THE ADSORPTION EFFICACY OF ACTIVATED CARBON FROM RICE HUSKS (*Oryza sativa* L.) AND SABA BANANA (*Musa acuminata* x *balbisiana*) PEELINGS FOR THE REMOVAL OF CHROMIUM FROM LABORATORY WASTEWATER

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ABSTRACT – One main issue in industrialization nowadays is the management of wastewater that causes negative impacts on both the environment and people. The Sustainable Development Goals states the importance of clean water and sanitation. This paper acts on the goal by finding a way on how to remove chromium (Cr^{2+}) found in laboratory wastewater using activated carbon from agricultural wastes, specifically banana peelings and rice husk. Varying ratios of activated carbon from rice husk (RHAC) and banana peelings (BPAC) were used for the adsorption experiments. Data from scanning electron microscope (SEM) showed that the activation of rice husk was more favorable than the activation of banana peelings due to the presence of more prominent adsorption sites on the surface of the RHAC than on BPAC. Results from Frontier-transform infrared - attenuated total reflection (FTIR-ATR) spectroscopy showed that there was a decrease in H_2O in BPAC; meanwhile there was complete loss of H_2O in RHAC. Atomic adsorption spectroscopy (AAS) showed that pure BPAC removed the lowest amount of Cr^{2+} at only 21 % while pure RHAC removed 61 %. The optimum dosage of activated carbon was determined to have a ratio of 6.67g BPAC:3.33g RHAC with 64 % of Cr^{2+} removed; exhibiting a synergistic effect of BPAC and RHAC. It was interpreted that this was due to the interaction of the Câˆ'O and Oâˆ'H bonds between RHAC and BPAC which increased the surface area of RHAC for Cr^{2+} attachment. The findings of this study present a safe, clean, and low cost means of wastewater management in a laboratory environment.

Keywords: banana peeling and rice husk activated carbon, laboratory wastewater, removal of chromium

INTRODUCTION

Nowadays, nations are striving for industrialization. However, despite all the innovations gained from it, many problems still arise such as pollution. One of the Sustainable Development Goals states that it is important to find ways for sustainable industrial development which will provide solutions to

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environmentally sound industrializations (Sustainable Development Goals, 2015). One main issue in industrialization is managing wastes such as carbon emissions, greenhouse gas emissions, material wastes, and wastewater. If not properly taken care of, these can cause negative impacts not only on the environment but also to people. In the Philippines, the Republic Act No. 9275 (An Act Providing for a Comprehensive Water Quality Management and for Other Purposes) or otherwise known as the Philippine Clean Water act of 2004, established strict policies for the maintenance of proper water quality standards.

Amidst the implementation of Republic Act No. 9275, the Philippines like other South-East Asian countries, suffers from water scarcity, poor water quality and inadequate sanitation including treatment of wastewaters (Napaldet and Buot, 2019). The country, being a developing nation could not afford the traditional and costly physico-chemical treatment; thus, should be looking into alternative means of clean-up such as phytoremediation and adsorption technology particularly for heavy metals. There had been some studies exploring the potential of phytoremediation in the country but only few for adsorption. This study aims to contribute to that data gap by documenting the efficacy of activated carbon from rice hull and banana peeling for the adsorption removal of chromium.

Activated carbon or activated charcoal is mainly used for the purification of water and of air and separation of gas mixtures. Unique for its "pores" or spaces with the size of molecules enclosed by carbon atoms, activated carbon gains its ability as an excellent adsorbent for purification of water. This ability is due to the intense van der Waals forces exhibited by the pores caused by the near proximity of carbon atoms. Only a couple of resources can be used to synthesize activated carbon such as coals, rice husks, peat, wood, fruit stones, nut-shells, coconut shells and also banana peelings according to recent researches (Achak et al., 2009). At present, different forms of activated carbon can now be found in the market for several industrial uses (Marsh & Reinoso, 2006).

Activated carbon purifies wastewater by adsorbing different pollutants such as heavy metals. Adsorption is the process of the adherence of particular molecules to an adsorbent. It has been widely used to remove pollutants from wastewater mostly involving solid adsorbents (Ahmaruzzaman, 2008; Ahmaruzzaman and Sharma, 2005). An ideal adsorbent should have the following properties: porous, large surface area, high hydrophobicity, and is selective in accumulating the pollutant from water onto its surface. The efficiency of adsorption can be ruled by the adsorbents properties, the nature of the adsorbent (solubility in water, hydrophobicity, size and molecular weight), and the solution chemistry (this includes the following pH, temperature, degree of polarity, availability of the other solutes competing for the adsorbent surface area and the concentration of the adsorbate.

