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Rosalina

Department of Industrial Waste
Treatment Polytechnic of AKA
Bogor, Bogor, Indonesia

Henny Rochaeni

Department of Analytical
Chemistry Polytechnic of AKA
Bogor, Bogor, Indonesia

Poppy Sri Lestari

Department of Industrial Waste
Treatment Polytechnic of AKA
Bogor, Bogor, Indonesia

Tun Tedja

Department of Chemistry,
FMIPA- Bogor Agricultural
University, Darmaga Bogor,
Indonesia

Etty Riani

Department of MSP, FPIK-
Bogor Agricultural University,
Darmaga Bogor, Indonesia

Sri Sugiarti

Department of Chemistry,
FMIPA- Bogor Agricultural
University, Darmaga Bogor,
Indonesia

Correspondence**Poppy Sri Lestari**

Department of Industrial Waste
Treatment Polytechnic of AKA
Bogor, Bogor, Indonesia

The influence of phosphoric acid activation of carbon from Bintaro fruit (*Cerbera odollam* Gaertn) on the adsorption of chromium in various conditions of pH

Rosalina, Henny Rochaeni, Poppy Sri Lestari, Tun Tedja, Etty Riani and Sri Sugiarti

Abstract

Activated carbon of bintaro fruit (*Cerbera odollam* Gaertn.) that is planted as a city shade tree comes from a poisonous bintaro mangrove plant. The form of bintaro fruit resembles coconut fibers and contain of lignin and cellulose more than the coconut plant has. Bintaro fruit was chemically activated using H_3PO_4 (20%, 4%, 60% & 80%) at 650 °C with activation time for 90 minutes. Proximate analyzes include moisture content, ash content, fly ash content, fix carbon content, and iodine absorption. Topography of activated carbon was analyzed by SEM, and functional group analysis was performed with FTIR as well as total chromium used AAS. Adsorption of chromium was carried out under acidic, neutral and alkaline pH conditions. The results showed that the activated carbon of bintaro fruit that was activated with 20% phosphoric acid at an alkaline pH was able to remove the chromium about 66.55%.

Keywords: bintaro fruit, activated carbon, phosphoric acid, chromium

Introduction

The Bintaro tree that is called Pong-pong or Indian suicide tree and has the Latin name *Cerbera odollam* Gaertn or *Cerbera manghas* Linn is a non-food plant. Bintaro includes mangrove plants originating from the tropics of Asia, Australia, Madagascar, and the islands of West Pacific Ocean. Bintaro is a drupe fruit (seed fruit) consisting of three layers of epicarp or exocarp (the outer shell of the fruit), mesocarp (middle layer of fiber like coco fiber), and endocarp (seed coated with seed coat or testa). Although it has a beautiful shape, but bintaro fruit cannot be consumed, because it contains substances that are toxic to humans ^[1].

Bintaro fruit contained about 58.5% lignin and 41.8% of the cellulose that has potential as raw material of activated carbon ^[2]. The studies of bintaro seeds were focused on the production of diesel oil and generate waste in the form of epicrap and mesocrap layer. One of the ways to manage the waste is to recycle the bintaro fruit waste into an activated carbon to treat heavy metal such as chromium from the process in the laboratory.

Material and Method**General experimental procedures**

Locations of bintaro fruit waste were taking place on Kol. Achmad Syam and Destrata streets, District of Bogor Utara, West Java, Indonesia. The chemicals used were concentrated nitric acid, chromium (III) nitrate, phosphoric acid 20%, 40%, 60% and 80% while for pH regulator used 4 N sulfuric acid and NaOH 4 N. The reagents used for proximate analysis of activated carbon were 0.1 N iodine solution, $K_2Cr_2O_7$ powder, $Na_2S_2O_3$ 0.1 N solution, 1% starch solution, 4 N H_2SO_4 solution, and 10% KI solution. All chemicals used were of analytical grade.

Sample preparation

Activated Carbon was made by burning 700 g waste of bintaro fruit for 5 hours at 500 °C and activated with phosphoric acid with concentration variations of 20%, 40%, 60% and 80%. After the activated carbon has been washed and dried, it was heated to the retort at 650 °C for 90 minutes.

