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Article

Biosorption of Indigo Carmine onto Duck Egg White-Modified Longan Peel Biosorbent: Effect of Particle Size and Contact Time

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Abstract. The increasing demand in the textile industry ideally requires effective wastewater treatment systems to prevent environmental pollution. However, in practice, textile effluents still contain significant amounts of synthetic dyes, particularly indigo carmine, due to low dye fixation efficiency during the dyeing process. These pollutants are resistant to degradation and pose serious risks to aquatic ecosystems and human health. To address this issue, sustainable and low-cost treatment methods are urgently needed. Biosorption using agricultural waste has emerged as a promising alternative; however its performance is often limited by insufficient active binding sites. Therefore, surface modification using natural protein sources is proposed to enhance adsorption capacity and efficiency. This study aims to develop and evaluate longan peel modified with duck egg white (LP-DEW) as an eco-friendly biosorbent for indigo carmine removal. The effects of particle size and contact time were investigated, and adsorption kinetics were analyzed using pseudo-first-order, pseudo-second-order, and intraparticle diffusion models. The results showed that LP-DEW exhibited a maximum adsorption capacity of 128.27 mg g^{-1} at an optimal particle size of 63 µm and equilibrium time of 90 min. The adsorption process followed the pseudo-second-order model, indicating chemisorption as the dominant mechanism. In conclusion, LP-DEW demonstrates high adsorption efficiency and favorable kinetics, highlighting its potential as a sustainable biosorbent for dye-contaminated wastewater treatment.

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1. Introduction

The textile industry in Indonesia has experienced rapid and continuous growth, leading to the generation of substantial volumes of industrial wastewater [1]. Textile effluents typically contain hazardous synthetic dyes originating from scouring, bleaching, dyeing, and finishing processes. These stages contribute approximately 70-80% of total water pollution within the textile sector [2]. During the dyeing process, the dye fixation effiency on textile fibers ranges from only 2% to 50%, while the remaining fraction is discharged into wastewater streams [3]. Textile dyes are synthetic aromatic compounds that are generally stable and resistant to environmental degradation. As a result, dye-containing effluents pose significant risks to aquatic ecosystems and human health. One commonly used dye is indigo carmine (IC), which exhibits considerable toxicity even at relatively low concentrations [4-5].

Indigo carmine is widely applied as a coloring agent in denim manufacturing [6]. In addition, it is utilized as an indicator in various analytical applications and in certain aquaculture practices [7]. The chemical structure of IC, an anionic dye, is presented in Figure 1. It has a molecular weight of 466.36 g mol^{-1}, and a maximum absorbance wavelength at 610 nm. Exposure to this dye may cause adverse health efffects, including corneal demage, skin irritation, dermatitis, and respiratory and digestive disorders [8].

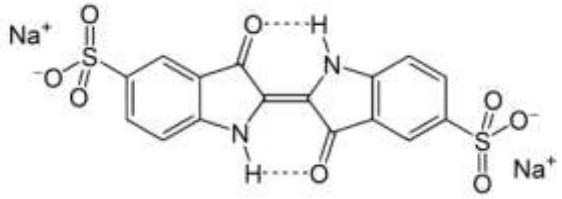


Figure 1. Molecular Structure of Indigo Carmine (IC) [5]

Biosorption is a physicochemical process that naturally occurs in certain types of biomass. Trough this mechanism, biological materials are able to passively accumulate and immobilize contaminants onto their cellular structures [9]. Biosorbents offer several advantages, such as environmental compatibility, low cost, ease of preparation, and the ability to reduce organic solid waste without requiring complex or expensive equipment [10-11]. Agricultural wastes are particularly attractive as biosorbents due to the presence of various functional groups, including carbonyl, carboxyl, phenolic, and amine groups, which facilitate effective binding of both cationic and anionic dyes [12]. Numerous studies have reported the utilization of agricultural waste-derived materials such as jengkol peel [13], durian peel [14], banana peel [15], matoa peel [16], and longan peel [17].

Previous investigations, have explored longan peel (*Dimocarpus longan* L.) as a biosorbent for Zn(II) adsorption; however, its biosorption perfomance relatively limited, with a maximum capacity of 28.41 mg g^{-1} [18]. This moderate perfomance has been attributed to its surface chemistry, which is

predominantly composed of polysaccharides (cellulose and hemicellulose), and lignin containing hydroxyl (-OH) and carboxyl groups (-COOH), while amine (-NH_2) functional groups are comparatively scarce. The limited availability of positively charged sites restricts effective binding of anionic dyes; therefore, surface modification is required to enhance its adsorption perfomance.

