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Remove organic matter and iron substances in water: the combination of eggshells using stirring and heating methods

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ABSTRACT

Chicken eggshell powder is a waste that contains $CaCO_3$ elements and is porous. The use of chicken eggshell waste combined with stirring as an alternative method reduces organic and Fe pollution in water. The presence of household waste and garbage can affect the quality of well water used by the community as a source of clean water. Leachate seepage can increase the organic matter and Fe content of well water used by the community as clean water. The use of chicken eggshell powder in research is intended as a natural medium to reduce levels of organic substances and Fe in water. Chicken eggshell powder is calcined to a temperature of 500°C for 5 h. Water containing organic substances and Fe was placed in the vessel of the stirrer chamber and given chicken eggshell powder as much as 10, 20, and 30 g, respectively and 0 g as a control. The heating temperature is 35°C with a stirring speed of 150 rpm, and the stirring time is 15 min. The results showed a decrease in organic matter of 80.6% and iron content of 74.4% with stirring intervention of 150 rpm, heating at 35°C, and eggshell mass of 30 g. It was concluded that chicken eggshell powder can reduce organic matter and iron levels through stirring and heating. The results of this study can be a solution to reducing the generation of chicken eggshell waste in water treatment technology to reduce organic substances and iron levels.

Keywords: Chicken eggshell; Adsorbent; Organic substance; Fe

1. Introduction

Well water is the main source of providing clean water in the Island of Java, Indonesia [1,2]. The declining quality of well water is an important concern in overcoming the crisis of clean water sources in the Java Islands, Indonesia [3,4]. Disposal of garbage that produces leachate can cause health problems in providing clean water. The community uses well water in addition to bathing and washing, it is also used as a source of drinking water and processing food. Leachate water absorption will increase organic matter and Fe contained in well water [5] The increased content of organic and Fe substances will cause changes in color, smell, taste, and pathogenic bacteria [6–8].

Some clean water treatment method that is able to bind particles contained in the water are by using the coagulant method. A coagulant is a method that is able to neutralize colloidal charges and bind particles. [9] On the other hand, common coagulants used are chemicals that have a negative impact on health and the environment. In addition, Saswita et al. [10] research revealed the fact that coagulation methods using CaO derived from quicklime did not have a significant effect on reducing Mn pollutants in water.

Adsorption is another method used as a medium in simple water treatment technology. In addition to raw

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materials easily obtained, the natural properties of materials have a positive impact on environmental sustainability. On the other hand, adsorption technology is widely applied on the grounds that it is cheap and efficient [11]. The adsorption method is one of the physical methods. Chicken eggshells are one of the wastes produced every day and have the potential to be used as adsorbents. Chicken eggshells have important components as adsorbents due to their $CaCO_3$ content and are porous. The chemical composition of chicken eggshells is reported to consist of calcium carbonate (94%), magnesium carbonate (1%), calcium phosphate (1%), and organic compounds (4%) [28]. The use of chicken eggshells as a natural adsorbent has been carried out by researchers from 2017 to 2021 to minimize contaminants in blood shells and used cooking oil. This study tested the potential of chicken eggshells in reducing heavy metals Hg, Cd, and Pb of blood mussels as well as the amount of peroxide and free fatty acids in used cooking oil. The results showed that chicken eggshells can reduce Hg, Cd, and Pb *Anadara granosa* significantly as adsorbents through a physical activation process by heating and chemical soaking using HCl solution [12,13].

Chicken eggshells can be used as catalysts to convert calcium carbonate into calcium oxide through shell burning which is a calcining process [14]. Calcined eggshells undergo a change in functional groups from calcium carbonate to calcium oxide [15]. Another study showed that calcined calcium oxide (CaO) reduced phosphate from water [16]. The presence of carbon and oxygen in chicken eggshells is one of the alternative adsorbents in reducing heavy metal levels and color in water [17]. The potential of chicken eggshells is thought to be able to minimize organic matter and Fe in dug well water. The study aims to analyze the potential of chicken eggshells to reduce organic and iron levels in well water through a stirrer chamber.

