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International Journal of Engineering Research & Technology (IJERT)
ISSN: 2278-0181
Vol. 14 Issue 07, July - 2025

Thermodynamic and Kinetic Study of the Dehydration of Ethanol using Potato Starch as a Biosorbent

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Abstract
This study thoroughly explores ethanol dehydration using potato starch as a biosorbent, analysing the thermodynamics, isotherms, and kinetics. The potato tuber was peeled, sliced into pieces and soaked in 0.2% sodium metabisulphite then the grinded. The resulting slurry was washed, filtered, sun dried, dehydrated and finely grinded. The potato starch was modified with sodium hypochlorite and allowed to stand for four hours. The slurry was then filtered and bone-dried in an oven at 50^{o}C and preserved in a desiccator. Two grammes of modified starch were weighed in a chemical weighing balance and placed in different conical flasks. 20ml of different concentration of ethanol-water mixture were measured and added to the content of the flasks. The flasks containing the solutions and the adsorbent were allowed to stand for 30min, the solution was then filtered and the refractive index of the filtrate checked using a refractometer. The final concentration of the corresponding refractive index was then determined. The concentration of the ethanol-water mixture was improved by stage-wise treatment with the modified starch. A four stage-wise contact treatment showed an improved quality (conc.) of ethanol-water mixture. A four stage treatment was able to raise the ethanol-water mixture concentration from 60% to 74.33%. The study demonstrated that the adsorption process is thermodynamically feasible, spontaneous and favoured at low temperatures (Gibbs free energy was negative; -4786.37 kj/mol.K), endothermic (change in enthalpy was positive; 2186 kj/mol) and occurred with a decreased order of disorderliness (change in enthalpy was negative; -5.2916 kj/mol.K) at adsorbent-adsorbate interface. The experimental data in the adsorption process indicated good correlations with the Langmuir isotherm and pseudo-second-order kinetic model. The research shows that potato starch can effectively dehydrate ethanol and has various other industrial uses. A stage-wise treatment process is recommended for the ethanol-water mixture. Modified potato starch offers technical and economic benefits, being eco-friendly, reusable, and biodegradable. Further studies using additional isotherm and kinetic models are suggested to better analyse adsorption data.

***Keywords*: Biosorbent, Thermodynamic, Kinetic, Isotherms, Starch-Based Adsorbents**

1.0 INTRODUCTION

Ethanol is one of the most used biofuels that contributes diminishing environmental effects of fossil fuels. Its properties and its renewable origin ensure environmental sustainability and process economy. The importance of ethanol as fuel and for other industrial uses has resulted in the need to produce it in a large quantity with particular reference to dehydration, (Okafor *et al*., 2014), and as such, a lot of research efforts continue to highlight on the improvement of the dehydration of the ethanol - water mixtures. Protection of the global environment and depletion of the conventional hydrocarbon fuel supplies have driven researchers to alternative fuels including hydro, wind, biofuels, solar and geothermal energy (Frolkova and Raeva, 2010; Kumar *et al.*, 2010),

Among these suggested alternatives, biofuels have drawn more attention (Okewale *et al*., 2015). Ethanol is commonly used as a fuel itself or an additive that helps improve the octane number and combustibility of gasoline (Diaz, *et al*., 2010). Anhydrous ethanol is one of the bio-fuels produced today; others include biodiesel, and biogas. One of the energy-efficient methods widely used for ethanol dehydration is adsorption process and adsorption by using starchy adsorbent was used due to its numerous benefits (Jeong *et al*., 2012)

Starch and its derivatives represent a cheap and environmentally safe source of raw material for the preparation of low-cost adsorbents (Zhang *et al.*, 2008). This biopolymer represents an interesting alternative as an adsorbent because it is an abundant, renewable and biodegradable raw resource (Baranwal et al., 2022).

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Starch, cellulose, hemicelluloses and starch – based materials have affinity for water and are able to be regenerated at temperature of 80 ^{O}C and lower. It has been shown that ethanol dehydration by adsorption requires far less energy than the conventional azeotropic distillation. Adsorption with biomass adsorbent is also less-energy consuming than adsorption with other adsorbents (Chrontira and Panarat, 2010). Biomass materials that have been investigated and found to be viable adsorbents include cassava starch, corn grits, potato starch, ligno-cellulosic, amylase, and corn starch (Mya, 2011). Adsorption isotherm is basically important to describe how solutes interact with adsorbents whose parameters express the surface properties and affinity of the sorbent at a fixed temperature and is critical in optimizing the use of adsorbents (Tan and Hameed, 2010).

Starch based adsorbents adsorb water by forming hydrogen bond between the hydroxyl groups (-OH) of the glucose unit and the water molecules (Beery and Ladisch, 2001), also water is by nature more polar than organic compounds such as alcohols. It however interacts with hydroxyl groups of glucose containing adsorbent with higher strength and faster rate. Consequently, aqueous alcohol solution passing through bed of starchy or cellulose material would improve the concentration of ethanol in the solution. Advantages of these starch based adsorbents in dehydration of ethanol-water mixtures include; non-toxicity, availability, biodegradable and renew ability (Okewale *et al.*, 2011).

