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Kinetics and Thermodynamics for Optimal Starch Acetylation Analysis

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ABSTRACT

This study investigated the effects of the degree of substitution (DS) on the characteristics of acetylated starches and reviewed the kinetics and thermodynamics of starch acetylation. Kinetics of starch acetylation may be highly correlated for zero, first, or second order. Also covered was the speculative use of pseudo pseudo-first and second order models. Negative entropy and enthalpy mathematical mechanisms were examined, and their use is pertinent in exposing the catalyst-dependent spontaneity of acetylation reactions. Following pseudo-first and second order kinetics with a correlation coefficient and the Arrhenius law to determine the effect of temperature on the rate constant, the process was considered a surface reaction in which hydroxyl groups might be substituted with acetate groups.Deep insights into the spontaneity of the starch acetylation reaction were provided by the application of strategic determination of thermodynamic parameters, which was evaluated equally hypothetically. Knowing these procedures makes it easier to optimize starch acetylation industrially, producing modified starches with better qualities for a range of uses.

Keywords: Acetylation, kinetics, hydroxyl, acetyl, thermodynamics

I. INTRODUCTION

Acetyl groups (-$COCH_3$) are added to the hydroxyl groups (-OH) of starch molecules in a chemical process known as starch acetylation. By changing the starch's physicochemical and functional characteristics, this alteration improves its suitability for a range of industrial, culinary, and medicinal uses [1].

Acetylaton alteration can improve the way starch works and how it can be used, particularly in food. Acetylated starch has the potential to be employed as a thickening agent due to its high hydration ability. According to several studies, acetylation modification has strong stability and resistance to syneresis and retrogradation, which means it might be used as a stabilizer in products that need to be stored at low temperatures [2,3].

Acetylation modification can also raise OAC, making it suitable for use as a filmmaking agent. According to Colussi et al. [4], Ackar et al. [5], Singh et al. [6], and others, acetylated starch is used in a wide range of food products, including frozen food, infant food, sauces, tinned pie fillings, retorted soups, and salad dressings.

Few studies have fully investigated the kinetics and thermodynamics of starch acetylation, despite the fact that they are crucial for process optimization. The bulk of the literature concentrates on reaction efficiency and the characteristics of modified starch, even though earlier studies indicate that the reaction follows pseudo-first-order kinetics[7], influenced by variables like time, temperature, and catalysts [8,9].

The process cannot be fully understood or optimized for large-scale applications due to the absence of thorough analysis of the kinetic and thermodynamic characteristics under various conditions.

Conceptual Clarification of Acetylation

Acetylation is the process by which an acetyl group is introduced to a chemical as a result of its replacement with an active hydrogen atom. Here, a CH_3 or methyl group on the end, together with an acetyl group made up of carbonyl atoms or a carbon atom bound in double pairs with oxygen, is added to produce CH_3CO, which is frequently denoted by the letter "R". A hungry nucleophile (NP) with additional electrons and a good leaving group (R) cause this reaction to occur in order to produce acetate [4,10].

An ester is created when the hydrogen atom from the alcohol group is swapped out for one from the acetyl group in an acetylation process. Numerous instances of potent acetylation reactions exist that can improve our understanding of acetylation in general. For instance, the acetyl group's facile connection with the additional electrons in nucleophilic oxygen facilitates the easy substitution of the hydrogen atoms in the phenol group during the acetylation of glucose [5,7].

According to IUPAC rules and norms, these reactions are classified as ethanoylation. It usually entails replacing the hydroxyl group in an acidic base with an acetyl functional group. The same IUPAC terminology also applies to the opposite chemical reaction, known as deacetylation, which eliminates the acetyl group entirely from an equation. With the aid of the acetylation reaction, they play a crucial role in drug biotransformation, which deals with how the body breaks down and eliminates medications. Conducting acetyltransferases can also be beneficial in the therapy of autoimmune disorders and cancer[5].

Acetylated Starch and Degree of Distribution (DS)

Acetylated starch (AS) is a chemically modified starch that is made by replacing the three free hydroxyl groups at C2, C3, and C6 of the starch with acetyl groups [10]. According to FDA guidelines, AS can theoretically have a degree of substitution (DS) of up to 3, while DS between 0.01 and 0.2 can be utilized in food [11]. As demonstrated by Hu et al. [12] and Lin et al. [10], AS with a low degree of replacement can effectively improve the quality of foods based on starch. According to reports, adding AS improves the quality of wheat flour, its gelatinization properties, and the stability of dough while cooking since acetyl prevents hydrogen bonds between starch chains. The degree of improvement increases with the amount of AS applied [1].