Characterization of Activated Carbon

The moisture, ash, and fly ash contents, as well as carbon fix and iodine absorption of activated carbon was identified and compared with SNI 06-3730-1995 on the requirement of technical activated carbon. Moreover, the functional group and the topography of the activated carbon surface were analyzed by the following tools:

- (1) Infrared spectrophotometer was used to determine the functional group changes due to the temperature activation and concentration of activator. This analysis was done by mixing the sample with KBr into pellet form, and then measured at wave number 600 - 4000 cm^{-1} .
- (2) Scanning Electron Microscope (SEM) was used to determine the topography and pores size of activated carbon surface by coating the sample with platinum. Samples inserted into SEM and electron devices which were detected were scanned with electron beam and then image was taken from the surface of the activated carbon [3].

Calculation of Chromium Removal Percentage

Concentration of adsorbed total chromium for each treatment was calculated from:

$$\text{Adsorbed chromium concentration} = (\text{Initial} - \text{Final}) \text{ Concentration}$$

$$C_{\text{adsorbed}} = C_{\text{initial}} - C_{\text{final}}$$

The percentage of total chromium removal was calculated by the formula:

$$\text{Chromium Removal (\%)} = \frac{C_{\text{adsorbed}}}{C_{\text{chromium waste initial}}} \times 100\%$$

Result and Discussion

Characterization of Activated Carbon

In Table 1, it is seen that none of the activated carbon having ash content meets the standard of technical activated carbon powder quality. The high level of ash was due to the oxidation process that occurs at high temperatures and soft material structure that consist of fiber. In addition, ash values also showed the amount of residual inorganic compounds from the combustion process in the form of mineral substances that were not lost during the combustion process. The increased of ash levels occurred due to the formation of mineral salts during the drying process and when the process continues it would form fine particles of the mineral salts [4]. The water content of activated carbon with phosphoric acid met the standards based on SNI 06-3730-1995 on the requirement of technical activated carbon. The highest water content was produced from activated carbon with 80% of H_3PO_4 and the lowest water content was obtained from activated carbon with 20% of H_3PO_4 .

Table 1: The result of characterization of phosphoric acid activated carbon

| Treatment | Water Content (%) | Ash Content (%) | Fly ash Content (%) | Fix Carbon Content (%) | Iodine Absorption (mg/g) | Yield (%) |
|-----------|-------------------|-----------------|---------------------|------------------------|--------------------------|-----------|
| A6W2S1 | 4.88 | 29.98** | 1.03 | 68.99 | 812.811 | 62.85 |
| A7W2S1 | 5.35 | 28.16** | 1 | 70.83 | 925.520 | 58.57 |
| A8W2S1 | 6.06 | 31.41** | 1.11 | 67.49 | 950.332 | 57.14 |
| A9W2S1 | 7.38 | 33.19** | 1.21 | 65.61 | 857.61 | 54.29 |
| Standard* | Max. 15 | Max. 10 | Max. 25 | Min. 65 | Min. 750 | |

Notes: *Standard = SNI 06-3730-1995 on the requirement of technical activated carbon

A6 = Activated by H_3PO_4 20 %

A7 = Activated by H_3PO_4 40 %

W2 = Activation time 90 minute

A8 = Activated by H_3PO_4 60 %

S1 = Activation Temperature 650oC

A9 = Activated by H_3PO_4 80 %

**Exceed the standard

Table 1 also showed that the higher concentration of activating agents caused higher levels of water content of activated carbon. That happened because the process of immersion with the activator was done to reduce the tar level, so the more concentrated the activator materials that were used the more decreased the levels of tar on the carbon, consequently the pores contained in activated carbon was bigger and much more. The bigger and more pores that were formed the larger the surface area of activated carbon. The increased of the surface area of the activated carbon will improve the hygroscopic properties, so that the absorption of water from the air by the activated carbon itself became increasing and the water content of the activated carbon was also increasing [5].

