Production of activated carbon from corn cobs and mango kernels via H_3PO_4 activation and mediated hydrothermal treatment

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Abstract. In this study, the activated carbon produced from mango kernels and corn cobs by impregnating the hydrothermally treated raw materials with 85% H_3PO_4 were characterize for their physical surface morphology and types of surface functional groups using SEM and FT-ir, respectively. Six samples of activated carbon were submerged for 1 hour, the second sample for 2 hours, and the third sample for 3 hours. SEM results showed that both KAC (Kernel Activated Carbon) and CAC (Corn Activated Carbon) had increasing roughness and irregularity along with residence time of the samples. Results from FT-ir (Fourier-transform infrared spectroscopy) testing of the mango kernels samples showed that a C-O stretch, C-H, C=O stretch, and C-N stretch on the surface. While corncobs consist of C-H bend, and O-H bend for the 1-HR sample. The 2-HR and 3-HR samples consist of C-O stretch, C-H wag, C-N stretch. Analysis of the relationship between residence time and adsorptive capacity was done using AAS via batch adsorption in a tri-metal solution of Cu(Copper), Ni(Nickel), and Pb(Lead) with results that showed CAC and KAC, with soaking time of 3 hours is a good adsorbent of Copper and Nickel, while soaking time of 2 hours yields the best adsorption conditions for both CAC and KAC.

1 Introduction

As stated by the United Nation's Human Rights Declaration (1947), it is the right of people to have access to clean drinking water. However, a report from the International Water Management Institute (2015) predicted that two-thirds of the world will have to survive under limited water supply by 2025, and that most of those that will be affected will be from developing countries, like the Philippines. With a population that is growing at 2% every year, access to water will be even harder in the years to come (National Statistics Board, 2010).

Carbon adsorption is one of the most versatile but expensive water treatment technologies that has the ability to remove a wide variety of compounds to nearly undetectable levels (Peng Li *et al.,* 2015). Activated carbon (AC) is a microcrystalline form of carbon with very high porosity and surface area. The major use of activated carbon is in solution purification and for the removal of taste, color, odors and other objectionable impurities from liquids, water supplies and vegetable and animal oils (Moustafa, 2014). According to Kwagher (2012), the challenge with activated carbon production is to produce very specific carbons which are suitable for certain application.

Liu et al. (2010) discussed that the adsorptive properties of activated carbon from biomass may be further improved by subjecting the material to hydrothermal treatment to change the precursor's chemical characteristics and increase its oxygenated functional groups since their study they were able to attain a 340% increase of OFG in their experiment on hydrothermally treated pinewood compared with pinewood char obtained from pyrolysis.

The study will help the researchers develop a better understanding on the mechanisms and kinetics of activated carbon and its improvement through the change of soaking time parameters. The resulting activating carbon can be used as an economical and environmentally friendly alternative for the activated carbon used in heavy metal treatment and handling.

2 Materials and methods

2.1 Materials and reagents

The study utilized corn cobs as one of the two materials for production and comparative analysis of properties of activated carbon. Samples were washed with distilled or de-ionized water and dried, then subjected to variable pre-treatment and activation procedures.

Mango seeds for this research were dried procured from the OZ canteen of Adamson University and Fruitas of SM Manila. Kernels were mechanically removed from within the seeds, washed with distilled or de-ionized water, dried, and then underwent same set of pre-

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treatment and activation procedures as that of the corn cobs.

The following chemicals used in this experiment were acquired from Belman laboratories: H_3PO_4 (85% sol'n) and Island Air Products Corporation: N_2 gas.

