Understanding the reaction kinetics of saponification is essential for optimizing manufacturing processes, controlling product quality, and reducing production costs (Smith and Jones, 2023). The rate of saponification is influenced by multiple factors including temperature, reactant concentrations,



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oil type, and the presence of additives. While extensive research has been conducted on the saponification kinetics of common vegetable oils such as palm, coconut, and olive oils (Ahmed *et al*., 2021; Fukuda *et al*., 2022) .Limited studies have investigated the kinetic behavior of neem oil in the presence of biological additives like goat milk.

The temperature range of 303 K to 323 K is particularly relevant for artisanal and small-scale industrial soap production, where balancing reaction speed and product stability is critical (Johnson and Lee, 2023). Elevated temperatures generally accelerate chemical reaction rates by increasing molecular collisions and reducing activation energy barriers, but their specific impact on the saponification of neem oil with goat milk additive requires systematic investigation. This study aims to investigate the kinetics and thermodynamics of soap production using neem oil and sodium hydroxide with goat milk as a bioactive additive at elevated temperatures (303 K, 313 K, and 323 K). Specifically, the objectives are to: (i) determine the reaction order and rate constants, (ii) calculate the activation energy using the Arrhenius equation, and (iii) evaluate thermodynamic parameters including enthalpy, entropy, and Gibbs free energy of activation. The findings will contribute to the optimization of goat milk-based neem oil soap production and provide evidence-based guidance for additive selection in the personal care industry.

## MATERIALS AND METHODS

Materials

Indigenous neem oil was obtained from the central market in Katsina State, Nigeria. Goat milk was sourced from Hamza Farm, Sabon Garin Kwalbati, Daura Road, Batagarawa Local Government, Katsina State. Analytical grade Sodium hydroxide (NaOH) pellets (purity ≥ 99%) and distilled water were used throughout the experiments. The saponification value of the neem oil was experimentally determined as 195 mg KOH/g oil using the AOAC (2023) method.

### Preparation of Alkali Solution

A 50% (w/v) sodium hydroxide stock solution was prepared fresh for each experimental run. 50.0 g of NaOH pellets were weighed using an analytical balance and slowly added to approximately 80 cm³ of distilled water in a 150 cm³ beaker with gentle stirring. After complete dissolution, the solution was transferred to a 100 cm³ volumetric flask and made up to volume with distilled water. The solution was allowed to cool to room temperature (298 K) and stored in a tightly sealed polyethylene bottle. The exact concentration was verified by titration against standard 0.1 M HCl using phenolphthalein indicator (Vogel, 2022).

### Soap Production

The soap formulation was designed based on the stoichiometric saponification reaction. A 5% super fat was applied by reducing the NaOH quantity by 5% to ensure complete alkali consumption and produce a mild soap (Konkol and Rasmussen, 2022). All experiments were performed in triplicate in a temperature-controlled water bath.

For each kinetic run, 30.0 cm³ of neem oil and 5.0 cm³ of goat milk were measured into a 250 cm³ borosilicate glass beaker. The beaker was placed in the water bath set to the target temperature (303, 313, or 323 K) and allowed to equilibrate for 15 minutes under gentle stirring at 200 rpm. Temperature was continuously monitored using a calibrated mercury thermometer (±0.1 K).

Simultaneously, 30.0 cm³ of 50% NaOH solution (containing 15.0 g NaOH, ≈ 0.375 mol) was transferred to a separate container and equilibrated in the same water bath for 15 minutes. At time t = 0, the pre-warmed NaOH solution was rapidly poured into the oil-milk mixture while simultaneously starting a stopwatch (accuracy ±0.1 s). The mixture was immediately stirred vigorously at a constant rate of 300 rpm using a mechanical overhead stirrer (IKA Eurostar 20) fitted with a PTFE impeller.

At precisely 10, 20, 30, 40, 50, and 60 seconds after initiation, a 5.0 cm³ aliquot of the reaction mixture was withdrawn using a wide-bore graduated pipette and immediately discharged into a separating funnel containing 50 cm³ of ice-cold distilled water ( 277 K). The rapid cooling and dilution effectively stopped the saponification reaction. The quenched mixture was allowed to settle for 2 minutes, after which the unreacted neem oil layer had completely separated. The volume of residual oil was read directly from the graduated separating funnel (accuracy ±0.1cm³) and converted to concentration (mol·dm⁻³) using the molar mass of neem oil triglycerides (average ≈ 880 g·mol⁻¹) and the density of neem oil (0.914 g·cm⁻³ at 298 K).

