Activated Charcoal from Coffee Dregs Waste as an Alternative Biosorbent of Cu(II) and Ag(I)

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Abstract: *This study examines the use of coffee dregs waste as biosorbents of Cu(II) and Ag(I). Coffee dregs waste still contains a high level of carbon and cellulose for biosorbents production. The production process was started with charcoal activation using H_3PO_4. The batch method was applied by variations of contact time, the mass of the biosorbent, and the initial concentration of metal ions. The results showed that Cu(II) and Ag(I) were optimally adsorbed at pH 6 and 4, respectively. The amount of adsorbed metal ions increased with adsorption contact time. The adsorption process of both metal ions reaches stability within 60 min and the optimum biosorbent mass is 1 g. Isothermal adsorption studies show that Cu(II) adsorption tends to follow Langmuir isotherm with an adsorption energy of 31.42 kJ/mol and Ag(I) adsorption follows Freundlich isotherms with an adsorption energy of 27.74 kJ/mol. Based on the results, the interaction between metal ions and adsorbents is a chemical adsorption process and coffee dregs charcoal has the potential to adsorb Cu(II) and Ag(I) metal ions.*

Keywords: *Ag(I); biosorbent; coffee dregs; Cu(II); isothermal adsorption*

■ INTRODUCTION

Nowadays, the silver industry is one of the small industrial centers that continues to grow. Silver handicraft production's gilding, coating, and rinsing release heavy metals waste, including copper (Cu) and silver (Ag) [1]. In addition, Cu and Ag are heavy metals that are commonly used in many industries [2]. The toxic Cu and Ag ions are difficult to be identified because of their presence in cations in the water. Direct discharging of wastewater containing heavy metals into the infiltration channels, soil, or into the surrounding environment causes ecological damage because of metal degradation difficulties [3]. Also, these two metal ions pose a significant risk if contaminated in animals and humans because of their high toxicity at low concentrations [4].

The regulation of the Republic of Indonesia Minister of Environment Number 5 of 2014 concerning Wastewater Quality Standards states Cu and Ag in wastewater should not be more than 0.5 mg/L [5]. Therefore, proper handling is needed to prevent environmental pollution due to industrial wastewater disposal into the surrounding environment.

Several methods have been applied in handling metal wastewater, such as adsorption [6-8], membranes [9], photoreduction [10], precipitation [11], ion exchange [12], and solvent extraction [13]. Adsorption is a well-known method with advantages because of simplicity, low cost, good performance at low concentrations, and recyclability [14].

Materials commonly used as biosorbents are activated charcoal, alumina, silica, chitosan, and zeolite [15-17]. Among them, activated charcoal is the most accessible material to obtain and provides the largest surface area [18], so its ability as an adsorbent is also greater compared to mesoporous silica [17]. Activated charcoal is a porous solid containing 85–95% carbon produced from materials containing carbon by heating at high temperatures.

One material that is potentially to be further used is coffee dreg waste. Coffee dregs can be considered new waste because they create an unpleasant odor, especially

during the rainy season. Coffee dregs still have a moisture content of around 75–80%, which easily for spoilage microbes to grow [19]. Caetano et al. [20] reported that coffee grounds provided 47.8–58.9% carbon, 1.9–2.3% nitrogen, 0.4–1.6% ash, and 8.6% cellulose. Therefore, coffee grounds have the potential to become activated charcoal. So far, the use of coffee grounds has not been widely developed.

The adsorption process is affected by several factors, such as temperature, contact time, surface area of the adsorbent, pore structure of the adsorbent, and the pH of the solution. The interaction between the adsorbent and the adsorbate under different conditions provides different adsorption results. Therefore, the optimum condition investigation of each treatment is essential for the adsorption effectiveness [21]. In this study, the utilization of waste from coffee dregs as a biosorbent activated by H_3PO_4 to Cu(II) and Ag(I) was investigated, which are the main pollutant materials in Indonesian waters on variations in biosorbent mass and adsorption contact time. In addition, an isothermal adsorption study and an adsorption test for Ag(I) and Cu(II) in a binary solution system were carried out simultaneously. The activation process was carried out under acidic or alkaline conditions. In this study, the H_3PO_4 activator was used, which is a relatively good degrading agent because H_3PO_4 provides three H^+, which is necessary to degrade residual impurities left on the pore surface of the adsorbent.