2.2 Experimental set-up

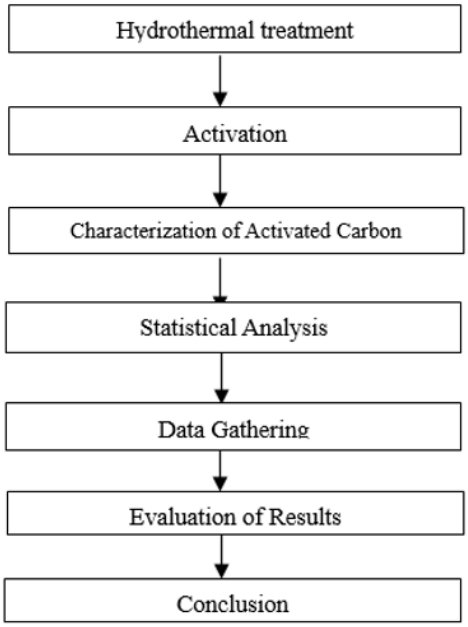

Figure 1. Process Flow Diagram of the Study

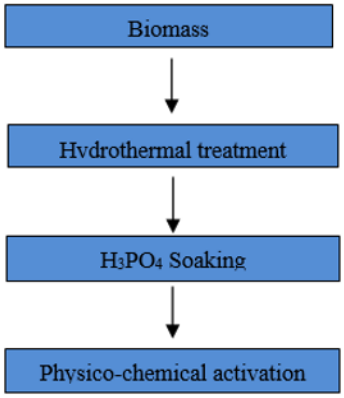

Figure 2. Treatment procedures prior to physical activation

3 Results and discussion

Experiments have been conducted to study the adsorptive capacity of the produced activated carbon on a mixture of heavy metals, namely: lead, copper, and nickel. The heavy metal mixture made use of a solution with 3ppm of each metal.

Comparing the SEM images of all three samples in **Fig. 3** at x 3,500 µm magnification it is noticeable that grooves that can be seen at the surface of samples appear to increase along with the number of hours of residence time. At x5000 magnification, seen on **Fig 4** it can be seen that 1 hour sample of the mango kernels possessed the most cavities, while 2 hours sample is the most grooved. Kuppusamy et al. (2015) credits the creation of pores to the removal of unreacted H_3PO_4 after the reaction and decomposition of organic volatiles from the biomass. Hameed et.al (2013) also described SEM images of their activated carbon as having rough and uneven surfaces which they considered as a factor for dye adsorption.

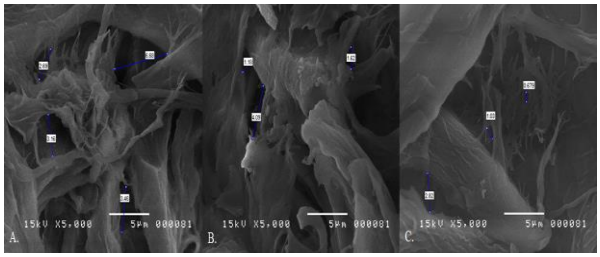

Figure 3. Mango kernel samples consecutively 1 hour, 2 hours, 3 hours X 3500 magnification

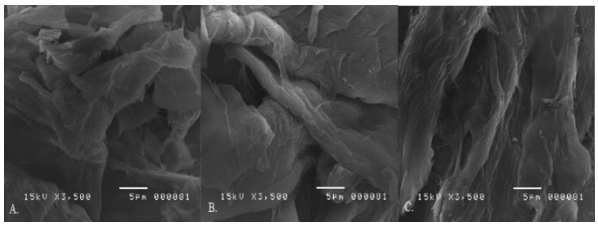

Figure 4. Mango kernel samples consecutively 1 hour, 2 hours, 3 hours X 5000 magnification

3.1. Activated carbon from corn cobs

Three samples of activated carbon were produced from the lignocellulosic material of corn cobs by impregnating the raw materials with 80% H_3PO_4, wherein the first sample was submerged for 1 hour, the second sample for 2 hours, and the third sample for 3 hours. All three samples were observed for the surface physical morphology with the use of a scanning electron microscope (SEM), composition with the use of a Fourier transform infrared spectrometer, and the adsorbance effectivity of lead, nickel, and copper with the use of an atomic absorption spectroscopy.

3.1.1. Fourier transform infrared spectroscopy results

In a similar way, Cob Activated Carbon (CAC) samples were tested for their surface functional groups. As tabulated in Table 3.2, a peak is observed at wavelength 2800.6 attributed to stretching of O-H and C-H bands. Peaks were also observed at 2346.3 cm^{-1} and 596.64 cm^{-1} due to C-H bend, O-H bend, and C-O stretch brought about by the presence of carboxyl- and carbonyl- as well as alkene aromatic groups.

