



## *The Kinetic Analysis and Adsorption Isotherm of Chicken Egg Shells and Membranes Against Synthetic Dyes*

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### ABSTRACT

Textile industry waste at this time is enough to worry the community and the environment. The presence of synthetic dyes in water is hazardous, even in small concentrations. These synthetic dyes are derivatives of aromatic compounds such as benzene, toluene, and naphthalene, which are more resistant and stable than natural dyes. The adsorption method is used because it is easier to do, has no side effects, and does not require complicated and expensive equipment. In this study, the shells and membranes of discarded chicken eggs became useful as an adsorbent of indigo carmine dye with an adsorption capacity of 6.399 mg/g. The adsorption reaction kinetics were analyzed from the optimal contact time data, and the reaction isotherm was analyzed from the adsorption optimal concentration data. The kinetic model that fits the research is the second pseudo-order with  $R^2 = 0.9998$ . The adsorption mechanism demonstrates that the adsorption capacity is proportional to the adsorbent's active sites. The adsorption isotherm model, with  $R^2 = 0.9748$ , is more closely related to the Freundlich isotherm model, indicating that adsorption occurs in several layers. From an economic point of view, chicken egg shells and membranes can be recommended as dye adsorbents that are eco-friendly, efficient, and simple to obtain while lowering organic solid waste.

## 1. INTRODUCTION

Along with the rapid population and industry growth, the volume of waste released into the environment is increasingly unstoppable. One of these wastes is textile waste in synthetic dyes that damage human health and the environment. For example, synthetic dyes can disrupt the ecosystem of aquatic biota, even in small concentrations. This is due to reduced sunlight, which can interfere with aquatic plants' photosynthesis and cause acute toxicity to aquatic life (Sharma, Dalai, & Vyas, 2017). Meanwhile, waste discharged into nature without being processed first will pollute the surrounding environment. For example, if wastewater is discharged to the ground, it will make the soil

become damaged and reduce fertility. In addition, industrial waste can pollute the river because of the many chemicals contained in it. Eye irritation, skin irritation, mutagenicity, and carcinogenicity are all side effects of these colours (Hevira and Zein, 2022; Wang et al., 2017)

Many methods for treating dyed wastewater have been reported, including chemical coagulation, activated sludge, biodegradation and magnetic separation (Rahmayeni, Arief, Jamarun, Emriadi, & Stiadi, 2017), and nanofiltration (Abid, Zablouk, & Abid-Alameer, 2012). However, this method is still not widely used because of the high cost and long process. Currently, the government is promoting and supporting the use of waste by providing other benefits to adsorb waste using green technology

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(Hevira, Ariza, & Rahmi, 2021). Meanwhile, from an economic perspective, the use of eggshells can reduce the cost of processing textile waste and also has the potential to be developed in Indonesia.

One of the appropriate methods is the adsorption method. The adsorption method is the most commonly used because it is reliable, has no harmful side effects, and does not necessitate complicated and expensive equipment. Biosorption is an adsorption method that utilizes natural adsorbents (Hevira, Zein, & Ramadhani, 2019) derived from plants such as orange peel (Arami, Limaee, Mahmoodi, & Tabrizi, 2005), melon peel (Djelloul & Hamdaoui, 2015), and banana peel (Abudi, Lattieff, Chyad, & Abbas, 2018). In addition, temporary animal waste can be utilized, such as chitosan from fish scales (Iqbal et al., 2011), crab shells (Fatombi et al., 2019), and chicken feathers (Bagaskoro et al., 2020).

Chicken egg shells are often not utilized and even become waste in large quantities, which are sent to landfills with high management costs. Whereas chicken egg shells contain mostly calcium carbonate and have 10,000-20,000 pores, they can be used as an adsorbent. Chicken egg shells can be utilized as a calcium supplement and green manure. The skin's membrane can also be used as cosmetics because they contain collagen (Faridi & Arabhosseini, 2018). Meanwhile, egg white has been used as a modifier to adsorb dyes (Rahmiana Zein et al., 2022). The ability of chicken eggshells to adsorb pollutants is because it has many pores and functional groups such as C-H, C-C, C-O, C=O, NH, and SH that can bind with cations or anionic compounds (Hevira, Rahmi, et al. 2020). The use of chicken egg shells is a low-cost adsorbent (Carvalho, Araujo, & Castro, 2011) that can absorb dyes in the textile industry. Besides, the eggshell membrane has amino acids that can interact with dyes.