■ EXPERIMENTAL SECTION

Materials

The materials used are coffee dregs obtained from coffee shops around Yogyakarta. Other materials include phosphate acid (H_3PO_4), copper(II) sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$), nitric acid (HNO_3), sodium hydroxide (NaOH), silver(I) sulfate (Ag_2SO_4), and other chemicals used in this study were the highest purity available from Merck and were used without further purification.

Instrumentation

A set of laboratory glassware, analytical balance (OHAUS), spatula, volumetric flask, hot plate, magnetic stirrer, oven, filter paper, Fourier transform infra-red (FTIR, Shimadzu Prestige-21), X-ray diffraction (XRD, Philips XRD X'Pert MS), scanning electron microscope (SEM, JSM-6510LA), and atomic absorption spectrophotometer (AAS, GBC Australia, at UGM Analytical Chemistry Laboratory) were used in this study.

Procedure

Preparation of H_3PO_4-activated coffee dregs charcoal

The coffee dregs were ground to increase the surface area, washed using distilled water to obtain a neutral pH, dried in an oven at 110 °C for 2 h, and carried out in a furnace at 400 °C for 3 h. The charcoal from the coffee dregs is then cooled, and the yield is calculated.

As much as 30 g of coffee dregs charcoal was immersed in 200 mL of H_3PO_4 1 M for 48 h. Then, it was filtered and washed using distilled water until neutral, dried at 110 °C for 90 min, and cooled in a desiccator for 30 min. Characterization by FTIR was applied before and after activation.

Contact time effect

As much as 10 mL of Cu(II) 10 mg/L was put into an Erlenmeyer, and 0.5 g of the developed biosorbent was added. Adjustment of pH 6 was applied to Cu(II) solution. The sample was stirred at room temperature for 30, 60, 90, and 120 min and then left for 5 min. The sample was then filtered using filter paper and analyzed using AAS. The same procedure was applied for Ag(I) adsorption using 50 mL of Ag_2SO_4 20 mg/L solution at pH 4.

Effect of mass variation of biosorbent

At pH 6, Cu(II) 10 mg/L were put into an Erlenmeyer and added H_3PO_4 1 M activated coffee dregs biosorbent in the amount of 0.5, 1.0, 2.0, and 3.0 g. The sample was stirred for 60 min at room temperature and then left for 5 min. Then, the sample was filtered using filter paper and analyzed employing AAS. The procedure was also applied for the adsorption of Ag(I) using 10 mL of Ag_2SO_4 20 mg/L solution at pH 4 in variations of the coffee dregs biosorbent, 0.25, 0.50, 0.75, and 1.00 g.

Metal ion concentration effect

A series of metal solutions were prepared, i.e., 4, 8, 12, 16, and 20 mg/L. Then, 0.5 g of the biosorbent was added to 50 mL of each solution and stirred at the optimum time. A blank experiment was also carried out under similar conditions. SSA analyzed the concentration of adsorbed metal.

Simultaneous adsorption study of Ag(I) and Cu(II) metals with coffee dregs biosorbent

As much as 0.5 mg of activated biosorbent H_3PO_4 1 M interacted with a mixed solution of Ag(I):Cu(II) (mol ratio of 1:1). The sample was stirred for 60 min at room temperature and left for 5 min. The sample was then filtered using filter paper and analyzed using AAS.

■ RESULTS AND DISCUSSION

Characterization of Coffee Dregs Activated Charcoal

Synthesis of activated charcoal started with the preparation of coffee dregs, then the furnace process or charcoal manufacture, and the charcoal activation using acid. Carbonization from coffee dregs was carried out at a temperature of 400 °C for 3 h, aiming to decompose the material and produce a material with absorption and a neat structure [22]. The next step was the activation of the coffee dregs charcoal using the H_3PO_4 as an activator to open the carbon pores covered by impurities trapped in the pores. Fig. 1 shows the mechanism of the activation reaction of charcoal with H_3PO_4. Fig. 1 shows that the H_3PO_4 as activator reacts with the available charcoal and then forms micropores on the surface, serving as a host of adsorption processes. The opening of the pores and increasing the surface area of activated charcoal can increase the ability of activated charcoal to adsorb [24].