Conventional ethanol dehydration methods, such as distillation, are highly energy-intensive, which significantly increases production costs. Distillation, a common technique for removing water from ethanol, requires large amounts of heat to separate the two substances due to their azeotropic nature. This results in substantial energy consumption, driving up operational expenses. The need for such intensive processes to achieve high-purity ethanol makes ethanol production costly, particularly in industrial applications where large volumes are needed (Tse *et al*., 2021)

In addition to these high production costs, countries that rely on ethanol imports face significant financial burdens. Ethanol is crucial for various sectors, including medical, pharmaceutical, research, and industrial fields. Importing ethanol involves shipping, logistics, taxes, and customs duties, all of which contribute to a substantial annual outlay. This dependence on external sources also exposes the country to price fluctuations in the global market, further increasing costs. Consequently, the combined impact of energy-intensive production and expensive imports results in high overall expenses for ethanol (Mizik, 2021).

Thus, the aim of this study was the dehydration of the ethanol-water solution using potato starch as biosorbent.

2.0 MATERIALS AND METHOD

The basic apparatus and standard chemicals (reagents) were used for this experiment. The potatoes used in this study were obtained at Anua market, Uyo, Akwa Ibom State, Nigeria. The experiment was conducted at the Chemical Engineering Laboratory at the University of Uyo, Nigeria.

2.1 Method of Starch Extraction

Starch was extracted from the potato tubers using a slight modification in (Anso, 2016). Tubers were manually peeled, cut into smaller pieces and soaked in 0.2% sodium metabisulphite for 10 minutes, and then the juice was extracted. The potatoes were then grinded, suspended in water and allowed to settle unhindered overnight. This process was repeated three times to eliminate residual sulphite. The resulting starch slurry was then filtered and a hydraulic press was employed to remove excess water from the starch. It was left under the press for 5 hours, then sun-dried. The starch was manually broken down by hand and filtered using a home available sieve then it was dehydrated locally with a stove and pan with constant stirring to remove any remaining moisture while retaining its whitish appearance. It was grinded again to achieve a fine starch powder.

2.2 Modification of Starch

Preparation of hypochlorite (oxidized starch): This was done using slight modification according to (Anso, 2016). One hundred grammes of the unmodified starch were dispersed in 500ml of de-ionized water in a glass beaker. The pH of the slurry was recorded to be 7.24 and then adjusted to 9.23 using 10% NaOH. Ten grammes of Sodium hypochlorite solution was added to the slurry slowly with constant stirring until a pH of 10.21 was obtained and then the resulting solution was allowed to stand for 4 hours. Finally, the pH of the mixture was recorded and then adjusted to 7.0 using 5% HCl and the slurry filtered through whatman No. 1 filter paper. The residue was washed four times with de-ionized water and then bone-dried in an oven at 50ºC, packed in an aluminium foil and stored in a desiccators.

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2.3 Dehydration Analysis

Ethanol concentration – Refractive index calibration curve was prepared using ethanol concentration ranges of 20%, 30%, 40%, and 60% using MS excel (2016). Two grammes of the sample of the modified starch was weighed on a chemical weighing balance and placed in different conical flasks. Twenty (20) ml of ethanol of different concentrations was measured and added to the content of the flasks. The flasks containing the solution and the adsorbent were intimately mixed and allowed to stand for 30 min. The solution was filtered and the refractive index of the filtrate checked using a refractomer. The final concentration of the corresponding refractive index was then determined.

2.4 Stage Wise Treatment Process

To further improve the quality (conc.) of the ethanol solution, a stage-wise contact treatment process was employed. A four stage-wise contact treatment showed an improved quality (conc.) of the ethanol-water mixture as the four stage treatment was able to raise the concentration of the ethanol solution from 60% to 74.33%.

2.5 Thermodynamic Study

Adsorption thermodynamic parameters were calculated from the temperature variations at a constant adsorbent weight and initial concentration. Thermodynamic parameters such as Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) were determined from conclusions of the analysis. Gibbs free energy (ΔG) was calculated using;

$$\Delta G^{\circ} = -RT \ln K \qquad \text{Equation 1}$$

Negative ΔG values indicated the feasibility and spontaneous nature of the adsorption process, positive value of change in enthalpy (ΔH) indicated that the adsorption was an endothermic process. Change in enthalpy (ΔH) was calculated using;

$$\Delta H_T = \Delta H_T^o + \int C_p dT \qquad \text{Equation 2}$$

A negative value for ΔS indicated decreased randomness. Change in entropy (ΔS) was calculated using;

$$\Delta S_T = \int C_p dT - \int \frac{R d_P}{P} \qquad \text{Equation 3}$$

2.6 Isothermal Study

Freundlich and Langmuir isotherm models were employed to conduct this study. For the Freundlich model, the Freundlich constant (K_F) represents adsorption capacity, therefore, the greater the value of K_F, the greater the adsorption capacity.