2. Method

2.1. Design study

Experimental research on a laboratory scale to determine the decrease in organic and Fe substances in water using eggshell powder as an adsorbent medium. The research was conducted at the Environmental Health Laboratory, Poltekkes Kemenkes Surabaya, Indonesia. The sample was divided into 4 groups with three replications. Each group was given adsorbent variation of chicken eggshell powder, namely, 10, 20, 30, and 0 g as controls. Tools used in the study include a stirrer chamber unit, a 1-L glass beaker, a 250 mL cylindrical measuring cup, a porcelain pestle and mortar, a 100 mesh and 120 mesh sieve, a glass funnel, a sample bottle, an analytical scale, and an ice box. The materials used in the experiment consisted of chicken eggshells, 0.1 M HCl, aquades and aquabides.

X-ray diffraction (XRD) was recorded using the PANalytical's *X'*Pert PRO diffractometer (Indonesia) with a tube voltage of 30 kV and a current of 40 Å.

The modified stirrer chamber unit consists of microcontroller module components, dry elements, stainless body, motor gear box, stirring iron, hinges, solid state relay, temperature sensor, timer, Liquid Crystal Display (LCD), temperature sensor circuit, and connector cable. The design of the stirrer chamber tool is shown in Fig. 1.

2.1.1. Working principle of the tool

Using heat-conducting elements through temperature control buttons. The stirring speed of the tool is set at 150 rpm. The first step is to put the ingredients to be processed into the pot. After that, position the stirrer into the pan. Press the power button after setting the time, temperature 35°C and speed 150 rpm. If the temperature and speed are appropriate, press the start button to start the processing process. Wait a few minutes and the process will stop on its own automatically. After that turn off the power.

2.1.2. Method

This work was carried out by a pre-posttest control group design and described the process of adsorbent production in detail.

2.2. Adsorbent manufacturing

1. Washing chicken eggshells thoroughly.
2. Soak in hot water, then cool.

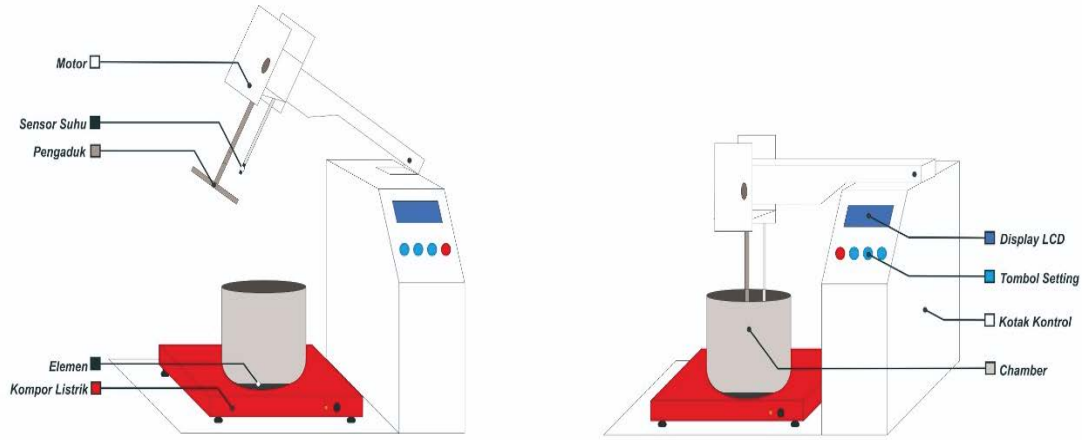

Fig. 1. Modified model of the tool "stirrer chamber".

3. Remove the membrane layer of chicken eggshells. Rinse the eggshell shell with aquades followed by rinsing with aquabides until clean.
4. Rinsed chicken eggshells, then drain using a container lined with absorbent paper.
5. Shell that has been drained, then put it in the oven to dry at 105°C for 2 h.
6. Chicken eggshells that have gone through the drying stage, are mashed using mortal pestle porcelain to obtain 100 to 120 mesh particle size eggshell powder.

Activate chicken eggshell powder by soaking in 0.1 M HCl solution for 48 h, then drained, filtered and washed with aquades and aquabides until the pH is neutral (pH = 7). After the pH is neutral, the chicken eggshell powder is calcined to a temperature of 500°C for 5 h (Fig. 2) [18].

2.3. Adsorption process

Adsorbents of 10, 20 and 30 g each treatment (stirring time 15 min before and after heating temperature 35°C and 2 replications) put into 200 mL of water sample. Activates the stirrer chamber by adjusting the temperature, speed and stir time. The samples were analyzed by permanganometric methods for organic substance analysis, and spectrophotometric methods for iron (Fe) levels.