The levels of fly ash content met the requirements of technical activated carbon. The highest level of flying substance was in activated carbon with 80% of H_3PO_4 and the lowest was in activated carbon with 40% of H_3PO_4 . It can be said that the higher the concentrations of the activated carbon, the higher the content of fly ash content. The high activator concentration caused a lot of ash formation from non-volatile salts while some volatile minerals became fly ash. The high level of fly ash content increasing from carbon to activated carbon also indicates that the surface of activated carbon contains a fly ash derived from the interaction of carbon with the activator [6].

As for the fix carbon parameter, it was known that the activated carbon of H_3PO_4 40% has the highest fix carbon content and activated carbon with H_3PO_4 80% has the lowest fix carbon content. The amount of fix carbon content produced not only influenced by high levels of fly ash and ash content but also by the content of cellulose and lignin materials that can be converted to carbon atoms [7]. This condition indicated the activated carbon of bintaro fruit has a relatively high adsorption. Data on low levels of fly ash and high levels of ash content of activated carbon support high levels of fix carbon. This relatively high fix carbon content indicated that the least carbon atoms reacting with water vapor produced CO gas, so that the carbon atom was reorganized to form a considerable hexagonal structure.

Iodine absorption has a correlation with surface area of activated carbon. The larger the iodine number, the greater the ability of activated carbon in adsorbing the adsorbent or solute [8]. If the activated carbon has a large surface area it would give a larger contact area between the adsorbent and its adsorbate, so that the adsorbate can be adsorbed more. It was known that high iodine absorption was obtained on activated carbon with 60% of H_3PO_4 and the lowest is 20% of H_3PO_4 . The low absorption rate of iodine was caused by the impurities that clog the pores of activated carbon and reduce the pores' surface area. Iodine absorption of activated carbon

from bintaro fruit shell fulfilled the requirement of technical activated carbon based on SNI 06-3730-1995 [9].

In Table 1 it can be seen that changes in the variation of phosphoric acid concentration will greatly affect the yield of the production of the activated carbon. The lowest yield of activated carbon was produced from activated carbon with 80% of H_3PO_4 concentration of 54.29% while the highest was produced at 20% of H_3PO_4 concentration of 62.85%. The greater the concentration of activating materials used the smaller the yield of the activated carbon produced. This was due to the fact that organic compounds and the relatively volatile substances in activated carbon decreased in number. All products that have been produced at the activation were gaseous, so that organic substances and volatile substances in activated carbon would be carried out by the gases. The more concentrated the phosphoric acid solution that was used the more gases were formed, so the more organic substances in the activated carbon would come out and the number would decrease. That condition resulted low yield of activated

carbon as H_3PO_4 concentration increases the total yield of the process decreases [10].

Identification of the topography of activated carbon with SEM

The topography of activated carbon with SEM at $500 \times$ magnification with $20 \mu m$ scale showed that activated carbon with phosphoric acid has enlarged pores, as seen in Figure 1. The activated carbon pore diameter with 20% of H_3PO_4 (Figure 1a) has the smallest pore diameter of $4.467 \mu m$, and the largest pore diameter of $12.51 \mu m$, while the activated carbon pore diameter with 40% of H_3PO_4 (Figure 1b) has pores with the smallest diameter of $5.807 \mu m$ and the largest of $21.00 \mu m$. The pore diameter on activated carbon with 60% of H_3PO_4 (Figure 1c) has the smallest pore diameter size of $10.72 \mu m$ and the largest pore diameter of $7.25 \mu m$, while the 80% of H_3PO_4 (Figure 1d) has the smallest pore diameter of $9,828 \mu m$ and the largest pore diameter of $16.98 \mu m$.