2.7 Kinetic Study

The kinetic models utilised here were the pseudo first order and pseudo second order models. The correlation coefficient of second order kinetic model should be greater than that of first order kinetic model indicating pseudo second order model might be best fit for this process.

3.0 RESULTS AND DISCUSSION

In this section, results obtained from the experimental data are treated using arithmetic, graphs and tables.

3.1 Calibration Curve

Ethanol concentration – Refractive index calibration curve was prepared using ethanol concentration ranges of 20%, 30%, 40% and 60% using Microsoft excel (2016). Two (2) grammes of the sample of the modified starch was weighed on the chemical weighing balance and placed in different conical flasks. Twenty (20) ml of Ethanol of different concentrations was measured and added to the content of the flasks.

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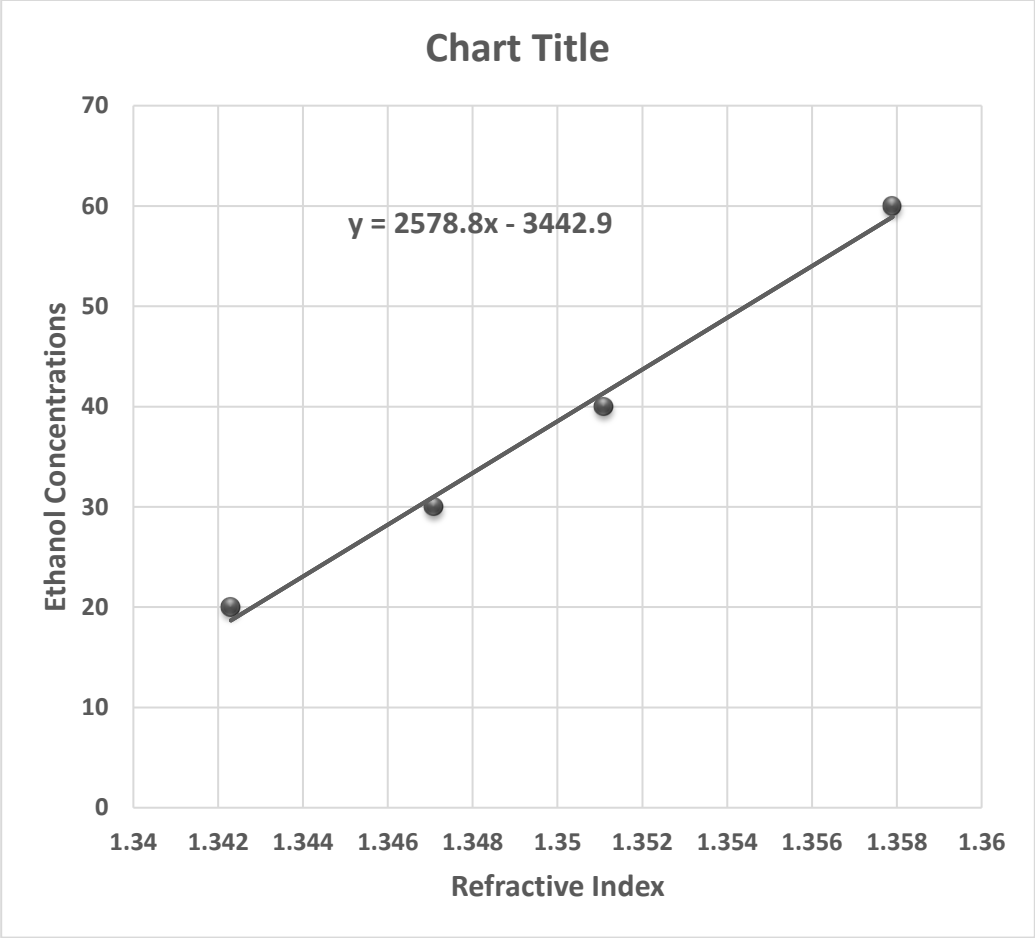

Figure 1: A Calibration Plot of Ethanol Concentration - Refractive Index

3.2 Stage-Wise Treatment Process

Using the best fit equation of the calibration curve in Figure 1 above;

$$y = 2578.8x - 3442.9 \qquad \text{Equation 4}$$

Table 1: Stage-wise treatment process of the dehydration analysis

Time (min)	Initial Conc. (%)	Amount of Starch used (g)	Volume of Ethanol used (ml)	Refractive index	Final Conc. (%)	Volume of Ethanol Recovered (ml)	Stage
30	60.00	10	100	1.3596	63.24	89	1
30	63.24	8	80	1.3608	66.33	70	2
30	66.33	6	60	1.3622	69.94	53	3
30	69.94	4	40	1.3639	74.33	39	4
30	74.33	2	20	1.3639	74.33	22	5

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From the table of result shown above, the optimum concentration of 74.33% was obtained when 4g of modified starch was contacted with 40 ml of ethanol at initial concentration of 69.94% and at contact time of 30 mins and the volume of ethanol recovered was 39 ml. Anso (2016) performed a stage-wise treatment of ethanol dehydration getting an optimum concentration of 74.07%. Thus, the obtained result was close to this.