2.3.1. Permanganometric method [19]

Permanganometry is one of the titration methods that uses the principles of reaction, reduction, and oxidation. This permanganometric uses $KMnO_4$ solution as a titrant. Permanganometry is commonly used to determine the content of iron(II). Permanganate ions will undergo reduction to produce Mn^{2+} while iron(II) will undergo oxidation to produce iron(III). The following is the oxidation–reduction reaction during titration. $MnO_{4\text{-}(aq)} + 8H^{+}_{(aq)} + 5Fe^{2+}_{(aq)} \rightarrow Mn^{2+}_{(aq)} + 4H_2O_{(l)} + 5Fe^{3+}$.

2.3.2. Spectrophotometric method [20,21]

Spectrophotometric techniques involve ligands that can selectively combine with iron (a particular redox state) to form a colored complex with high molar absorptivity. The initial ligands that were used for iron determination were 2,2′,2″-tripyridyl, 2,2′-bipyridyl, and thiocyanate. Total dissolved iron was determined after the reduction of Fe(III) and these ligands were used to bind Fe(II). Later, more selective and sensitive ligands for iron complex, such as 1,10-phenanthroline, 2,4,6-tripyridyl-1,3,5-triazine, and bathophenanthroline, were used for iron spectrophotometric determination.

2.4. Data analysis

Laboratory test data on organic substances and Fe levels based on eggshell powder mass, stirring speed and heating temperature are processed, made in table form and then explained descriptively. The data were analyzed using statistics to determine the difference in values between treatments, so that the value of the adsorption capacity of chicken eggshells from several variant groups could be determined using the Two-Way ANOVA test through the SPSS Statistics 20 system.

The X-ray diffraction method is used to determine the characteristics of activated chicken eggshell powder particles. The resulting diffraction yield pattern is matched with the pure diffraction pattern of the Joint Committee on Powder Diffraction Standards (JCPDS). The XRD test results were analyzed using the MATCH application to provide a descriptive analysis of the particle structure of the chicken eggshell powder used.

2.5. Research results

Table 1 explains that unstirred treatment (P_0) yields an average of organic substances, and Fe is higher than stirring treatment (P_1). Testing the difference between organic substances and Fe of dug well water before and after stirring intervention produces test statistics with significance values of > alpha (5% or 0.05), so that H0 is accepted. Therefore, it can be stated that there is no significant difference in organic substances, and Fe water dug wells before and after stirring intervention.

Table 2 shows that unheated treatment (T_0) yields an average of organic substances, and higher Fe than treatment with 35°C heating (T_1). Based on the average value that after the heating intervention produced the average value of organic substances, and the Fe of dug well water

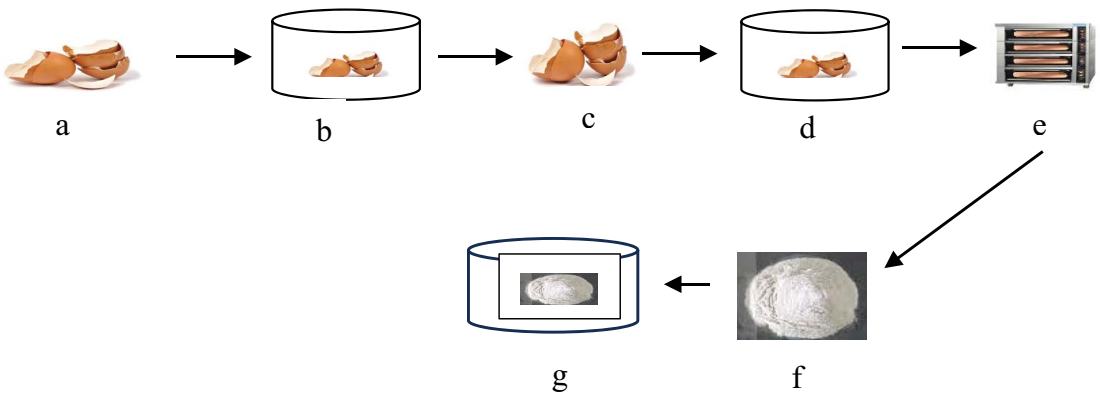

Fig. 2. Pembuatan adsorbent.

