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A Kinetic and Thermodynamic Study of CI Fluorescent Brightener 113 on Cotton

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ABSTRACT

Fluorescent brightening agents are dyestuffs in which adsorption is the primary mechanism involved in the dyeing process. They are commonly used to improve the brightness in various industrial products, especially textiles. FBA 113 is widely used in the dyeing process for cotton. This study aims to evaluate the optimum dyeing process of FBA 113 on cotton to assess the dyeing behavior, the adsorption isotherm model, and thermodynamic parameters. The study examined various parameters, including dyeing times, dyeing temperature, the amount of dye, and auxiliary. Variations in conditions were observed to define the dyeing equilibrium time, the Langmuir and Freundlich adsorption isotherm, the pseudo-first-order, pseudo-second-order dyeing kinetic model, and the thermodynamic parameters, such as enthalpy (ΔH^0), entropy (ΔS^0), and Gibb's energy (ΔG^0). The results show that the appropriate dyeing was FBA 113 0.25 % owf with 20 g/L KCl as an auxiliary, the best dyeing temperature was 323 K, and the equilibrium time was 120 min. This dyeing reflected an adsorption behavior that displayed multilayer adsorption that corresponded to the Freundlich isotherm. Also, the adsorption model had a pseudo-second-order of 0.9874 using linear regression. The dyeing reaction was exothermic, with ΔH^0 -26.58 kJ/mol, ΔS^0 34.19 J/mol, and the spontaneous process had a negative ΔG^0 value. The fiber and dye interacted physically. The study can enhance our understanding of FBA 113 dyeing behavior and identify situations in which it can be used in other dyeing process models, which will help optimize further dyeing studies. Prog. Color Colorants Coat. 15 (2022), 225-233© Institute for Color Science and Technology.

1. Introduction

Fluorescent brightening agents (FBAs) or optical brightening agents (OBAs) are organic compounds. They are primarily colorless dyestuffs, but they can be slightly self-colored substances that act as substitutes for the chromophore system of a dye, as they have their fluorescing system. The substitute dye has an affinity for a specific type of fiber used in this study [1-3]. FBAs are applied to substrates and absorb the light in the near-ultraviolet (UV) region of the electromagnetic spectrum (360-380 nm), invisible to the human eye. They re-emit the light at lower energy radiation in the visible light spectrum at about 400-500 nm [2, 4, 5]. The energy difference is lost in the form of kinetic energy to enable the molecules to perform their function, and certain construction principles are an

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essential prerequisite. These can be determined empirically [3]. This means that fluorescence occurs when the ultraviolet component of sunlight is absorbed and subsequently reflected as additional blue light. When FBAs are applied to substrates, they cause the material to reflect more blue light, increasing whiteness and making us see a much brighter material [6]. The interaction of FBA mechanisms and their absorption on the substrates has been examined using many techniques. The methods include liquid chromatography, zeta-potential measurements, calorimetry, capillary electrophoresis, fluorescence spectroscopy, UV–Vis spectroscopy, light scattering, and thin-layer chromatography [1, 4, 7, 8].

FBAs are widely used as additives in industrial processes to enhance color visualization via detergent, textile, paper, and other industries such as plastics, paint, food, and cosmetics [4, 9, 10]. There are also reports on the international optical brighteners market, and as key market indicators show. FBAs worth approximately $ 249.93 million and were used in the paper industry in 2016. Their value will be $ 439.35 million in 2021 in consumer products, with the European market enjoying a compound annual growth rate (CAGR) of 8.06 %. In this study, CAGR is defined as the implied yearly growth rate of an investment over a targeted duration of time that must be longer than one year [1].

