

Kinetic Study and Thermodynamic Properties of Thermal Decoloration of *Raphia sese* Pulp Oil from Congo

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Abstract The aim of this study was to investigate the kinetics and thermodynamic properties of thermal bleaching of *Raphia sese* oil. The effectiveness of temperature in bleaching *Raphia sese* oil was investigated in this work. The result suggests that increasing temperature increases the bleaching rate of the oil. Two models were used to conduct the kinetic study of oil bleaching, the pseudo-first order and the pseudo-second order. The study revealed that pseudo-second-order is the kinetic model that effectively describes the experimental data of the *Raphia sese* oil bleaching process with rate constant values equal to $5.2548 \cdot 10^{-4}$, $6.1065 \cdot 10^{-4}$ and $6.7170 \cdot 10^{-4}$ g/mg.min respectively for temperatures of 150, 180 and 210°C. The model fits better as heating temperature increases. Thermodynamic properties were evaluated using parameters such as free energy variation (ΔG^0), enthalpy variation (ΔH^0) and entropy variation (ΔS^0). The enthalpy, entropy and activation energy were determined to be $875 \cdot 10^{-3}$ kJ/mol, $-244 \cdot 10^{-3}$ kJ/mol and 2.505 kJ respectively. The free energy ranged from 104.274 to 118.940 kJ/mol. The result indicates that a bleaching rate of up to 96% can be achieved with a temperature of 210°C for an oven heating time of 7 h, i.e. 420 min.

Keywords: kinetics, thermodynamics, thermal bleaching, *Raphia sese* oil

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1. Introduction

Vegetable oils play an essential role in our diet. That's why they have always played such an important role in the daily lives of people, who use them to perfume, flavour or even treat themselves. They make a major contribution to the body's energy supply and normal functioning. They are also a source of essential fatty acids, in particular linoleic and α -linolenic acids, and fat-soluble vitamins (E and D) not synthesized endogenously. Secondly, they provide the body with other constituents of nutritional interest, such as phytosterols and phenolic compounds with antioxidant activity [1]. Their availability and security of supply are essential elements in the choice of using these fats. A number of studies have been carried out in this area, given the undeniable importance of oils in various economic sectors, including the pharmaceutical, cosmetics and food industries. In order to give vegetable

oils a mild flavor and a pleasant color for the consumer, they are subjected to various operations that are part of the refining process, such as degumming, neutralization, decoloration and deodorization [2]. Decolorization is an essential step in oil refining, the main purpose of which is to eliminate undesirable coloration of the oil caused by pigments (chlorophylls, carotenoids) [2]. They also contribute to improving the organoleptic quality of foodstuffs, giving them a smooth, creamy, melting texture, a glossy appearance and a specific flavor. Oils also perform technological functions, in particular as a heat transfer medium in cooking, a coating and release agent, or as a carrier for lipophilic flavors and colorants [1]. Finally, *Raphia* oil is an edible vegetable oil of high nutritional quality. However, its quality depends on its physico-chemical and organoleptic characteristics, and on the content of natural components such as chlorophyll, polyphenols, carotenoids and fatty acids (mainly oleic and linoleic acids).

The Republic of Congo is a country rich in oil-producing plants. However, these resources are still

exploited on a small-scale. National production of *Raphia Sese* oil is essentially small-scale, despite the presence of a few industrial palm oil production units such as Eco-oil énergie, Congolaise de l'Industrie Agricole et du Tourisme and Sangha palme. These units only produce red oils, which do not necessarily attract consumers. This leads us to believe that artisans do not master the oil decoloration process, which involves heat treatment. The aim of this study was therefore to investigate the kinetics and thermodynamic properties of thermal decolorization of *Raphia sese* oil produced by Congolese artisanal producers.

2. Materials and Methods

2.1. Plant Material

Raphia sese oil (figure 1) from the Makotipoko district in the Plateaux department was purchased at the Ouenzé market in Brazzaville. This oil is known in the Republic of Congo as 'Mafuta kolo', and is used to prepare food and massage newborn babies.



Figure 1. *Raphia sese* oil

2.2. Determination of Chemical Indices and Fatty Acid Composition of the Oil Studied

The AFNOR NF T60-204 and NF T60-203 standards [3] were used respectively in the determination of the acid and saponification indices.