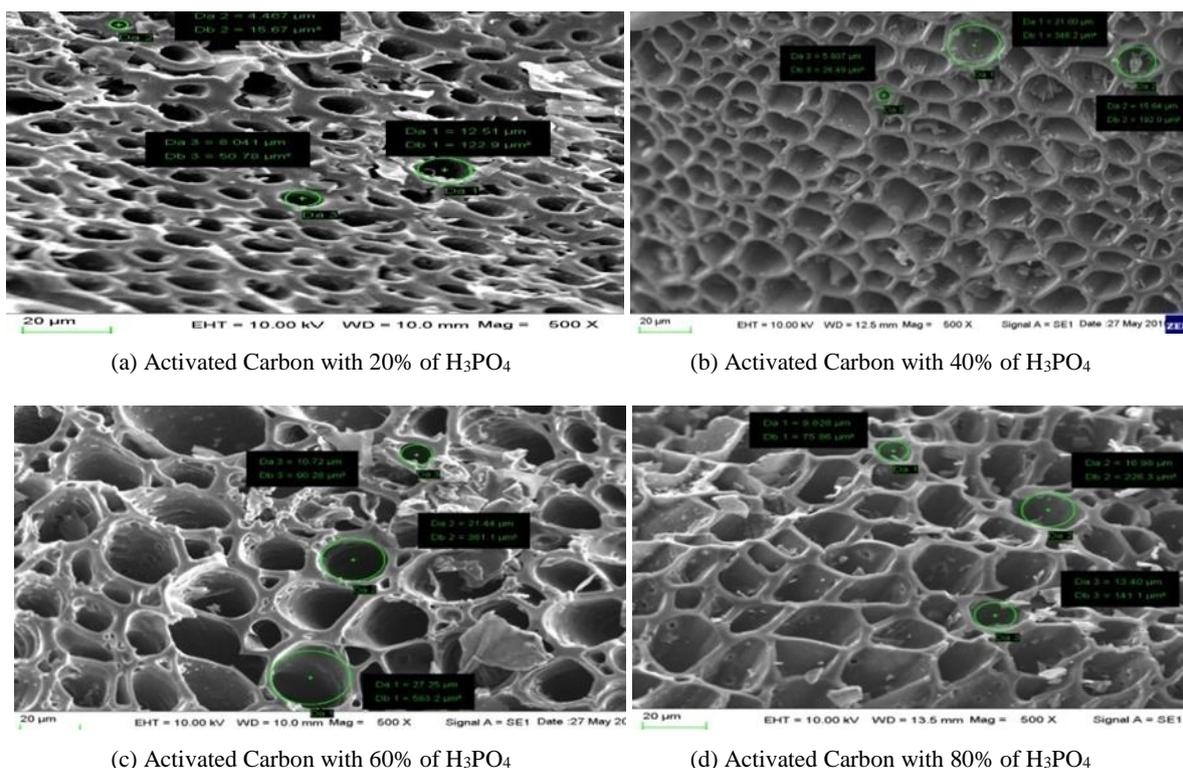


Fig 1: Topography of Phosphoric Acid Activated Carbon

The structure and pore size of the characterization with SEM indicated that during the activation process the irregularly carbonized crystallite plates have a friction, so that the crystallite surface became exposed to the activating gas which could drive the hydrocarbon residues. In addition, the solution of H_3PO_4 as an activator also affects the size of the pore diameter as the H_3PO_4 solution was acidic, thus capable of removing hydrocarbon or impurity compounds. This caused the formation of pores on the carbon surface. Based on the SEM test results, the increased concentration of phosphoric acid would increase the pore diameter. However, when the concentration was magnified to 80%, the pore diameter became smaller. This happened because the phosphoric acid activator would react immediately with the cellulose when it was mixed and would form a hydrolyzed glycoside bond. Moreover, when the concentration of phosphoric acid mixed was too high it would cause more formation and caused clogging of the pores [11]. The SEM test results were suitable

with iodine absorption data, which indicated the iodine absorption capacity of activated carbon decreased at concentration of phosphoric acid activator by 80%.

Functional Groups of Activated Carbon

In Figure 2, the absorptions in the wave number 885, 840 cm^{-1} and the wave number 775 cm^{-1} were the deformation of the C-H group with the out-of-plane mode tendency for different substitutions of the benzene ring. The spectra between 1300 cm^{-1} and 900 cm^{-1} of wave numbers indicated the C = O stretching group of acids, alcohols, phenols, ethers, and esters due to the presence of oxidized carbon. However, it was needed to define the characteristic of carbon phosphorus compounds because the phosphoric acid activated carbon was between the wave numbers 900 cm^{-1} and 1300 cm^{-1} [12]. Spectrums in wave numbers 1300-900 cm^{-1} are allocated specifically to phosphor species such as P = O, O-C stretching in P-O-C from aromatics and P = OOH. Spectrums in the

wave numbers 696 cm^{-1} and 850 cm^{-1} were allocated for aromatic substitution by aliphatic functional groups and the spectrum in the 609 cm^{-1} wave number was intended for O-H bending vibrations in out of plane.