3.3 Adsorption Studies and Analysis

Adsorption study was performed by the batch adsorption method by varying different parameters including temperature, initial concentration of sorbate and contact time, to find conditions best suited for the removal of water from ethanol-water solution.

3.3.1 Effect of temperature

The effect of temperature on adsorption was studied by using temperature range 30℃, 40℃ and 55℃ with pH 7. Adsorption of water onto potato starch was found to decrease with increase in temperature. This decrease is possible because the increase in thermal energy may induce higher mobility of the adsorbate causing the water molecules to break out from the sorbent surface. This finding is in line with results from Ebisike *et al.,* (2018).

3.3.2 Effect of Initial Concentration

Initial concentration of sorbate is another important parameter, which affects the adsorption phenomenon. For this purpose, initial concentration of water was varied in ranges of 40%, 60% and 70% by keeping all other parameters constant. Increase in adsorption capacity was observed initially for adsorption. So, rise in concentration also raised adsorption of sorbate on available sites. This result is in correlation with findings by Fozia *et al.,* (2018).

3.3.3 Effect of Contact Time

Contact time was varied from 10 to 30 minutes under neutral conditions with constant amount of sorbent (10g), initial concentration (40%). Maximum adsorption was achieved at 30 minutes time interval and no significant increase found by further increase in time. Fozia *et al.,* (2018) found that adsorption of cadmium on *Saccharum arundinaceum* was maximum at 60 minutes time interval and no significant increase found by further increase in time.

3.3.4 Thermodynamic Study

The thermodynamic parameters ΔG° (standard free energy), ΔH° (enthalpy change) and ΔS° (entropy change) were calculated to determine the characteristic energy change of the sorbent material and the sorption mechanism after adsorption. The values of ΔH° and ΔS° obtained from the slope and intercept of the linear plot of ($\ln K$) against (1/T).

Table 2: Values for plot of $\ln K$ against 1/T

K	ln K	T (K)	1/T ℃
6.67	1.9	303	0.0033
5.69	1.74	313	0.0032
3.83	1.34	328	0.00304

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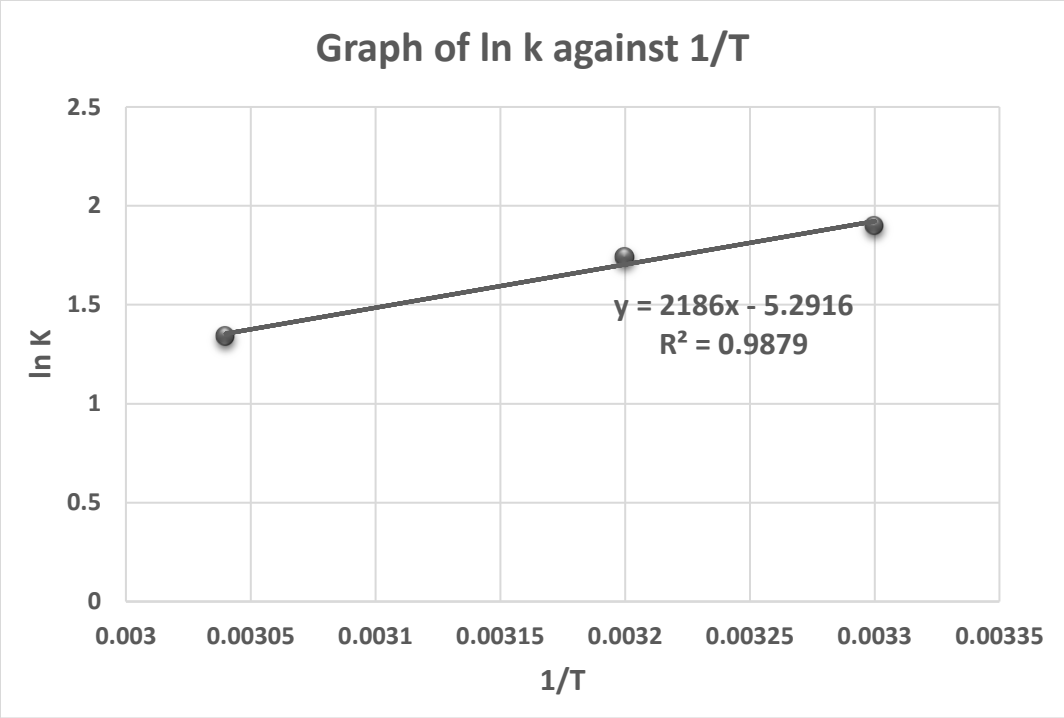