One promising strategy involves protein-based modification. Previous studies have demonstrated that protein incorporation significantly improves biosorption performance toward anionic dyes, including methyl orange [19]. Egg white protein, which contains ovalbumin, ovotransferrin, ovomucin, and lysozyme, is rich in amine and carboxyl functional groups that play crucial roles in adsorption mechanism. Under acidic conditions, amine groups become protonated, resulting in a positively charged surface capable of eletrostatically interacting with negatively charged species such as anionic dyes [20]. Therefore, protein-based modification is expected to increase both the number and effectiveness of active binding sites, strengthen surface interactions, and enhance adsorption capacity.

However, despite these advancements, several limitations remain in previous studies. Most existing biosorbents exhibit relatively low adsorption capacities, limited stability, or require complex modification processes. In addition, the utilization of natural protein sources as eco-friendly modifying agents is still insufficiently explored, particulary in combination with longan peel for anionic dye removal. Furthermore, studies investigating the combined effect of operational parameters, such as particle size and contact time, along with adsorption kinetics, are still limited. Therefore, there is a clear research gap in developing a simple, sustainable, and efficient protein-modified biosorbent with improved performance for indigo carmine removal. This study aims to develop and evaluate longan peel modified with duck egg white (LP-DEW) as a biosorbent for indigo carmine removal from aqueous solutions. The effects of particle size and contact time on adsorption performance are investigated, and the adsorption mechanism is analysed using kinetic modeling.

2. Experimental Section

2.1 Materials

The experimental apparatus included standard laboratory glassware, ultrasonic cleaner (Laboratte 17), sieves (Save Shaker AS 200 control), a mechanical shaker (VRN-480), pH meter (Methrom 744), an analytical balance (ABS 220-4), filter paper, magnetic stirrer (MR Hei Standard), a mortar and pestle, and spray bottles. Fourier transform infrared (FTIR) spectra were recorded using a PerkinElmer instrument equipped with a Universal ATR sampling accessory (735 B). Dye concentrations were determined using a UV-Vis spectrophotometer (Agilent 8435, model G1103A). The materials employed in this study included longan peel, duck egg white, distilled water, indigo carmine (1000 mg L^{-1}), ethanol (96%), sodium hydroxide (NaOH, 5 M), and nitric acid (HNO_3, 5 M).

2.1.1 Modification Longan Peel with Duck Egg White

Longan peel was thoroughly washed with distilled water to remove surface impurities and subsequently air-dried at ambient temperature. The dried material was then ground using a mortar and pestle sieved to obtain particle size ≤45, ≤63, ≤125, and ≤180 μm. Chemical activation was performed by immersing the longan peel powder in 0.01 M nitric acid (HNO_3) solution for 2 h, following a prevously reported methode [5]. This treatment was intended to enhance pore accessibility and activate surface functional groups. After activation, the material was filtered and rinsed with repeatedly distilled water until neutral pH was achieved. The biosorbent was then air-dried at room temperature prior to further use.

Fresh duck eggs were used as the source off egg white, wich was separated and air-dried under ambient conditions for one week. The dried egg white was then ground and sieved to obtain particle size ≤45, ≤63, ≤125, and ≤180 μm. Subsequently, 9 g of longan peel powder was mixed with 9 g of dried egg white, followed by the addition of 75 mL ethanol. The mixture was subjected to

ultrasonication for 15 min to enhance dissolution and disrupt micron-sized colloidal particle aggregates. The modified biosorbent was then filtered, air-dried, and prepared for subsequent experiments. The resulting material was designated as LP-DEW [5].

2.2 Adsorbent Characterization

LP-DEW was characterized using several analytical techniques to evaluate its physicochemical properties before and after adsorption process. The functional groups involved in the adsorption process were dentifed by Fourier Transform Infrared spectroscopy (FTIR).