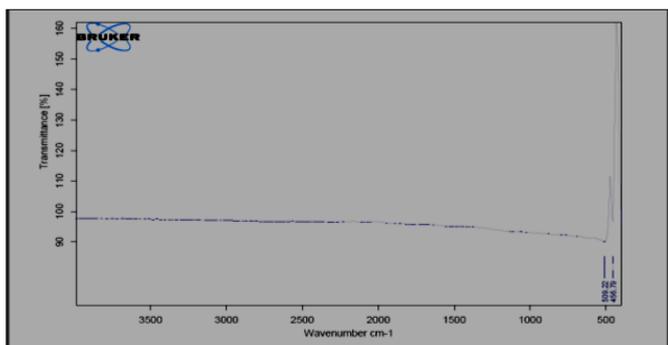


Fig 2: Infrared absorption spectra of phosphoric acid activated carbon

Percentage of Removal Chromium

The chromium (VI) which was highly dependent on the pH value might be in the form of dichromate ($\text{Cr}_2\text{O}_7^{2-}$), hydrochromate (HCrO_4^-), chromate (CrO_4^{2-}) and potassium chromate (KCrO_4^-) as shown in Figure 3. The chromium (III) might also be in the form of trivalent chromium hydrate, $\text{Cr}(\text{H}_2\text{O})_6^{3+}$, and the chromium hydroxide complex, $\text{Cr}(\text{OH})(\text{H}_2\text{O})_5^{2+}$ or $\text{Cr}(\text{OH})_2(\text{H}_2\text{O})_4$ [13].

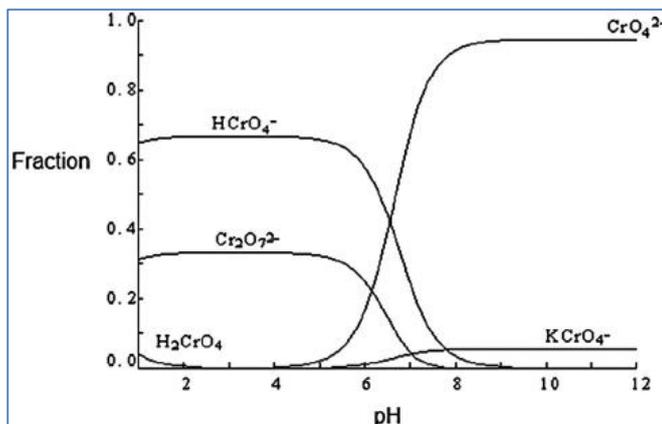


Fig 3: Chromium (VI) in various pH values [14]

Based on Figure 3 it can be seen that the percentage of chromium removal of an adsorbent can be affected by the pH of the solution. It was because pH could affect the solubility of metal ions in the solution and affect the charge on the surface of the adsorbent during the reaction which corresponds to the protonation or deprotonation of the active side surfaces of the adsorbent, so that it can be positively or negatively charged (Yakout and El-Deen 2011). The pH will affect the surface load of the adsorbent, the degree of ionization and type of specimen that can be absorbed in the adsorption [15]. The pH value can also affect the chemical equilibrium, either on the adsorbate or on the adsorbent. The existence of this pH variation causes the possibility of the occurrence of chemical bond between adsorbent and adsorbate.

In Figure 4 it is known that the highest removal percentage of chromium at $\text{pH} = 9.78$ is 66.55% and the lowest is 24.91%. The highest removal percentage was obtained from activated carbon by H_3PO_4 20% and the lowest was H_3PO_4 40%. It was proved that the concentration of the activator influenced the

percentage of chromium removal in metal ion adsorption. At the low pH (acid) the highest removal chromium (54.45%) was obtained on activated carbon with H_3PO_4 40% and chromium adsorption decreased by 49.11% in activated carbon with H_3PO_4 60%, then rose again by 54.09% in activated carbon with H_3PO_4 80%. The adsorption/desorption of chromium ions and protonation/deprotonation of the cell wall of the functional group depends on the pH of the solution. The higher total chromium removal occurred at low pH values ($\text{pH} 1$ and $\text{pH} 2$) due to strong protonation of functional groups, thus making the biomass more positively charged and creating electrostatic attraction with Cr (VI) specimens.