Figure 2: Graph of ln K against 1/T

Table 3: Thermodynamic parameters

Parameters	Temperature (K)	Results
Gibbs Free Energy (kj/mol.K)	303	-4786.37
	313	4527.97
	328	-3654.17
Enthalpy (kj/mol)	303	2186
	313	3630.2
	328	5363.24
Entropy (kj/mol.K)	303	-5.2916
	313	-3.8474
	328	-2.1144

These results indicate that the adsorption of water onto potato starch is spontaneous and greatly favourable ($\Delta G° < 0$) and endothermic ($\Delta H° > 0$) in nature. Also, increase in $\Delta G°$ value as temperature increase is an indication that the sorption of water onto potato starch was favoured at lower temperature. The negative $\Delta S°$ value denotes the decreased randomness at the solid–solution interface during the adsorption process. This finding is in line with results from Ebisike *et al*., (2018) who investigated the adsorption of cadmium and nickel onto chitosan.

3.3.5 Isothermal Study

Adsorption isotherms were investigated for 30 – 60% initial ethanol concentrations using 10g of the raw adsorbent added to 100 ml of the solution. Model employed for this study were the Freundlich and Langmuir model. Isothermal study was conducted at a constant temperature of 30℃.

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3.3.5.1 Freundlich Model

Freundlich isotherm was obtained by plotting ln C_e against ln q_e (Mustapha *et al.*, 2019). K_F and *n* are constants obtained from intercept and slope, respectively. Freundlich adsorption capacity (K_F) is an indicator of a system, whether it is favourable for adsorption or not. Adsorption is considered promising if value of K_F is found in range of 1–20, and results reveal that in the present study, K_F was 2.55. Similarly, adsorption intensity represented by *n* indicates fitness of model for adsorption purposes if value of *n* is above 1 and results reveal that in the present study, n was 1.35. Value of R^2 obtained from the plot is significant (0.9982) representing good fitness of this model.

Table 4: Values for the plot of ln Ce against ln qe

Adsorbate Concentration (%)	C_e (ml)	q_e (ml)	ln C_e	ln q_e
40	24	1.6	3.18	0.47
60	38	2.2	3.63	0.79
70	44	2.5	3.78	0.92