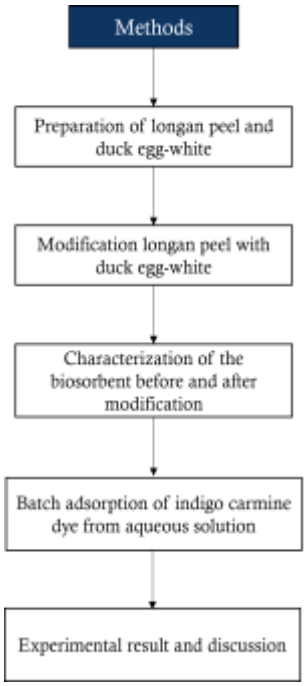


Figure 2. Flowchart of the Research Methodology

2.3 Batch Biosorption Experiment

Biosorption experiments were conducted using the batch technique. The effects of particle size (≤45, ≤63, ≤125, and ≤180 μm) and contact time (30, 60, 90, 120, 150, 180, and 210 min) were investigated. All experiments were performed at pH 2 with an initial dye concentration of 1200 mg L^{-1} under continuous agitation at 150 rpm.

$$qe = \frac{(C_e - C_0)\, V}{m}$$

3. Results and Discussion

3.1 Functional Group Analysis

FTIR spectroscopy was employed to identify changes in the surface functional groups of the biosorbent before modification , after modification with duck egg white, and following the adsorption of indigo carmine dye. The FTIR spectra of LP, LP-DEW, and LP-DEW + IC are presented in Figure 3.

The FTIR spectrum of raw longan peel (LP) was recorded within the wavenumber range of 4000-600 cm^{-1}. A broad absorption band observed at 3286 cm^{-1} is attributed to O-H stretching vibrations

associated with hydroxyl groups [21]. The peak at 2923 cm^{-1} corresponds to aliphatic C-H stretching, indicating the presence of lignocellulosic components [22]. The band detected at 1616 cm^{-1} is assigned to aromatic skeletal vibrations of lignin and/or conjugated carbonyl (C=O) groups [23]. Additionally, absorption bands at 1235 and 1023 cm^{-1} are related to C-O and C-O-C stretching vibrations, characteristic of glycosidic linkages in cellulose and hemicellulose [24].

Following modification with duck egg white (DEW), notable spectral changes were observed in LP-DEW. The broad band around 3278 cm^{-1} is associated with overlapping O-H and N-H stretching vibrations [25], reflecting the incorporation of protein-derived functional groups. The appearance of bands at 1628 and 1524 cm^{-1} correspond to amide I (C=O stretching) and amide II (N-H bending and C-N stretching) vibrations [26], confirming successful modification of egg white protein onto the LP surface. The band at 1239 cm^{-1} is attributed to C-N stretching further supporting protein attachment [26]. The C-H stretching band around 2926 cm^{-1} remains characteristic of the lignocellulosic backbone of LP [27].

After adsorption of indigo carmine (LP-DEW + IC), additional spectral variations were detected, suggesting interactions between the dye molecules and the modified surface. The O-H/N-H band shifted to 3227 cm^{-1}, indicating possible hydrogen bonding and electrostatic interactions involving protonated amine groups [28]. A slight displacement of the amide II band from 1524 to 1526 cm^{-1} further supports the participation of functional groups during dye uptake [28]. Moreover, the emergence of an absorption band around 1161 cm^{-1} is assigned to S=O stretching vibrations of sulfonate groups, confirming the attachment of sulfonated dye species onto the biosorbent surface [29].

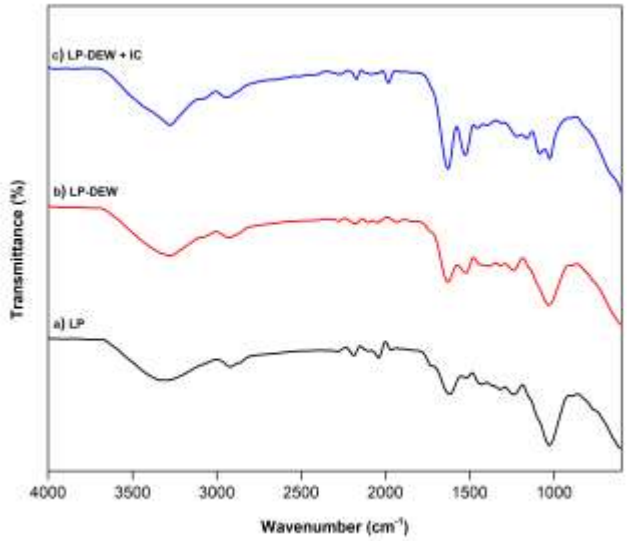


Figure 3. FTIR Spektrum of (a) Longan peel (LP), (b) Longan peel modified with duck egg white (LP-DEW), (c) Longan peel modified with duck egg white after contact with indigo carmine (LP-DEW + IC)

3.2 Standard Curve Preparation of LP-DEW

A calibration curve was constructed prior to the determination of optimum conditions to ensure accurate concentration analysis of indigo carmine [30]. In accordance with the Lambert-Beer law, absorbance increased proportionally with increasing concentration [31]. The regression equation obtained was $y = 0.0221x + 0.0011$ with a coefficient of determination of $R^2 = 0.9997$, indicating excellent linearity. The corresponding calibration curve is presented in Figure 4.