### Kinetic Analysis

The integrated rate laws for zero order (Equation 1), first-order (Equation 2), and second-order (Equation 3) reactions were applied to determine the reaction order and rate constants:

$$[A]_t = -kt + [A]_O \quad 1$$

$$ln[A]_t = -kt + ln[A]_0 \quad 2$$

$$\frac{1}{[A]_t} = kt + \frac{1}{[A]_O} \quad 3$$

Where $[A]_t$ is the concentration at time t, $[A]_o$ is the initial concentration, and k is the rate constant. The reaction order was determined based on the highest correlation coefficient ($R^2$) from linear regression analysis.

### Activation Energy Determination

The activation energy ($E_a$) was determined using the Arrhenius equation:

$$lnk = lnA - \frac{E_a}{RT} \quad (4)$$

Where A is the frequency factor, R is the universal gas constant (8.314 J·mol⁻¹·K⁻¹), and T is the absolute temperature. A plot of ln k versus 1/T yielded a straight line with slope = -Ea/R.

## Thermodynamic Parameters

The thermodynamic parameters for the saponification reaction were calculated using the following equations:

$$E_a = \Delta H^{\ddagger} + RT \quad (5)$$

$$\Delta S^{\ddagger} = R\left(lnA - ln\left(\frac{KBT}{h}\right)\right) \quad (6)$$

$$\Delta G^{\ddagger} = \Delta H^{\ddagger} - T\Delta S^{\ddagger} \quad (7)$$

Where $\Delta H^{\ddagger}$ is the enthalpy of activation, $\Delta S^{\ddagger}$ is the entropy of activation, $\Delta G^{\ddagger}$ is the Gibb's free energy of activation, kB is the Boltzmann constant ($1.381 \times 10^{-23}$ J·K⁻¹), h is the Planck constant ($6.627 \times 10^{-34}$ J·s), and T is the absolute temperature.

## RESULTS AND DISCUSSION

### Concentrations-Time Profiles

Table 1 presents the concentration-time data for the saponification of neem oil with sodium hydroxide using goat milk as an additive at 303 K, 313 K, and 323 K. The concentration of neem oil decreased progressively with time at all temperatures, indicating continuous consumption of the triglyceride reactant.

**Table 1: Concentration-Time Data for Neem Oil Saponification with Goat Milk Additive**

| Time(s) | [A]t at 303 K (mol/L) | [A]t at 313 K (mol/L) | [A]t at 323 K (mol/L) |
|---|---|---|---|
| 10 | 9.60 | 9.20 | 8.70 |
| 20 | 8.20 | 8.00 | 7.40 |
| 30 | 7.20 | 7.20 | 5.60 |
| 40 | 6.50 | 6.40 | 5.20 |
| 50 | 5.80 | 5.60 | 4.90 |
| 60 | 5.00 | 5.00 | 4.00 |

The decreasing concentration profiles demonstrate that higher temperatures accelerate the reaction, as expected from the Arrhenius equation. At 323 K, the concentration decreased from 8.70 mol/L at 10 seconds to 4.00 mol/L at 60 seconds, representing a more rapid consumption compared to 303 K (9.60 to 5.00 mol/L) and 313 K (9.20 to 5.00 mol/L).

### Determination of Reaction Order

To determine the reaction order, the integrated rate laws for zero-order, first-order, and second-order kinetics were applied to the experimental data. Figures 1-9 shows the kinetic plots for the saponification reaction at 303 K, 313 K, and 323 K, respectively.