Table 1
Levels of organic substance, Fe, based on stirring

Variable	Stirring	*N*	Mean	Std. deviation	Minimum	Maximum
Organic matter	P_0	24	31.2754	12.15229	9.76	45.06
	P_1	24	30.3163	12.28347	8.61	44.68
Fe	P_0	24	3.1792	1.03961	1.13	4.50
	P_1	24	3.0904	1.10653	1.06	4.81

Table 2
Levels of organic substances, and Fe based on heating

Variable	Temp.	*N*	Mean	Std. deviation	Minimum	Maximum
Organic matter	T_0	24	39.5704	5.11968	31.39	45.06
	T_1	24	22.0213	10.59004	8.61	38.04
Fe	T_0	24	3.7079	0.80399	2.57	4.81
	T_1	24	2.5617	0.98864	1.06	3.68

Table 3
Organic substance levels, and Fe-based on adsorbent mass addition

Variable	Mass	*N*	Mean	Std. deviation	Minimum	Maximum
Organic matter	W_0	12	40.7942	3.86057	36.47	45.06
	W_1	12	34.1717	9.59671	24.61	44.11
	W_2	12	27.6767	11.49583	15.88	40.07
	W_3	12	20.5408	11.80204	8.61	32.23
Fe	W_0	12	3.9992	0.61573	3.24	4.81
	W_1	12	3.8883	0.50776	3.16	4.50
	W_2	12	2.7458	0.51992	2.16	3.34
	W_3	12	1.9058	0.82105	1.06	2.93

was lower than before the heating intervention showed that heating effectively reduced organic substances, and the Fe of dug well water.

Table 3 shows that treatment without the addition of chicken eggshell powder mass (W_0) produces an average of organic substances, and Fe is higher than treatment with addition of chicken eggshell powder mass. The lowest levels of organic substances and Fe in the addition of chicken eggshell powder mass 30 g (W_3).

Based on the interaction of 150 rpm stirring treatment for 15 min, heating 35°C and adding variations in chicken eggshell mass obtained results as shown in Fig. 1.

Testing differences in organic substances, and Fe of dug well water before and after the intervention of chicken eggshell mass produced test statistics with a significance value of < alpha (5% or 0.05), so that H_0 was rejected. It is stated that there are significant differences based on the mass of chicken eggshell powder, stirring and heating the content of organic substances, and Fe in water.

Based on Table 3, it can be seen that treatment without the addition of chicken eggshell powder mass (W_0) produces an average of organic substances, and Fe is higher than treatment with the addition of chicken eggshell powder mass. The lowest levels of organic substances and Fe in the addition of chicken eggshell powder mass 30 g (W_3).

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2.6. Characterization of chicken eggshell particles

The XRD test results of chicken eggshell powder before activation can be shown in Fig. 3.

Fig. 3 shows the crystal structure of calcium carbonate calcite with a crystal system formed in the form of trigonal (hexagonal axes) with a lattice constant value a = 4.99150 Å c = 17.08800 Å. Orientation of the diffraction plane (*hkl*) at an angle of 2θ = 29.4522° with crystal size = 2,632.3 nm.

The XRD pattern of chicken eggshell powder after activation can be shown in Fig. 4.

Fig. 4 shows that there is a crystal structure of calcite with a crystal system formed in the form of trigonal (hexagonal axes) with a lattice constant value of a = 4.98440 Å,