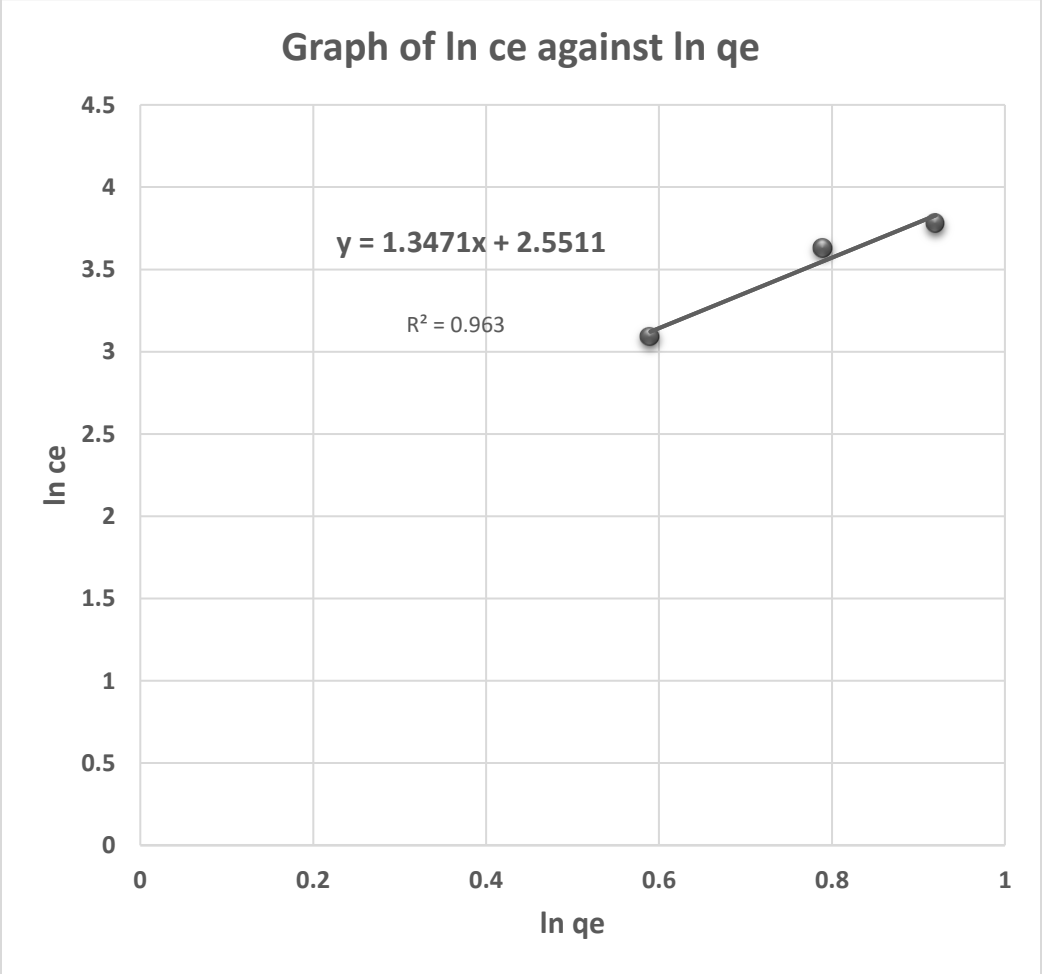

Figure 4.3 Graph of ln C_e against ln q_e

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3.3.5.2 Langmuir Model

Langmuir isotherm was obtained by plotting C_e/q_e against C_e (Mustapha *et al.,* 2019). Langmuir constant (K_L) and maximum adsorption capacity (q_{max}) are constants obtained from intercept and slope, respectively. Where q_{max} is the inverse of slope i.e. 1/slope and K_L is $1/q_{max} \times$intercept. R^2 value (0.9645) obtained for plot was found satisfactory showing fitness of model on the adsorption experiment. K_L was found to be 0.01 and q_{max} was gotten as 6.65. A dimensionless constant R_L is calculated by using Langmuir constant, and initial concentration represents model fitness for a particular system.

$$R_L = \frac{1}{(1+K_L C_E)} \qquad \text{Equation 5}$$

If value of R_L falls between 0 and 1, the system is considered appropriate for adsorption purpose and table below shows results which are in this range. The R_L in the range of 0 –1 decreased with increasing initial metal ion concentration which indicates favourable uptake of water. For the Langmuir model, the Langmuir constant (K_L) represents affinity between adsorbent and adsorbate, therefore, the greater the value of K_L, the greater the affinity. The Langmuir isotherm showed better fit to the experimental data with higher correlation coefficients for all concentrations.

Table 5: Values for the plot of C_e/ q_e against C_e

Adsorbate Concentration (%)	C_e (ml)	q_e (ml)	C_e/ q_e	R_L
40	24	1.6	15	0.69
60	38	2.2	17.27	0.6
70	44	2.5	17.6	0.56

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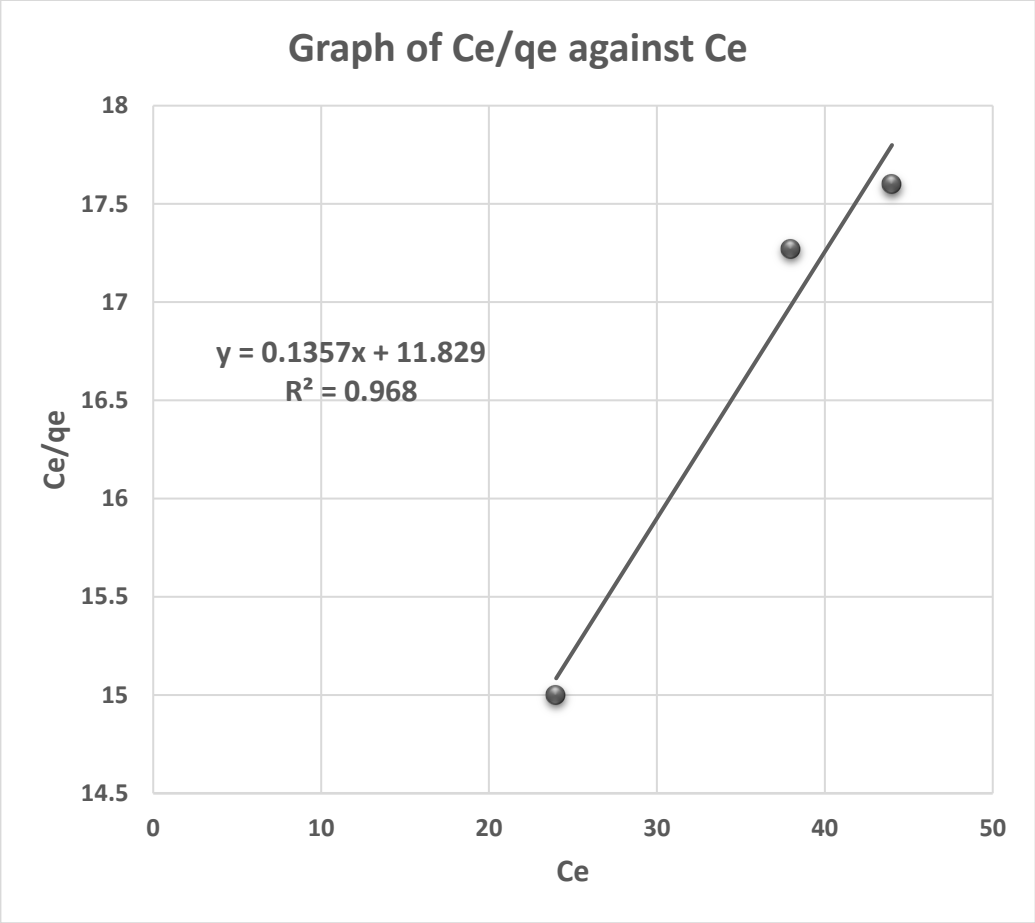

Figure 4: Graph of Ce/qe against Ce

The Langmuir isotherm showed better fit to the experimental data with higher correlation coefficients for all concentrations. The results showed a correlation of values to the experimental data. This result is in line with findings by Mustapha *et al.,* (2019) who investigated the adsorption of lead, cadmium, zinc and copper from aqueous solutions using *Albizia lebbeck* pods.