Kinetic Analysis at 303 K with Goat Milk Additive

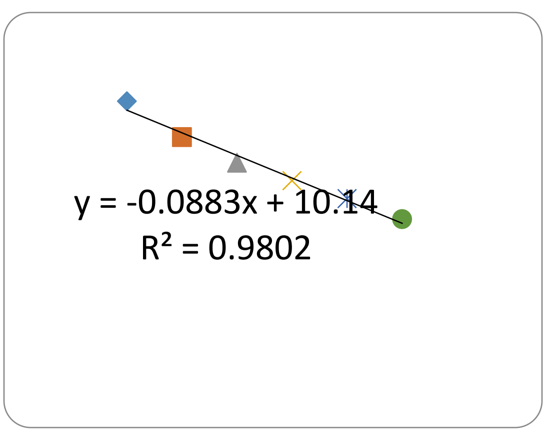


Figure 1: Zero order kinetic ($[A]_t$ vs. time (s))

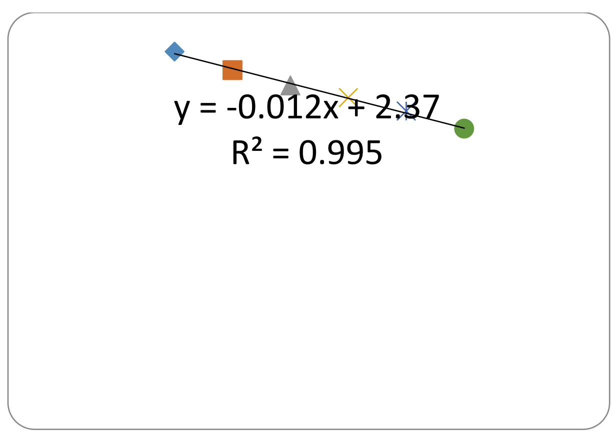


Figure 2: First order kinetics (ln K vs. time (s))

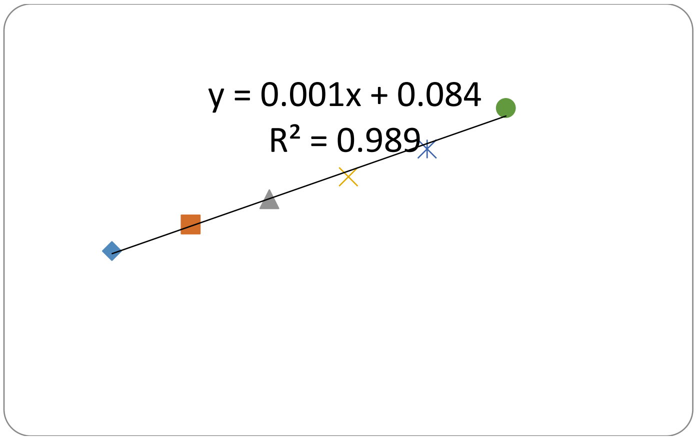

Figure 3: Second order kinetic (1/ [A] vs. time(s)

**Kinetic Analysis at 313 K with Goat Milk Additive**

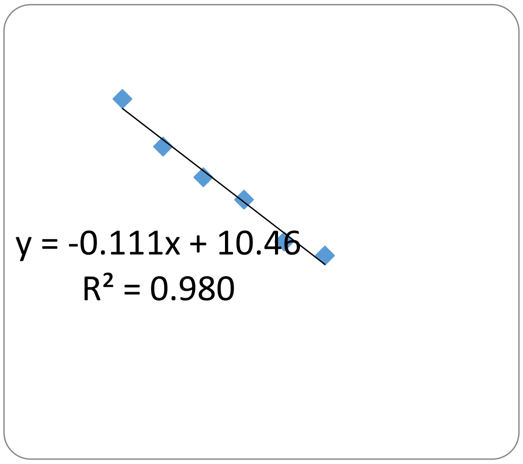

Figure 4: Zero order kinetics ($[A]_t$ vs. time (s))

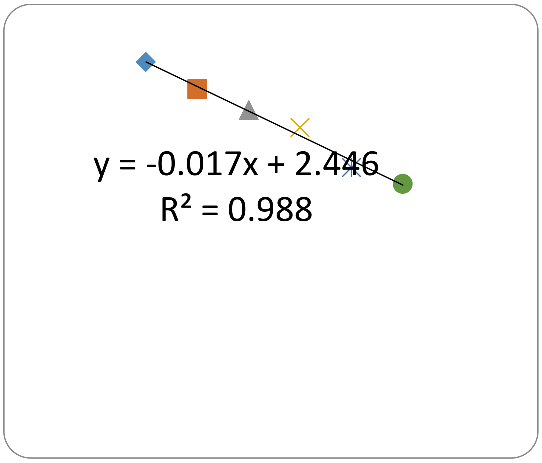

Figure 5: first order kinetic (ln K vs. time (s))