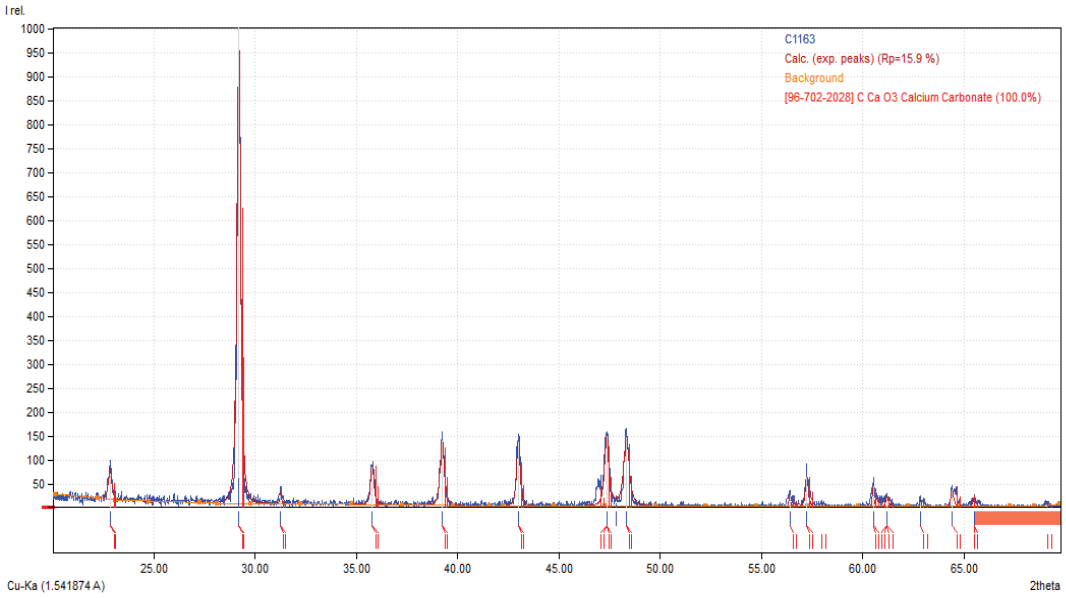

Fig. 3. X-ray diffraction pattern graph of chicken eggshell adsorbent sample before activation (C1163).

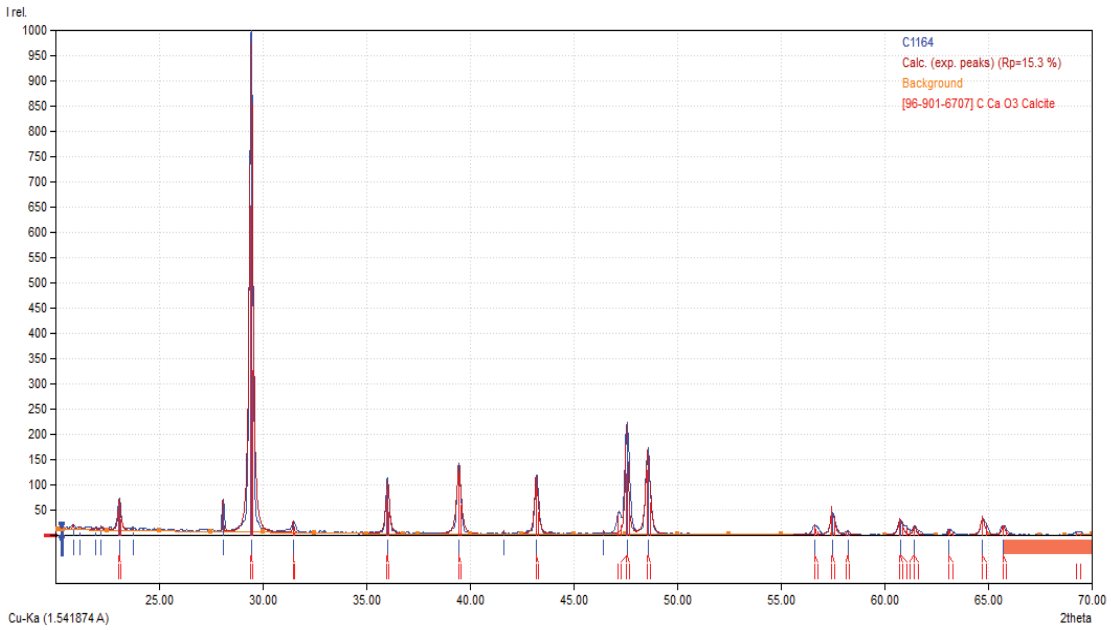

Fig. 4. X-ray diffraction pattern graph of chicken eggshell powder after activation (C1164).

c = 17.03760 Å. Orientation of the diffraction plane (*hkl*) at an angle of 2θ = 29.4691° with crystal size = 69343.7 nm.

The peak pattern produced in Figs. 3 and 4 is a series of diffraction peaks with relative intensity varying along the value of 2θ. Both of these patterns indicate the formation of trigonal crystal structures. The peak growth with the highest intensity is shown in Fig. 4, namely chicken eggshell powder has been activated. Shell powder that has not gone through the activation process, contains the more dominant element calcium carbonate, but after going through activation, calcium carbonate is converted into CaO elements.

