



## PREPARATION OF ACTIVATED CARBON FROM *Syzygiumcumini* SEED FOR THE REMOVAL OF CHROMIUM (II) ION FROM AQUEOUS SOLUTION

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### ABSTRACT

**The most effective and low cost adsorbent produced from biomass materials is to adsorb heavy metal ions from aqueous solutions. The study aimed at utilizing chemically activated carbon from *Syzygiumcumini* seed plant for adsorption of chromium (II) ion from aqueous solution. The chemically produced *Syzygiumcumini* seed activated carbon using phosphoric acid ( $H_3PO_4$ ) was characterized using Fourier Transform Infrared Spectroscopy and Scanning Electron Microscope for its functional groups and morphology respectively. Batch adsorption was investigated to determine the optimum parameters such as pH, adsorbent dosage, and initial concentration and contact time of the produced activated carbon from the seed plant. The optimum adsorption was observed at contact time of 60 min, dosage of 1g and pH of 9. The result of the research shows that *Syzygiumcumini* seed plant has promising percentage removal of Chromium (II) ion from aqueous solution.**

**Keywords: Activated carbon, pH, *Syzygium*,  $H_3PO_4$ , Chromium (II) ion**

### INTRODUCTION

Industrialization is the major source of inclusion of heavy metals ions into the environment especially water bodies all over the world. It is well known that heavy metals such as chromium (II) ion amongst others can cause serious damages to the nerves, liver and bones (Nourbaksh *et al.*, 2005). Also, acute systemic poisoning can result from high exposure to hexavalent chromium (Ulmanu *et al.*, 2003). The presence of chromium (II) ion in surface and ground water is very hazardous to the environment due to its high potentiality in contaminating drinking water sources. Therefore, the use of activated carbon produced from local sources such as plant materials will be of help in removing these heavy metals from water bodies.

Activated carbon also called activated charcoal is a form of carbon processed to

have small low- volume pores that increases the surface area available for adsorption or chemical reaction. Activated carbon is sometimes substituted with active due to its high degree of micro-porosity, one gram of activated carbon has a surface area in excess of 3.00m as determined by gas adsorption (Bailey *et al.*, 1999). A variety of activated carbons are commercially available but very few of them are selective for heavy metals and most of them are very costly (Mohan *et al.*, 2005).

Black plum (*Syzygiumcumini*) is a plant that originated from India, it is widely used by people to treat diarrhea, inflammation and diabetes (American Diabetes Association, 2009). The seed of *Syzygiumcumini* are moderately rich in protein (6.3 – 8.5%) and various phytochemicals along with flavonoids quercetin and rutin a well – known antioxidants (Ranjan *et al.*, 2011).



g. 1: Black plum before crushing



Fig. 2: After crushing

Adsorption is an effective and friendly method for removing both organic and inorganic pollutants from waste water. This solves the problems of sludge disposal and renders the system more economically viable, especially if low cost adsorbents are used (Ovasif *et al.*, 2013). Therefore, this paper aimed at studying the potential of chemically produced activated carbon from black plum for waste water treatment.

## MATERIALS AND METHOD

### Sample Collection

The samples of black plum were collected from Okehi Local Government Area of Kogi State, Nigeria.

### Sample Preparation

The collected samples were washed with distilled water and air dried at room temperature in the laboratory. Thereafter, the samples were oven dried at 110°C for 24 hours, after which they were pulverized into fine powdered form using grinding machine at a frequency of 35.4HZ for one hour. The samples were sieved using a standard sieve size of 125µm shaken by a sieve shaker for 20 minutes. The sieved samples were then used for the preparation of activated carbon.

### Production of Activated Carbon

The one step pyrolysis method was used in preparing activated carbon. Exactly, 30g of

the sample was weighed for the first part on an electronic Ohaus weighing balance and poured into the crucibles. The initial weight of the crucible and crucible with samples was taken. 10% of phosphoric acid was added to the sample that's 50ml of phosphoric acid onto 50g of the sample in a crucible. The impregnated sample was dried with the aid of an oven at 110°C for one hour and then was transferred to a muffle furnace maintained at 500°C for carbonization. The hot crucible with the carbonized sample was removed from the muffle furnace and was allowed to cool in a desiccator. The pyrolysed carbons was leached with 2% hydrochloric acid for one hour and was washed several times with distilled water to remove the ash, residual  $H_3PO_4$  and HCl from the prepared carbon until it was neutral (Dada *et al.*, 2012). The wash carbon collected was dried in an oven at 110°C for two hours. The weight of the activated carbon was noted after activation to estimate the loss in weight after carbonization. The dried activated carbon was weighed to get the percentage yield and it was stored in an airtight plastic sample bottle to prevent adsorption of dust and other particles from the atmosphere.