Regarding the fatty acid composition, 3 drops of oil are introduced into the flask using a Pasteur pipette. Add 3 pumice stones and 3 mL of sodium methoxide. Place the saponification cane on the flask and heat for 10 minutes at the thermostat. Then add 3 mL of acetyl chloride until the phenolphthalein discolors, and heat for 10 minutes and leave to cool. After cooling, add 8 mL of hexane then 10 mL of water. A separation of the two phases (aqueous and hexanic) is observed. 1 mL of the hexanic phase is collected in a vial and injected into a Focus brand CPG. Fatty acids are identified by comparison with calibration chromatograms.

2.3. Discoloration Kinetics of *Raphia sese* Oil

Discoloration kinetics is the monitoring of oil discoloration as a function of time and temperature. This study was carried out using a Memmert UN30 natural

convection oven. 10 g of crude oil were placed in vials numbered 1 to 15, then subjected to the oven at temperatures of 150, 180 and 210°C for 24h. Readings of the optical densities at 520 nm of the oils were obtained at 30-minute intervals until the optical density was almost constant. 0.05g of oil was diluted in 10 mL of carbon tetrachloride. Optical densities were measured using a JENWA 7205 UV/Visible spectrophotometer. The rate of oil bleaching at each heating time interval was defined as follows (equation 1) [4]:

$$\%bleaching = \frac{(OD)_0 - (OD)_1}{(OD)_0} \quad (1)$$

With $(OD)_0$: optical density of unbleached oil;
 $(OD)_1$: optical density of bleached oil.

2.3.1. Kinetic Models Used

In the present work, two empirical kinetic models such as pseudo-second-order and pseudo-first-order were used in their linear form to describe the thermal bleaching process of *Raphia sese* oil.

2.3.1.1. Pseudo-First-Order Kinetic Model

The expression for the pseudo-first-order velocity is given as (equation 2) :

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

Where q_e and q_t represent in this work the bleaching rate (in %) at equilibrium time and at any instant t , respectively; k_1 is the Pseudo-first-order thermal bleaching rate constant (in min^{-1}), and t is the heating time or duration (in min). Plots of $\log(q_e - q_t)$ versus t were used to express pseudo-first order at different temperatures from which q_e and k_1 were evaluated from the intercept and slope, respectively.

2.3.1.2. Pseudo-Second-Order Kinetic

The expression for the pseudo-second-order linear form is (equation 3):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (3)$$

Where q_e and q_t represent in this work the bleaching rate (in %) at equilibrium time and at any instant t , respectively; k_2 is the Pseudo-second-order thermal bleaching rate constant (in $\text{g/mg} \cdot \text{min}$), and t is the heating time or duration (in min). The pseudo-second-order rate constant k_2 and q_e for all three temperatures were determined from the slope and intercept of the plot of t/q_t versus t .

2.4. Estimation of Thermodynamic Parameters (ΔH , ΔS , ΔG°) and Activation Energy (E_a)

Thermodynamic parameters for thermal bleaching of *Raphia sese* oil were determined following the approach first described by [5] and later used by [6] using the following equation (4):

$$\ln\left(\frac{k_i}{T}\right) = \left[\ln\left(\frac{k_s}{h}\right) + \left(\frac{\Delta S}{R}\right) \right] - \left[\left(\frac{\Delta H}{R}\right) \left(\frac{1}{T}\right) \right] \quad (4)$$

Where k_i is the reaction rate constant, k_s is Boltzmann's constant ($1.38 \times 10^{-23} \text{ J.K}^{-1}$), h is Plank's constant ($6.63 \times 10^{-34} \text{ J.s}$) and R is the gas constant.

From this equation (4), we can determine: ΔH , ΔS and ΔG° by regressing $\ln\left(\frac{k_i}{T}\right)$ as a function of $\frac{1}{T}$.

The linear form of the Arrhenius equation is used to determine the activation energy of the bleaching process by plotting $\ln k$ as a function of $1/T$ [7].

2.5. Comparison of Model Accuracy

The values of two statistical parameters were used to compare the fit of the data predicted by the kinetic models with the curves of the experimental data. The two parameters used are the coefficient of determination (R^2) and the chi-square test (χ^2).

The coefficient of determination (R^2) is a measure of the closeness of the statistical data to the regression line [8]. The R^2 value is the first criterion for selecting the most suitable model for the kinetic study [9]. When comparing model fit to experimental data curves, the best-fitting model should have the highest R^2 values, and conversely, it should have the lowest χ^2 values [10] and [11].

The statistical parameter R^2 was calculated automatically using originpro2018 software.