Previous research showed that the use of egg shells and their membranes has a good adsorption capacity (Pramanpol & Nitayapat, 2006) (Hevira, Rahmi, et al., 2020). Therefore, this time the author will look at the reaction kinetics tested using the first pseudo-order, second pseudo-order, and equations of intra-particle diffusion.

Meanwhile, the Langmuir and Freundlich isotherm equations are used to perform adsorption analysis.

## 2. METHODS

### 2.1. Adsorbent Preparation

Chicken egg shells and membranes (CESM) were collected in Bukittinggi, West Sumatra, Indonesia. Nitric acid, NaOH, indigo carmine dye, and aquadest. Instruments: shaker, pH meter, spectrophotometer UV-Vis. Procedures: CESM washed with flowing water, dried, and in a blender using (Turbo 8099). After that, it was sieved with a particle size of 36  $\mu\text{m}$  (Hevira, Rahmi, et al. 2020). Then 0.1 g of CESM with a particle size of 36  $\mu\text{m}$  was added to 50 mL dye solution with variation concentration, then stirred with a shaker at 100 rpm with the respective optimum contact time. After that, the solution was filtered, and the filtrate was analyzed by UV-vis spectrophotometer at max.

### 2.2. Activation of an Adsorbent

In 75 millilitres of 0.01 N  $\text{HNO}_3$ , 25 grams of CESM was immersed for 2 hours to open the pores of CESM and activate the active adsorbent (Sopiah, Prasetyo, and Aviantara 2017). After that, the CESM was rinsed with distilled water until the pH returned to normal. After drying, CESM was ready for use.

### 2.3. Determination of Optimal Contact Time for Adsorption

At the optimum pH previously determined, 0.1 g CESM is added to 25 mL indigo carmine at a concentration of 10 mg/L into erlenmeyer 50 mL. The addition of nitric acid and base is used to change the pH level. The stirring time ranged between 15 and 60 minutes at 100 rotations per minute. The adsorption process was then filtered, and the concentration was determined using a UV-Vis spectrophotometer.

Adsorption capacity against indigo carmine calculated by equation 1:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

Where  $C_0$  and  $C_e$  denote the initial and equilibrium dye concentrations (mg/L),  $m$  denotes the mass of CESM (g), and  $V$  denotes the volume of the indigo carmine reaction mixture (L).

#### 2.4. Reaction Kinetics

Reaction kinetics analysis predicts the speed at which adsorbate is transferred from solution to adsorbent. In this research, the reaction kinetics can be seen from the reaction kinetics of the indigo carmine in CESM. The first pseudo-order (Lagergren) and second pseudo-order adsorption kinetics models were used to calculate the adsorption kinetics equations (Ho & Mc Kay).

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

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

Where :

$q_e$  = equilibrium adsorption capacity in mg / g

$q_t$  = time  $t$  adsorption capacity in mg/g

$k_1$  denotes the first pseudo-order reaction rate constant in minutes<sup>-1</sup>

$k_2$  denotes the second pseudo-order rate constant in g/mg.min

The intra-particle diffusion model was used to explain the adsorption process. As shown in equation 4, the Weber and Morris equations express intra-particle diffusion.

$$q_t = K_{ip} t^{0.5} + C \quad (4)$$

Where  $K_{ip}$  ( $\mu\text{mol/ g. min}^{0.5}$ ) is the constant rate intra-particle diffusion, and  $C$  ( $\mu\text{mol/g}$ ) is the thickness layer limit, with plotting  $q_t$  versus  $t^{0.5}$  then we get the slope and intersection of straight lines in the form of  $K_{ip}$  and  $C$  (Marcel et al. 2019).

#### 2.5. Determination of the Initial Dye Concentration

The same procedure used to evaluate the time available was used to determine the early concentration of indigo carmine. The dye studied, in this case, has an initial concentration of 10-50 mg/L.