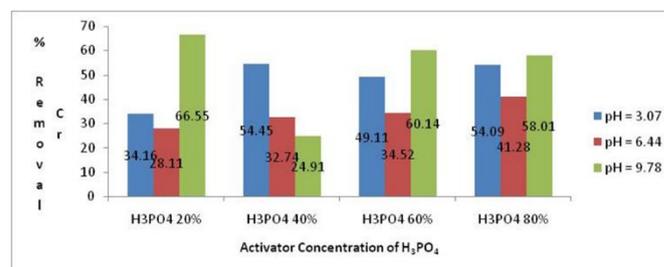
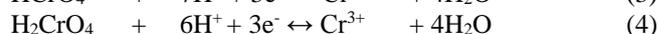
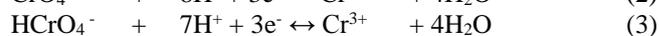
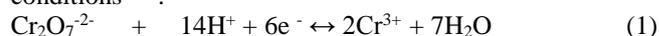


Fig 4: Percentage of chromium removal on various pH condition

However, the pH condition of chromium liquid waste was greater than two so that chromium removal did not well proceed. In addition, the longer the contact time it could also release chromium into the solution; that was due to the electron repulsion between the positively charged functional groups of the cell wall and Cr^{3+} cation specimen resulting from the reduction of hexavalent chromium. The decrease of adsorption at 60% activator concentration was due to repulsion between Cr (III) specimen and activated carbon cell wall. However, the higher the concentration of the activator causes the protonation of the functional group to rise again so that it increased the adsorption capacity of $\text{Cr}_2\text{O}_7^{2-}$. The following was chromium (VI) reactions under acidic conditions [13].



At the acid pH, activated carbon that was activated with phosphoric acid 20%, 60% and 80%, the product of hydroxide ions concentration and chromium metal was less than Ksp of $\text{Cr}(\text{OH})_3 = 6.1 \times 10^{-31}$ so that most chromium ions were dissolved. As for activated carbon that was activated with 40% phosphoric acid, the product of hydroxide ions concentration and chromium (III) ions exceeds the Ksp $\text{Cr}(\text{OH})_3$ value, resulting deposit of $\text{Cr}(\text{OH})_3$ which caused the decrease of adsorbed chromium ions concentration and many chromium deposits were retained on the filter paper.

The increased of the solution pH enhance the negative charge on the cell surface because deprotonation of the metal bond place will attract Cr (III) ions that were produced from Cr (VI) reduction. At neutral pH it was indicated that the bigger the activator concentration, the higher the adsorption efficiency. The largest chromium removal (41.28%) occurred in activated carbon with 80% H_3PO_4 . At neutral pH, dominant specimens were HCrO_4^- , CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$. All those chromium specimens presented with oxidation of +6, Cr (VI). In this case, Cr (VI) ions behave as oxoanions in an aqua solution with an overall charge (-1) or (-2). This charge might

bind a positively charged functional group (eg. amino functional group) when it was protonated. In this case, electrostatic attraction might contribute significantly to the adsorption mechanism because at neutral pH some amino functional groups were protonated [14].

At the alkaline pH the highest percentage of chromium removal of 66.55% was occurred in activated carbon with H₃PO₄ 20%. The adsorption ability of the activated carbon inclined to decrease at larger activator concentrations. Above pH 6, adsorption decreased because there was competition between OH⁻ and chromium ions (CrO₄²⁻). The positive charge potential of the adsorbent surface has decreased as the pH of the solution increased, thus weakening the repulsive charge electrochemical force between the adsorbate and the adsorbent thereby decreasing the chromium absorption capacity. However, that condition was not occurred in small concentrations of activators because there were still activated carbon functional groups that were protonated and have a positive charge so that the attraction between adsorbate and adsorbent increased the adsorption capability.