In the textile industry, FBAs are used to preserve and enhance the brightness or whiteness of dyed and printing fabrics, to give cleaner and more appealing textiles, especially for hospital or hotel linen, towels, and cloths [1, 11]. Nowadays, FBAs are no longer restricted to white materials, and they are also used to improve the appearance of subdued pastel shades such as faded blue, red, violet, and green colors [3]. FBAs are classified into two main groups according to their method of use. Firstly, the brightener is a predominantly water-soluble substance used for natural fibers and polyamides. Secondly, dispersed brighteners are typically water-insoluble substances that are used to improve the coloration of polyester and acetate [2, 12]. Typically, 80 % of FBAs produced are stilbene derivatives, and various substituents rely on the fiber types and affinities, such as CI optical brightener 113 (FBA 113), which is suitable for cotton dyeing. Accordingly, at least 12 out of the 18 stilbene derivatives are triazine-stibene derivatives, and 8 benzoxazoline-based and 11 diazo-based substances have been registered by REACH [1].

Dyeing textiles is a complex process and involves many aspects from the field of physical chemistry. Researchers need to determine the kinetics and thermodynamics of adsorption on substrates using various factors, including pH, concentration, temperature, and time [13, 14]. These factors are related to the state of the dyes, the interaction in the dye-fiber, thermodynamics, and the adsorption kinetics of dyes [4, 15-18], as well as dyeing mechanisms, mass transfer and diffusion of dyes in fiber bath solutions [4-5, 17, 19-23]. The results of our study have made it possible to improve our understanding of the adsorption kinetics of the phenomena that occur during the dye-fiber interaction [4, 15-17, 19-31].

This work aimed to study the dyeing behavior of FBA 113 on cotton fabrics using dyeing conditions, the dyeing equilibrium time, and the Langmuir and Freundlich adsorption isotherm. The pseudo-first-order, pseudo-second-order dyeing kinetic models and thermodynamic parameters such as enthalpy (ΔH^0), entropy (ΔS^0), and Gibb's energy (ΔG^0) was also determined.

2. Experimental

2.1. Cotton fabric preparation

The dyeing material was a single thread 100 % plain weave grey cotton fabric with a fabric weight of 157.22 g/m^2, which was acquired from a retail store in Bangkok, Thailand. The material was put through a one-bath process of scouring and bleaching to remove the impurities and make the fabric appear whiter at 80 °C for 60 minutes in a solution of 8 g/L of hydrogen peroxide, two g/L of sodium hydroxide, and four g/l of Tanawet RCN. The fabric was then pressed in a Laboratory PadMangle (Newave Lab Gear, Taiwan) and dried at room temperature. The specimens had a CIE-Whiteness of 84.74 and were considered ready for the following dyeing process.

2.2. FBA 113 purification

CI fluorescent brightener 113 (FBA 113) was obtained from the Phisit Group, a trader of textile dyes and chemicals in Thailand. The brightener was purified before using a precipitation process using a 20 % w/v potassium thiocyanate solution (Merck, Germany) to obtain a purified version of FBA 113. A 0.001 % w/v FBA 113 solution was then prepared to measure adsorption by ultraviolet rays in the wavelength range of 200 to 450 nm using a 2-beam spectrophotometer UV-Vis (UV-160A, Shimadzu Corporation). The concentration of a solution from the result of the optical measurement of adsorption in terms of the molar extinction coefficient (ε) was calculated using Lambert's law (Equation 1).

$$\varepsilon(M^{-1}cm^{-1}) = \frac{A}{LC} \quad (1)$$

where A is the amount of light absorbed by the sample for a particular wavelength, L is the distance that the light travels through the solution in cm, and C is the absorption per unit volume concentration. The molecular weight of FBA 113 was 992.20 g/mol.

2.3. Dyeing procedure

The specimens were dyed in an automated laboratory IR dyeing machine (Starlet DL-6000+), Korea) with a liquor ratio of 25:1. The study was performed to evaluate the dyeing behavior at the various stages.