The characterization of acid-activated coffee dregs and coffee dregs was carried out using an FTIR spectrophotometer. Fig. 2 shows the spectra of coffee dregs before and after activation with H_3PO_4, and Table 1 shows the interpretation of the spectra.

Table 1 shows that the two FTIR spectra have almost the same absorption of functional groups. A few shifting occurred at 1570 to 1573 cm^{-1} for the absorption of aromatic C=C groups. Also, the absorption of the C–H

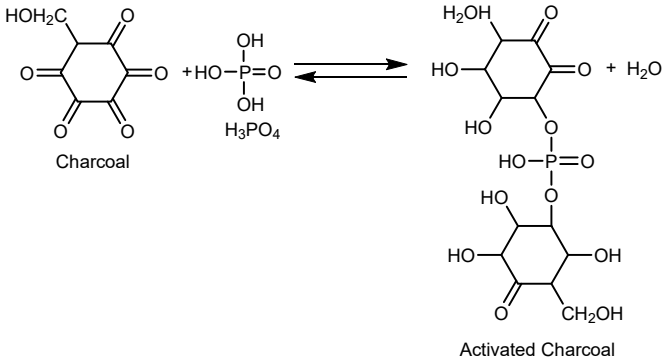

Fig 1. Charcoal activation reaction with H_3PO_4 [23]

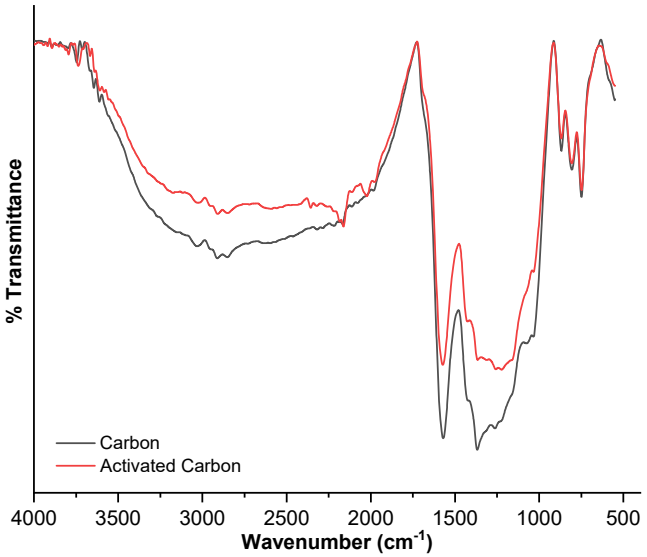

Fig 2. FTIR spectra of coffee dregs charcoal

functional group at wave number 2911 shifted to 2909 cm^{-1}. The shifting occurred because the pores in the coffee dregs charcoal became more open due to the loss of impurities after the activation process with acid. The spectra of H_3PO_4-activated coffee dregs activated charcoal showed a characteristic absorption at 1223 cm^{-1} as P=O absorption of H_3PO_4 [25].

Fig. 3 shows the morphology of H_3PO_4-activated coffee dregs charcoal using SEM. The morphology of H_3PO_4-activated coffee dregs charcoal becomes more open, so it is expected to be more effective in adsorbing heavy metal ions. In line with a study conducted by Purwiandono et al. [26], which showed that the ability of an acid-activated zalacca peel biosorbent to adsorb Cu(II) was much better than biosorbents without activation. The results of this study are also in line with

Table 1. FTIR spectra interpretation results of coffee dregs charcoal

Wavenumber (cm^{-1})		Functional group interpretation
Coffee dreds	Activated coffee dregs by H_3PO_4	
2911	2909	C-H (Csp^3)
1570	1573	C=C aromatic
-	1223	P=O of H_3PO_4