At pH > 7.0 activated carbon that was activated with phosphoric acid 20%, 40% and 80% made Cr (III) ions precipitated as Cr(OH)₃ exceed the K_{sp} value of Cr(OH)₃ in the solution, so the amount of adsorbed ions decreased and many chrome deposits were filtered. At high pH, the hydroxide group was a more effective ligand than the carboxylate active group and amine, so that the metal ions were more interested in binding to the hydroxide ligand and made the precipitation process more dominant than the adsorption process in the solution. As for activated carbon

that was activated with 60% phosphoric acid, the K_{sp} Cr(OH)₃ value was greater than the product of hydroxide ion and chromium (III) ion concentration, causing a lot of dissolved chromium were adsorbed by the activated carbon.

Based on Table 2 it was known that the pH value of liquid waste of chromium which met the quality standard of waste water was on the waste adsorbed with activated carbon of 40% phosphoric acid at a neutral condition of 6.67 and the alkaline condition of 7.11. The pH value of chromium liquid waste after adsorption with activated carbon with 40% phosphoric acid can meet the environmental quality standard (EQS) because in the neutral and alkaline waste conditions, the concentration of 40% phosphoric acid activator was not strong enough to cause a drastic change in the pH value of liquid waste. At low pH the surface of the adsorbent was surrounded by H⁺ ions (due to the functional groups present in the protonated adsorbent), so that many H⁺ ions were read at the pH meter that caused the lower pH value [16]. Moreover, since the activated carbon was made with a phosphoric acid activator, the higher the activator concentration would cause a drastic change in the pH conditions of the measured waste after adsorption.

Based on the data in Table 2 the measurement of active carbon pH dissolved in aquadest showed that the higher concentration of phosphoric acid activator, pH value inclined to decrease. The low pH value of wastewater was caused by activated carbon that adsorbed more chromium and organic materials contained in the liquid waste so that the absorption of H ions was not maximal [17].

Table 2: The result of the adsorption of phosphoric acid activated carbon on chromium in waste water from the laboratory

| Treatment of Chromium Waste + activated carbon | pH of adsorption result | pH of activated carbon with aquadest | Chromium (ppm) |
|--|-------------------------|--------------------------------------|----------------|
| Acid | 3.07 | 3.05 | 2.58 |
| A6W2S1 | 2.88 | 6.62 | 1.85 |
| A7W2S1 | 3.72 | 5.97 | 1.28 |
| A8W2S1 | 2.18 | 1.96 | 1.43 |
| A9W2S1 | 2.68 | 3.81 | 1.29 |
| Neutral (initial) | 6.44 | 6.67 | 2.81 |
| A6W2S1 | 4.13 | 6.64 | 2.02 |
| A7W2S1 | 6.67 | 5.89 | 1.89 |
| A8W2S1 | 2.30 | 2.15 | 1.84 |
| A9W2S1 | 3.61 | 4.87 | 1.65 |
| Alkaline | 9.78 | 9.83 | 2.78 |
| A6W2S1 | 4.06 | 8.39 | 0.94 |
| A7W2S1 | 7.11 | 7.71 | 2.11 |
| A8W2S1 | 2.41 | 3.63 | 1.12 |
| A9W2S1 | 4.18 | 5.91 | 1.18 |
| Standard* | 6 - 9 | | Max. 0.5 |

Notes: *Standard = Regulation of Environmental Minister No 5 Year 2014 about Standards of Waste Water for activities that do not have a defined waste water standards

A6 = Activated with H₃PO₄ 20 %
W2 = Activation Time 90 minute
S1 = Activation Temperature 650 oC

A7 = Activated with H₃PO₄ 40 %
A8 = Activated with H₃PO₄ 60 %
A9 = Activated with H₃PO₄ 80 %

Conclusion

Based on the result of the research, it concluded that bintaro fruit shell waste can be made into a good quality activated carbon because the result of activated carbon proximate almost fulfill all of the SNI requirement of technical activated carbon except for ash content. The chromium waste from the laboratory can be treated with activated carbon from bintaro fruit which was activated with 20% phosphoric acid at an alkaline pH with the best chromium removal percentage of 66.55%.

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