3. Discussion

Organic substances are complex and varied mixtures of aromatic and aliphatic hydrocarbons with functional groups such as amides, carboxyls, hydroxyl and ketones and others in smaller quantities. These organic compounds come from natural sources such as soil or compost or from sanitary landfill generation. Dissolved organic matter can migrate to water from landfills or biosolids to soil (farmland, forests and grasslands [22]. The presence of organic substance levels is positively correlated with the Fe content in water in an aquatic environment [23]. In waters, high levels

of iron (Fe^{2+}) correlate with high organic levels or high levels of iron found in water derived from deep groundwater.

An increase in organic substances can be indicated by a low level of acidity. Research by Novianti et al. [24] shows a decrease in the pH value of water caused by an increase in organic substances. This increase in organic substances is known from the treatment group without the addition of eggshells as filter media. Organic substances in the form of humus acid come from the decomposition of organic matter such as leaves, trees or wood. The treatment group given a chicken eggshell filter showed a higher pH increase. This study shows that the addition of chicken eggshells has the potential to raise the pH which can reduce organic substances. Chicken eggshells contain 98.41% $CaCO_3$ when dissolved in water, will cause calcium carbonate to decompose. Research Novianti et al. [24], the decomposition of calcium carbonate is proven when reacted with water will release hydroxide ions (OH^-), this makes the number of hydroxide ions in water more and more. The increase in hydroxide ions in the water, making peat water alkaline. This reaction equation is in line with the acid–base theory derived from the Arrhenius theory. Arrhenius' theory explains that bases are compounds that release hydroxide ions in water so that peat water which was originally acidic after being reacted with $CaCO_3$ produces alkaline compounds, namely $Ca(OH)_2$ and makes the pH of peat water increase. Therefore, by turning chicken eggshell granules into filter media can increase the pH of water in peat water [24].

The presence of Fe metal in well water can have an impact. Iron metal (Fe) is an essential heavy metal whose presence in certain amounts is needed by living organisms, but if the amount of heavy metal enters the body in excess amount, it will change its function to be toxic to the body. Levels of organic substances in waters are sources of organic matter derived naturally such as from soil or from atrophic sources derived from composting processes (biosolids) and from seepage from landfills [22]. Organic substances are substances that are generally part of animals or plants with the main components being carbon, protein, and fat. Organic substances are easily decayed by bacteria by using dissolved oxygen [25].

The heating treatment, stirring and mass material of chicken eggshell powder contributes to a decrease in organic substances, and iron, heating or activating the adsorbent will increase the absorption of adsorbents against adsorbate causing the pores of the adsorbent to be more open, heating that is too high causes damage to the adsorbent so that its absorption ability decreases. Al-Essa and Khalili [26] explain that the influence of temperature on absorption is very important not only because it affects the absorption rate, but also because of the fact that the absorption temperature provides information about possible interactions between adsorbents. His research evaluated the absorption of Pb(II) isotherms into both modified kaolinites at 25°C, 35°C and 45°C, with specific capacities. The results of the study of the effect of Pb absorption temperature in this study show that the absorption/adsorption process increases with an increase in temperature, so that this absorption process is endothermic. This condition is also thought to occur in the adsorption process of eggshell powder to organic zar, and iron content.

The use of stirring speeds above 90 rpm will make the bond between adsorbent and adsorbate particles detached. In addition, too fast stirring makes the adsorbent not have time to form a strong bond with the particles. As a result, only a small amount of Fe is able to be absorbed. It can be said that the speed of 90 rpm is the effective stirring speed for Fe adsorption [27]. The study showed that the stirring speed had a better effect on the adsorption process of the heavy metal Fe compared to the length of the stirring time.

The use of chicken eggshells as an adsorbent is one of the methods used in minimizing heavy metal pollutants. Research by Narwati and Suryono [12] explained the potential of chicken eggshell powder as an adsorbent to reduce blood clam Cd levels by 82.1% using 50 g adsorbent stirring treatment speed 250 rpm 65°C. These results confirm that chicken eggshells can be used as an environmentally friendly and inexpensive medium for adsorbent heavy metal pollutants Cd. The potential of chicken eggshell powder is thought to be due to the calcination process of 500°C within 5 h of chicken eggshell powder. The capacity of shell powder in removing heavy metal levels is also stated in Faridi and Arabhosseini [28] research, with the higher mass of calcined chicken eggshell powder has the ability to minimize pollutants in making biodiesel from used cooking oil through the transesterification process.