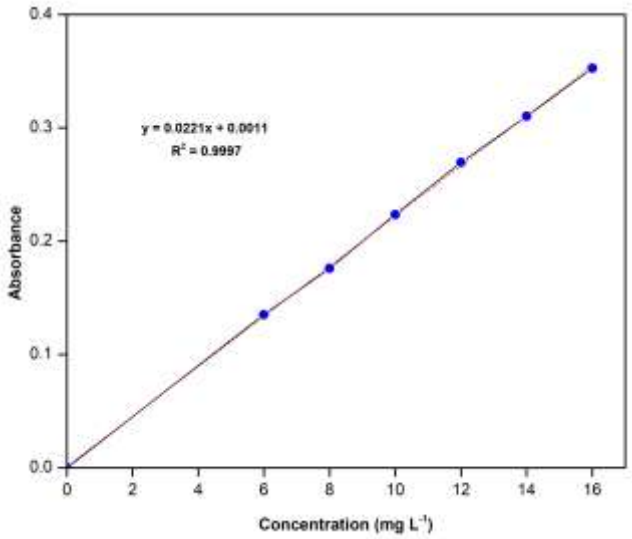


Figure 4. Preparation of Indigo Carmine Calibration Curve

3.3. Effect of Factors on Biosorption

3.3.1 Effect of Particle Size

Adsorption experiments were conducted to evaluate the effect of particle size using 0.2 g of LP-DEW with particle fractions ≤45, ≤63, ≤125, and ≤180 µm. The experiments were performed in 25 mL of indigo carmine solution (1200 mg L^{-1}) under constant agitation at 150 rpm. The results indicate that particle size significantly influences adsorption performance. The highest adsorption capacity was obtained at 63 µm, suggesting that particle size plays a critical role in adsorption efficiency.

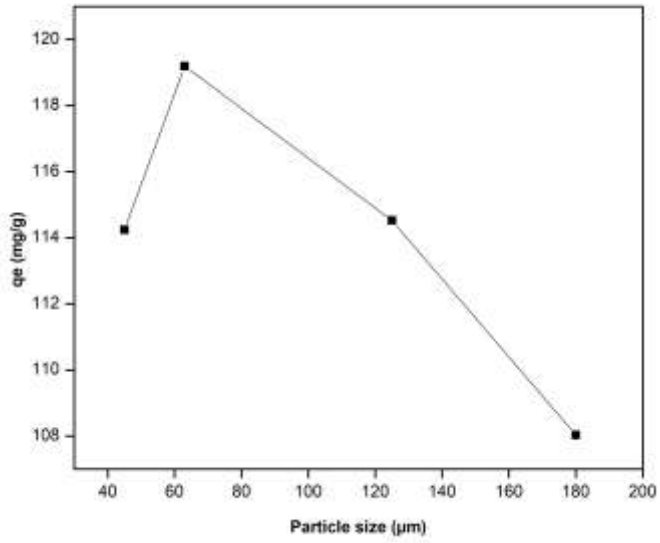


Figure 5. Effect Particle Size of LS-DEW on IC Dye Biosorption

Quantitatively, the adsorption capacity increased from 114.24 mg g^{-1} at 45 µm to 128.27 mg g^{-1} at 63 µm. This improvement is attributed to the increased specific surface area and the greater availability of active binding sites at smaller particle sizes. However, further reduction to 45 µm resulted in

decreased adsorption capacity. This behavior can be explained by particle aggregation and pore blockage, which limit dye diffusion and reduce the effective surface area [32].

In contrast, larger particle sizes (125 μm) exhibited lower adsorption performance due to reduced surface area and limited accessibility of internal pores. These limitations restrict the interaction between dye molecules and active sites [33]. This indicates that adsorption is governed not only by surface area but also by mass transfer efficiency, where excessively small or large particles negatively affect performance.

These findings are consistent with recent studies on lignocellulosic biosorbents, which reported that an optimal particle size is required to balance surface area and diffusion limitations. Excessive reduction in particle size may lead to particle aggregation and decreased adsorption efficiency due to restricted mass transfer and pore accessibility [9]. Overall, the optimal particle size of 63 μm provides an optimal balance between surface exposure and diffusion pathways, resulting in improved adsorption performance.