According to Choy, May, & Small [13], Kang et al. [14], Lin et al. [10], and Mi et al. [15], adding 8% AS to the production process of bread, noodles, and raw dumpling wrappers inhibits the hydrogen bonding between linear starch molecules, has good hydrophilicity and water holding capacity at low degrees of substitution, and further improves the dough network structure, which could improve cooking quality, texture, digestion resistance, and storage stability. These properties may also be influenced by the wheat starch variety.

Determination of Degree of Distribution (DS)

The number of hydroxyl groups per glucose monomer that have been swapped out for acetyl moieties is known as the degree of substitution (DS) in starch acetylation. 0 denotes no acetylation, while 3 denotes complete replacement. The functional behavior of the modified starch is determined by its DS value, which ranges from industrial polymers at high DS to food-grade thickening agents at low DS[16].

Table 1. Typical Ranges & Applications of DS

DS Range	Approximate Value	Typical Applications
Low DS	< 0.2 (or sometimes < 0.1)	Food industry—improves thickening, paste clarity, reduces retrogradation
Medium DS	0.2–1.5	Broader functions—still food, some packaging, enhanced solubility
High DS	1.5–3.0	Non-food applications—thermoplastics, adhesives, biodegradable films, pharmaceuticals

Experimentally, the procedure for obtaining the parameters for determining the DS of a reaction is outlined in the figure 1.

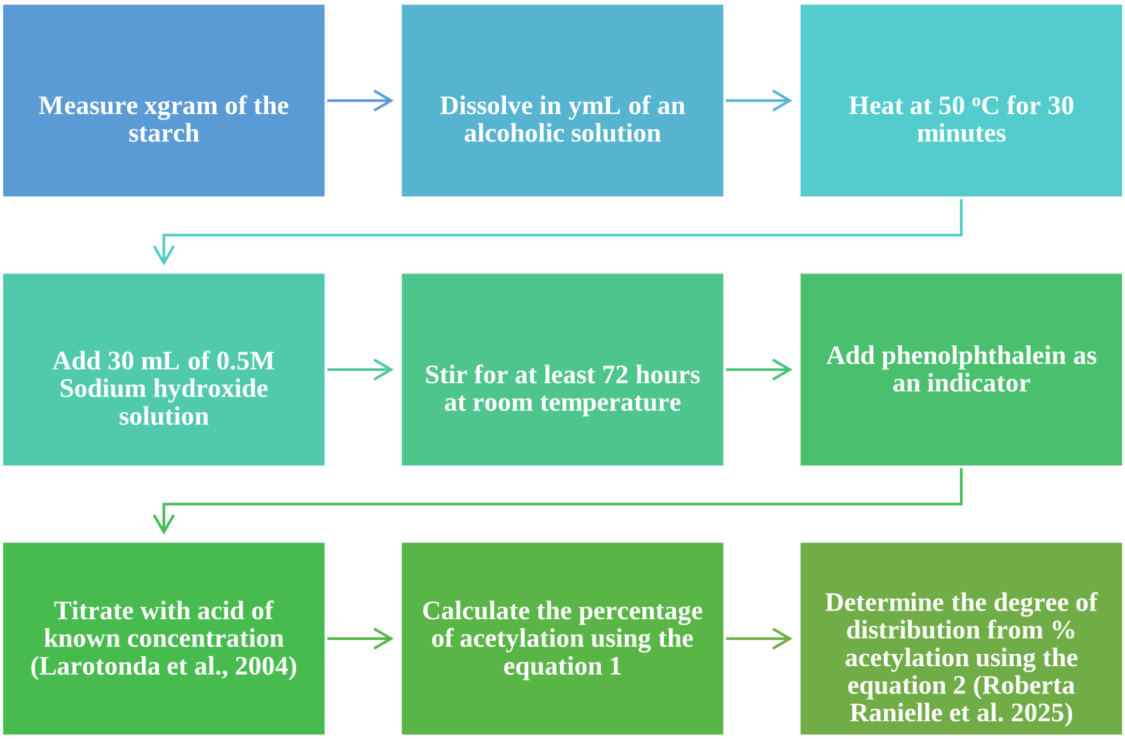


Figure 1. Experimental determination of percentage acetylation for DS

Percentage of acetylation (%A) = $\frac{(V_0-V_n) \text{ x N x } 0.043 \text{ x } 100}{M}$ 1

where

V_o = volume of the acid used to titrate reference sample

V_n = volume of acid used to titrate acetate sample

N = HCl used normality

43 = molar mass of acetyl group (g/mol)

M = mass of weighed sample

While the DS can be calculated using [16]**:**

$$DS = \frac{162 \text{ x } \%A}{4300-(42 \text{ x } \%A)} \quad 2$$

Starch Acetylation Kinetic Analysis

The reaction rate and rate constant are ascertained by performing acetylation kinetics. Equation 3 has been utilized [16] to calculate the proportion of hydroxyl group consumption as a percentage for each DS produced, and the rate law is assessed based on this consumption along the starch chain:

$$\%OH \text{ consumption} = 100 - \left[\left(\frac{DS}{3}\right) \text{ x } 100\right] 3$$

In kinetic studies of higher-order reactions, conditions were often used in which these reactions exhibited a kinetic behavior of first, second, or even zero order.