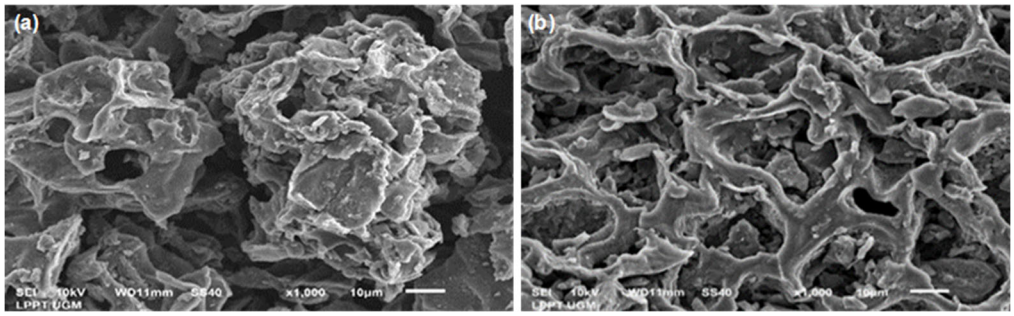

Fig 3. SEM photo of coffee dregs charcoal (a) before and (b) after activation process at magnification 1000×

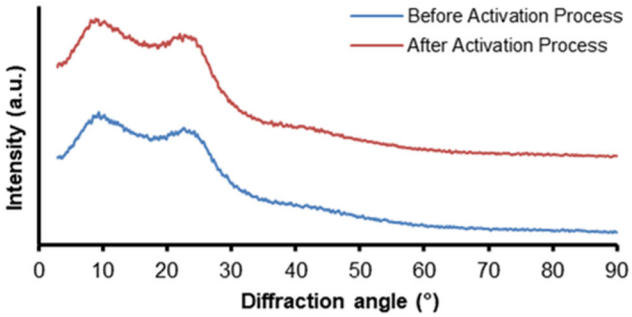

Fig 4. Diffractogram (XRD) of coffee dregs charcoal before and after activation with H_3PO_4

the results obtained by previous researchers [22]. It is because the activation treatment was able to release impurities that partially cover the biosorbent's pores, so the pores become larger to adsorb metal ions.

Further characterization was carried out using XRD. The diffractogram of coffee dregs charcoal before and after activation with acid is shown in Fig. 4. The diffractogram of coffee dregs charcoal before and after activation performed insignificant differences in intensity and diffraction patterns. It proves that the activation process did not change the charcoal structure, which has an amorphous structure. This result is in line with previous research [27]. The charcoal peaks at an angle of 2θ around 20° and 40° show typical graphite characteristics identified as basal spacing d_{002} and d_{100}, respectively [18]. A shift of the peak to a smaller angle from 23.32° to 22.88° was identified in activated charcoal with basal spacing values of 0.38 and 0.39 nm, respectively. It indicates that the charcoal structure after activation is more open than before activation, which is in line with the results of SEM and FTIR analysis. Peaks at an angle of 2θ around 9° on both diffractograms indicate the presence of an amorphous cellulose structure in the activated charcoal [18].

Adsorption of Cu(II) and Ag(I)

Effect of contact time

Metal adsorption by coffee dregs biosorbent was carried out isothermally at a constant temperature of 25 °C. Fig. 5 shows that the increased contact time helped the amount of adsorbed metal ions, and the adsorption process reached an equilibrium. After 60 min of stirring,

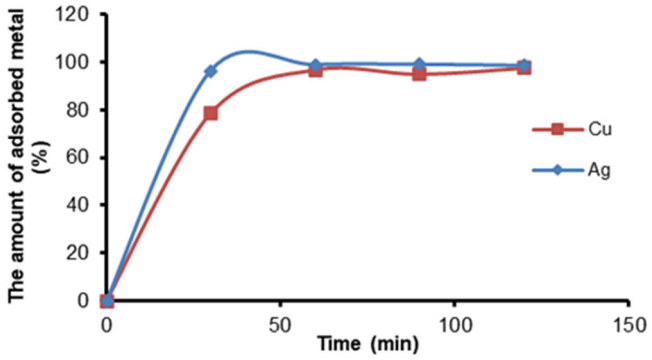

Fig 5. Effect of contact time on Cu(II) and Ag(I) adsorption

the amount of adsorbed metal ions relatively did not increase. It can be concluded that the equilibrium occurred after a stirring time of 60 min.

Effect of mass variation of biosorbent

The adsorption process on the mass variation of the biosorbent was carried out at room temperature with an adsorption contact time of 60 min, as the results indicated that the adsorption equilibrium occurred after a stirring time of 60 min. Fig. 6 shows the results on the mass variation of this biosorbent.