- **The exhaust dyeing process:** Exhaustion was defined as the ratio of the concentration of dye taken up by the cotton fabric to the total initial concentration of dye in the bath. The cotton fabric was dyed using 0.25 % owf, and purified FBA 113 powder and KCl 20 g/L by varying the dyeing temperature between 323 and 363 K and the dyeing time from 5 to 240 min. The exhaustion percentage acquired is expressed in Equation 2.

$$\%\, exhaustion = \frac{(C_0 - C_t)}{C_o} \times 100 \quad (2)$$

where C_o (mg/L) is the initial FBA 113 concentration, C_t (mg/L) is the concentration of FBA 113 at time t, W (mg) is the weight of the cotton specimen, and V (L) is the volume of the FBA 113 solution. Meanwhile, C_e is the concentration of the FBA 113 solution at equilibrium (mg/L), and the FBA 113 equilibrium adsorption (q_e) is expressed in Equation 3.

$$q_e = \frac{(C_0 - C_e) * V}{W} \quad (3)$$

- **Adsorption isotherm model:** The fabric was dyed using 0.25 % owf purified FBA 113 powder at the equilibrium dyeing condition obtained from the previous study, but KCl concentration was varied as 0, 5, 10, 20, and 40 g/L.

2.4. Measurement of cotton fabric whiteness

The CIE whiteness index value (CIE whiteness) shows the most common form of white shadow caused by the surface reflection of the object. The CIE whiteness value was evaluated using the Spectraflash SF 600PLUS CT (Datacolor international), using the Data Match Color software. The light source was set as D65, with a viewing angle of 10^0.

3. Results and Discussion

3.1. Molar extinction coefficient

FBA 113 is a greenish-yellow powder with a non-ionic charge. It can be used for dyeing on cotton due to its structural similarly structure to direct dyes. The structure can be classified as an insoluble compound, like aniline (R1) and the N,N'-Diethanol group (R2), which are highly water-soluble but not ionized. Both groups are connected to the Triazine group, and FBA 113 is slightly water-soluble, adheres well to fibers, and is suitable for use in the exhaustion dyeing process [32]. The structure is shown in Figure 1.

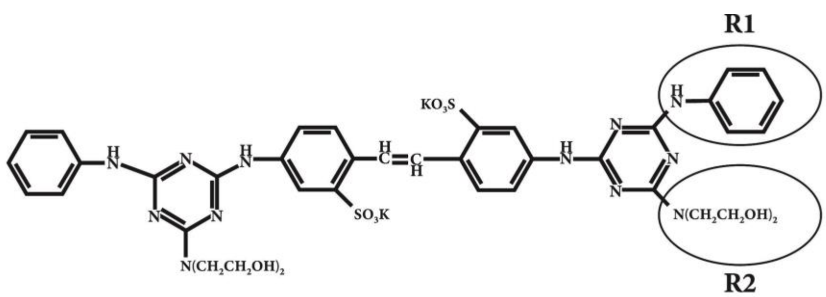


Figure 1: Chemical structure of FBA 113.

Figure 2 the comparative UV adsorption pattern of the original FBA 113, purified FBA 113 purified, and the FBA 113 purified to which 20 g/L KCl has been added. All patterns showed the same maximum wavelength position at 353 nm, although the ABS intensities were different. The first part of the experiment between the original FBA 113 and purified FBA 113 found that the purified FBA113 had a greater intensity, implying that the molar extinction coefficient (ε) was also high. We found that potassium thiocyanate reacts with iso-thiocyanate (-N=C=S) in the hydrogen atom at the lower ring of the compound structure. Thus the particle molecules were activated through the absorption of radiation, electromagnetic or photoluminescence, resulting in a release of fluorescent energy [1]. The molar extinction coefficient increased from 42,665 to 62,509 $M^{-1}cm^{-1}$, which is close to the industrial fluorescent isothiocyanate level, at 73,000 $M^{-1}cm^{-1}$ [33]. The cotton fabrics were then dyed at 363 K for 120 minutes, with the result that the CIE-whiteness value increased from 132.15 to 154.77, confirming that purified FBA 113 is appropriate for this study.