#### 2.6. Isotherm of Adsorption

The isotherm of adsorption is determined by the relationship between the concentration of the adsorbed solute on the solid and the concentration of the solution. Langmuir isotherm is used to determine whether adsorption occurs in the monolayer or not, while the isotherm Freundlich assumed that adsorption occurs in a multilayer manner. The formula used in this case is the Langmuir and Freundlich equation.

$$\frac{1}{q_e} = \frac{1}{q_m k_L} \frac{1}{C_e} + \frac{1}{q_m} \quad (5)$$

$$k_L = \frac{1}{q_m a} \quad (6)$$

$$R_L = \frac{1}{1+(1+k_L C_e)} \quad (7)$$

$$\log q_e = \log k_F + \frac{1}{n} \log C_e \quad (7)$$

Where :

$q_m$  : capacity adsorption maximum

$q_e$  : capacity adsorption in state balanced

$C_e$  : ion concentration in state balanced

$k_L$  : Langmuir's constant Among sorbent with sorbate

$R_L$  is the separation factor, where  $R_L$  in the form of bad ( $R_L > 1$ ), linear ( $R_L = 1$ ), beneficial ( $0 < R_L < 1$ ), and irrecoverable ( $R_L = 0$ ).

$k_F$  : Constant of Freundlich denoting capacity adsorption

$1/n$  : Intensity adsorption

### 3. RESULT AND DISCUSSION

#### 3.1. The Influence of Agitation Time

The Influence of agitation time greatly affects whether or not adsorption occurs. In Figure 1, we can see that as the agitation time of the reaction between the indigo carmine and CESM increases, the adsorption capacity decreases. Although the reduction is minimal, the capacity adsorption of The optimum CESM against indigo carmine occurred at a 15 minutes contact time. This fast time is possible because indigo carmine is an anionic dye that will quickly interact with eggshell membranes which contain lots of amino acids. The same thing also happened during the adsorption of indigo carmine using Ketapang shells; the adsorption capacity was low initially (Hevira, Zilfa, et al. 2020). Then if it is modified with egg white which contains

rich amino acids, the adsorption capacity will increase (Rahmiana Zein et al., 2022). This is also the advantage of this research compared with Rahmiana Zein's studies. This research is better because it is more effective and efficient in terms of time and cost, besides it is also easy to implement, as explained in part 3.5. However, there are still shortcomings; the amount of  $\text{CaCO}_3$  contained in eggshells can affect the hardness of water (Aidha, 2013).

Meanwhile, suppose the contact time between the adsorbate and the adsorbent continues to be increased after reaching the optimum capacity. In that case, it will potentially inhibit the adsorption of the adsorbate and even release the bonds between the adsorbate and the adsorbent so that the adsorption capacity will decrease.

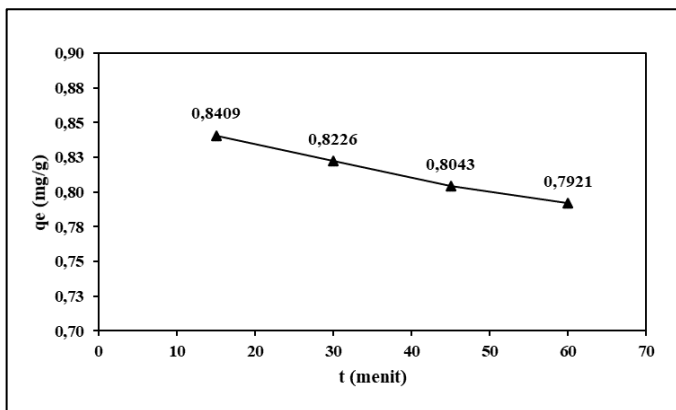


Figure 1. Effect of contact time on adsorption capacity of CESM to adsorb indigo carmine

### 3.2. Reaction Kinetics

The first pseudo-order model was derived from the Lagergren reaction rate equation for liquid and solid adsorption based on solid capacity. While the second pseudo-order is suggested that the number of available sites on the adsorbent determines the adsorption ability. (Liu et al. 2016).