3.3.6 Kinetic Study

The adsorption kinetics and rate constants were determined from kinetic models including the pseudo-first-order and pseudo-second-order models. The pseudo-first-order and pseudo-second-order adsorption kinetics based on equilibrium adsorption are represented as follows:

$$\ln(q_e - q_t) = \ln q_t - k_1 t \qquad \text{Equation 6}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad \text{Equation 7}$$

Where q_e and q_t are the amounts of water adsorbed onto the potato starch at equilibrium and at a time, *t*, respectively. k_1 and k_2 are the rate constants for pseudo-first-order and pseudo-second-order kinetics, respectively. The first-order constant (min^{-1}) was determined in linear form by plotting ln (q_e –q_t) against t. A plot of t/q_t against t was used to determine pseudo-second-order constant (mg/g min) (Mustapha *et al.,* 2019). The correlation coefficient of second order kinetic model was greater than that of first order kinetic model indicating pseudo second order model might be best fit for this process. Kinetic study of adsorption of water onto potato starch was conducted at varying contact times of 10 mins, 20 mins and 25 mins, constant ethanol concentration of 60% and 10g of adsorbent.

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Table 6: Values for kinetic study plot

t	q_t (ml)	$\ln(q_e - q_t)$	t/q_t
10	0.5	0.0953	20
20	1.1	-0.693	18.18
25	1.4	-1.609	17.86