3. Results and Discussion

3.1. Physicochemical Characterization and Fatty Acid Composition of *Raphia sese* Oil

Unbleached *Raphia sese* oil was analyzed to determine its physicochemical properties and chemical composition. The results are reported in Table 1.

The saponification number provides information on the length of the carbon chain making up the oil. Determination of the saponification value is based on the molecular weight and average length of the fatty chain, to which it is inversely proportional (the higher the molecular weight and average length of the fatty chain, the lower the saponification value). Crude *Raphia sese* oil has a saponification value of 234.42 mg KOH/g oil. this result corroborates that of silou et al. [4]. This saponification value indicates that *Raphia sese* oil can indeed be used in soap production.

Acid value and acidity are oil quality criteria. It provides information on the degree of alteration following chemical or enzymatic hydrolysis under the right conditions [12]. Acid value or acidity is a fundamental characteristic of oil quality. This index is also linked to the sanitary freshness of the crushed fats, and to the mastery of the technological process used to preserve, store and process these oils. Increasing the acid value or acidity index leads to fat degradation. The higher the value, the more the oil is degraded. The acid value determined for the *Raphia sese* oil studied is 19.63 mg KOH/g oil, i.e. an acidity of 9.88%. This value is well above 4 mg KOH/g oil, the maximum value for an edible oil according to the Codex Alimentarius commercial standard [13].

The *Raphia sese* oil studied consists mainly of the common fatty acids palmitic, oleic and linoleic. In this respect, it is similar to palm oil [4]. However, *Raphia sese* oil has the advantage of a slightly higher mono- and di-unsaturated fatty acid content [4]. Linolenic acid content is less than 2%, the limit for frying oils. These characteristics give *Raphia sese* oils the status of a frying oil source of essential fatty acids.

3.2. Kinetic Study of Thermal Bleaching of *Raphia sese* Oil

3.2.1. Effects of Heating Time and Temperature on Bleaching Efficiency

Figure 2 shows the evolution of the bleaching rate of *Raphia sese* oil as a function of heating time at temperatures of 150°C, 180°C and 210°C. These curves characterize the overall behavior of carotenoid degradation during the heating process. The three curves show that thermal bleaching of *Raphia sese* oil increases very rapidly over the first 30 minutes, then slows down progressively. After initial rapid degradation of the carotene pigments, bleaching efficiency increases at a much slower rate until it becomes constant. This trend has also been observed by several authors who have studied the bleaching process in the presence of adsorbents. In the latter case, several researchers explain this phenomenon in the gradual reduction in the number of vacant adsorbent sites [14,15,16,17].

Examination of figure 2 shows that increasing temperature increases bleaching efficiency, i.e. the bleaching rate increases with temperature.

Bleaching efficiency is less significant between 150 and 180°C, but very significant between 180 and 210 °C. This trend can be explained by undesirable structural changes in the oil molecule (carotenoids) through degradation, oxidation and isomerization [18]. Mohammed et al. [19] reported a similar trend.

Table 1. Analysis of crude (unbleached) *Raphia sese* oil

Saponification value (mg KOH/g huile)						Acid value (mg KOH/g huile)				
234,42						19,63				
Composition en acides gras (%)										
C14:0	C16:0	C16:1	C16:2	C18:0	C18:1 (n-9)	C18:2 (n-6)	C20:0	C18:3 (n-3)	AGS	AGI
0,20	41,14	0,14	0,09	10,18	11,88	35,47	0,19	0,71	51,71	48,20

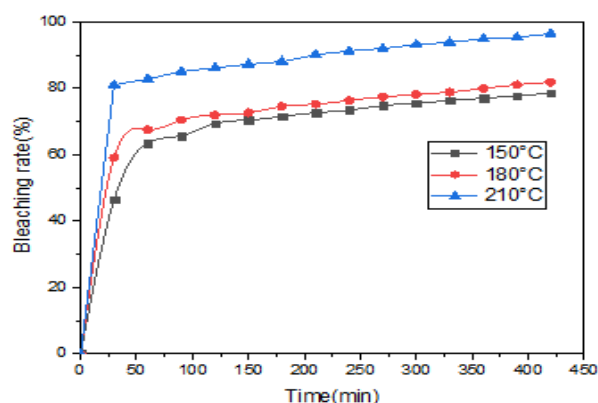


Figure 2. Effect of time on the bleaching efficiency of palm oil

3.2.2. Kinetic Models

Figure 3 plots $\log(q_e - qt)$ as a function of time t . This figure expresses the pseudo-first order at different temperatures. The equilibrium bleaching rate (q_e) and rate constant (k_1) were evaluated from the intercept and slope respectively. (Table 2)