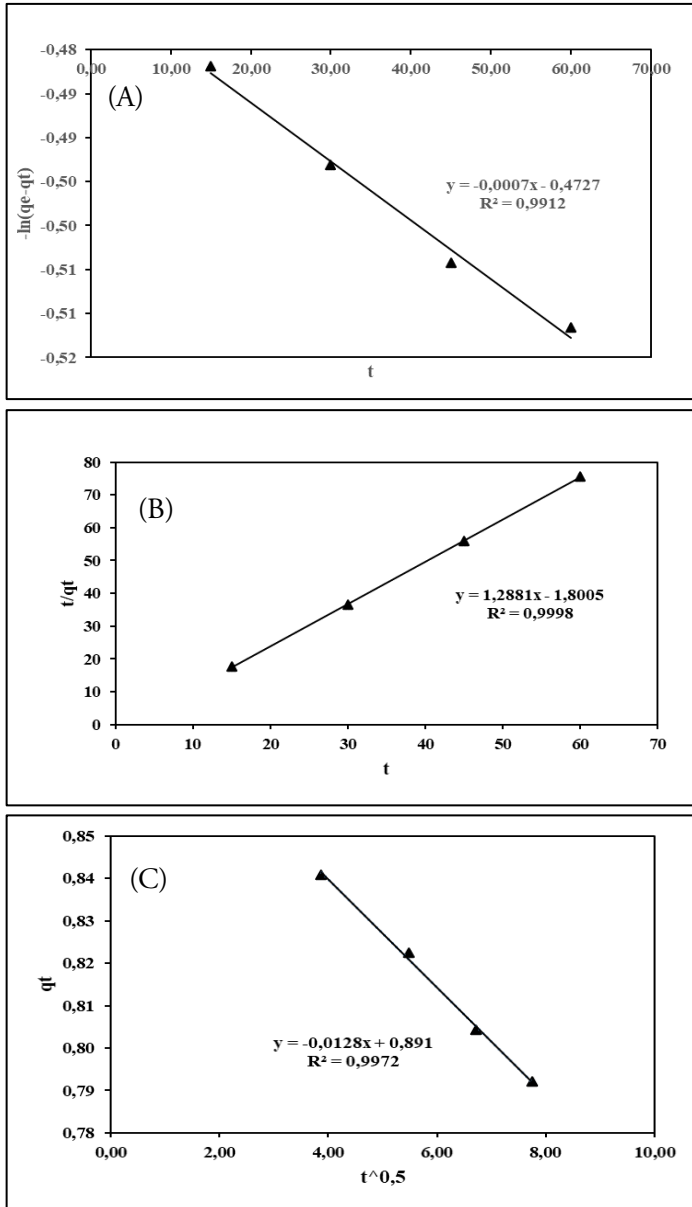
Figures 2A and 2B show that the  $R^2$  value of the first-order pseudo equation is 0.9912, while the coefficient of determination of the second pseudo-order is 0.9998. Thus the second pseudo-order kinetic model is a better way to explain the reaction rate for indigo carmine in CESM (Budnyak et al. 2018).

Figure 2C shows that the adsorption process in the intra-particle diffusion equation begins with the passage of particles from outside the adsorbent to the adsorbent's surface, followed by the diffusion of molecules into the pores. (Holle, Wuntu, and Sangi 2013). For dye adsorption by CESM, the coefficient of determination ( $R^2$ ) is 0.9972, which means that the only model that restricts the adsorption rate is the intra-particle diffusion model.

Table 1. CESM's Kinetic Parameter of IC Adsorption

Variant Parametric	Variant Parametric	CESM +IC
Trial Data	$q_e (exp)$ (mg/g)	0.8409
	$k_1$ ( $\text{min}^{-1}$ )	0.0007
First pseudo-order	$q_e (calc)$ ( $\text{mg}\cdot\text{g}^{-1}$ )	1.6043
	$R^2$	0.9912
	$k_2$ ( $\text{g}\cdot\text{mg}^{-1}\cdot\text{minute}^{-1}$ )	0.9215
Second pseudo-order	$q_e (calc)$ ( $\text{mg}\cdot\text{g}^{-1}$ )	0.7763
	$R^2$	0.9998
	C	0.8910
Intra- particle diffusion	$K_{diff}$ ( $\text{mg}\cdot\text{g}^{-1}\cdot\text{min}^{-0.5}$ )	0.0128
	$R^2$	0.9972

Table 1 shows that the value of  $q_e$  in the treatment and  $q_e$  in the pseudo-second-order equation are closer, namely 0.8409 with 0.7763. This slightly adjacent value is confirmed by an  $R^2$  value of 0.9998. Meanwhile, the value of  $q_e$  obtained in the experiment and  $q_e$  in the pseudo-first-order equation is further, namely 0.8409 and 1.6043. This value is confirmed by the value of  $R^2$ , which is 0.9912. The same result also happened to the adsorption of indigo carmine by *Ocimum gratissimum* (Adewumi O. Dada et al. 2020).