Rice husk and banana peelings, two of the identified materials that activated carbon can be extracted, are common in the Philippines and are often considered as waste. Activated carbon from rice husks is deemed reliable adsorbent to heavy metals such as chromium (Bishnoi et al., 2004; Singh and Singh, 2012). On the other hand, Boniolo (2008) found that banana peelings are effective in adsorbing heavy metals. "Saba" bananas (Musa acuminate x balbisiana), is one of the most common type of banana fruit in the Philippines. When consumed, its peelings are usually thrown away as waste. These often-considered waste materials could help in the solution of heavy metal contamination from wastewater, provided that thorough studies are conducted on them.

The study aims to determine the most effective ratio of activated carbon made from rice husks and banana peelings to treat laboratory wastewater from chemical oxygen demand (COD) analyses before disposal. Specifically, it aims to observe the ultrastructure of the activated carbons to explain their adsorption

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capability and determine the effect of the varying specific ratios of activated carbon from rice husks and banana peelings on the removal of heavy metal Cr^{2+}, and on the pH level. The study also. The resulting ratio will be used in laboratory adsorption treatments which, in the end, results in a safer and cleaner way of wastewater management in a laboratory environment.

METHODOLOGY

Collection and Preparation of Activated Carbon

Rice Husk

Dry rice (Oryza sativa) husk was obtained as waste from a rice mill from Bay, Laguna. The samples were grinded and sieved for a particle size of 0.3 mm. For chemical activation, the procedure of Hui and Zaini (2015) was adapted. The samples were soaked in 1M KOH in 1:1 ratio for 24 hours. They were then dried to remove excess KOH and carbonized at approximately 300oC. The samples were stored in an air-tight container to prevent adsorption. Approximately 30% of the weight of the rice husks was lost upon activation. A sample of the RHAC was analyzed using FTIR-ATR.

Banana Peelings

'Saba' banana (Musa acuminate x balbisiana) peelings were gathered from the local market in Los Banos, Laguna. It was washed with distilled water to remove dirt and then oven dried at 70oC. The samples were grinded and then carbonized at the optimal temperature of 450oC for 1 hour as adapted from the procedure of Abdulfatai et al (2013). They were sieved for a particle size of less than 0.3 mm. For the start of chemical activation, 100 g of the samples was treated with 0.5M concentration of H_2SO_4 in a 1:1 ratio. The sample was mixed and left without agitation at room temperature in order for activation to take place. The sample was dried to remove excess H_2SO_4 and carbonized at approximately 300oC. The samples were stored in an air-tight container to prevent adsorption. The weight of banana peel was reduced by approximately 60% upon drying and 50% upon activation. A sample of the BPAC was analyzed using FTIR-ATR.

Wastewater from COD Analyses

Four (4) L of COD wastewater was acquired from the government-owned laboratory in CALABARZON. It was observed for visible coloration and solids. A liter of wastewater was taken and filtered to remove solid wastes. Its pH was measured. The pH was adjusted through addition of NaOH and HCl to comply with the allowed pH for the AAS spectrophotometer (for measuring Cr). The pH level was approximately adjusted to 4. After adjusting the pH, the sample was filtered again.

The model of the AAS spectrophotometer used for chromium analysis is Perkin Elmer AAnalyst 400. Samples were prepared by adding 2.5 g of each into 25 mL of Diethylenetriaminepentaacetic acid (DTPA). The mixture was shaken in a mechanical shaker for 30 min, then filtered using Whatman grade 42 filter paper. The filtrate was used in the quantification of Cr using AAS. Two samples of wastewater (~50 mL each) were used as controls for the experiment.

Preparation of Adsorbents (Ratios of Activated Carbon)

Varying ratios of prepared rice husk and banana peeling activated carbon, with a total weight of 10 grams each, were placed in containers with their appropriate labels. The researchers identified the following ratios of rice husk to banana peelings respectively as follows: 1:0; 2:1; 1:1; 1:2; and 0:1. Two batches of the samples were analyzed. The total number of activated carbon ratio samples was ten.