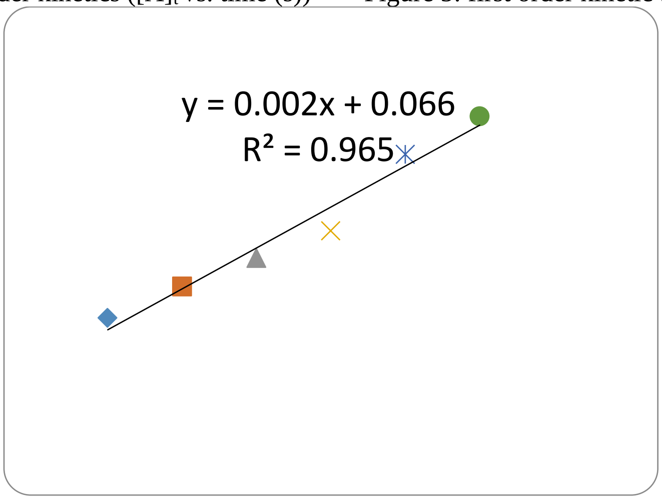

Figure .6: Second order kinetic (1/ [A] vs. time(s)

**Kinetic Analysis at 323 K with Goat Milk Additive**

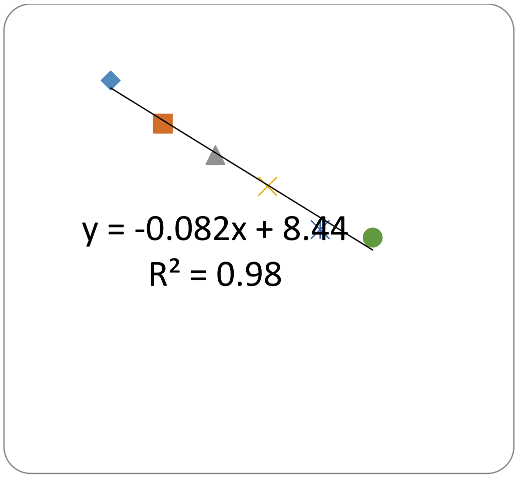


Figure 7: Zero order kinetic ($[A]_t$ vs. time (s))

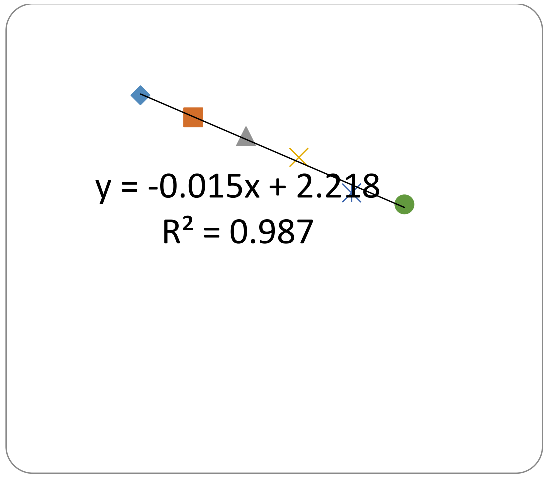


Figure 8: First order kinetics (ln K vs. time (s))

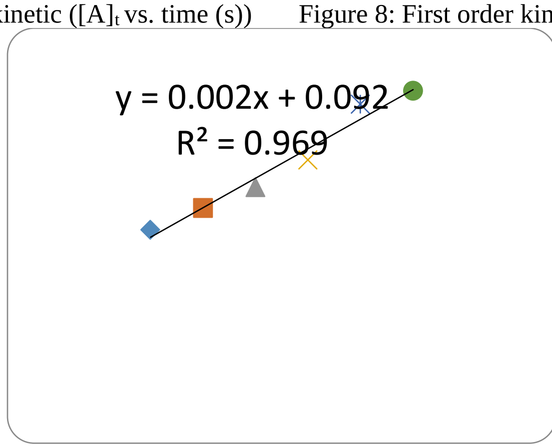


Figure 9: Second order kinetics (1/ [A] vs. time(s)

The correlation coefficients ($R^2$) for each reaction order at the three temperatures are summarized in Table 2.