3.3.2 Effect of Contact Time

The effect of contact time on indigo carmine adsorption was investigated over a range of 30–210 min at 30 min intervals. The experiments were conducted using 0.2 g of LP-DEW in 25 mL of dye solution (1200 mg L^{-1}) under constant stirring at 150 rpm. The adsorption capacity increased rapidly during the initial stage and reached a maximum value of 128.27 mg g^{-1} at 90 min. This result indicates a fast adsorption rate and efficient utilization of available binding sites.

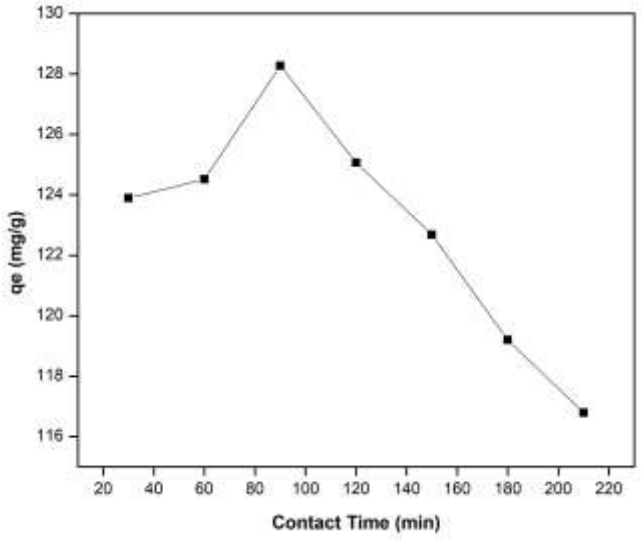


Figure 6. Effect Contact Time on IC Dye Biosorption

Quantitatively, the adsorption process reached equilibrium within 90 min, indicating relatively rapid kinetics compared to similar adsorption systems. For instance, adsorption using Bi_2O_3-doped MgO nanoparticles reached equilibrium at approximately 74 min with a comparable adsorption capacity of 126 mg g^{-1} [34]. In contrast, lignocellulosic biosorbents such as kapok husk generally require around 60 min but exhibit lower adsorption capacities [35]. These comparisons suggest that LP-DEW provides a favorable balance between adsorption rate and capacity.

The rapid adsorption observed at the initial stage can be attributed to the abundance of accessible active sites and the strong concentration gradient between the solution and the biosorbent surface, which enhances mass transfer [36]. This indicates that external surface adsorption and film diffusion

dominate the early stage of the process. As contact time increases, the availability of active sites decreases, leading to a gradual reduction in adsorption rate.

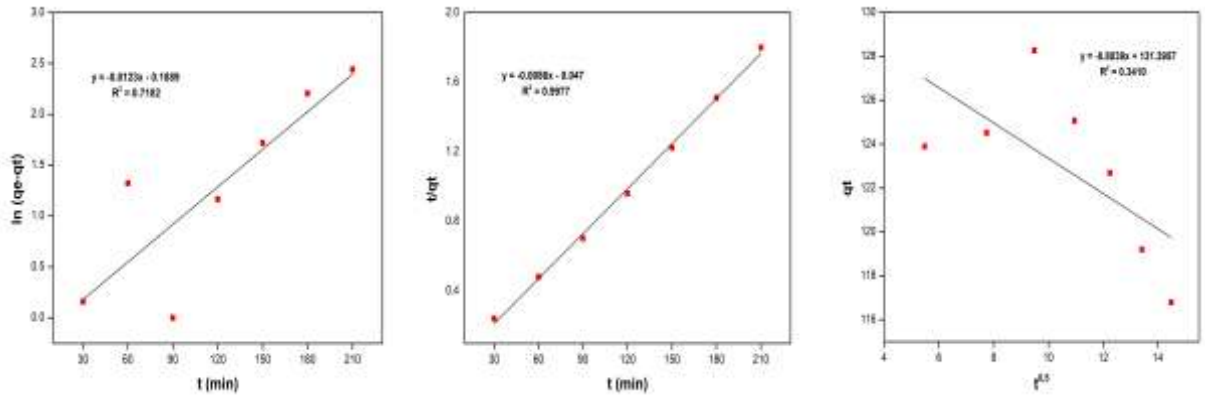
Beyond 90 min, a slight decrease in adsorption capacity was observed up to 210 min, indicating the establishment of adsorption–desorption equilibrium. This behavior suggests that surface sites become saturated and the rate of adsorption decreases due to limited active sites. Additionally, partial desorption or redistribution of dye molecules may occur on the biosorbent surface, contributing to the observed decline in adsorption capacity [37]. Overall, LP-DEW demonstrates efficient adsorption kinetics and strong potential for indigo carmine removal.