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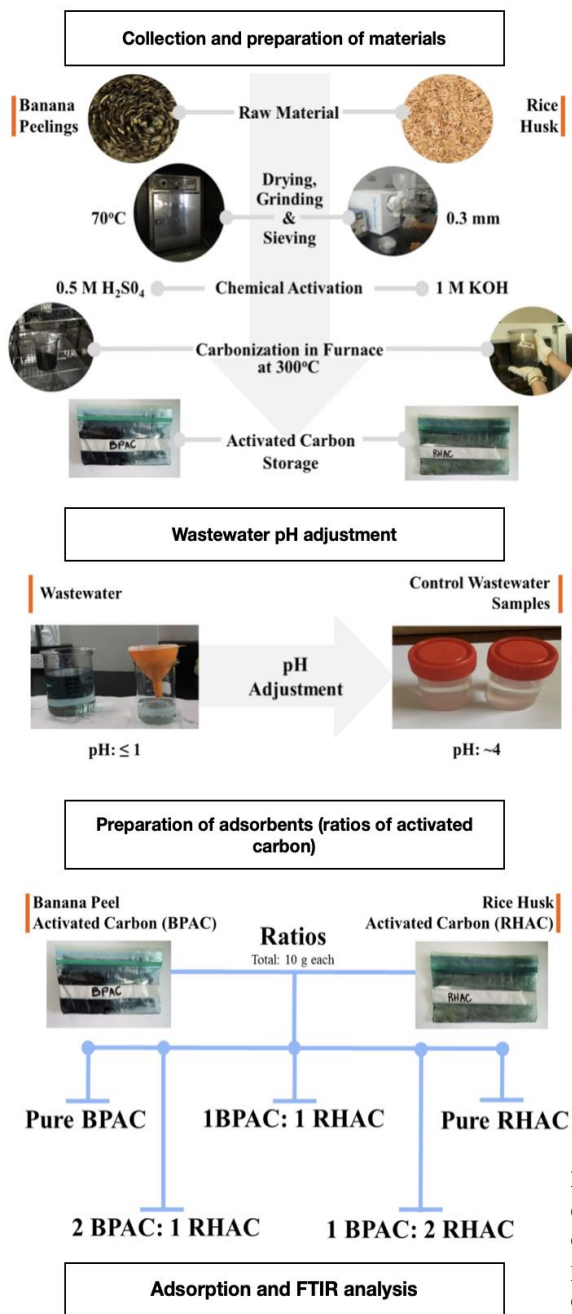from Laboratory Wastewater

Figure 1. Adopted methodology and layout of experiment. Different ratios of activated carbon from banana peel and rice husk were prepared and tested for adsorption of chromium in laboratory wastewater.

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Adsorption Analysis

One hundred mL of the filtered and pH adjusted wastewater sample was added to each container of activated carbon ratio. Two batches of 5 samples each were placed in a rotary shaker for 30 mins. After shaking, the samples were filtered to remove the adsorbent. The filtrate from each sample was placed and labeled in separate containers ready for AAS analysis for Cr^{2+}. The adsorbents from each sample were dried and placed inside air-tight containers for FTIR-ATR analysis. Samples of the activated carbons were carefully placed on the instrument diamond stage before scan. Fourier transform infrared spectroscopy (FTIR, Thermo Scientific Nicolet 6700) equipped with attenuated total reflectance (ATR) accessory with a total of 32 scans being co-added at a resolution of 8 cm-1 was used in the analysis. The flowchart of the methodology and layout of the experiment was shown (Fig 1).

RESULTS AND DISCUSSION

Structure of Rice Husk and Banana Peel Activated Carbons

SEM images showed that the RHAC, after reacting with KOH, had prominent micro-pores creating a bigger surface area for substances to adhere on its surface. This resulted in honeycomb-like structures. The BPAC, however, after reacting with H2SO4, had pores smaller compared to the RHAC.
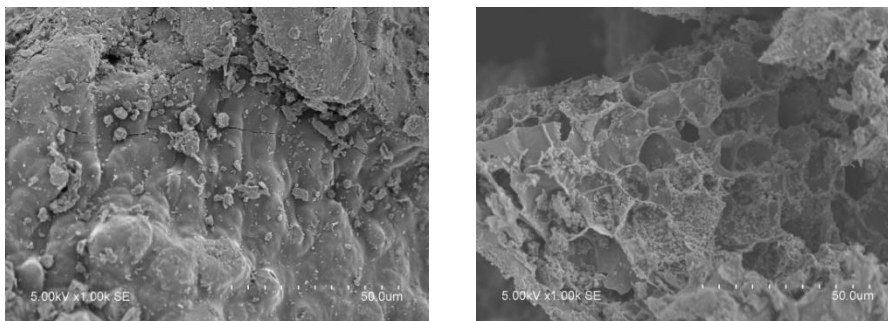

Figure 2. SEM images of rice husk before and after activation. Bigger micropores on the surface of the rice hull were evident after reacting it with KOH which makes it more suitable for adsorption.

