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Characterization charcoal and activated carbon from starry triggerfish (*Abalistes stellaris*) bone activated using zinc chloride to reduce Cr^{6+}

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ABSTRACT

Starry triggerfish bone is the leftovers of fish products, and the utilization as carbon active is still minimal. Fish bones are organic waste containing carbon; therefore, they could be utilized as adsorbents (charcoal and activated carbon). This study aims to determine the characteristics of charcoal and activated carbon produced from starry triggerfish bone. The study about making charcoal and carbon active from starry triggerfish bone waste has been conducted using combined physics and chemical activation. physics activation is conducted at temperatures of 500°C, 600°C, 700°C, 800°C (CFB-500; CFB-600, CFB-700 and CFB-800), then the chemical activation using $ZnCl_2$ 15% (AC-500, AC-600, AC-700 and AC-800). Finding charcoal and carbon active characteristics such as surface area, analysis composition chemistry and morphology using BET-Ads, X-ray diffraction (XRF), and scanning electron microscopy (SEM). Based on chemical composition, CFB-600 has a better proportion of impurities than other CFBs and ACs. The activated CFB-500 with an activator (AC-500) experienced a significant increase of surface. It was better compared to others that reached 73.07 m^2/g, while in the other treatments, the increase of surface area from CFB to AC ≤40 m^2/g. Thus, it can be concluded the increase in temperature is not directly proportional to the increase in surface area. The temperature of 500°C is the optimal temperature obtained in this study and has a significant increase in surface area and absorption efficiency of Cr^{6+} of 87.47%.

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INTRODUCTION

One of many types of fish found in the waters of Aceh is starry triggerfish, or in the Aceh, language is called “Leubim.” Starry triggerfish is a fish considered a pest by fishermen because it has low economic value. This type of fish is also not consumed by the people of Aceh and is often thrown away or sold very cheaply for animal feed. However, over time, the starry triggerfish is used as a raw ingredient for processed fish, such as meatballs, jerky, nuggets, sausages, and empek-empek, with only the meat part are taken while the thick skin and hard bones thrown away as waste. According to previous reviews, as much as 15.3% of the total body weight of the fish will be wasted in the form of bones (Thirukumaran et al. 2022), as presented in Figure 1.

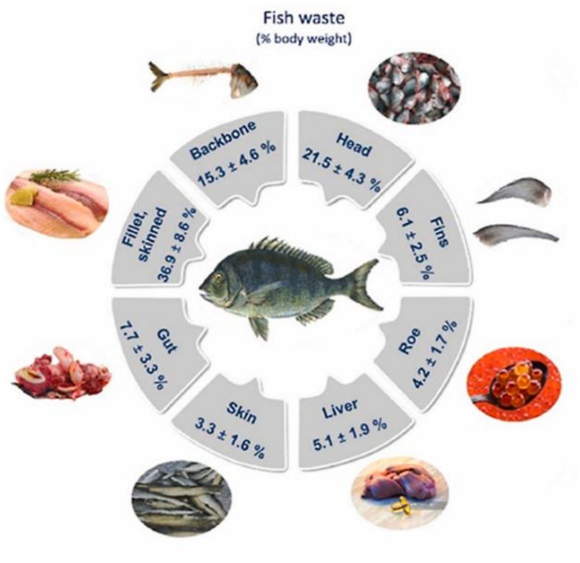

Figure 1 The percentage of the amount of fish waste produced based on each part of the fish

Nowadays, fish bones have become one of the targeted research to reuse waste as material and polymer resources like calcium (Sriuttha et al. 2014, Handayani and Syahputra 2017a, Nemati et al. 2017, Restari et al. 2019, Asmaini et al. 2020) gelatin (Rosida et al. 2018) activated carbon (Nurhadi et al. 2018, Risna et al. 2020, Nurhayati et al. 2021, Thaib et al. 2021) glue (Rohmah et al. 2015). Several researchers have utilized starry triggerfish bone waste in charcoal form as fish feed, but the research is not focusing on the characterization (Mekbungwan et al. 2004, Thu et al. 2010, Pirarat et al. 2015, Najmudeen et al. 2019). The characterization of starry triggerfish bone waste as activated carbon (AC) in various temperatures has still not yet been conducted. This study is essential because Physical activation steps affect the adsorbent's characteristics produced by charcoal and activated carbon.