3.4. Kinetics Modeling

The biosorption process can be interpreted using kinetic models to clarify the adsorption rate and the governing mechanism involved. The physicochemical properties of the biosorbent, together with mass transfer phenomena, strongly influence the overall biosorption behavior. A kinetic model is considered appropriate when the tcoeffcient of determination (R^2) approaches unity. In this study, the adsorption kinetics were evaluated using the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models.

The kinetic analysis indicates that the pseudo-second-order model provides the best fit to the experimental data, as evidenced by the highest R^2 value compared to the pseudo-first-order and intraparticle diffusion models. This suggests that the adsorption process is predominantly governed by chemisorption, involving electron sharing or exchange between the functional groups of LP-DEW and indigo carmine molecules. In contrast, the pseudo-first-order model showed a lower correlation, indicating that physisorption alone is insufficient to describe the adsorption mechanism.

Quantitatively, the calculated adsorption capacity obtained from the pseudo-second-order model is in close agreement with the experimentally obtained value of 128.27 mg g^{-1}, confirming the reliability of this model. Similar findings have been widely reported in previous studies, where indigo carmine adsorption using various biosorbents and carbon-based materials followed pseudo-second-order kinetics, indicating chemisorption as the dominant mechanism [36-39]. For instance, adsorption using *Terminalia catappa* shell and activated carbon also showed that the pseudo-second-order model provided the best fit to experimental data, confirming the involvement of surface functional groups in the adsorption process [36-37].



(a) (b) (c)

Figure 7. Pseudo-First-Order (a) Pseudo-Second-Order (b), and Intra-Particle Diffusion (c) Kinetics Plot for IC adsorption onto LP-DEW

The intraparticle diffusion model exhibited a lower R^2 value and did not pass through the origin, indicating that intraparticle diffusion is not the sole rate-limiting step. Instead, the adsorption process likely occurs through multiple stages including external surface adsorption followed by gradual diffusion into internal pores. The intercept constant (C) also suggests the presence of boundary layer effects, where a higher C value reflects a greater contribution of surface resistance during the adsorption process [37-38].

These results are consistent with recent studies, which reported that adsorption of indigo carmine using modified biosorbents typically involves multiple menchanism, including chemisorption and diffusion processes [36-39]. Overall, the dominance of the pseudo-second-order model, combined with the relatively high adsorption capacity, indicates that LP-DEW exhibits efficient adsorption kinetics and strong interaction with indigo carmine.

Comparison of kinetic fitting parameters revealed that the pseudo-second-order model exhibited superior correlation coefficients relative to the pseudo-first-order model. This finding suggests that indigo carmine uptake onto LP-DEW is governed predominantly by chemisorption involving surface chemical interactions.

Table 1. Kinetic Parameters for Indigo Carmine Adsorption onto LP-DEW

Kinetic Models	Equation	Parameters	Result
Pseudo-firs Order	$\ln(qe - qt) = -lnqe + k_1 t$	qe_{exp} (mg g^{-1})	128.7
		k_1 (min^{-1})	0.0283
		qe_{calc} (mg g^{-1})	0.78
		R^2	0.7182
Pseudo-second Order	$\frac{t}{qt} = \frac{1}{k_2 qe^2} + \frac{t}{qt}$	k_2 (g.mg^{-1}. min^{-1})	0.000924
		qe_{calc} (mg g^{-1})	151.52
		R^2	0.9977
Intra-particle diffusion	$qt = k_{diff}\, t^{0.5} + C$	C (mg.g^{-1})	139.357
		K_{diff} (mg g^{-1}.$min^{-0.5}$)	4.803
		R^2	0.3410

4. Conclusion

Duck egg white-modified longan peel (LP-DEW) demonstrated strong potential as an effective biosorbent for indigo carmine removal from aqueous media. Optimal adsorption performance was achieved at pH 2 using a particle size of 63 μm, resulting in a maximum adsorption capacity of 128.27 mg g^{-1}. Kinetic analysis indicated that the adsorption behavior was best described by the pseudo-second-order model, suggesting that chemisorption governs the dye uptake mechanism.

5. Acknowledgement

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