Similar findings on the characterization of the BPAC were found on researches conducted by Mohammad et al. (2015) and Kamsonlian et al. (2011) along with orange peels. Mohammed et al. (2015) prepared nanoscale BPAC using mechanical ball mill RWTCH Planetary Ball mills type (RM400). SEM images showed the morphology of BPAC to have large numbers of intra-aggregate pores which are good sites for trapping ions.

Kamsonlian et al. (2011) used are a variety of instruments and tests in characterizing the two bioadsorbent particularly the SEM and the Fourier Transform Infrared (FTIR) Spectroscopy. The bioadsorbents were then dried, cleaned and sieved for a particle size of 0.89 mm to 0.97 mm. SEM images from Kamsonlian et al. (2011) showed that the bioadsorbent from the banana peel has a porous and irregular surface. The bioadsorbent from orange peel also have these properties. It can be interpreted that the surface of the bioadsorbents are ideal in adsorption experiments and are effective in adsorbing heavy metals.
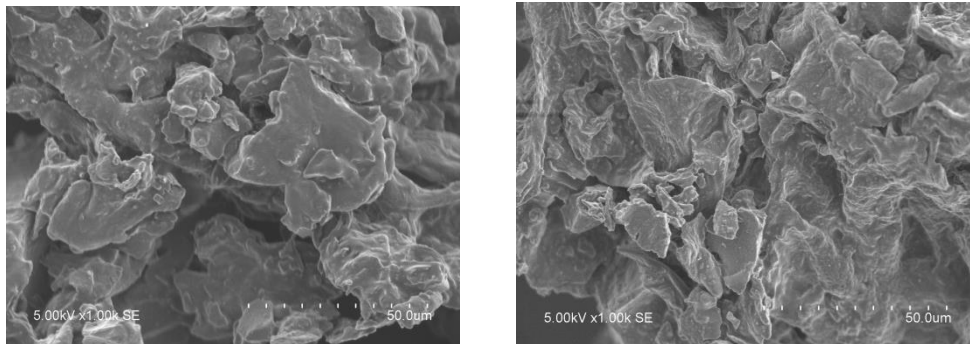
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Figure 3. SEM images of banana peel before and after activation. Banana peels developed smaller
micropores after reacting with H_2SO_4 which makes it suitable for adsorption.

Likewise, Mukoko et al. (2015) found RHAC SEM surface morphology to contain a highly porous
structure with clearly identifiable pores suitable for adsorption. RHAC was prepared using 85 % phosphoric
acid.

The FTIR-ATR spectra of raw banana peel (BP) and raw rice husk (RH) before and after activation
using H_2SO_4 are shown in Figures 3 and 4. Based on the FTIR-ATR spectra, raw BP contains O−H, C−H,
C=C, and C−O groups. There are noticeable changes in the peaks of O−H, C=C, and C−O. There is a
noticeable decrease in peak intensity of O−H stretching due to loss of water at 3269 cm^{-1} (raw) and 3248
cm^{-1} (activated). There is also a decrease for O−H bending at 1375 cm^{-1}. This is an indication of carbon
properly activated and ready to be utilized for adsorption. A shift of peak is also visible at 1014 cm^{-1} and
1107 cm^{-1}. This shows the formation of C−O stretching groups upon activation.

Based on the FTIR-ATR of raw RH before and after activation, RH contains O−H, C−O, C=C, and
C−H groups. There are noticeable changes in the O−H, and C−O groups. A loss of the peak of the O−H
group at 3298 cm^{-1}(raw) and 3300 cm^{-1}(raw) is shown in the figure above. This shows proper activation of
carbon. There is a shift of peak for C−O stretching from 1022 cm^{-1} (raw) to 1055 cm^{-1} (raw). Unlike BP,
there are no O−H bending groups present in RH.

