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Article

Synthesis and Characterization of Activated Carbon Derived from *Pennisetum glaucum* Millet Waste for Adsorption Properties

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Abstract: Activated carbon has been widely developed as an adsorbent to adsorb heavy metal in waste water treatment due to its high efficiency and low cost. The present work reported the adsorption of Cr (VI) from aqueous solutions on activated carbon prepared from millet waste. The millet waste was dried and mill. The powder thus obtained was activated with H_2SO_4 and KOH and carbonized at 300, 400 and 500ºC for 30, 35 and 40min in an inert atmosphere. The effect of activation temperature shows an optimum temperature of 300ºC at the time of 30mins. Further studies revealed that the best impregnation ratio of carbon to potassium hydroxide (KOH) was 3:2. The results of the characterization show that the values of bulk density, ash content, moisture content, volatile content and fix carbon of the activated carbon show that millet waste especially the leaf and husk activated carbon are good precursor. The best adsorption performance is at the temperature 300ºC with 85, 88, 80 and 98% respectively. However the lowest adsorption performance was found to be at temperature of 500^0C with % removal of 50, 82, 50 and 78. For spike, stalk, leaf and husk respectively from the (AAS) Analysis results. Though they have low performance at 500ºC but the % removal is good.

Keywords: *Activated carbon, Adsorption, Wastewater, Millet waste and Heavy metals*

1. Introduction

World consumption of activated carbon was expected to rise with increasing the demand of industries. The largest applications of activated carbon were used in industries for purification and separation process such as removal of organic compounds, heavy metals, improving water treatments and raising the environmental standards [1]. Activated carbon has been a popular choice used as adsorbent compared to zeolites or other adsorbent types, because it has well developed pore structure, high adsorptive capacity and available in large quantity. Activated carbon can be manufactured from many types of raw materials which have high carbonaceous compound. In recent years, biomass waste materials with high cellulose and lignocelluloses promise a well-started material for preparation of bio-activated carbon [2]. [3]. Heavy metals such as Mercury, Lead, Copper, Cadmium and Chromium are toxic even in small amounts. Chromium (Cr), is one of the toxic metals, is used in many applications which include nuclear power plant, electroplating, water cooling and chromate preparation [4, 5]. Cr is consists of two stable oxidation states such as trivalent state Cr(III) and hexavalent state Cr(VI) in natural aqueous environment. It is well known that Cr(III) is an important materials for living organisms, however Cr(VI) is more toxic, carcinogenic and mutagenic [6-7]. The maximum levels permitted in wastewater are 5 mg·dm-3 for the trivalent Cr and 0.05 mg·dm-3 for the hexavalent Cr [8]. There are several processes for removal of metal contaminated wastewaters, including chemical precipitation, membrane filtration, reverse osmosis, ion exchange, and adsorption. But, they have a limited use as a result of some demerits [9]. Adsorption has been proved to be one of the most efficient methods for the removal of heavy metals from aqueous solution [10]. Activated carbon has shown tremendous potential for the removal of many inorganic, organic pollutants and radio nuclides removal due to properties such as large surface area, micro porous structure, and high adsorption capacity [11, 12]. It has also been used as an adsorbent to remove heavy metals from aqueous solutions and/or wastewaters. However, the low adsorption capacity of Cr(VI) on activated carbon has restricted its wide application. Recently, surface modification of activated carbon has been recognized as an attractive approach to improve the removal of heavy metals from wastewater [13, 14]. Millets are a group of highly variable small seeded grasses, widely grown around the world as cereal crops or grains for fodder and human food [15]. Millet are indigenous to many parts of the world [16]. The mostly widely grown millet is the *Pennisetum glaucum* also known as pearl millet, which is an important crop in India and part of African [17]. Generally *Pennisetum glaucum* are small grained, annual, warm weather cereals belonging to the family. They have high tolerant of drought and other extreme weather conditions and also have a similar nutrients content to the major cereals [18]. In this work *Pennisetum glaucum* millet waste was investigated it activated carbon yield and properties for use as raw material in water treatments for removal of Chromium metal.

2. Materials and Methods

2.1. Sample Collection and Treatments

Samples of the millet waste (stalk, leaf, spike and husk) were obtained from Bunza LGA in Matseri area close to the stream between Mungadi and Matseri in Kebbi State, Nigeria. The sample was washed with water several times to removal oil, solid particles, and followed by flushing with di-ionized water to remove all traces of impurities such as dust, sand and other smaller particles and then sun dried for 72 hours to remove the excess water. The sun dried sample was pulverized using mortar and pestle and then was allowed to pass through 0.2 mm sieve. 40g of the dried sample of (stalk and spike) was impregnated with 1.5M of sulphuric acid and (leaf and husk) 0.5M of potassium hydroxide was to impregnate at the acid and base/precursor ratio of 3:2(wt basis) respectively. The impregnated samples were then oven dried at a temperature of 85ºC for 5days.Then the impregnated dried samples was carbonized in a muffle furnace at a temperature of 500ºC, 400ºC and 300ºC for 30, 35,and 40min respectively and likewise for samples impregnated with KOH. And then it was cooled at an ambient temperature, the carbonized samples was washed with de-ionized water several times until pH of 6-7 was attained. It was then filtered using whatman filter paper. The activated carbon was oven dried at temperature of 105ºC for 24hr.