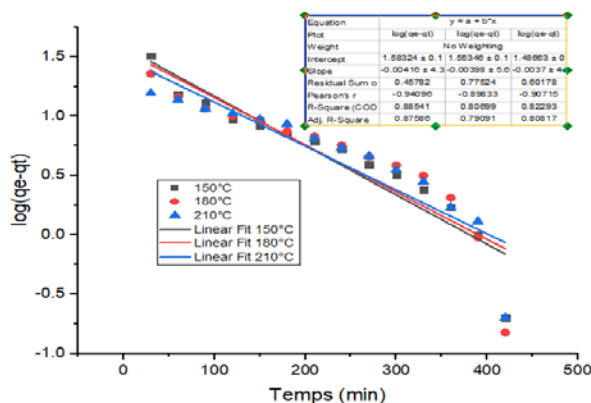


Figure 3. Pseudo-first-order kinetic diagram of thermal decolorization of *Raphia sese* oil

The coefficients of determination (R^2) show values below 0.990, indicating poor correlation. The thermal decolorization process at temperatures of 150, 180 and 210 °C of *Raphia sese* oil does not follow the pseudo-first-order kinetic model.

Table 2. Calculated kinetic parameters of the bleaching process

Kinetic models	Temperature (°C)		
	150	180	210
Pseudo-first-order			
k_1	$9,5805 \cdot 10^{-3}$	$9,1659 \cdot 10^{-3}$	$8,5211 \cdot 10^{-3}$
q_e calculated	38,3036	35,7651	30,6641
q_e experimental	78,70	81,95	96,60
R^2	0,8854	0,8070	0,8229
R^2_{adj}	0,8759	0,7909	0,8082
Pseudo-second-order			
k_2	$5,2548 \cdot 10^{-4}$	$6,1065 \cdot 10^{-4}$	$6,7170 \cdot 10^{-4}$
q_e calculated	81,8331	84,0336	98,3284
q_e experimental	78,70	81,95	96,60
R^2	0,9994	0,9987	0,9988
R^2_{adj}	0,9993	0,9986	0,9987

The equilibrium bleaching rate (q_e) and rate constant (k_2) of the pseudo-second-order thermal bleaching of *Raphia sese* oil at different temperatures were determined from the intercept and slope of the plot of t/qt versus time t (Figure 4).

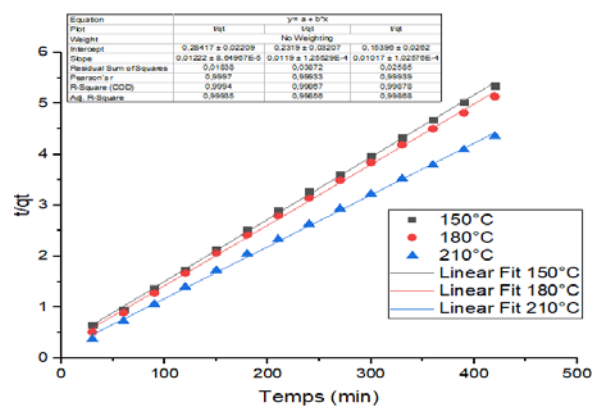


Figure 4. Pseudo-second-order kinetic diagram of thermal decolorization of *Raphia sese* oil

The correlation or determination coefficient (R^2) has very high values, above 0.997 for thermal bleaching. This suggests a very good correlation of the thermal bleaching process of *Raphia sese* oils. Similarly, the values of the equilibrium bleaching rate calculations (q_e) were very close to the experimental values. Consequently, the pseudo-second-order model is satisfactory and applicable to the thermal bleaching of *Raphia sese* oil.

Table 2 shows the calculated kinetic parameters of the *Raphia sese* oil bleaching process at three temperatures: 150, 180 and 210°C. Two kinetic models were studied: pseudo-first-order and pseudo-second-order. The coefficient of determination R^2 values for the two models studied at different temperatures ranged from 0.8070 to 0.8854, and from 0.9987 to 0.9994, for pseudo-first-order and pseudo-second-order respectively. The pseudo-second-order model at all three study temperatures proved the best fit to the experimental data, with coefficient of determination values close to 1. The calculated q_e was found to be close to the experimental q_e . It has been reported that if the calculated q_e values were not close to the experimental q_e values, the process would be assumed not to have followed the kinetic model even if it has a high R^2 correlation coefficient value [20] et [17].