Adsorption of Chromium by the RHAC and BPAC

The data obtained from the AAS (Table 1), showed a relationship between the ratios of activated
carbon from rice husks and activated carbon from banana peels. Comparing the chromium absorption of 10g
of RHAC and 10g of BPAC reported in Table 1, it can be observed that the absorbance of the 10g RHAC
was better than the absorbance of 10g BPAC to the value of absorbance of the control samples. The same
cannot be said in the data for the two 2:1 ratios of activated carbon as 6.67g BPAC: 3.33g RHAC seemed to
have better absorbance than 6.67g RHAC: 3.33g BPAC. Meanwhile, equal amounts of RHAC and BPAC
are shown to be more effective than 10 BPAC but not quite as effective as the other ratios. Likewise, the
concentration of chromium in 6.67 BPAC: 3.33 RHAC is seen to have the lowest value at 0.946 mg/L (~ 21
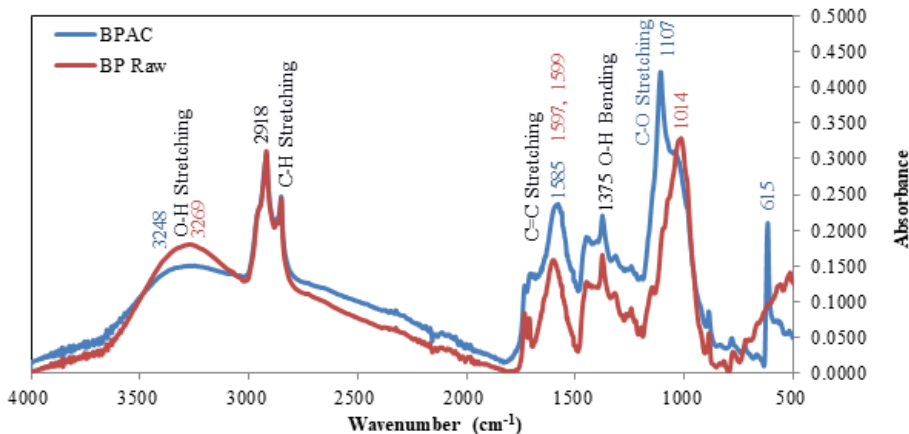%).

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Figure 4. FTIR-ATR Spectra of banana peels before and after activation. There are noticeable changes in the peaks of O−H, C=C, and C−O after activation indicating formation of new bonds.
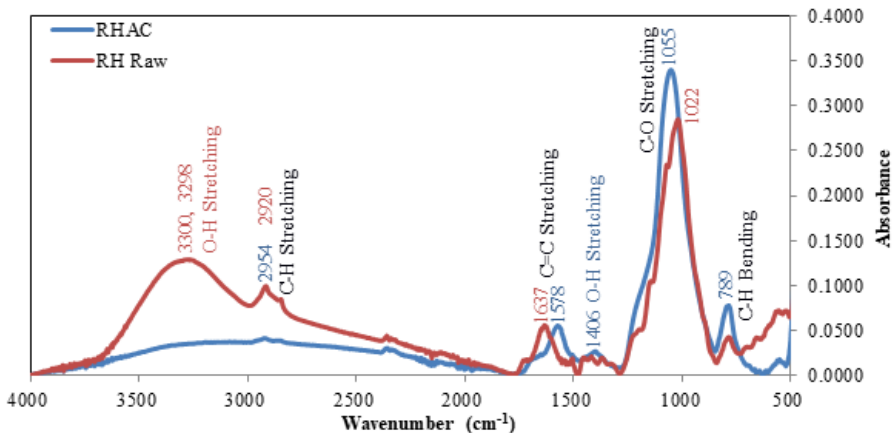

Figure 5. FTIR-ATR Spectra of rice husks before and after activation. There are noticeable changes in the O−H, and C−O groups after activation indicating formation of new bonds.

A similar study conducted by Bishnoi et al. (2004) proved the effectiveness of RHAC in adsorbing chromium. It was stated in their study that the maximum removal of chromium using rice husks is at pH level 2. In another study by Singh and Singh (2012), RHAC prepared using H_3PO_4 showed increasing percentage removal of chromium with respect to time and contact time. In their study, the percent removal increased with increasing adsorbent dose wherein 1200mg/L of adsorbent dose nearly removes the chromium entirely.

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The banana peel is also known to be a versatile adsorbent for various pollutants. The study conducted by Abdulfatai et al (2013) titled "Development of Adsorbent from Banana Peel for Wastewater Treatment" investigated the use of banana peel for the remediation of Pb, Zn and Cr from industrial wastewater. The results of their study showed that banana peels activated with 0.5 M H_2SO_4 is ideal to remove Cr^{2+} from industrial wastewater with 88.9 % percentage removal of chromium ion at a pH of 6.