$$YC = \frac{W_f}{W_i} \times 100\% \text{ -----Equation (1)}$$

Where: YC = the percentage yield of carbon.  
 W<sub>f</sub>= the weight of carbon after activation.  
 W<sub>i</sub> = the weight of carbon before activation.

**Characterization of the Activated Carbon.**

**Scanning Electron Microscopy (SEM)**

Exactly, 1g of the activated carbon was coated with gold at 1mm for 1second to make it electrically conductive. The sample was then glued into an aluminum pin type sample holder designed for high resolution (≤17 nm) and placed at 2 mm working distance. The sample holder was placed in a chamber where it is irradiated with an acceleration voltage of 15 KV. The electrons released were detected by the Back Scattered Detector full (BSD full). The BSD full then transmitted the electrons current onto another detector on a monitor mounted PC.

**Batch Adsorption Experiment**

The batch adsorption was carried out by varying the dosage from 0.25 to 1.25g per 50mL of 100ppm Cr (II) and all the samples were mechanically agitated at 250rpm for different experiments carried out under room temperature. The adsorbent was

filtered using whatman filter paper and the filtrate was taking for analysis using Atomic Adsorption Spectrophotometer (AAS). The effect of pH, the effect of adsorbent dosage, effect of the contact time duration were measured in the same manner. The removal percentage (%R) of chromium (II) ion was measured using the following formula:

$$\%R = \frac{C_1 - C_2}{C_1} \times 10 \text{ -----}$$

**Equation(2)**

Where: C<sub>1</sub> = Initial Concentration of Chromium (ii) Ion in the Solution.  
 C<sub>2</sub> = Final Concentration of Chromium (ii) Ion in the Solution.  
 (% R) = Removal Percentage

**RESULTS AND DISCUSSION**

The result of the activated carbon using H<sub>3</sub>PO<sub>4</sub> showed twelve elements on the activated carbon and is only the carbon that has highest elemental composition as shown in **table 3.1** below:

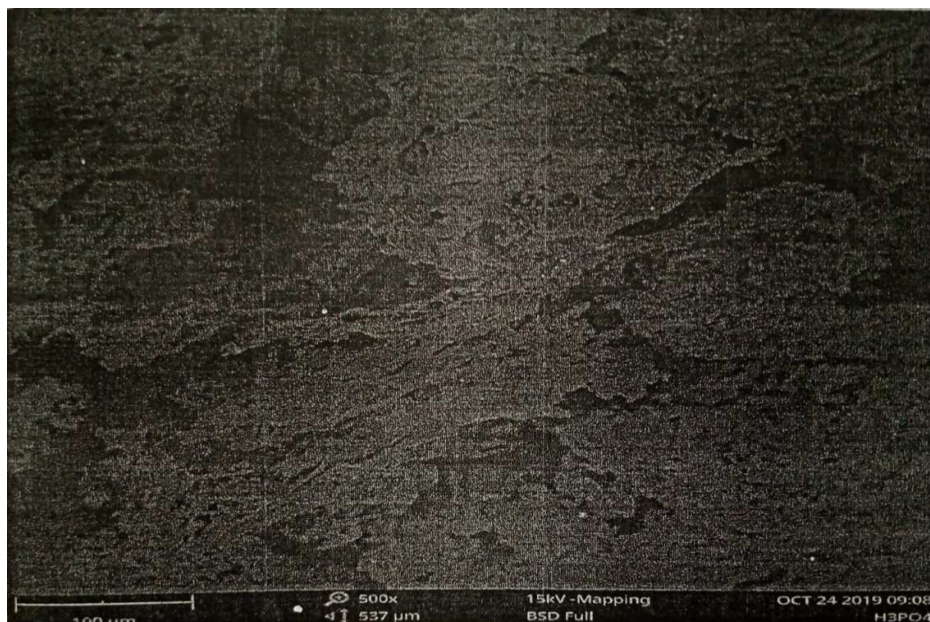
**Table 3.1:**Result of Scanning Electron Microscope of ASCS using H<sub>3</sub>PO<sub>4</sub>