AC can work as an adsorbent/absorbent material because it has a large specific surface area, capacity adsorption size, structure, type of pore, and reactivity on various surfaces (Park et al. 2004). AC manufacturing stages consist of 2 stages: carbonization and activation. Each stage has a different purpose; carbonization removes organic compounds, and activation aims to enlarge porosity and surface area AC (Rahmawati et al. 2010). Choosing the type of activator also affects the quality of the AC. Some researcher state that AC activation using $ZnCl_2$ produces AC with excellent adsorption power (Wang et al. 2010, Yulianti et al. 2010, Pambayun et al. 2013). Using $ZnCl_2$ as an activator in manufacturing AC from oil palm oilcake waste to produce AC with a large surface of 86.62 m^2/g with carbon content reached 87.15% (Wirasnita et al. 2015). This study discusses the removal of lignin in raw palm oil material so that the activator can work optimally to increase the surface area. As a result, charcoal without chemical treatment has large pores (macropores), while in charcoal with chemical treatment, the pores turn into mesopores in a more significant amount. Thus, the surface area of activated charcoal becomes larger (Wirasnita et al. 2015).

Thus, it is necessary to conduct research aimed at utilizing abalistes stellaris fish bones into activated charcoal using temperature variations and $ZnCl_2$ activators and studying their characteristics including chemical composition analysis using X-ray Fluorescence (XRF), pore size analysis and surface area analyzed using *Brunauer-Emmett-Teller* (BET) method, surface morphology analysis using Scanning Electron Microscopy (SEM) and its application as a heavy metal adsorbent. The percentage of removal tested is for Cr^{6+} ions.

MATERIALS AND METHODS

Preparation of AC

Starry triggerfish bone waste (FBs) gathered from the Fish Landing Site (FLS) Lampulo, Banda Aceh, Indonesia, washed and then boiled to remove the meat residue that still stick to the bone. After cleaning the meat from sticking, the bones are dried for 4 days until they are dehydrated. Then 50 grams of fish bone waste is carbonized at different temperatures, i.e., 500°C, 600°C, 700°C and 800°C for 40 minutes, called bio charcoal. Each sample was labeled CFB-500 (charcoal fish bone with carbonization at T = 500°C), CFB-600, CFB-700 and CFB-800. This stage is called activation by the physics method that uses high temperature.

The next step in creating AC from the triggerfish bone is activation by chemical using $ZnCl_2$ 15% activator. The method used slightly modified from previous studies (Handayani et al. 2020b, 2020a). Each obtained bio charcoal from different temperature-activated chemicals. CFB-500 was soaked using the $ZnCl_2$ (3:1) ratio of activator and sample for 2 hours. The mixture is filtered, and the residue is carbonized at 700°C for 10 minutes. After reaching room temperature, the sample is neutralized (pH = 7) and dried using an oven at 105°C for 6 hours. The result was AC, labeled AC-500 (AC made through the carbonization process at a temperature of 500°C and activated using $ZnCl_2$). The same steps were also carried out on samples of other bio charcoal labeled accordingly AC-600, AC-700, and AC-800.

Characterization of Starry Triggerfish Bone and AC

Analysis characterization of FBs, CFB, and AC includes chemical composition analysis using X-ray fluorescence (XRF) instrument (Bruker), The surface area and pore size were measured using the Brunaeur-Emmet-Teller (BET) and Barret-Joyner- Halenda (BJH) methods with the Surface area analyzer (SAA) instrument (NOVA Quantachrome 11.0 version), and surface morphology was analyzed using Scanning Electron Microscopy (SEM) instrument (JEOL-6510 LA) (Handayani et al. 2020a, 2020b)

Adsorption of Cr^{6+}

The method used to calculate the % removal of Cr^{6+} follows similar research methods that have been done previously (Handayani et al., 2020b). Testing the adsorption of heavy metal ion Cr^{6+} is done with contact as much 0.3 grams of FBs, CFB, and AC to 20 ml of Cr^{6+} test solution 25 mg/l for 20 hours. Then, the absorbance is measured using a spectrophotometer (Biobase Model BK-UV1000) at the maximum wavelength (max). The Cr^{6+} solution is made from stock solution Cr^{6+} 1000 mg/l (which was made by dissolving 2.8 g $K_2Cr_2O_7$ into 1000 ml of distilled water). Then, the stock solution is diluted to get the test solution with a concentration of 25 mg/l. Whereas for looking for λ_{max} of spectrophotometer instrument, the standard solution Cr^{6+} is used with a concentration of 5 mg/l made of stock solutions. The measurements absorbance performed on a long-range wave (λ) 490–600 nm. After λ_{max} is obtained, the next step is to look for a standard curve created with measure absorbance standard solution (concentration range 0.04 to 0.2 mg/l) at λ_{max} 540 nm.