After the dyeing process had been completed, the purified FBA 113 with 20 g/L potassium chloride added showed a similar curvature of FBA 113 shapes in graphic form (Figure 2). This case indicates that the electrolyte was not significant in its molar extinction coefficient. Still, it had developed a CIE-whiteness value, which means that FBA 113 with high concentrations of sulfonate groups shows good solubility in water and demonstrated electrostatic repulsion between the negatively charged ions of the cellulose surface and the anionic charge of FBA 113. Meanwhile, adding the electrolyte (KCl) introduced cations to reduce the contribution of electrostatic repulsion to the interaction between ionic FBA 113 and negatively charged cellulose surfaces. The Van der Waals interaction was reduced [4], which meant that more FBA 113 molecules were absorbed on the fabric surface. After the process, the CIE-Whiteness value was more significant than 163.43.

3.2. Exhaust dyeing process

The exhaust technique is the most common method of applying dyes, as it is resistant to small changes in system variables. The procedure involves gradually transferring dye from a dye bath to the textile material using the same equipment. This study set the dyeing condition at 323-363 K for 5-240 min, and the exhaustion percentage is shown in Table 1.

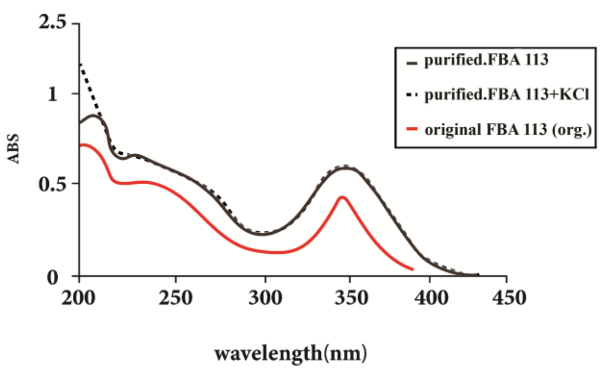


Figure 2: UV absorption patterns of FBA 113.

Table 1: Exhaustion percentage of the FBA 113 on cotton.

T (K)	% Exhaustion							
	5 min	15 min	30 min	45 min	60 min	90 min	120 min	240 min
323	31.70	36.01	45.44	55.92	69.48	87.92	92.31	92.45
333	33.15	42.17	52.36	61.44	72.21	85.22	88.72	89.43
343	37.59	45.85	55.6	63.59	73.57	81.62	82.83	84.96
353	42.36	51.85	61.94	70.16	78.27	78.28	78.32	78.34
363	47.72	63.06	72.73	74.62	74.81	75.7	75.73	75.98

3.3. Adsorption isotherm model

The equilibrium adsorption isotherm describes the interaction between the solute and the adsorbent. It is a fundamental theory that explains the adsorption mechanism of FBA 113 on cotton fabric. The two most frequently used adsorption isotherm models are the Langmuir and the Freundlich models. The Langmuir isotherm model was developed to explain the chemistry occurring at different adsorption sites with the same adsorption energy, away from the surface coverage, and with no contact between the adsorbed molecules. The model assumes a monolayer deposit of the adsorbent on the surface with a limited number of similar sites. This relationship is shown as a linear expression in Equation 4.

$$\frac{1}{q_e}=\frac{1}{Q}+\frac{1}{QbC_e} \qquad (4)$$

where Q is the maximum FBA 113 per unit weight of cotton needed to form a complete monolayer coverage on the surface at the equilibrium FBA 113 concentration of C_e. q_e is the amount of FBA 113 per unit weight of cotton at equilibrium, and b is the Langmuir constant related to the affinity of the binding site. The linear plot of $1/q_e$ against $1/C_e$ is shown in Figure 3.