Nwabanne et al. [17] also found the good fit of the pseudo-second-order model with a maximum R^2 value of 0.999 in palm oil bleaching using activated Nando clay at different temperatures compared to the pseudo-first-order and intra-particle diffusion models.

The pseudo-second-order kinetic model was found to be best suited to the thermal bleaching process of *Raphia sese* oil, with rate constant values equal to $5.2548 \cdot 10^{-4}$, $6.1065 \cdot 10^{-4}$ and $6.7170 \cdot 10^{-4}$ for temperatures of 150, 180 and 210°C respectively. The better the model fits, the lower the heating temperature. $R^2 = 0.999$ for discoloration at 150°C. As can be seen from Table II, the values of the rate constants k (bleaching rate constant) and equilibrium bleaching rates (q_e) increase with temperature.

Figures 5, 6 and 7 show, respectively, the fits of the pseudo-second-order kinetic model with experimental data for temperatures of 150, 180 and 210°C.

Figures 5, 6 and 7 show the dispersion of the experimental data around the line representing the calculation data. The lower the temperature, the closer the dispersion to this line. This indicates that the pseudo-second-order model best describes the thermal discoloration behavior of *Raphia sese* oil at a temperature of 150°C. A good correlation between experimental and predicted data can be observed (Figure 5).

From these results, we can conclude that the pseudo-second-order kinetic model better represents the thermal decolorization process in the oven at 150°C of *Raphia sese* oil than at temperatures of 180 and 210°C, and even better than the pseudo-first-order kinetic model.

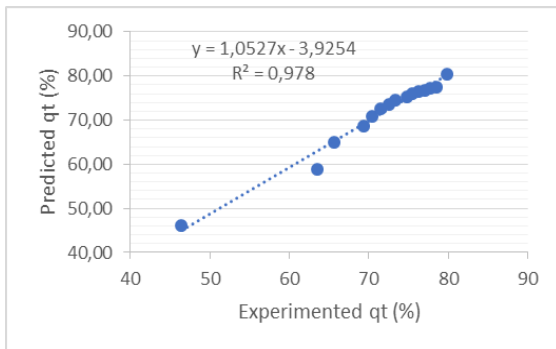


Figure 5. Experimental data at 150°C and estimated bleaching rate, calculated by the pseudo-second-order model

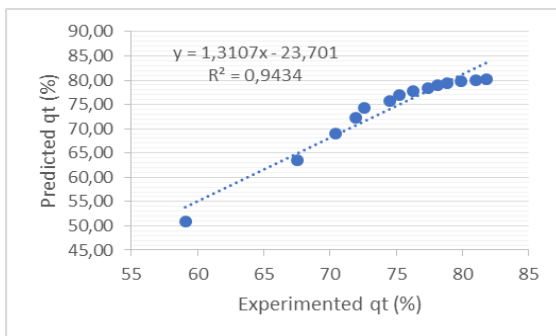


Figure 6. Experimental data at 180°C and estimated bleaching rate, calculated by the pseudo-second-order model

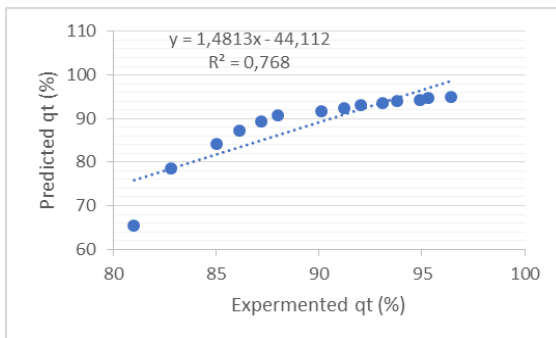


Figure 7. Experimental data at 210°C and estimated bleaching rate, calculated by the pseudo-second-order model

3.3. Thermodynamic Study of Thermal Decolorization of *Raphia sese* Oil

Thermodynamic properties were evaluated using

parameters such as free energy variation (ΔG^0), enthalpy variation (ΔH^0) and entropy variation (ΔS^0). ΔG^0 determines whether the process is feasible and spontaneous or not, ΔH^0 determines whether the process is exothermic or endothermic, and ΔS^0 determines the increase or decrease in randomness of the process. Reactions occur spontaneously at a given Temperature if ΔG^0 is a negative quantity [21].