From the FTIR results in Figure 3, BP had noticeable changes in the peaks of O−H, C−H, C=C and C−O. Particularly, there is a loss of H_2O due to the decrease of O−H stretching and O−H bending due to the activation of the carbons from its glucose structure. Upon activation, the oxygen from the BPAC becomes more prominent as its electronegativity attracts the chromium molecules. On the other hand, the activation of rice husk had a noticeable loss of O−H which resulted in a more significant loss of H_2O than in the activation of banana peel. Additionally, the activation of rice husk resulted in a slight shift in its C−O group. Similar to BP, the oxygen molecules became more prominent and attracted chromium molecules from the wastewater.

Table 1. Average concentration of chromium in the different wastewater samples after adsorption is determined using AAS. the concentration of chromium in 6.67 BPAC: 3.33 RHAC is seen to have the lowest value at 0.946 mg/L (~ 21 %).

Sample	Initial Concentration	After Treatment Concentration	% Removal
10 g BPAC	2.753	2.183	20.70
10 g RHAC	2.753	1.061	61.46
5 g RHAC: 5 g BPAC	2.753	1.659	39.73
3.33 g RHAC: 6.67 g BPAC	2.753	1.010*	63.31
6.67 g RHAC: 3.33 g BPAC	2.753	1.025	62.77

*Lowest value or concentration of Cr

There must be a synergistic effect between the activated carbon from banana peels and the activated carbon from rice husk in the ratio 3.33g RHAC:6.67 g BPAC causing the lowest concentration of chromium left. The absorbance of RHAC is significantly improved with the addition of BPAC and vice versa. It is possible that the mix of 6.67g BPAC and 3.33g RHAC made for more adsorption sites that chromium can attach to since there is a less uniform but larger surface area upon mixing the two activated carbons before the adsorption experiment. The researchers theorized that this may be due to an increase in surface area of the RHAC because of BPAC. The succeeding paragraphs further explain this finding.
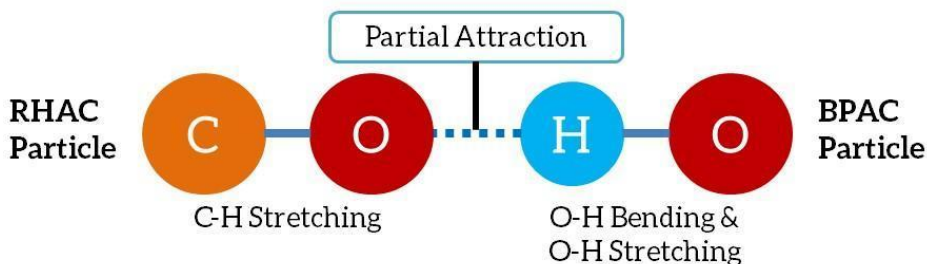
Figure 6. Proposed mechanism of adsorption. The particles of RHAC and BPAC were attracted through hydrogen bond interactions of their C−O and O−H bonds, respectively, resulting in an increase of surface area.

According to the research conducted by Shao-Yun Fu et al. (2008), when particles are crushed into smaller sizes, surface attraction becomes stronger between particles. The larger the difference in size after grinding the stronger the attraction between surfaces. This causes clumping of particles leading to a decrease in surface area, such as the case with the adsorbent made with pure RHAC. According to the FTIR results, RHAC consisted of a lot of C−O bonds and almost no O−H bonds. This is due to the sturdy beta ring structure of glucose present in rice husk, with O−H removed. Meanwhile, BPAC contained O−H bending and stretching bonds. It is possible that a particle of BPAC, which has a lot of O−H bonds left, unlocked a clump of RHAC particles, which has almost no O−H bonds but a lot of C−O bonds, through hydrogen bond interactions of their C−O and O−H bonds. Li (2005) mentioned that finer particles tend to stick to larger particles in mixing; therefore, it is also possible that the finer particles of RHAC that was detached from one another attached to a larger particle of BPAC. Thus, the RHAC particles separating lead to an increase of surface area.