Zero order

Generally, for zero order reactions, rate is instantly equal to K, which is independent of reactant concentration. Consider the acetylation reaction in which OH is consumed below;

$n[\%OH^-] \rightarrow$ Acetylated Starch

The rate of OH^- consumption for zero order acetylation = $-\frac{d[OH]}{dt} = K[\%OH]^0$

$$- \quad d[\%OH^-] = Kdt \quad 4$$

Integrate equation 4:

$$- \quad \int_{[\%OH]_o}^{[\%OH]_t} d[OH] = \int_0^t Kdt \quad 5$$

$$-[\%OH]_t - [\%OH]_0] + C = K[t-0] + C$$

(where C= integration constant)

$$[\%OH]_0 - [\%OH]_t = Kt \quad 6$$

$$[\%OH]_t = -Kt + [\%OH]_0 \quad 7$$

$$K = \frac{([\%OH]_0 - [\%OH]_t)}{t}$$

First order Case II starch-acetylation kinetics

Consider the acetylation reaction as presented in Atkins [17]:

$n[\%OH^-] \rightarrow$ Acetylated Starch

The rate of OH^- consumption for first order acetylation = $-\frac{d[OH]}{dt} = K[\%OH]^1$

$$-\frac{d[\%OH]}{dt} = K_1[\%OH]$$ 8

$$-\frac{d[\%OH]}{[\%OH]} = K_1 dt$$ 9

Integrate equation 9

$$-\int_{[\%OH]_o}^{[\%OH]_t} \frac{1}{[\%OH]} d[\%OH] = \int_0^t K_1 dt$$ 10

$$-[\ln\%OH]_{[\%OH]_o}^{[\%OH]_t} + C = K_1[t]_0^t + C$$

$$-[\ln[\%OH]_t - \ln[\%OH]_0] = K_1[t-0] + C - C$$ 11

$$\ln[\%OH]_0 - \ln[\%OH]_t = K_1 t$$ 12

$$K_1 = \frac{\ln[\%OH]_0 - \ln[\%OH]_t}{t}$$ 13

$$K_1 = \frac{1}{t}.\ln\left(\frac{[\%OH]_0}{[\%OH]_t}\right)$$ 14

First order Case II starch-acetylation kinetics

Using the reaction: $n[\%OH^-] \rightarrow$ Acetylated Starch

Let the initial concentration of [OH]be $[\%OH]_0$

So that at time t = 0 we have $[\%OH]_t = 0$

At time t = t, we have [OH] $=([\%OH]_0 - [\%OH]_t)$

The rate expression for the disappearance of [%OH] will be:

$$\frac{d[\%OH]_t}{dt} = K_1([\%OH]_0 - [\%OH]_t)$$ 15

$$\frac{d[\%OH]_t}{([\%OH]_0 - [\%OH]_t)} = K_1 dt$$ 16

$$\int \frac{d[\%OH]_t}{([\%OH]_0 - [\%OH]_t)} = K_1 \int dt$$ 17

$$\int([\%OH]_0 - [\%OH]_t)^{-1} d[\%OH]_t = K_1 \int dt$$ 18

$$-\ln([\%OH]_0 - [\%OH]_t) = K_1 t + C$$ 19

To find the value of C in the equation above, we have that at time:

t=0, $[\%OH]_t$=0, C = $-\ln[\%OH]_0$. Hence, equation 12 becomes:

$$-\ln([\%OH]_0 - [\%OH]_t) = K_1 t - \ln[\%OH]_0$$ 20

$$\ln[\%OH]_0 - \ln([\%OH]_0 - [\%OH]_t) = K_1 t$$ 21

$$K = \frac{1}{t}.\ln\left(\frac{[\%OH]_0}{([\%OH]_0 - [\%OH]_t)}\right)$$ 22

Second order starch-acetylation kinetics

The second order for starch-acetylation kinetics can be derived as thus:

$n[\%OH^-] \rightarrow$ Acetylated Starch

The rate of OH^- consumption for second order acetylation = $-\frac{d[OH]}{dt} = K[\%OH]^2$

$$-\frac{d[\%OH]}{[\%OH]^2} = Kdt$$ 23

Integrate equation 23

$$-\int_{[OH]_o}^{[OH]_t} \frac{1}{[\%OH]^2} d[OH] = \int_0^t K_2 dt$$ 24

$$-\int_{[OH]_o}^{[OH]_t} [\%OH]^{-2} d[OH] = \int_0^t K_2 dt$$

$$-\left(-[[\%OH]^{-1}]_{[OH]_o}^{[OH]_t}\right) + C = K_2[t]_0^t + C$$

$$-[[\%OH]_o^{-1} - [\%OH]^{-1}] = K_2[t-0] + C - C$$

$$\frac{1}{[\%OH]_t} - \frac{1}{[\%OH]_o} = K_2 t$$ 25

$$K = \frac{1}{t}\left(\frac{1}{[\%OH]_t} - \frac{1}{[\%OH]_o}\right) \text{ in } mol^{-1}L^{-1}s^{-1}$$ 26

Pseudo OrdersAcetylation Kinetics

i. Pseudo-First order

It is often referred to as the Lagergrenmodel. It explains the first-order technique for adding an acetyl group or removing [OH]. Under low pressure, it is also diverse. The plot of $\ln([\%OH]_e - [\%OH]_t)$ versus t gives a straight-line graph. The slopes and intercepts of the linear plots can be used to calculate the theoretical equilibrium acetylation capacities$[\%OH]_e$ (theoretical) and rate constants K_1 at various concentrations and temperatures [18]. The equation of pseudo-first order is provided by Enajeme [19].

$$\ln([\%OH]_e - [\%OH]_t) = \ln[\%OH]_e - K_1 t$$ 27

where $[\%OH]_t$= Concentration of [OH] removed at time t.

$$[\%OH]_e = \frac{(C_0 - C_t)V}{m}$$ 28

where C_0, C_t, V and m are initial,[OH] at time t, volume of the solution and mass of acetyl, respectively.

$[\%OH]_e$= Equilibrium acetylation capacity (mg/g)

The plots of $\ln([\%OH]_e - [\%OH]_t)$ versus t give a straight line graph with slope K_1 and intercept lnq_e. Consider figure 2, a hypothetical pseudo-first order acetylation isotherm:

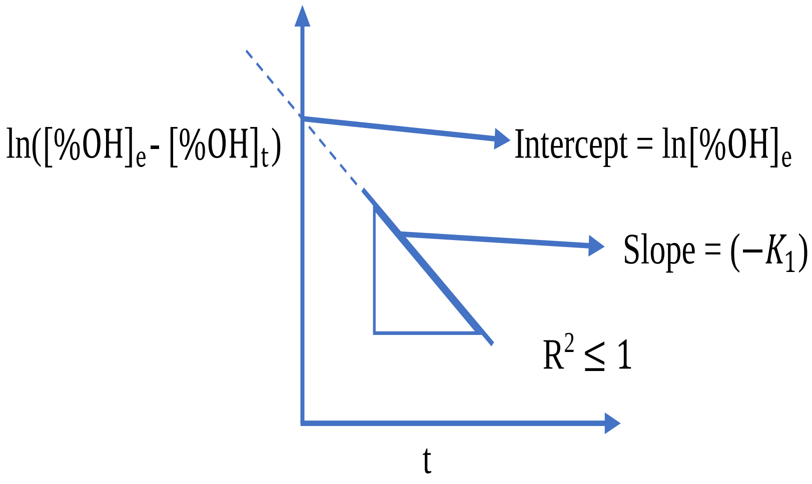


Figure 2. Hypothetical graph for Pseudo-first-order acetylation

Pseudo-Second order

This model assumes that the rate of removal of hydrogen in theOH group is proportional to the rate of formation of ester. The reaction rate is dependent on the amount of acetyl added and the driving force ($[\%OH]_e$ - $[\%OH]_t$) is proportional to the number of active acetyl molecules available [20, 21]. The equation can be represented as:

$$\frac{t}{[\%OH]_t} = \frac{1}{K_2[\%OH]_e^2} + \left[\frac{1}{[\%OH]_e}\right]t$$

29

The chemisorption step is assumed to be the rate-determining step in the pseudo-second order. The plot of $\frac{t}{[\%OH]_t}$ versus t gives a straight-line graph. Values of K_2 and $[\%OH]_e$ can be calculated from the slope, $\left[\frac{1}{[\%OH]_e}\right]$ and intercept, $\frac{1}{K_2[\%OH]_e^2}$. A hypothetical graph for pseudo-second order acetylation isotherm can be represented in figure 3.