The standard solution is made from the stock solution. A standard curve is made to measure the concentration of Cr^{6+} using controlled absorbance. The measurement procedure of heavy metal ion Cr^{6+} is by APHA procedures (APHA 1985), which involves complexing the Cr^{6+} ion until the color purple using 1.5-diphenylcarbazide 0.5% (pH regulated up to 2 using H_2SO_4 and H_3PO_4 as buffer). Measurement of test solutions before adsorbed and after adsorbed using FBs, CFBs, and ACs. The percentage removal of metal ions Cr^{6+} is calculated using the equation (1).

$$C(\%) = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

Where: C (%): % Cr^{6+} removal; C_0 = initial concentration; C_e: obtained concentration after treatment

RESULTS AND DISCUSSION

Characterization of FBs, CFB, and AC

Samples such as the flour from starry triggerfish bone (FBs) or raw material, starry triggerfish (CFB) bio charcoal on various temperatures (CFB-500, CFB-600, CFB-700, CFB-800) and activated from starry triggerfish bone using $ZnCl_2$ activator 15% with different carbonization temperature (AC-500, AC-600, AC-700, and AC-800) were characterized as follows:

Chemical composition analysis

XRF is an element or oxide metal technique analysis that forms a material with x-ray interaction with analyte material. XRF is generally used to analyze contained elements in minerals or rocks. The element could be determined directly without particular standards. The results obtained for samples of AC and charcoal from the starry triggerfish bone produced use different temperatures presented in Table 1.

The content analysis results show that there are still several impurity substances, such as calcium (Ca) and phosphorus (P), which are dominant elements and other elements with small amounts. So, its presence in the pores makes them clogged, resulting in limited surface area (Ketaren, 1986). The impurity substances could blockage FBs, CFB, and AC pores. Based on the XRF analysis result, it is visible that the compound oxides that are present in AC, in general, are CaO, SO_3, and P_2O_5. This thing happens because the AC is not immersed in solid acid. Previous research in making active charcoal from tuna fish bones has one step of immersion using H_2SO_4 12 % for 30 minutes; therefore, it dissolves CaO and MgO that fill pores and increase the AC surface area (Siswati et al. 2015).

Based on the data presented in Table 1, the AC-600 has a composition with lower impurities compared to other treatments. At the same time, the AC-700 and AC-800 have started to form white ash, which is expected to be calcium oxide (CaO). At the moment of the CO2 decomposition (700-800°C), much carbon content is lost, so the carbon content and some other impurities are also decreased. Meanwhile, for charcoal (before activation of $ZnCl_2$), CFB-600 has a better composition than other CFBs because it contains little impurities but has the highest ZnO content. Increasing Zn content decreases specific surface area and pore volume data (Fatimah et al., 2016).

Surface area and pore size analysis

Surface area and pore size are significant to assess active charcoal quality. The surface area of an adsorbent is closely related to its contact area of adsorbate; therefore, a larger surface area will make the adsorbent's contact area larger, increasing the adsorption process. The loss of impurities in the activation stage will increase the surface area of the adsorbent because it opens the pores that were previously filled with compound impurities. Similar statements were also expressed in research on making activated carbon a mask layer (Dizbay-Onat 2023).

Table 1 Chemical analysis of FBs, CFB, and AC

sample	Chemical composition (%)								
	Ca	Cl	Al	S	P	K	Sr	Ag	Zn
FBs	83.24	0.34	0.27	0.85	13.86	0.38	0.62	0.45	-
CFB-500	82.87	0.38	-	0.86	14.25	0.40	0.62	0.43	0.20
CFB-600	52.20	0.7	0.3	0.1	15.50	-	0.4	0.4	30.4
CFB-700	81.10	0.26	0.31	0.51	16.32	0.45	0.52	0.36	0.18
CFB-800	81.04	0.30	0.27	-	17.21	0.32	0.52	0.35	-
AC-500	81.80	0.31	0.31	0.51	15.57	0.36	0.54	0.42	0.18
AC-600	53.62	0.46	0.35	0.14	16.22	-	0.36	0.43	28.41
AC-700	57.21	0.45	0.32	-	16.37	-	0.38	0.34	24.94
AC-800	58.61	0.39	0.34	0.06	16.56	-	0.36	0.35	23.34

Table 2 Surface area and pore characteristics

Sample	Physical properties			
	Surface area (m^2/g)	Pore radius (Å)	Pore volume (cc/g)	Surface area micropores (m^2/g)
FBs	27.97	21.77	0.03	28.48
CFB-500	6.76	41.73	0.14	7.86
CFB-600	32.45	63.57	0.10	45.55
CFB-700	76.46	55.86	0.21	105.71
CFB-800	83.46	62.34	0.26	116.41
AC-500	79.83	56.51	0.23	110.34
AC-600	71.13	68.73	0.24	98.85
AC-700	83.21	65.88	0.27	114.15
AC-800	87.13	71.67	0.31	119.44