**Figure 2.** Adsorption kinetics of indigo carmine dye utilizing (A) first pseudo-order kinetics, (B) second pseudo-order kinetics, (C) intra-particle diffusion model

### 3.3. The Influences of the Early Dye Concentration

The influences of the early dye concentration were investigated in the 10 to 50 mg/L range. The adsorption capacity of CESM against indigo carmine increases as the concentration of the dye indigo carmine increases. With increasing concentrations of both dyes, the amount adsorbed at equilibrium increases. This is because enhancing the initial dye level enhances the diffusion's primary driver, i.e., concentration gradient, which favours

the velocity at which dyes migrate from solutions to the surface of the adsorbent (Marcel et al. 2019). At a concentration of 50 mg/L, Figure 3 demonstrates that CESM had an optimal adsorption capacity against indigo carmine of 6.3963 mg/g. The adsorption capacity is quite high compared to fluoride adsorption on eggshells (Bhaumik et al. 2012).

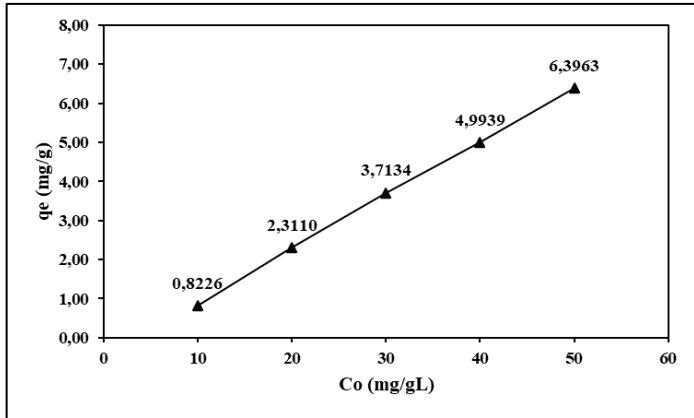
Meanwhile, the greater the starting concentration of indigo carmine, the more active side of the adsorbate or CESM will be saturated so that the adsorption capacity will decrease (Hevira and Zein, 2022).

### 3.4. Adsorption Isotherm

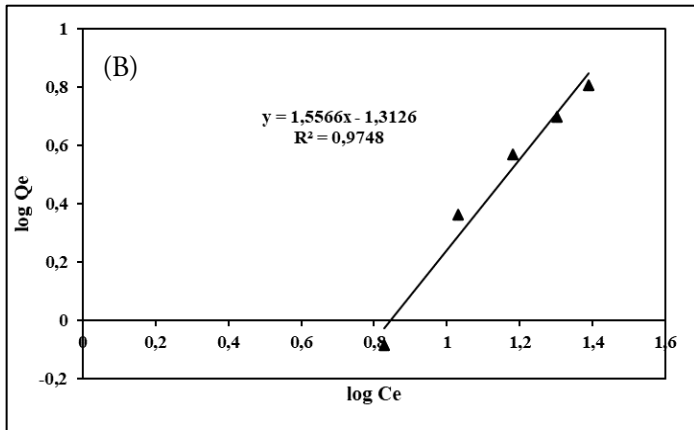
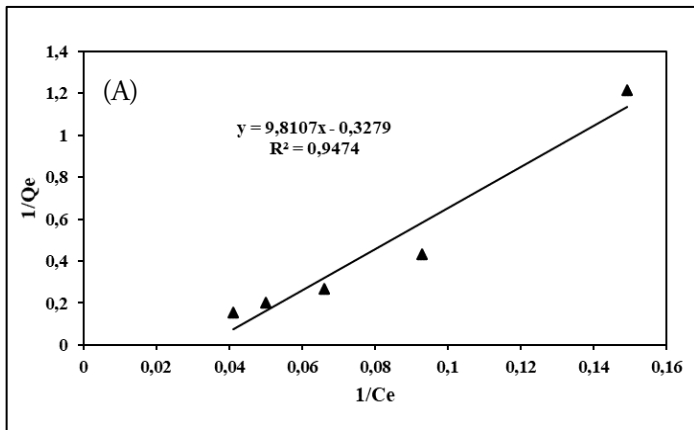
Isotherm Adsorption is a connection among many components absorbed by the adsorbent when the state is in equilibrium at a constant temperature. Isotherm adsorption is a function of concentration substance solute that is adsorbed on the solid to concentration solution.

In Figure 4, it can be seen that the coefficient of determination of the Langmuir equation used is 0.9474. While the value of the coefficient of determination using the Freundlich isotherm is 0.9748. Since the value is closer to 1, this data indicates that the Freundlich equilibrium model is more appropriate for the data gathered from the experiment. It demonstrated that indigo carmine could be absorbed using CESM more dominant in multilayer. The isotherm of Freundlich assumes the surface of the adsorbent was heterogeneous, and each molecule has potency different adsorption. The same thing also happened to the adsorption of metal ions using the Ketapan shell (Hevira, Munaf, and Zein, 2015). The value of the adsorption intensity or  $n$  from the Freundlich isotherm model is 0.6424. It suggests that the intensity has increased of adsorption of the dye with the biosorbent went well because it was in the range of 1-10.