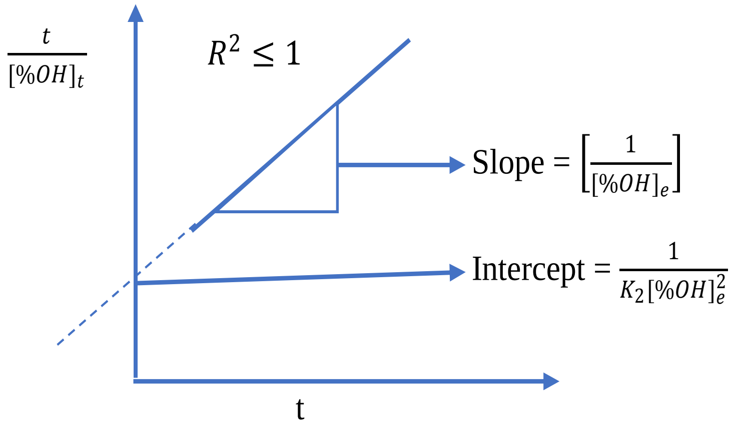


Figure 3. Hypothetical graph for Pseudo-second-order acetylation

When the acetyl concentration is low, the equation explains the adsorption mechanism more than any other kinetic model. However, at high initial concentration, pseudo first order model is favoured [22].

Hypothetical Application of Arrhenius Law on Effect of Temperature

Arhenius law on effect of temperature on equilibrium constant states that:

$$\frac{dlnK_c}{dT} = \frac{\Delta E}{RT^2} 30$$

Where K_c = expressed in terms of equilibrium constant, ΔE = energy change accompanying the acetylation reaction, R = gas constant and T = temperature. For experimental and graphical applicability, equation 30 is reduced to equation 31 through to 16 presented below:

Considering a second order acetylation reaction as thus:

$$\text{R-OH} + \text{CH}_3\text{COCl} \underset{K_{-1}}{\overset{K_1}{\longleftrightarrow}} \text{R-O-COCH}_3 + \text{HCl}$$

Forward rate = K_1[R-OH][CH_3COCl] so that K_1= rate constant for forward reaction

Backward rate = K_{-1} [R-O-$COCH_3$][HCl] so that K_{-1}= rate constant for backward reaction

At equilibrium we will have;

$$K_1[\text{R-OH}][\text{CH}_3\text{COCl}] = K_{-1}[\text{R-O-COCH}_3][\text{HCl}]31$$

$$\frac{[R-O-COCH3][HCl]}{[R-OH][CH3COCl]} = \frac{K_1}{K_{-1}} = K_c 32$$

Substitute the value of K_c into equation 32;

$$\frac{dlnK_c}{dT} = \frac{\Delta E}{RT^2} \qquad 33$$

$$\frac{dln\left({}^{K_1}/_{K_{-1}}\right)}{dT} = \frac{\Delta E}{RT^2}34$$

$$\frac{dlnK_1}{dT} - \frac{dlnK_{-1}}{dT} = \frac{\Delta E}{RT^2}35$$

Equation 35 may be split as two equations such that;

$$\frac{dlnK_1}{dT} = \frac{E_1}{RT^2} + C36$$

$$\frac{dlnK_{-1}}{dT} = \frac{E_{-1}}{RT^2} + C \qquad 37$$

Where $E_1 - E_{-1} = \Delta E$ and C = integration constants. Experiments show that C can be set equal to zero (Enajeme, 2024), so that equation 36 or 37 may be written as;

$$\frac{dln\,K_{ads}}{dT} = \frac{E_a}{RT^2} \equiv lnK_{ace} = \frac{E_a}{RT^2}\,\text{dt}38$$

$$\int dlnK_{ace} = \frac{E_a}{R}\int T^{-2}\text{dt} \equiv lnK_{ace} = \frac{\mathrm{E_a}}{\mathrm{R}}\left[\frac{\mathrm{T}^{-2+1}}{-2+1}\right] + \mathrm{A}$$
39

$$\mathrm{lnK_{ace}} = \frac{\mathrm{E_a}}{\mathrm{R}}\left[\frac{\mathrm{T}^{-1}}{-1}\right] + \mathrm{A} \equiv \mathrm{lnK_{ace}} = -\frac{\mathrm{E_a}}{\mathrm{R}}\left[\frac{1}{\mathrm{T}}\right] + \mathrm{A}\ 40$$

$$\mathrm{lnK_{ace}} = -\frac{\mathrm{E_a}}{\mathrm{RT}} + \mathrm{A} \equiv \mathrm{K_{ace}} = \mathrm{A}e^{-\frac{\mathrm{E_a}}{\mathrm{RT}}}$$
41

where$\mathrm{lnK_{ace}}$ is the rate constant for the acetylationreaction at T; A = frequency factor for the reaction (Adewumi et al., 2025). A hypothetical graph of the effect of temperature on acetylation reaction rate is presented in figure 4.