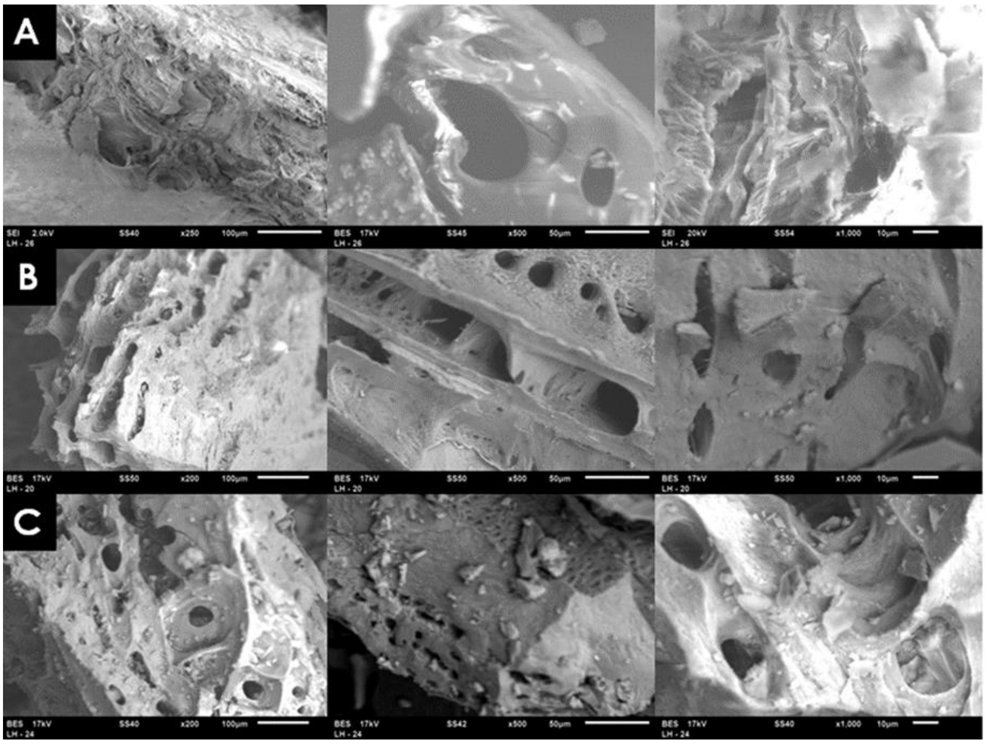

Figure 2 SEM Image of (A) FBs (magnified: x250, x500, x1000). (B) CFB (magnified: x200, x500, x1000). (C) AC (magnified: x200, x500, x1000)

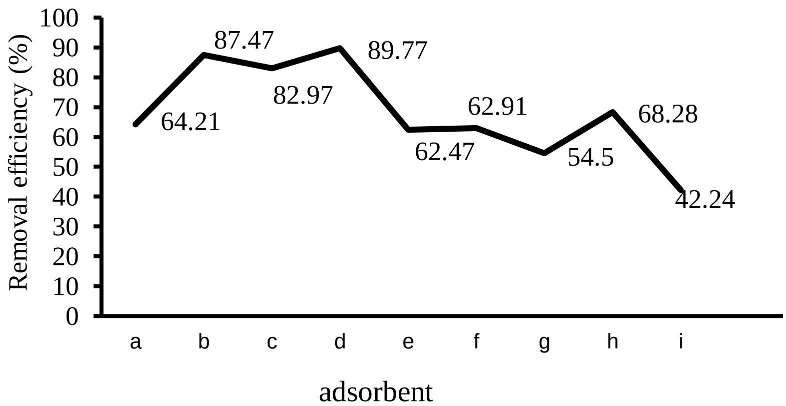

Figure 3 Removal efficiency of Cr6+ by (a) FBs, (b) CFB-500, (c) CFB-600, (d) CFB 700, (e) CFB-800, (f) AC-500, (g) AC-600, (h) AC-700, (i) AC-800