3.2. Effect of residence time

The residence time of the adsorbent in the activating agent is an important parameter because it affects the

concentration of the counter ions on the functional group of the adsorbent which effectively affect the solubility of the metal ions. Using a trimetal solution of Lead, Copper, and Nickel, the adsorption data gathered were as follows:

3.2.1. Cob activated carbon

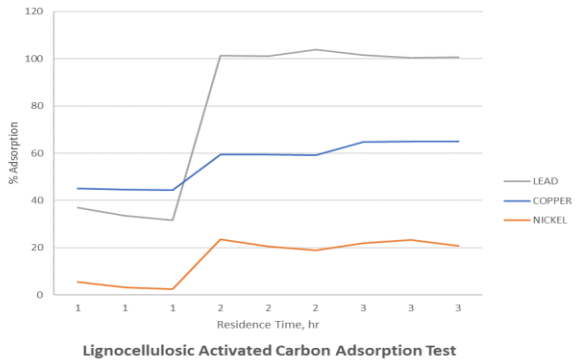

Figure 5. Variation of Residence Time vs % Adsorption of Cu, Ni, and Pb for CAC

Based on **Fig. 5**, it can be observed that percent adsorption of Copper, Nickel, and Lead, for tri-component adsorption increases proportionally with increased residence time. Percent adsorption for Nickel exhibits the highest % removal among the three.

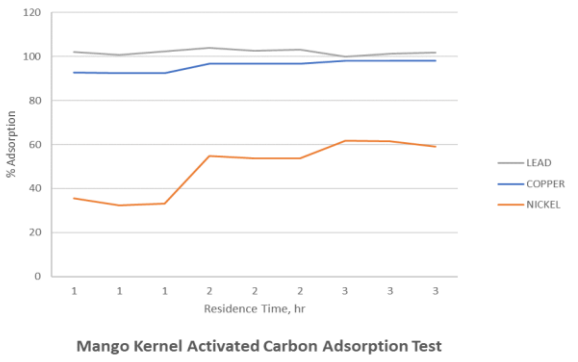

Figure 6. Variation of Residence Time vs % Adsorption of Cu, Ni, and Pb for KAC

Based on **Fig. 6**, it can be observed that percent adsorption of Copper, Nickel, and Lead,, for tri-component adsorption increases proportionally with increased residence time. Percent adsorption for Nickel exhibits the highest % removal among the three.

4 Conclusion

At x3,500 magnification both KAC and CAC showed increasing roughness and irregularity along with increasing residence time of the sample, which increases the physical adsorptive capacity of the samples for particles at this range of sizes. For KAC at x 5,000 magnification, sample 1 exhibited the most cavities but sample 2 was observed to have the most roughness and irregularities.

Fourier Transform infrared spectroscopy for KAC reveals stretching and bending of C-H and O-H bends as well as C-O at 1179.82 cm^{-1} and 982.83 cm^{-1}. Peaks for CACA were observed at 2346.3 cm^{-1} and 596.64 cm^{-1} due to C-H bend, O-H bend, and C-O stretch. This indicates the relative abundance of alkene aromatics, carbonyl-, and carboxyl- functional compounds on the materials' surfaces.

Atomic adsorption spectroscopy via batch adsorption was done using tri-metal solution of Cu, Ni, and Pb. A plot of % adsorbed versus Residence Time gives the following: maximum %adsorption for CAC is 64.91% (Cu, 3-HR), 22.04% (Ni, 3-HR), and 102.14% (Pb, 2-HR). For KAC, 98.00% (Cu, 3-HR), 60.79%(Ni, 3-HR),and 103.10% (Pb, 2-HR). This indicates that CAC and KAC with soaking time of 3 hours is a good adsorbent of Copper and Nickel, while soaking time of 2 hours yields the best adsorption conditions for both CAC and KAC.

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