The average pH of samples after adsorption was also determined and reported in Table 2. As the amount of rice husk in the ratio increases the pH also increases. It becomes more basic due to the increase in hydroxyl group after adsorption.
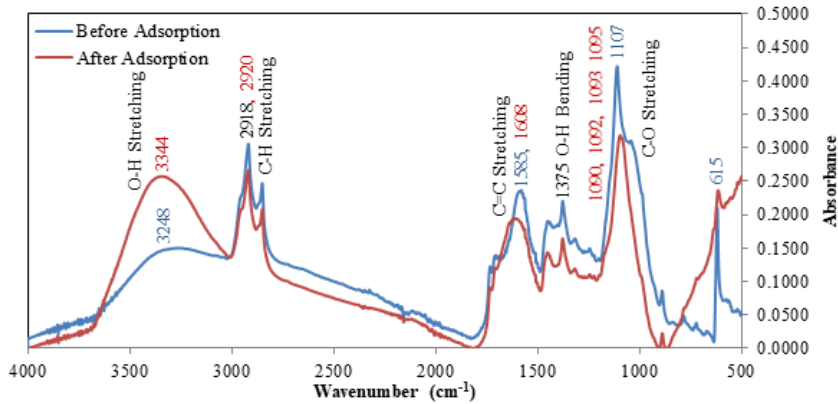
Table 2. Average pH of samples at approximately 23°C.

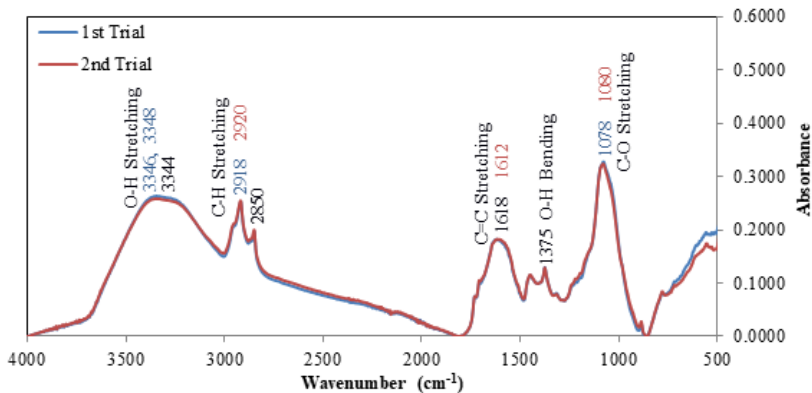| SAMPLES	Before	After pH
10 g BPAC	4.44	6.98
10 g RHAC	4.44	8.75
5 g BPAC: 5 g RHAC	4.44	7.65
6.67 g ACBP: 3.33 g ACRH	4.44	7.40
6.67 g ACRH: 3.33 g ACBP	4.44	8.14

The adsorption of pollutants from water is based on four steps, the first step being the movement of the pollutant molecules towards the external boundary layer. After that, the pollutant molecules will move to the adsorbent's active molecules' surface sites and will be eventually adsorbed. Lastly, the adsorbed pollutants will diffuse into the pore's surfaces.

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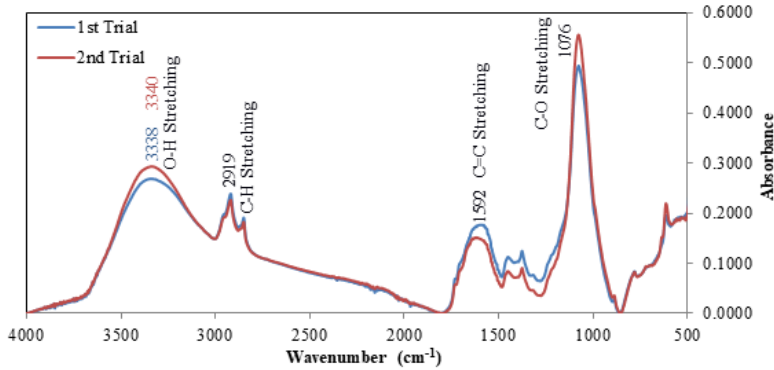### Structure of the RHAC and BPAC after adsorption

A significant increase of the hydroxyl groups particularly O−H stretching was seen in the FTIR-ATR spectra (Figure 7a) of pure BPAC before and after adsorption. Additionally, there was a decrease in C−O stretching groups. Meanwhile, it was worth noting in the FTIR-ATR spectra of pure RHAC before and after adsorption that there was an increase in O−H stretching groups and a decrease in C−O stretching groups which is similar to the comparison of pure BPAC before and after adsorption.
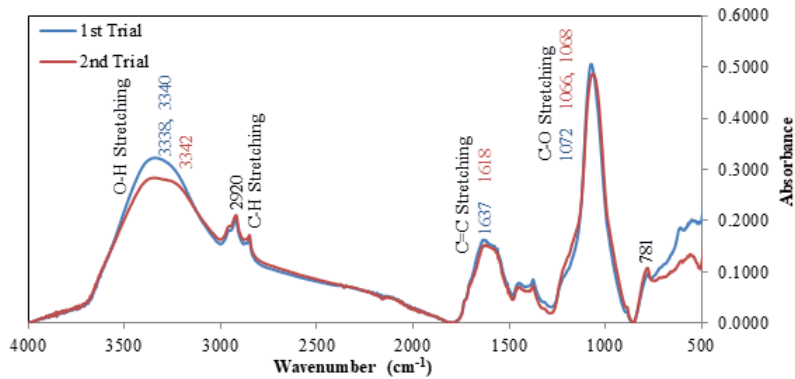