Fig. 6 shows that the increase in adsorbed metal ions is directly proportional to the amount of biosorbent used. The more biosorbent used, the more pores available and the wider surface area, affecting higher interaction between the active side of the pores of the coffee dregs activated charcoal and metal ions. Thus, the number of adsorbed metal ions increases [28]. The results show that the maximum absorption of Cu(II) and Ag(I) occurred using 1 g of coffee dregs biosorbent. After reaching equilibrium, the ability of the adsorbate to bind to the adsorbent decreases. Fig. 6 shows a decrease in metal ions adsorbed on biosorbents above 1 g. Because the amount of metal ions in solution is not proportional to the number of biosorbent particles available, the surface of the biosorbent has reached its saturation point and the absorption efficiency has decreased.

Adsorption Isotherms of Cu(II) and Ag(I)

This research investigated the adsorption isotherms by calculating for each treatment according to both Langmuir and Freundlich adsorption isotherms. The Langmuir isothermal adsorption equation is $1/q_e = 1/(X_m.K.C_e) + 1/X_m$ and the Freundlich equation is $\log q_e = \log k + 1/n \log C_e$ with q_e as the concentration of the adsorbate at equilibrium (mol/g), C_e is the adsorbate concentration in the aqueous phase (mol/L), X_m is the maximum adsorption capacity (μmol/g), and k is the equilibrium constant. Parameters obtained from Langmuir and Freundlich isothermal analysis are presented in Table 2.

Table 2 shows that Cu(II) provided a higher level of isothermal graphic linearity than the Langmuir model compared to the Freundlich model. The opposite occurred for Ag(I). The adsorption process for Cu(II) metal ions tends to follow the Langmuir isothermal model. The adsorption process occurred in a monolayer, assuming that maximum adsorption occurred when the adsorbate fills all the active sites of the adsorbent to form a monolayer layer. Meanwhile, the Ag(I) metal ion tends to follow the Freundlich isothermal model. The adsorption process occurred in the multilayer sheet.

The adsorption energy equation can be written as $E_{ads} = -\Delta G°$. The value of ΔG can be measured in the standard state, while for any other state, the value of the Gibbs free energy (ΔG) is: $\Delta G = \Delta G° + RT \ln K$, as R is the general gas constant (8.314 J/K mol), T is the temperature (K), and K is the adsorption equilibrium value. Huang et al. [29] state that the minimum limit for chemical adsorption energy is 5 kcal/mol or 20 kJ/mol.

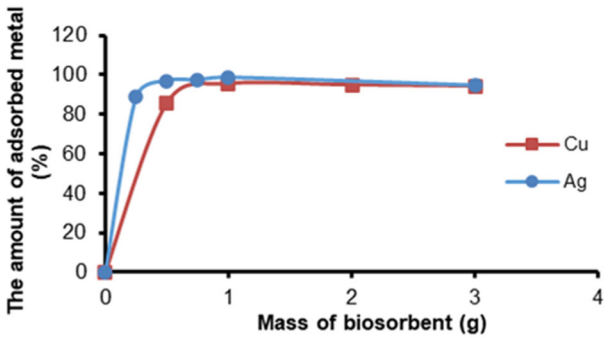

Fig 6. The effect of the mass of the biosorbent on the adsorption of Cu(II) and Ag(I)

Table 2. Adsorption isothermal parameters determined from the Langmuir and Freundlich equations

Ion	Adsorption parameter						
	Langmuir				Freundlich		
	X_m (mg/g)	$K \times 10^6$ (L/mol)	ΔG (kJ/mol)	R^2	ΔG (kJ/mol)	K (L/mol)	R^2
Cu(II)	0.4136	1.0270	31.420	0.9720			0.9007
Ag(I)		0.1949		0.0901	27.740	3.3962	0.9219

The adsorption energy value of the two metal ions is above 20 kJ/mol, so the interaction between the metal ion and the coffee dregs biosorbent can be seen as a chemical adsorption process. Coffee dregs provide active sites in the form of hydroxy groups (–OH) from cellulose. These active sites can interact with metal ions. It is understood that these OH groups can donate electron pairs to metal ions, forming coordination. This can be enhanced by activating the coffee dregs charcoal so that the pores of the charcoal become more open, making it possible to trap more metal ions effectively. An illustration of the interaction model between metals and biosorbents is shown in Fig. 7 [30].