This model shows two stages of FBA113 dyeing: potassium chloride (0 g/L KCl) and potassium chloride (5-40 g/L KCl) conditions. The dyeing condition without KCl showed a straight line corresponding to the Langmuir adsorption isotherm, indicating that FBA 113 was absorbed onto cotton fabric as a single layer. Meanwhile, the other process of dyeing shows the exponential profiles, indicating that the adsorption has no saturation point and occurs as a multilayer process. At this point, the Freundlich adsorption isotherm (Figure 4) becomes more relevant.

The Freundlich isotherm investigates the diverse nature of the adsorbate and the exponential structure of the active site. It also refers to reversible adsorption and multilayer adsorption. The linearized form of the Freundlich adsorption isotherm expresses in Equation 5.

$$\ln q_e = \ln K + x \ln C_e \qquad (5)$$

where K is the adsorption capacity (mg/g), and x is the adsorption intensity. The x value shows the favorability of an adsorption process and the importance of the Freundlich isotherm displayed in Table 2.

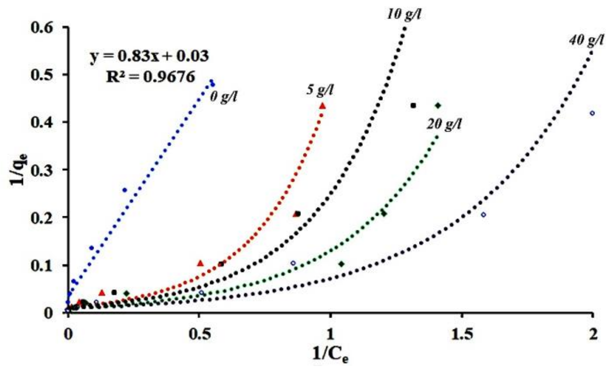


Figure 3: The Langmuir adsorption isotherm.

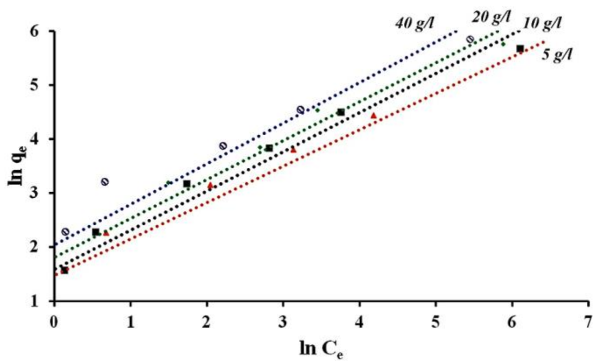


Figure 4: The Freundlich adsorption isotherm.

Table 2: Effect of KCl on Freundlich adsorption isotherm.

KCl (g/L)	ln K	x	R^2	CIE-Whiteness
5	0.86	0.56	0.9909	157.43
10	0.90	0.61	0.9893	160.74
20	1.01	0.59	0.9942	163.43
40	1.02	0.67	0.9945	163.52

In Table 2, the x value was between 0.56 and 0.67, depending on the concentration of KCl, suggesting that the adsorption process is more favorable for the Freundlich isothermal model (0 < x <1.0). This study, therefore, matches previous dyeing studies on multilayer dyeing adsorption [15, 20] in which the dyes and fibers had the same change following the dyeing mechanism for natural fiber fixation [6] as previous studies [35, 37]. This type of adsorption is usually seen in large molecular structures containing several chemical groups, facilitating the aggregation process due to forces attracting dyes and fibers in various forms, including hydrogen bonds and Van der Waals forces. FBA 113 adheres to the cotton fabric during the adsorption process, filling the first layer, and the dyeing continues, causing FBA 113 molecules in solution to continue superimposing. Meanwhile, the log K value shows electrolytes have released ionizers to a positive potassium ion (K^+), which allows the negative ions of FBA 113 and the negative ions on the surface of the cotton fabric (COO^-) to mix [4]. The CIE whiteness values become more prominent when the concentration of KCl is increased. This study concludes that the amount of electrolyte influences the dyeing adsorption process and that 20 g/L KCl is most appropriate for FBA 113 dyeing.