S/No	Element Name	Atomic Conc. (mg/L)	Weight Conc. (mg/L)
1	Carbon	95.37	91.75
2	Oxygen	2.24	2.87
3	Phosphorus	0.41	1.02
4	Sodium	0.44	0.81
5	Calcium	0.19	0.63
6	Sulfur	0.23	0.59
7	Silicon	0.25	0.55
8	Magnesium	0.25	0.49
9	Aluminum	0.21	0.45
10	Chlorine	0.15	0.42
11	Nitrogen	0.20	0.23
12	Potassium	0.06	0.19

**KEYS:** ASCS Activated *Syzygiumcumini* Seed

The result of Scanning electron microscope (SEM) for morphology of the black plum (*Syzygiumcumini*) seed using phosphoric acid activation was mainly

uniform agglomeration with the average range of particle size distribution of 100pm at a magnification of 537 and 269\* as in **fig. 3** below:



**Figure 1: Scanning Electron Microscope of ASCS at 537µm**

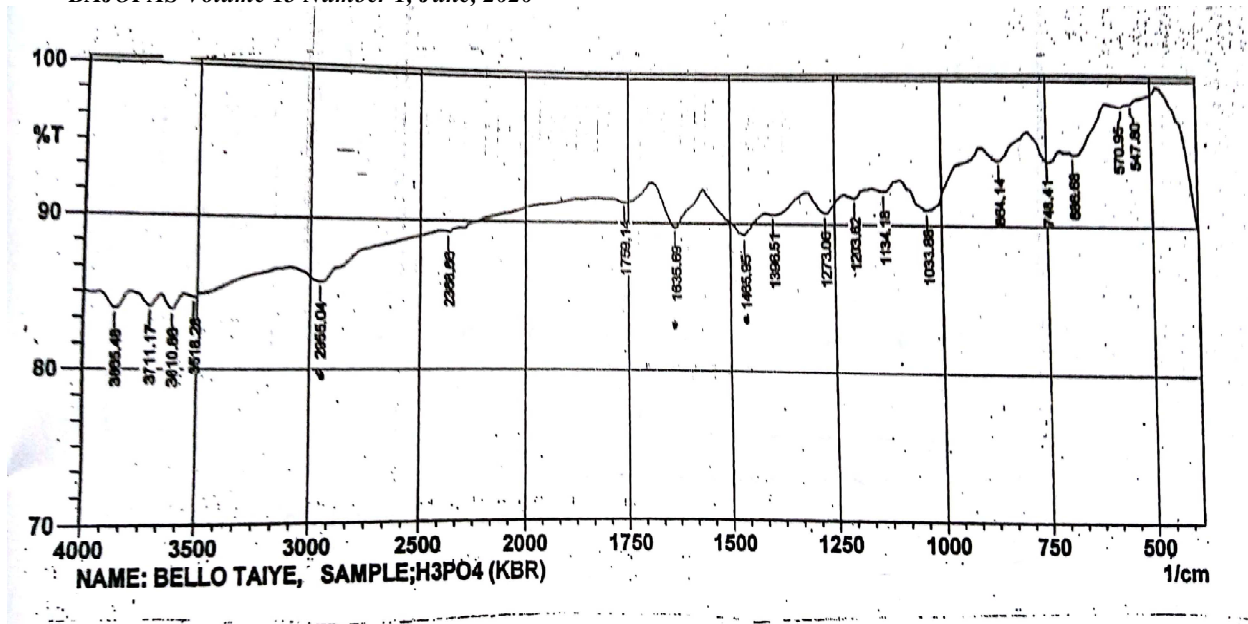
**Fig. 3:** Scanning electron microscope of the activated *Syzygiumcumini* Seed

The FTIR analysis permitted Spectrophotometer observation of *Syzygiumcumini* Seed in range of 4000 – 500cm<sup>-1</sup> and serve as a direct means for the identification of the surface functional groups as shown in table 3.2. The intensity of the peak decrease with increasing activation temperature. The position of 2955.04 – 1465.95 for ASCS is due to C – H

bonded groups of the alkanes. The peak of 1635.69 cm<sup>-1</sup> is attributed to N – H which indicate the presence of primary amines, the peak of 1277.06 is attributed to C-X indicate the presence of alkyl halides and lastly the peak of 1033.88 is attributed to C-O indicate the presence of alcohol on the functional group.