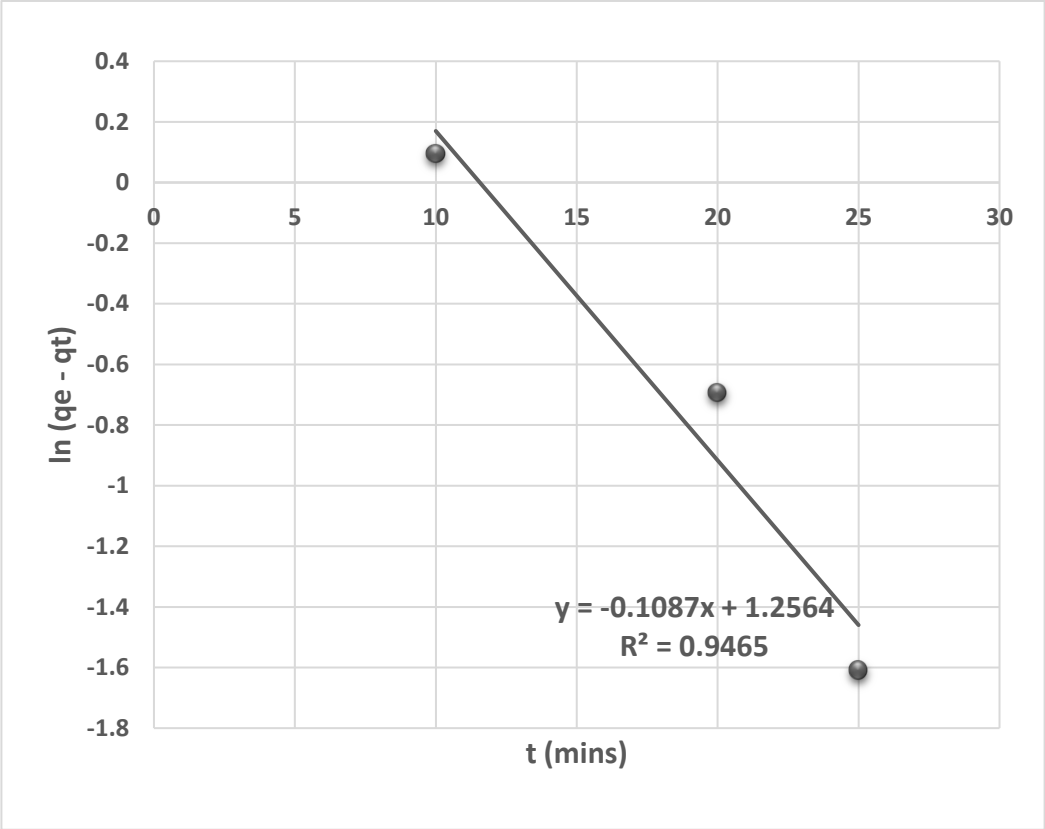

Figure 5: Graph of ln ($q_e - q_t$) against time

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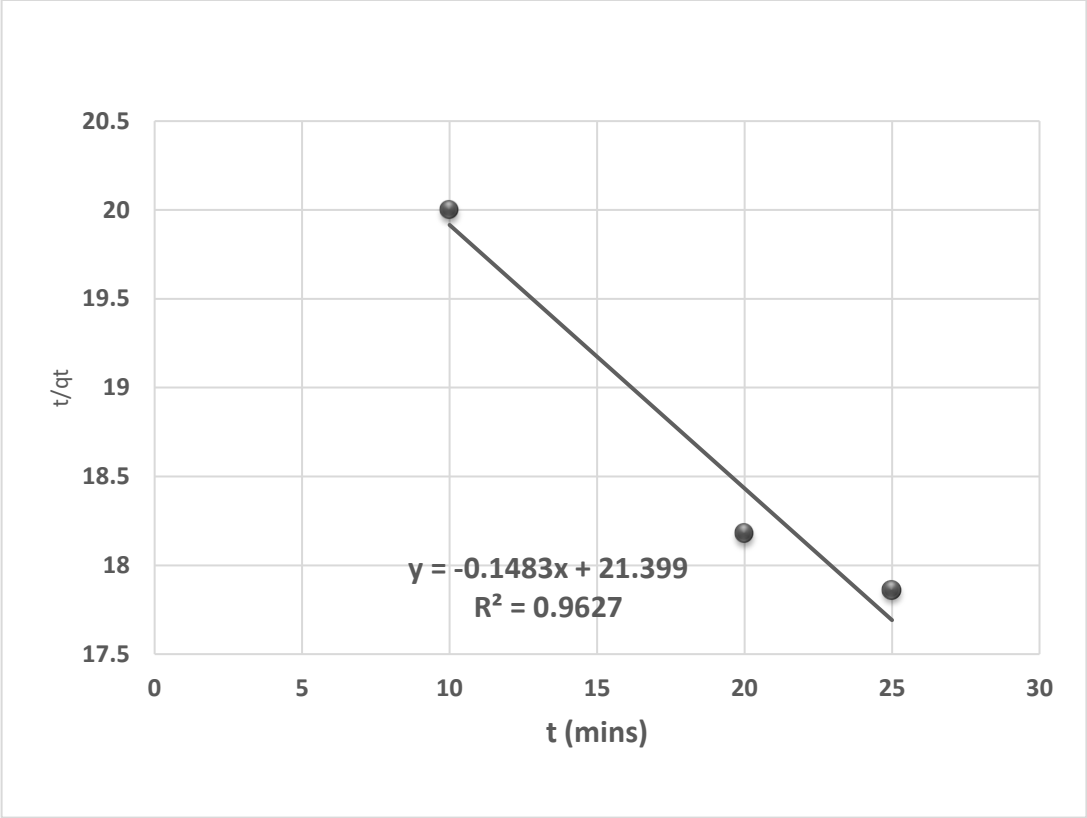

Figure 6: Graph of t/q_t against time

The correlation coefficient of second order kinetic model (0.9627) is greater than for first order kinetic model (0.9465) indicating that the pseudo second order model is better fit for this system. This result is in line with findings by Mustapha *et al.*, (2019).

4.0 CONCLUSION

Based on the findings of the research, it is evident that potato starch can be utilized for the dehydration of ethanol, along with various other industrial applications. It is advisable to develop a stage-wise treatment process for dehydrating the ethanol-water mixture. The modified potato starch demonstrates both technical and economic advantages, as it is environmentally friendly, capable of regeneration, and biodegradable

The four stage-wise contact treatments showed an improved quality (conc.) of the ethanol-water mixture, as the concentration was raised from 60% to 74.33%. The potato starch modified with sodium hypochlorite was found as a promising adsorbent for the ethanol-water solution dehydration. It is clear from this study that potato starch adsorbent is capable of adsorption of water from ethanol-water solution. Effect of temperature on the adsorption was investigated by thermodynamic analysis, and it was found to decrease with increase in temperature. The study demonstrated that the adsorption process is thermodynamically feasible and spontaneous, endothermic and occurred with a decreased order of disorderliness at adsorbent-adsorbate interface. The experimental data in the adsorption process indicated good correlations with the Langmuir isotherm and pseudo-second-order kinetic model.

The use of potato starch as an adsorbent for ethanol dehydration offers a greener alternative to traditional methods, aligning with the growing demand for environmentally friendly techniques. This study paves the way for the development of integrated bio refineries, where ethanol dehydration is a critical step in producing biofuel, chemicals, and other valuable products from biomass. In addition, this study investigates the underlying mechanisms of ethanol dehydration, the study sheds light on the role of potato starch in facilitating this reaction, revealing new insights into the thermodynamic and kinetic processes at play. The research also provides information for optimizing

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dehydration conditions, examining the effects of temperature ethanol and starch concentrations on dehydration kinetics.

This knowledge can be used to improve the efficiency and cost-effectiveness of ethanol production, making it a crucial contribution to the development of sustainable technologies. It is recommended that more isotherm and kinetic models be employed to investigate the data from the adsorption process.

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