3.4. Dyeing kinetic models

A kinetic model is related to the motion of material bodies and forces and the associated energy, which includes the surface adsorption and diffusion into the pores. Moreover, the adsorptions revealed by mass transfer, diffusion control, and chemical reaction are essential for investigating the dyeing procedures. In this study, a kinetic model of the dyeing process was investigated by pseudo-first-order and pseudo-second-order models. The model widely used to analyze the adsorption of dye through the material surface means that the coefficient of determination (R^2) can be examined to test the authenticity of the selected model [17].

The pseudo-first-order model is a simple kinetic analysis of adsorption, which describes the absorption process of a species in an adsorbent particle using the ordinary first-order differential equation shown in Equation 6:

$$\frac{dq_t}{dt}=k_1(q_e-q_t) \quad (6)$$

where k_1 is the rate constant for the pseudo-first-order adsorption (min^{-1}), q_t is the amount of FBA 113 adsorbed per gram of cotton (mg/g), and q_e is the equilibrium adsorption (mg/g). At the initial condition $q_t = 0$ at $t = 0$ and $q = q_t$ at $t = t$, this was applied by integration as follows:

$$ln(q_e - q_t) = lnq_e - k_1 t \quad (7)$$

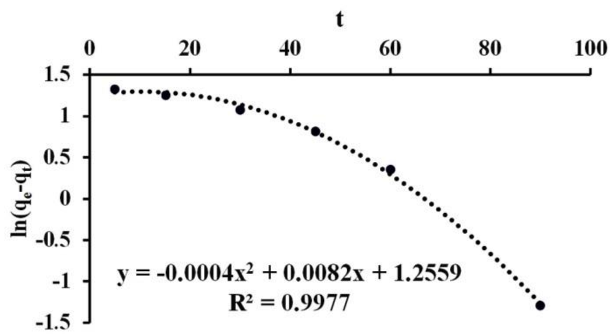


Figure 5: The pseudo-first-order model.

Figure 5 shows the relationship between $ln(q_e-q_t)$ and t from the dyeing of FBA 113 on cotton. The model did not fit the pseudo-first-order model and described a polynomial curve with an R^2 value of 0.9977. From this, we can conclude that the pseudo-first-order model does not adequately describe the adsorption of FBA 113 on cotton [17, 21, 22].

The pseudo-second-order model was employed to describe the dyeing process, as expressed as Equation 8:

$$\frac{dq_t}{dt}=k_2(q_e-q_t)^2 \quad (8)$$

The integration accounts for the boundary conditions, and the integrated liner is shown in Equation 9:

$$\frac{t}{q_t}=\frac{1}{k_2 . q_e^2}+\frac{t}{q_e} \quad (9)$$

where k_2 is the rate constant for the pseudo-second-order adsorption (g/mg.min). Meanwhile, the plot of the dyeing time (t) against the amount of FBA 113 adsorbed onto each gram of cotton (q_t) is shown in Figure 6.

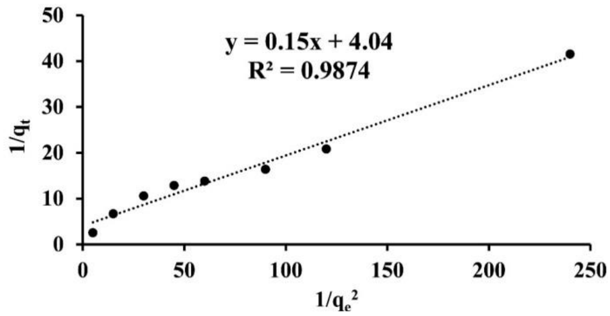


Figure 6: A pseudo-second-order model.