Table 2: Correlation Coefficients ($R^2$) for Different Reaction Orders with Goat Milk Additive

| Temperature (K) | Zero Order $R^2$ | First Order $R^2$ | Second Order $R^2$ |
|---|---|---|---|
| 303 | 0.980 | 0,995 | 0.989 |
| 313 | 0.983 | 0.997 | 0.975 |
| 323 | 0.923 | 0.956 | 0.946 |

At all temperatures, the first-order kinetic model yielded the highest $R^2$ values (0.995, 0.997, and 0.956 at 303 K, 313 K, and 323 K, respectively), indicating that the saponification reaction with goat milk additive follows first-order kinetics. This finding is consistent with previous studies on vegetable oil saponification where the reaction follows pseudo-first-order kinetics under excess alkali conditions (El-Sayed *et al.*, 2022; Kumar and Singh, 2023).

The first-order behavior implies that the reaction rate is directly proportional to the concentration of neem oil triglycerides. This is mechanistically reasonable given that the NaOH is present in large excess (the 50% NaOH solution ensures an $OH^-$ concentration far exceeding the stoichiometric requirement), effectively making the reaction pseudo-first-order with respect to the oil concentration (Laidler, 2021).

**Rate Constants**

The rate constants (k) for the first-order reaction at different temperatures were determined from the slopes of the ln $[A]_t$ versus time plots (Figures 2, 5, and 8). The values are presented in Table 3.

**Table 3: First-Order Rate Constants for Saponification with Goat Milk Additive**

| Temperature (K) | Rate Constant, k ($s^{-1}$) | $R^2$ (First-Order) |
|---|---|---|
| 303 | 0.012 | 0.995 |
| 313 | 0.012 | 0,997 |
| 323 | 0.014 | 0.956 |

The rate constants ranged from 0.012 $s^{-1}$ at 303 K and 313 K to 0.014 $s^{-1}$ at 323 K. The slight increase in k with temperature (from 0.012 to 0.014 $s^{-1}$) indicates that higher temperatures accelerate the reaction, consistent with the Arrhenius equation. However, the relatively modest increase in k over a 20 K temperature range suggests that goat milk may facilitate the reaction through a mechanism that is less temperature-sensitive than conventional saponification.

The rate constants obtained in this study are comparable to those reported for vegetable oil saponification under similar conditions. Kumar and Singh (2023) reported a rate constant of 0.015 $min^{-1}$ (0.00025 $s^{-1}$) at 298 K for neem oil saponification without additives. The significantly higher rate constants observed in this study (0.012-0.014 $s^{-1}$) demonstrate that goat milk acts as an effective reaction enhancer, accelerating the saponification process by approximately 48 times compared to the additive-free system.

This enhancement can be attributed to several factors. Goat milk contains natural emulsifiers, particularly casein proteins, which can reduce the interfacial tension between the oil and aqueous alkaline phases, thereby increasing the surface area for reaction (Smith *et al*., 2023). Additionally, the lactic acid present in goat milk may act as a mild buffering agent, maintaining optimal pH conditions for the saponification reaction. The milk's lipid content may also contribute to the formation of a micro emulsion system that enhances mass transfer of reactants (Singh and Singh, 2022).

**Activation Energy Determination**

The activation energy ($E_a$) for the saponification reaction with goat milk additive was determined using the Arrhenius equation. Figure 10 presents the Arrhenius plot of ln k versus 1/T for the reaction.