In the meantime, the correlation coefficient value of the Langmuir model is 0.9474, as shown in Table 2. Its worth is also quite good, indicating that the adsorption also occurs in a monolayer. This is confirmed by the RL value of 0.5631, where if the RL is greater than 0 and smaller than 1 at equilibrium, it indicates that the adsorption is going well (A. O. Dada, Ojediran, and Olalekan 2013).



**Figure 3.** The effect of early dye concentration at the start on CESM's ability to eliminate indigo carmine



**Figure 4.** (A) Langmuir, (B) Freundlich isotherm linear equation of indigo carmine dye adsorption at CESM

### 3.5. Analysis of Adsorption Costs

A cost analysis of the colour waste treatment was also conducted from an economic viewpoint. In many studies, adsorbents used as activated carbon generally have

a high absorption capacity, such as chitosan-activated carbon (Fatombi et al. 2019) and date palm-derived activated carbon (Khadhri et al. 2019). However, if the adsorbent is activated with acid, the absorption capacity is almost the same as that of the activated carbon adsorbent. This was also proven by Gupta et al. when negatively absorbing charged Cr(VI) metal ions, similar to the absorption of indigo anionic dyes. The absorption cost of 1 g Cr(VI) using acid-modified bagasse is 11 times cheaper than the absorption of Cr(VI) using bagasse-activated carbon with almost the same adsorption capacity (Gupta, Gupta, and Kharat 2018). A similar result also proves that eggshells have a better adsorption capacity than activated carbon (Misfadhila et al., 2018).

Table 3 shows the estimated cost for treating adsorbents before contact with dyestuffs, excluding using reagents and other definite means with the same treatment to adsorb indigo carmine dye. We can see that adsorption using egg shells at an IC concentration of 50 ppm is very cheap. However, when compared with the adsorption of iC using egg white with crosslinker N, N'-Methylenebis (acrylamide) can adsorb anionic dyes (indigo carmine) with an adsorption capacity at an IC concentration of 50 ppm is 26.25 mg g<sup>-1</sup> (Oymak & Esra Bağda, 2018). Although the adsorption capacity value is quite high, the use of acrylamide is still dangerous and the costs used are high.

**Table 2.** Isotherm Model for IC adsorption by CESM

Isotherm	Parameter	CESM+IC
Langmuir	$k_L$ (L /mg)	0.0334
	$q_m$ (mg/g)	3.0497
	ALL	0.5631
	$R^2$	0.9474
Freundlich	$k_F$	0.0487
	N	0.6424
	$R^2$	0.9748

**Table 3.** Comparison of The Cost of Processing Adsorbent with Other Adsorbents

No.	Material (100 g)	price (SGD) in sigmaaldrich.com	price (IDR) raw material and processing services	adsorption capacity (mg/g) in 50 ppm dye solution	reference
1	1 kg activated carbon peanut shell	-	10,000 (estimation)	20.11	(Fatombi et al. 2019)
2	1 kg dried eggwhite as crosslinker		20,000	12.35	(Rahmiana Zein et al. 2022)
3	1 kg ketapang shell	-	10,000	5.46	(Hevira, Zilfa, et al. 2020)
4	1 kg g N,N'- methylenebis (acrylamide) as crosslinker	97,40	103,244 (10 % usage estimate)	26.25	(Oymak and Esra Bağda 2018)
5	1 kg eggshell and its membrane	-	2000	6.3963	This study

#### 4. CONCLUSION

From the research data analysis, it can be concluded that CESM (chicken eggshell and membrane) can adsorb indigo carmine dye. The absorption mechanism shows that the kinetic model is more suitable with the second pseudo model, where the adsorption capacity is proportional to the number of active sites of the adsorbent. Meanwhile, intra-particle diffusion was revealed to be the only one that limits the adsorption rate. The adsorption capacity of CESM was 6,3963 mg/g, while the adsorption isotherm follows the Freundlich isotherm model. It means that the adsorption takes place in a multilayer manner. From an economic point of view, CESM has the potential to be recommended as a dye adsorber in reducing textile industry waste because the cost is low, easy, and fast.

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