Figure 6 shows a good linearity match and offered good coefficients of linear regression at 0.9874, which confirms that a pseudo-second-order model describes the dyeing behavior of FBA 113 on cotton. This result corresponds with several studies on dyes that were adsorbed, such as Reactive Red II on natural cocoa shells [35, 36] and Atrazine on Apricot shells [35, 37].

3.5. Thermodynamic parameter of FBA 113 dyeing

Thermodynamic parameters are essential to our understanding of the effect of temperature on the adsorption process, which can be explored using the Van't Hoff equation, which relates changes in the equilibrium constant (K_{eq}) to temperature in a reaction. The Equation was expressed as Equation 10:

$$\ln K_{eq}=\frac{\Delta S^0}{R}-\frac{\Delta H^0}{RT} \quad (10)$$

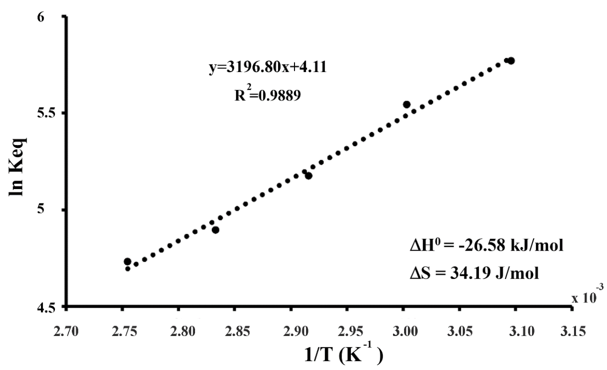
where K_{eq} (l/g) is the distribution coefficient for the adsorption, T is the absolute temperature (K), and R is the gas constant (8.314 J/mol K).

From the Equation, we can acquire the value of enthalpy (ΔH^0) and entropy (ΔS^0) from the intercept and slope of the Van't Hof plot from $\ln K_{eq}$ against 1/T. Subsequently, Gibb's energy can be calculated from the relationship expressed in Equation (11) and shown in the data in Table 3:

$$\Delta G^0=\Delta H^0-T\Delta S^0 \quad (11)$$

This study obtained a negative value ΔH^0 of -26.58 kJ/mol, confirming that FBA 113 was adsorbed onto cotton by an exothermic reaction corresponding to other dyeing processes [17, 21-23]. However, the entropy (ΔS^0) was positive at 34.19 J/mol.K, showing that the fiber arrangement was random in terms of adsorption [20, 24]. We can see that the universe's entropy, including systems and the environment, can increase because the absorbing reaction was not an isolated process [24]. In addition, the negative value of ΔG^0 shows that the adsorption of FBA 113 onto cotton was a spontaneous process in which temperature was a dominant factor in controlling the adsorption mechanism. The ΔG^0 value decreased as the temperature rise, imparting a lower rate of adsorption [24]. Generally, the ΔG^0 value can be used to predict the state of the mechanism, the physical reaction $-20 < \Delta G^0 < 0$ and the chemical reaction $-400 < \Delta G^0 < -80$ [15]. Therefore, this study showed that the dominant mechanism is the physical reaction.

Table 3: The thermodynamic parameters of adsorption FBA 113 on cotton.



Temp. (K)	323	333	343	353	363
ΔG^0 (kJ/mol)	-37.62	-37.96	-38.31	-38.65	-38.99

4. Conclusion

The process of FBA 113 dyeing reached a dyeing equilibrium at 323 K for 120 minutes when adding 20 g/KCl to enhance the adsorption ability and obtain a high CIE-whiteness value. We can conclude that the adsorption of FBA 113 was best fitted with the Freundlich adsorption isotherm. Meanwhile, the pseudo-second-order kinetic model was best at describing the kinetic result of FBA 113 adsorption onto cotton, and the thermodynamic parameters imply that the process is spontaneous, random, and exothermic. Finally, FBA 113 can be used to determine the whiteness achieved in the textile dyeing process.

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