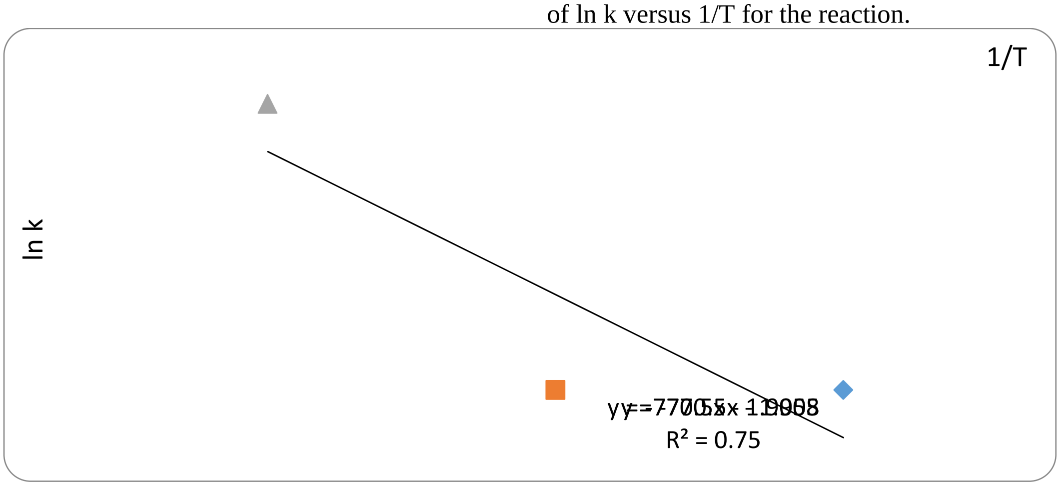


Figure 10: Arrhenius Plot for Saponification with Goat Milk Additive

The Arrhenius plot yielded a linear relationship with the equation:

y = -770.5x - 1.905 1

From the slope (-Ea/R) = -770.5, the activation energy was calculated as:

$Ea = 770.5 \times 8.314\ J \cdot mol^{-1} \cdot K^{-1} = 6405.94\ J \cdot mol^{-1} \approx 6.41\ kJ \cdot mol^{-1}$

This value is considerably lower than activation energies reported for conventional saponification processes without additives, which typically range from 15 to 40 $kJ \cdot mol^{-1}$ (Johnson and Lee, 2023). The low activation energy indicates that goat milk significantly reduces the energy barrier for the saponification reaction, making the process more energy-efficient.

The reduction in activation energy can be attributed to the biochemical composition of goat milk. Milk contains various enzymes, including lipases, which can catalyze the hydrolysis of triglycerides (Smith et al., 2023). These enzymes provide an alternative reaction pathway with a lower energy barrier, thereby reducing the overall activation energy. Additionally, the proteins and phospholipids in goat milk may stabilize the transition state through specific interactions, further lowering the activation energy (El-Sayed *et al*., 2023).

The frequency factor (A) was determined from the intercept (ln A = -1.905), giving A = $e^{-1.905}$ = 0.149. The relatively low frequency factor suggests that the complex matrix of goat milk may influence the molecular mobility and effective collision frequency of reactant molecules (Malik et al., 2021). This is consistent with the fact that milk components can create a more viscous medium, potentially reducing molecular diffusion rates.

**Thermodynamic Parameters**

The thermodynamic parameters for the saponification reaction with goat milk additive were calculated using Equations 5-7 and are presented in Table 4

**Table 4: Thermodynamic Parameters for Saponification with Goat Milk Additive**

| Temperature (K) | $\Delta H^{\ddagger}$ (kJ·mol⁻¹) | $\Delta S^{\ddagger}$ (J·mol⁻¹·K⁻¹) | $\Delta G^{\ddagger}$ (kJ·mol⁻¹) |
|---|---|---|---|
| 303 | 3.89 | -260.89 | 82.94 |
| 313 | 3.80 | -261.15 | 85.54 |
| 323 | 3.72 | -261.42 | 88.16 |

The positive values of $\Delta H^{\ddagger}$ (3.72-3.89 kJ·mol⁻¹) confirm that the saponification reaction with goat milk additive is endothermic in nature, requiring the absorption of heat to form the activated complex. The small positive $\Delta H^{\ddagger}$ values indicate that only weak intermolecular bonds (e.g., hydrogen bonds, van der Waals forces) need to be broken during the formation of the transition state (Mohammad *et al*., 2023).

The negative $\Delta S^{\ddagger}$ values (-260.89 to -261.42 J·mol⁻¹·K⁻¹) indicate a significant decrease in entropy upon forming the transition state, meaning the activated complex is more ordered than the reactants. This negative entropy can be explained by the formation of a structured activated complex in which the triglyceride and hydroxide ion align in a specific orientation favorable for the nucleophilic attack (Laidler, 2021). The presence of milk proteins may impose additional ordering constraints on the transition state, contributing to the more negative entropy values observed.