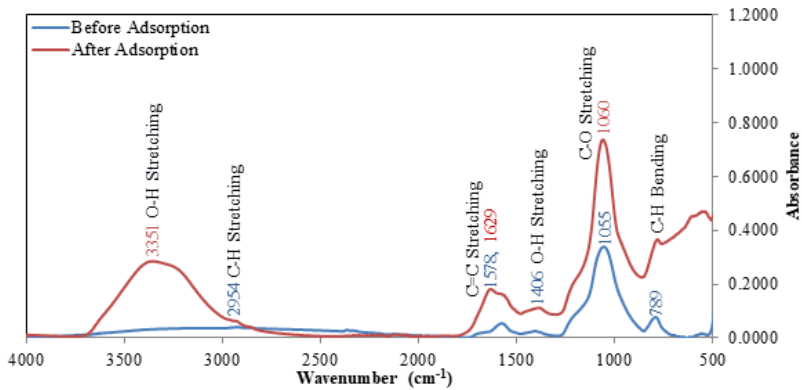a) Pure BPAC Ratio Before and After Adsorption

b) 1 RHAC: 2 BPAC Ratio After Adsorption

c) 1 RHAC: 1 BPAC Ratio After Adsorption

d) 2 RHAC: 1 BPAC Ratio After Adsorption

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e) Pure RHAC Ratio Before and After Adsorption

Figure 7. FTIR-ATR Spectra of the different ratios of RHAC and BPAC after adsorption.

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FTIR-ATR spectra (Figure 7) shows that the hydroxyl groups present in the different ratio of adsorbent increased after adsorption. This is due to the adsorbent being submerged in the wastewater. The increase of the hydroxyl groups and the increase in pH as indicated in Table 2 led to attachment of the H^+ ions from the wastewater to the surface of the adsorbent leading to a partial release of Cr^{2+}. The chromium present therefore became more susceptible to attach to the exposed carbon on the surface of the adsorbent.

The study conducted by Annadurai et al (2003) which focused on the adsorption of heavy metals Cu^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}, and Pb^{2+} in the concentration range of 5-25 mg/L from water using adsorbents made from banana and orange peels also pointed out that the adsorption of heavy metals depends on the properties attributed to the adsorbent surface and species solution. Annadurai et al (2003) interpreted that there is a competition for exchange sites between H+ and heavy metals at lower pH. This usually results in partial release of the metal; and in cases where the conditions are extremely acidic, the metal is completely released. It was also stated that the minimum pH for the minimum amount of adsorption is 2 which is due to the higher concentration and mobility of H+. The amount of adsorption also increases as pH increases up to the point where the metal precipitates.

CONCLUSION

The surface area of the waste materials increased due to the formation of micro-pores upon carbonization, providing more adsorption sites. According to the FTIR-ATR results, the most prominent functional group in RHAC was carboxyl with little to no hydroxyl groups present. Meanwhile, hydroxyl groups in BPAC still remained noticeable. Based on the results from AAS, the 2 BPAC: 1 RHAC ratio was the most effective ratio in removing chromium, with approximately 64% of chromium removed. It performed better than pure BPAC and pure RHAC. Even though the pure BPAC had the lowest concentration removal with 2.183 ppm of chromium remaining, the 2: 1 ratio still performed better with only 1.010 ppm of chromium remaining than pure RHAC with 1.061 ppm left. It is proposed that this behavior is caused by the hydrogen bonding interactions between hydroxyl and carboxyl groups.

STATEMENT OF AUTHORSHIP

All the authors conceptualized the study and did the literature review. **Frances Ann Aguda, Laurice Elaine Mercado,** and **Paulo Martin Santos** performed the collection and preparation of activated carbons and wrote the final capstone paper. **Karl Calungsod Ondoy and Aprhodite Macale** addressed issues in the paper and reviewed the final paper. **Karl Calungsod Ondoy** coordinated with institutions for the instrumental methods of analysis. **Aprhodite Macale** undertook the writing and formatting of the manuscript.

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