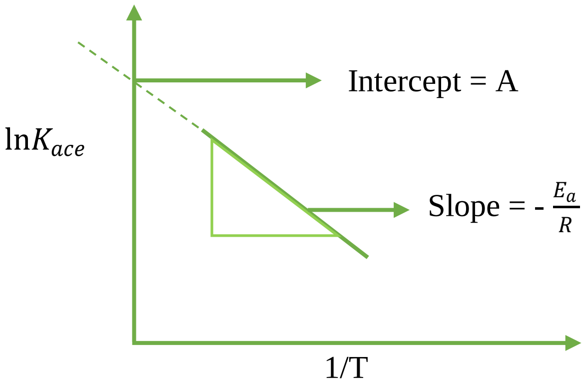


Figure 4. Effect of temperature on acetylation rate constant

Thermodynamic Analysis for Starch Acetylation

The spontaneity, viability, and energetic requirements of the reaction are all shown by the thermodynamic analysis of starch acetylation. To evaluate the path toward equilibrium and comprehend how temperature and reaction conditions affect acetylation efficiency, important factors including enthalpy (ΔH), entropy (ΔS), and heat capacity (Cp) have been studied. These thermodynamic analyses determined the energy favorability of acetyl group attachment to starch, which facilitated the optimization of reaction conditions [23].The spontaneity of the reaction is intimately associated with the entropy value (ΔS). A low level of disorder in the starch acetylation process is indicated by a negative ΔS. However, the overall Gibbs free energy (ΔG), which takes into account both entropy (ΔS) and enthalpy (ΔH), is what ultimately determines spontaneity. It has been claimed that even when there is a negative ΔS, the reaction is nevertheless driven ahead by its exothermic nature (negative ΔH). Despite the unfavorable entropy change, the catalyst was essential in breaking through the activation energy barrier and enabling the reaction.Finding the ideal temperature and pressure for a chemical reaction's products is one of the most significant and productive uses of thermodynamics, which is applied in the analysis of chemical equilibria [24]. Without a comprehensive thermodynamic understanding of the chemical reactions—including adsorption—no industrial process would ever be started.

Obtaining Thermodynamic Parameters

To calculate the thermodynamic parameters, the following equations may be employed:

$$\ln\theta_T = -\frac{\Delta H}{RT} + \frac{\Delta H}{RT_0} + \ln\theta_0 \qquad 42$$

$$\Delta H = \int_{T_1}^{T_2} C_P\, dT = C_P(T_2 - T_1) \qquad 43$$

$$\Delta S = C_P \ln\left(\frac{T_2}{T_1}\right) \qquad 44$$

Where ΔHis the heat of starch acetylation, T_0 is critical temperature of acetylation, T is temperature, R is the universal gas constant, C_P is heat capacity, θ_0 is critical degree of starch acetylation, andΔS is the entropy variation value[16].

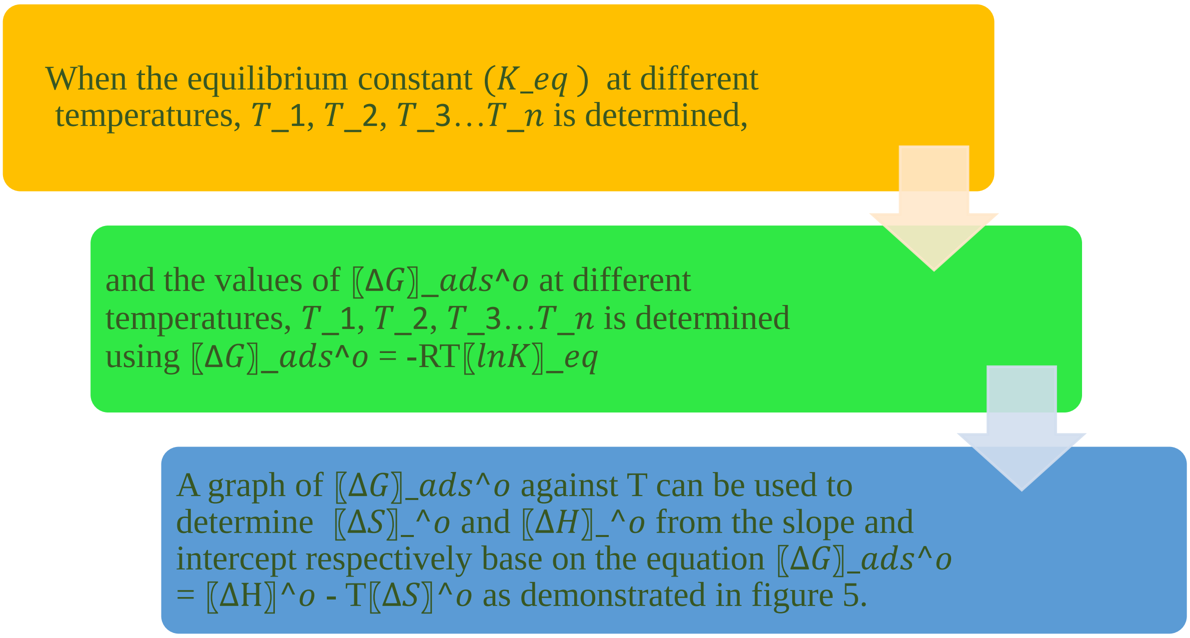
Graphical Dimensions for determining Thermodynamic parameters