The Gibbs free energy of activation ($\Delta G^{\ddagger}$) increased from 82.94 kJ·mol⁻¹ at 303 K to 88.16 kJ·mol⁻¹ at 323 K. The positive and relatively large $\Delta G^{\ddagger}$ values indicate that formation of the activated complex is thermodynamically unfavorable and requires significant energy input. The increase in$\Delta G^{\ddagger}$ with temperature is mathematically dictated by the equation $\Delta G^{\ddagger} = \Delta H^{\ddagger} - T\Delta S^{\ddagger}$: Since $\Delta S^{\ddagger}$ is negative, the term $-T\Delta S^{\ddagger}$ becomes increasingly positive as temperature increases, outweighing the slight decrease in $\Delta H^{\ddagger}$

The thermodynamic parameters obtained in this study are consistent with previous reports on saponification reactions. Onyango *et al*. (2022) reported positive $\Delta G^{\ddagger}$ values for vegetable oil saponification under standard conditions, with the reaction proceeding efficiently in practice due to non-standard conditions in the reaction mixture. The negative $\Delta S^{\ddagger}$ values are typical for bimolecular reactions where two reactant species must come together to form a single activated complex (Atkins and de Paula, 2023).

**Mechanistic Implications**

The kinetic and thermodynamic data provide insights into the mechanism of goat milk-enhanced saponification. The first-order kinetics with respect to neem oil suggests that the rate-determining step involves the attack of the hydroxide ion on the triglyceride ester bond. The presence of goat milk facilitates this step through several mechanisms:

1. Emulsification: Milk proteins (casein) act as natural emulsifiers, reducing the oil-water interfacial tension and increasing the interfacial area for reaction (Smith *et al*., 2023).
2. Catalysis: Milk lipases may catalyze the hydrolysis reaction, providing an alternative pathway with lower activation energy (El-Sayed *et al*., 2023).
3. Transition State Stabilization: Milk phospholipids and proteins may stabilize the transition state through specific molecular interactions, reducing the energy barrier (Malik *et al*., 2021).
4. pH Buffering: The lactic acid in milk provides buffering capacity, maintaining optimal pH conditions for the reaction (Singh and Singh, 2022).

The low activation energy (6.41 kJ·mol⁻¹) and positive enthalpy of activation (3.72-3.89 kJ·mol⁻¹) indicate that the reaction proceeds through a mechanism requiring minimal energy input, making goat milk an effective facilitator for saponification.

**CONCLUSION**

This study has successfully investigated the kinetics and thermodynamics of soap production using neem oil and sodium hydroxide with goat milk as a bioactive additive at elevated temperatures (303 K, 313 K, and 323 K). The following conclusions can be drawn:

The saponification reaction follows first-order kinetics with respect to neem oil concentration at all temperatures studied, as evidenced by the highest correlation coefficients ($R^2 \geq 0.956$) for the first-order model.

The rate constants ranged from 0.012 $s^{-1}$ to 0.014 $s^{-1}$, with higher temperatures yielding slightly faster reaction rates. These values are significantly higher than those reported for additive-free saponification, confirming goat milk's role as an effective reaction enhancer.

The activation energy was determined to be 6.41 $kJ\cdot mol^{-1}$, substantially lower than conventional saponification processes (15-40 $kJ\cdot mol^{-1}$), indicating that goat milk reduces the energy barrier for the reaction.

Thermodynamic analysis revealed positive enthalpy of activation (3.72-3.89 $kJ\cdot mol^{-1}$), negative entropy of activation (-260.89 to -261.42 $J\cdot mol^{-1}\cdot K^{-1}$), and positive Gibbs free energy of activation (82.94-88.16 $kJ\cdot mol^{-1}$). These parameters confirm the endothermic nature of the reaction, the formation of a more ordered transition state, and the energy requirement for activated complex formation.

The kinetic and thermodynamic data suggest that goat milk facilitates saponification through emulsification, catalysis, transition state stabilization, and pH buffering mechanisms.

These findings provide valuable insights for optimizing goat milk-based neem oil soap production in terms of reaction temperature, energy efficiency, and product quality. Future studies should investigate the effects of varying goat milk concentration, explore the use of other milk types, and evaluate the physicochemical properties of the resulting soap products.

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