Plotting $\ln(k/T)$ as a function of $1/T$ (absolute temperature) in Figure 8, the quantities ΔH and ΔS were calculated from the slope and intersection respectively. The results are given in Table 3.

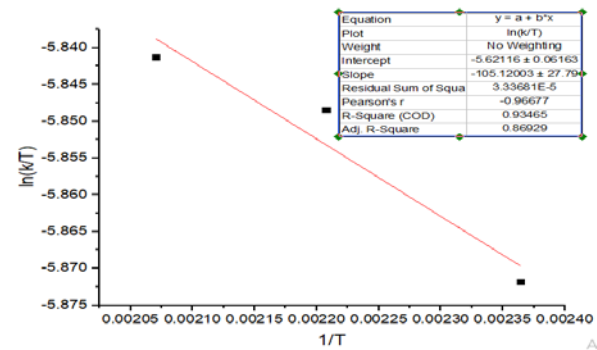


Figure 8. Thermodynamic diagram for bleaching *Raphia sese* oil

The enthalpy and entropy values calculated using equation (4) are shown in Table 3.

The enthalpy of activation of oil decolorization is 875.10^{-3} kJ/mol. Positive values of ΔH indicate the endothermic nature of the bleaching process, which leads to an increase in carotenoid degradation with increasing temperature [22]. This implies that more heat is required to trigger the degradation reactions of pigment molecules. In an endothermic process, the total energy adsorbed in bond breaking is more than the total energy released in the creation of other bonds. Consequently, more energy is required as the process progresses.

Table 3. Enthalpy and entropy of activation for the decolorization of *Raphia sese* oil

Parameters	Temperature (K)		
	423	453	483
ΔG^0 (kJ/mol)	104,274	111,607	118,940
ΔH^0 (kJ/mol)	875.10^{-3}	-	-
ΔS^0 (kJ/mol/K)	-244.10^{-3}	-	-

The activation entropy for the decolorization of *Raphia sese* oil is -244.10^{-3} kJ/mol. Negative entropy values indicate that the transition state has less structural freedom than the reactant (the reacting molecule) [23]. The positive enthalpy value and negative entropy value is a common value for thermal reaction [5,22] et [24].

Gibbs free energy is defined as the difference between the energies of the reactants and the activated state and is generally used as a measure of the spontaneity of the process [22]. Positive ΔG^0 values indicate that thermal discoloration of the oil leading to thermal degradation of the carotenoid pigments is a non-spontaneous reaction.

Figure 9 shows the plot of $\ln(k)$ versus T^{-1} for thermal bleaching of *Raphia sese* oil at the three temperatures when the pseudo-second-order model was used. Activation energy is calculated from the slope.

The activation energy for thermal bleaching of *Raphia sese* oil is 2.505 kJ. This is the energy required to initiate or start the bleaching process.

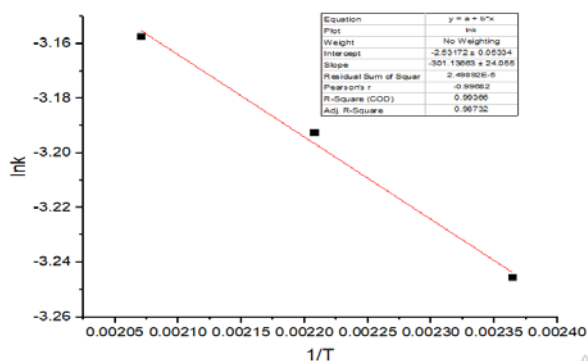


Figure 9. Curve of the linear form of the Arrhenius equation

With an R^2 value equal to 0.9936 for the determination of the activation energy of the thermal decoloration of *Raphia sese* oil in an oven, based on the Arrhenius diagram $\ln(k)$ as a function of $1/T$, it makes sense to consider this decoloration process as following the pseudo-second-order model. This value of the activation energy is therefore a satisfactory translation of the discoloration process.

4. Conclusion

In this work, decolorization of *Raphia sese* oil was carried out by oven heating at three different temperatures, namely 150, 180 and 210°C. Pseudo-first-order and pseudo-second-order kinetic models were used to describe the decoloration process. Of these two models, only the pseudo-second-order model was able to effectively describe the experimental data on the decolorization process of *Raphia sese* oil.

The thermodynamic study revealed that the thermal discoloration process is endothermic, non-spontaneous and entropy-depleting.

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