The thermodynamic parameters can be obtained using two graphical dimensions based on two equations. The equations 45 and 46 are used in the first graphical dimension.

$$\Delta G^{o}_{ads} = -RT\ln K_{eq} \qquad 45$$

$$\Delta G^{o}_{ads} = \Delta H^{o} - T\Delta S^{o} \qquad 46$$

Where R is gas constant (8.314 $Jmol^{-1}K^{-1}$) and T is absolute temperature in Kelvin, R is the gas constant and K_{eq} can be obtained from $\frac{q_e}{C_e}$[18]. The modality for obtaining the first graphical dimension thermodynamic parameters is represented in figures 4 and 5.



When the equilibrium constant (K_eq) at different temperatures, T_1, T_2, T_3…T_n is determined,

and the values of 〖ΔG〗_ads^o at different temperatures, T_1, T_2, T_3…T_n is determined using 〖ΔG〗_ads^o = -RT〖lnK〗_eq

A graph of 〖ΔG〗_ads^o against T can be used to determine 〖ΔS〗_^o and 〖ΔH〗_^o from the slope and intercept respectively base on the equation 〖ΔG〗_ads^o = 〖ΔH〗^o - T〖ΔS〗^o as demonstrated in figure 5.

Figure 4: First dimension-1 for determining thermodynamic parameters [18]