The pores and surface area characteristics of the resulting samples can be seen in Table 2. The activation stage has influenced the surface area and pore radius for either resulting samples activated by physics (CFBs) or activated with the addition of chemical activators (ACs). The highest surface area increase is found in the sample that is activated by physics at a temperature of 500 C (CFB-500) and then reactivated by chemical with the addition of $ZnCl_2$ activator (AC-500). The increase is 73.07 m^2/g, from 6.76 m^2/g to 79.83 m^2/g. This proves that the chemical activator affects the resulting sample's surface area and pore radius. In addition, another reason is that the $ZnCl_2$ activator works optimally at a temperature of 500ºC, as previously conducted (Wirasnita et al. 2015). This is reinforced by other data obtained in Table 2, which shows that the surface area of all ACs is higher than that of CFB. The AC's more significant surface area and pore radius are due to the bonding of compound impurities on the surface and inside active charcoal pores by the activator. However, the pore volume of CFB and AC is still minimal because the impurities in the sample fish bones, like calcium (Ca) and phosphorus (P), have not all been lost because no vital acid immersion treatment could dissolve the impurities. This is conveyed by previous research characterizing activated charcoal from tuna bones (Siswati et al. 2015). For charcoal base ingredients that contain lots of Ca and Mg elements, in addition to using the active ingredients mentioned above in the activation process, it is necessary to soak H_2SO_4 with a 10-15% content for 0.5 hours. Immersion aims to dissolve CaO and MgO components that fill the pores and open closed pores to expand the absorbent surface (Ketaren, 1986).

Surface Morphology

Morphological analysis of the surface of the adsorbent is carried out to know the depiction of the surface and pores formed. It is conducted using SEM instruments. The samples analyzed for the surface morphology are FBs, CFB-600, and AC-600. Based on previous characterization, the treatment with a temperature of 600°C creates a better adsorbent composition than other treatments. FB samples are analyzed for surface morphology as a comparison. The inside active charcoal pores by the activator. However, the pore volume of CFB and AC is still minimal because the impurities in the sample fish bones, like calcium (Ca) and phosphorus (P), have not all lost (Siswati et al. 2015) because there are no morphology analysis results of the three adsorbents presented in figure 2.

Based on the analysis of the results presented in Figure 2. The morphology surface of each sample is different. FB pores are visible but not yet open because impurities still cover them. This is caused by oxides metal like CaO, MgO, SiO, P_2O_5, Al_2O_3, Fe_2O_3, protein, and fat. However, on the CFB-600 surface, the pores seem more open and cleaner even though the number of pores formed is still limited. The components of these oxide compounds fill the pores and cause the clogging of the pores to reduce the absorbent surface (Ketaren, 1986). The activeness of absorption from activated charcoal depends on the amount of carbon compounds. The surface of activated charcoal carbon is relatively free from the build-up of oxide compounds so that it can actively absorb (Siswati et al. 2015).

The evaporation of organic compounds that filled the pores makes the CFB-600 surface have visible pores. This is based on the results of XRF and BET-Ads testing. The CFB-600 sample has lower types and levels of impurities; however, the surface area is still low because of the impurities bound firmly to the pores' walls. This is similar to the AC-600, which has a better morphology surface compared to CFB-600 because some of the impurities that fill wall pores are lost in the activation stage. We can see from the picture that the AC-600 has more pores than the CFB-600. The detail surface seems like ununiform cotton. The surface of CFB-600 and AC-600 is visibly finer, with form pores that are more visible and relatively regular than FBs. If the pores are adsorbent and covered by impurities, then the adsorption ability will decline.

Adsorption of hexavalent chromium Cr^{6+}

The samples have been tested by adsorbing the Cr^{6+} ions.

Based on Figure 3, CFB-700 has the best adsorption ability compared to others. It can absorb up to 89.77%. This is due to its characteristics, such as a good surface area, pore radius, and pore volume compared to others. Meanwhile, AC-800 has the lowest adsorption ability, only 42.24%. This is due to damage to the wall's pores on AC-800, which is caused by too high a temperature during the process. A high temperature also causes the CaO to form, characterized by the appearance of white powder. Calcium carbonate ($CaCO_3$) contained in fish bones will start decomposing Becomes CaO at ≥700°C, and decomposition will stable at ≥900°C temperature, and can be seen by the change of all fish bones powder to white color (Mohamed et al. 2012, Handayani and Syahputra 2017b, 2017a).

CONCLUSIONS

Starry Triggerfish bone waste can be utilized as an adsorbent. Based on chemical composition, CFB-600 has a better proportion of impurities than other CFBs and ACs. Activated CFB-500 with an activator (AC-500) experienced a significant increase in surface area (reaching 73.07 m^2/g) compared to others (averaging ≤40 m^2/g). Thus, it can be concluded that the increase in temperature is not directly proportional to the increase in surface area. The temperature of 500°C is the optimal temperature obtained in this study and has a significant increase in surface area and absorption efficiency of Cr^{6+} of 87.47%.

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