This study shows the amount of adsorbed metal increased with the increasing number of biosorbents used and the results of adsorption investigation in a solution consisting of both metal ions. It was seen that the coffee dreg biosorbent tended to absorb more Ag(I) compared to Cu(II). The ability of biosorbents to adsorb multi-metals is also essential to determine the level of interference from the presence of co-cations in wastewater and the efficiency of biosorbents in removing these ions from wastewater [31].

This study also investigated the adsorption of Ag(I) and Cu(II) simultaneously in the binary solution system, detailed in Fig. 8. Fig. 8 shows that more Ag(I) was adsorbed by the coffee dregs biosorbent than Cu(II) even though the adsorption process was carried out simultaneously. It means that metal ions interact with each other synergistically. The interaction between metal ions and biosorbents from coffee dregs also occurred physically, where metal ions were trapped in the pores of the activated charcoal. Thus, this adsorbent does not show a high selectivity towards Ag and Cu ions compared to the composite adsorbents reported in previous studies [32]. The suitability of the size of the metal ion with the pore size of the biosorbent is one of the factors affecting the adsorption process [33]. The Ag(I) size is more suitable with the pore size of the coffee dregs charcoal, affecting more abundant ions Ag(I) could be absorbed.

This study shows that the H_3PO_4-activated coffee grounds biosorbent can potentially adsorb Cu(II) and Ag(I) metal ions. Compared to previous studies using

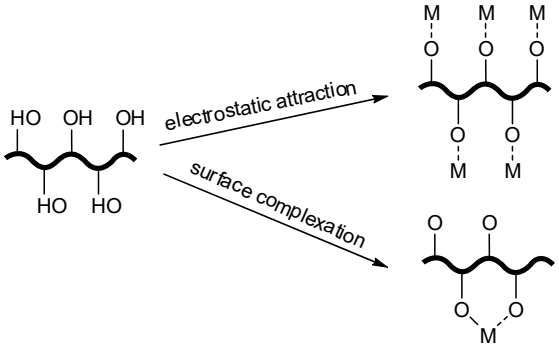

Fig 7. Model of interaction between metals and active groups from biosorbents

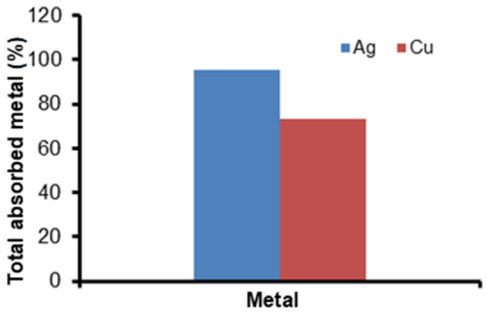

Fig 8. Amount of adsorbed metal in binary solution system

biosorbent from cassava peel [34], coffee dregs biosorbent can absorb Cu(II) metal ions at lower concentrations. Compared to research conducted by Mariana et al. [22], the adsorption process of this study requires a shorter adsorption time. The results of this study are also in line with the results obtained by previous researchers [35].

■ CONCLUSION

The study showed that coffee dregs charcoal activated with H_3PO_4 could be applied as a biosorbent for Cu(II) and Ag(I). The optimal adsorption contact time was 60 min. Adsorption isothermal investigation shows that Cu(II) tends to follow the Langmuir isotherm with adsorption energies of 31.42 kJ/mol. In contrast, Ag(I) tends to follow the Freundlich isotherm with adsorption energies of 27.74 kJ/mol. The adsorption energy of coffee dregs charcoal biosorbent is more than 20 kJ/mol. This means that the interaction between metal ions and the biosorbent can be viewed as a chemical adsorption process. It was observed that coffee

dregs charcoal was a more effective biosorbent for the removal of Ag(I) than Cu(II). The functional groups of cellulose charcoal play an important role in adsorption abilities. In general, coffee dregs charcoal biosorbent can be regarded as a potential biosorbent in heavy metal wastewater treatment applications.

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■ AUTHOR CONTRIBUTIONS

Susy Yunita Prabawati: Conceptualization, methodology, resources, formal analysis, writing–original draft, writing–review & editing, supervision, project administration, funding acquisition. Priyagung Dhemi Widiakongko: Formal analysis and editing. Mohammad Ahsani Taqwim: Methodology, formal analysis, investigation, visualization, and editing.

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