**Table 3.2:**Result of Fourier Transform Infrared Spectroscopy of ASCS using H<sub>3</sub>PO<sub>4</sub>

S/No	Peaks(cm <sup>1</sup> )	Bond	Functional Group
1	2955.04	C-H	Alkanes
2	1635.69	N-H	1 <sup>0</sup> Amine
3	1465.95	C-H	Alkanes
4	1273.06	C-X	Alkyl halides
5	1033.88	C-O	Alcohol



**Figure 4: Fourier Transform Infrared Spectroscopy**

**Effect of Adsorbent Dosage**

A series of adsorption experiments were carried out with different adsorption dosages varying from 0.25 to 1.25g at initial concentration of 50ml aliquots of a 100 mgL<sup>-1</sup>. The effect of carbon dose for the uptake adsorption by activated carbon from

black plum was found to decrease by increasing the adsorbent dosage. It was observed that adsorbent dosage of 1g has the highest percentage removal and adsorbent dosage of 0.25g has the lowest percentage removal as shown in table 3.3 below:

**Table 3.3:**Result of Effect of Adsorbent Dosage for Chromium (II) ion

Dosage	C <sub>1</sub> (mg/L)	C <sub>2</sub> (mg/L)	Removal (%)
0.25	21.7321	15.3479	29
0.50	26.6517	7.3348	72.4
0.75	41.0051	3.9329	90.4
1.0	41.2602	2.8869	94
1.25	44.3603	2.2255	93

**KEY:** C<sub>1</sub> = initial concentration and C<sub>2</sub> = final concentration

**Effect of pH**

The optimum pH absorption onto ASCS was found to be 9. pH9it was selected as the optimum value in the pH range. The sharpest increase in final concentration was observed between pH 7 and 9. These result

show suitability of the adsorbent for the treatment of basic wastewater. It was observed that pH 9 has the highest percentage removal and pH 3 has the lowest percentage removal as shown in table 3.4 below:

**Table 3.4:** Result of Effect of pH for Chromium (II) ion

pH	C <sub>1</sub> (mg/L)	C <sub>2</sub> (mg/L)	Removal (%)
3	46.7928	10.6414	22.8
5	37.3056	12.5388	66.3
7	26.6372	14.2725	46.7
9	13.3710	17.3258	78.6
11	20.49	15.9024	22.3

**KEY:** C<sub>1</sub> = initial concentration and C<sub>2</sub> = final concentration

**Effect of Contact Time**

The adsorption on activated carbon prepared from *Syzygiumcumini* increases with increasing agitation time, it assists to find out the time required for equilibrium attainment for Chromium. The removal rate increase within the first 50min then increases rapidly and gradually diminished

to attain equilibrium within 60min beyond which there was no significant increase in the removal rate. i.e. at 60 min the adsorption took place. It was observed that contact time of 60 min has the highest percentage removal and contact time of 10min has the lowest percentage removal as shown in table 3.5 below:

**Table 3.5:** Result of Effect of Contact time for Chromium (II) ion

Time (min)	C <sub>1</sub> (mg/L)	C <sub>2</sub> (mg/L)	Removal (%)
10	27.7517	14.4496	47.9
20	30.7227	13.8554	54.9
30	35.7272	12.8545	64
40	41.7413	11.798	71.2
50	45.3063	11.6517	72
60	45.3063	10.9387	75.8
70	45.3063	10.9387	75.8
80	45.3063	10.9387	75.8

**KEY:** C<sub>1</sub> = initial concentration and C<sub>2</sub> = final concentration

**CONCLUSION**

The research work investigated the potential of chemically activated carbon of black plum(*Syzygiumcumini*) seed for the removal of chromium (II) ion from aqueous

solution. The adsorption of Chromium (II) ion onto ASCS was found to be optimum at contact time of 60min, adsorbent dosage of 1g and pH 9.

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