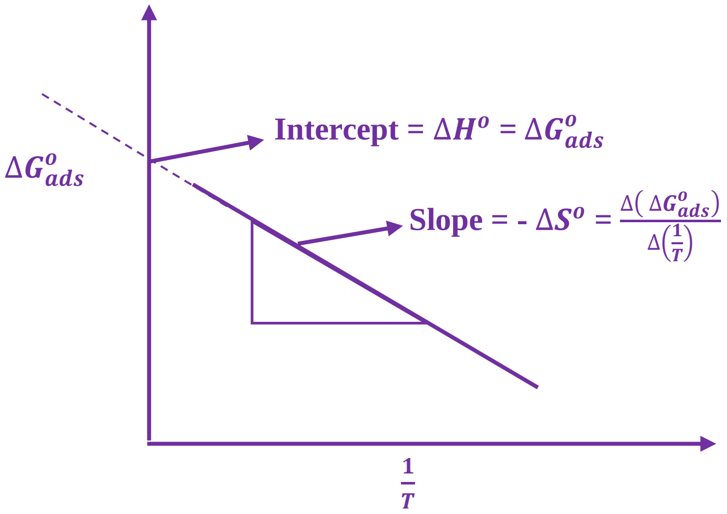


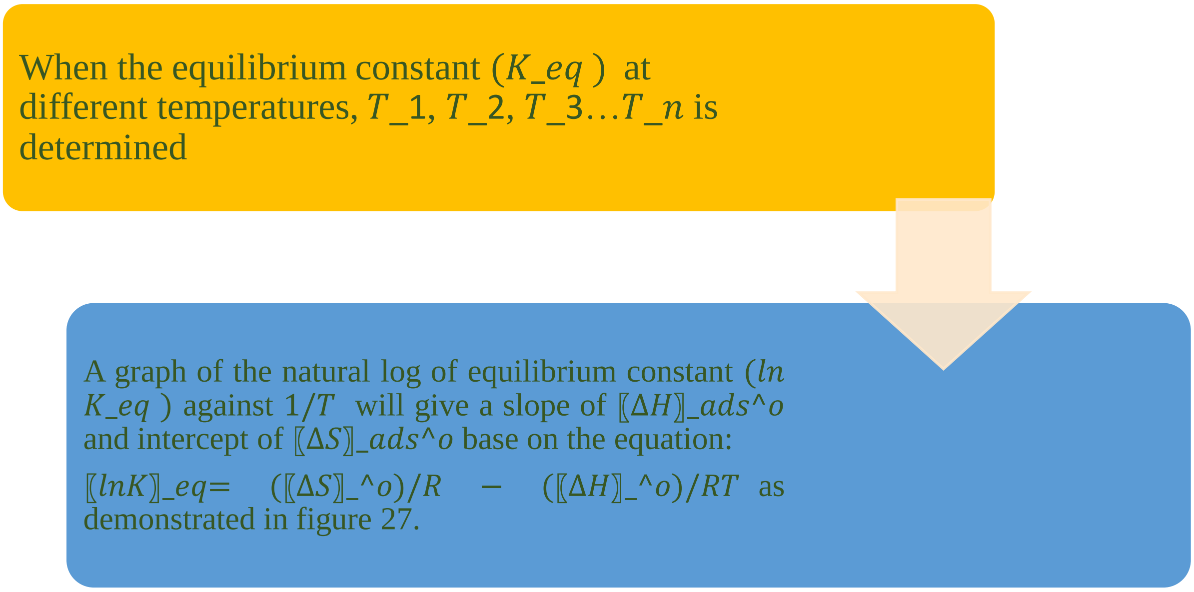
Figure 5: Graphical dimension-1 for determining thermodynamic parameters[18]

The second graphical dimension also utilizes two equations (47 and 48):

$$\Delta G^{o}_{ads} = -RT\ln K_{eq} \qquad (47)$$

$$\ln K_{eq} = \frac{\Delta S^{o}}{R} - \frac{\Delta H^{o}}{RT} \qquad (48)$$

Where R is gas constant (8.314 $Jmol^{-1}K^{-1}$) and T is absolute temperature in Kelvin, R is the gas constant and K_{eq} can be obtained from $\frac{q_e}{C_e}$[18]. The modality for obtaining the second graphical dimension thermodynamic parameters is represented in figures 6 and 7.



When the equilibrium constant $(K\_eq\ )$ at different temperatures, T_1, T_2, $T\_3 \ldots T\_n$ is determined

A graph of the natural log of equilibrium constant $(ln\ K\_eq\ )$ against $1/T$ will give a slope of $\llbracket \Delta H \rrbracket\_ads\hat{}o$ and intercept of $\llbracket \Delta S \rrbracket\_ads\hat{}o$ base on the equation:

$\llbracket lnK \rrbracket\_eq= \quad (\llbracket \Delta S \rrbracket\_\hat{}o)/R \quad - \quad (\llbracket \Delta H \rrbracket\_\hat{}o)/RT$ as demonstrated in figure 27.

Figure 6: Dimension-2 for determining thermodynamic parameters[18]

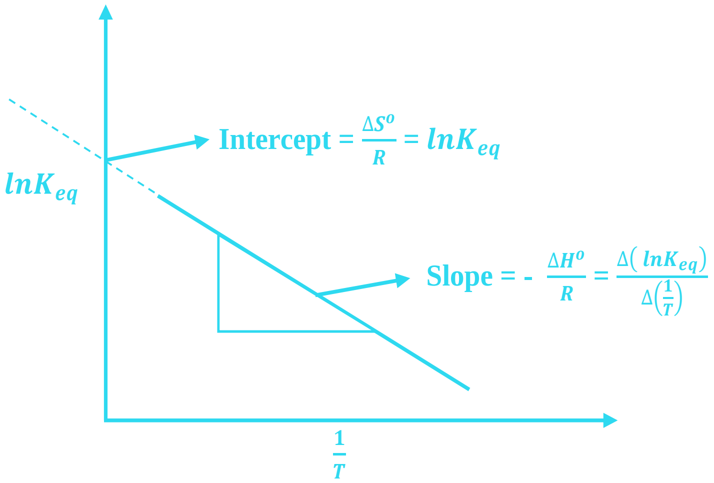


Figure 7. Graphical dimension-II for determining thermodynamic parameters[18]

II. CONCLUSION

This study examined the thermodynamics and kinetics of starch acetylation. The procedure was considered a surface reaction, with pseudo-first and second order kinetics, the ability to substitute acetate groups for hydroxyl groups, and the use of the Arrhenius law to determine the correlation coefficient and the impact of temperature on the rate constant. The strategic application of thermodynamic parameter determination was evaluated hypothetically and offered profound insights into the starch acetylation reaction's spontaneity. Understanding these processes makes it simpler to optimize starch acetylation on an industrial scale, resulting in improved modified starches for a variety of applications.

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