The research of Ajala et al. [17] through the Fourier-transform infrared spectroscopy (FTIR) analysis method resulted in the observation of chicken eggshell patterns, namely the O–H/N–H bond showed weak stretching vibrations while the aliphatic C–H alkane bond from the eggshell was in accordance with the carbonyl stretching of the amide group. Other groups observed are alkane and alkene groups. The presence of various clusters on the eggshell of this chicken makes it a potential adsorbent.

Research by Supriyanto et al. [15] observed the characterization of eggshells after the calcination process indicating a change in structure between the original sample and samples that had gone through the calcination process. FTIR results show changes in calcium carbonate functional groups to calcium oxide due to the calcination process. It was explained again that the scanning electron microscopy picture in uncalcined samples has a variable particle size due to agglomeration at the time of precipitation after the milling process. However, in a calcined sample, the particles look more homogeneous with an even particle size and result in irregular crystal structures into interconnected structures. Calcined samples have more porosity than non-calcined samples due to the release of CO_2 during the calcination process [15].

Shell waste consisting of calcium carbonate ($CaCO_3$) accounts for 94% of the total mass. Commercial nano-$CaCO_3$ used leads to abundant mesopores with acid leaching. The research of Shi et al. [29] found that the surface microporous volume of nano calcium carbonate is comparable to the volume of C-800 micropores (carbon with a physical activation temperature of 800°C), which is 0.23–0.31 vs. 0.23 cm^3/g. This shows that cheap and environmentally friendly eggshell waste can be used as an agent that can be activated as porous carbon that is more economical and ecological. On the other hand, in addition to $CaCO_3$, eggshell

waste also contains some organic components such as collagen and glucosamine. During carbonization, this organic material can help incorporate oxygen, nitrogen, and other heteroatoms into the final carbon, forming a beneficial surface function for adsorption. Shi et al.'s [29] research indicates that while $CaCO_3$ plays a major role for pore formation in carbon, carbon shows its potential to act as an adsorbent in removing various contaminants from both aqueous and non-aqueous solutions.

The difference in levels of organic substances, and Fe using HCl-activated chicken eggshell powder provides better adsorption ability than those that are not activated. Bungas' [30] research resulted in the adsorption ability of an HCl-activated adsorbent to have the ability to absorb Fe and better color with a mass of 10 g compared to 5 g. Better adsorption ability based on adsorbent mass because the greater the mass of the adsorbent has implications for the greater the surface area of the adsorbent pores, resulting in greater adsorption power against Fe and color. The ability of the shell to adsorb pollutants of organic compounds, and Fe is influenced by physical activity in the form of heat treatment. Ajala et al. [17] explained that through activation treatment with heat treatment, it can increase the microporous area of eggshells. This increased expansion of eggshell micropores can be used in the adsorption process.

The results of X-ray diffraction analysis showed chicken eggshell powder showed the formation of calcium oxide after the calcination process. The characteristics of eggshell powder through the X-ray diffraction method are also explained by Ajala et al. [17] that eggshells through the energy dispersion X-ray method obtained 51.40% carbon elements, 39.94% oxygen, 0.2% chlorine and 8.46% calcium. This high carbon content in eggshells and little oxygen content is the potential for eggshells to become efficient adsorbents. Wong and Ang [31] further explain that calcium oxide peaks appear with high crystallinity and exhibit calcium hydroxide components prior to calcination. However, calcium hydroxide is not completely converted to calcium oxide under calcination at 800°C. This indicates that if a calcining temperature of less than 800°C is used, calcium oxide cannot be fully formed completely from changes in calcium hydroxide. Based on this, it can be said that the transformation of calcium oxide requires a longer calcination time.

4. Conclusion

The stirring treatment, heating and addition to the powder mass of chicken eggshells contribute to significant differences in the levels of organic substances, and Fe, before treatment. Chicken eggshell powder can be used by the public in minimizing organic substances and Fe water as one of the adsorbent materials.

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