3. Results and Discussion

3.1. Results

The experimental results are listed in tables below.

Table 3.1: Result of the physical parameter of activated carbon derived from millet spike using H_2SO_4 Activating agent

Parameters	**Temperature °C**		
	300	**400**	**500**
Yield%	**62**	**58**	**56**
Moisture content%	**16**	**12**	**11**
Ash content%	**20**	**21**	**23**
Volatile matter%	**14**	**15**	**19**
Fixed carbon content	**50**	**52**	**47**
Bulk density	**0.4**	**0.6**	**0.7**

Table 3.2: Result of the physical parameter of activated carbon derived from millet stalk using H_2SO_4 Activating agent

Parameters	**Temperature °C**		
	300	**400**	**500**
Yield%	**64**	**59**	**52**
Moisture content%	**20**	**17**	**13**
Ash content%	**15**	**17**	**20**
Volatile matter%	**10**	**13**	**16**
Fixed carbon content	**55**	**53**	**51**
Bulk density	**0.6**	**0.78**	**0.9**

Table 3.3: Result of the physical parameter of activated carbon derived from millet leaf using KOH Activating agent

Parameters	**Temperature °C**		
	300	**400**	**500**
Yield%	**83**	**81**	**75**
Moisture content%	**8**	**6**	**5**
Ash content%	**14**	**16**	**18**
Volatile matter%	**5**	**7**	**9**
Fixed carbon content	**73**	**71**	**68**
Bulk density	**0.41**	**0.52**	**0.64**

Table 3.4: Result of the physical parameter of activated carbon derived from millet husk using KOH activating agent

Parameters	**Temperature °C**		
	300	**400**	**500**
Yield%	**86**	**80**	**60**
Moisture content%	**3**	**4**	**7**
Ash content%	**11**	**13**	**14**
Volatile matter%	**2**	**4**	**5**
Fixed carbon content	**84**	**79**	**74**
Bulk density	**0.52**	**0.57**	**0.68**

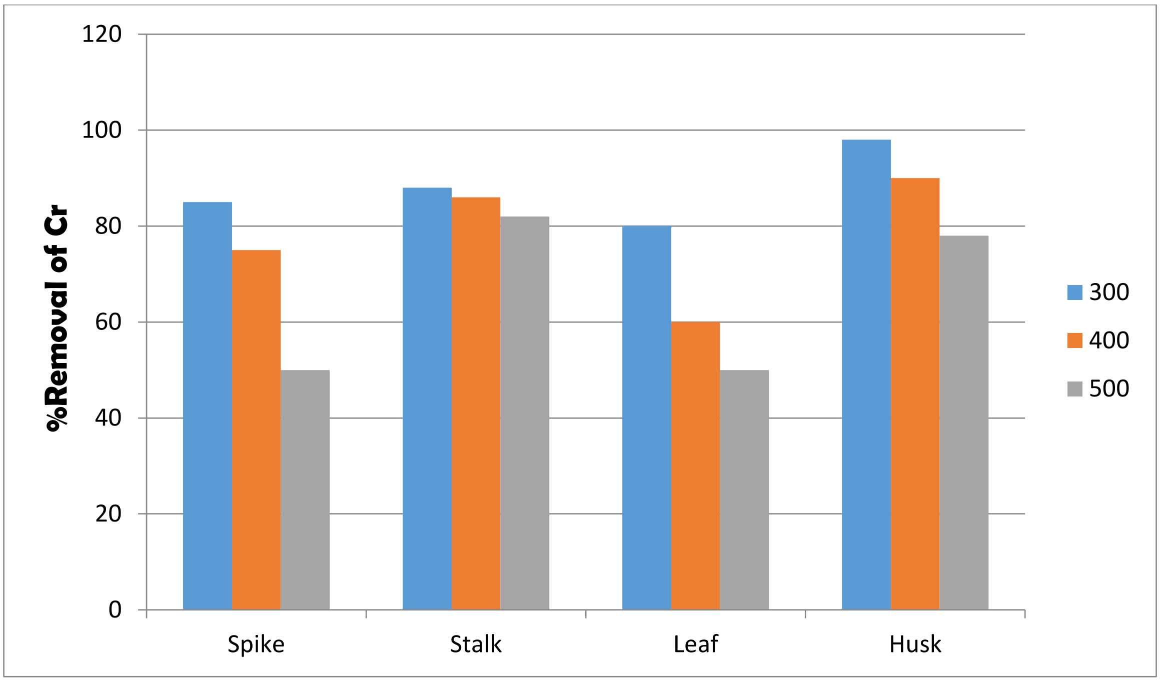

Figure 1: Percentage removal of chromium from Atomic adsorption Spectroscopy (AAS)

3.2. Discussion

3. 2.1. Yield

The results of the proximate analysis of millet waste (spike, stalk, leaf and husk) from table 3.1, 3.2, 3.3, and 3.4 at temperature of 300, 400, and 500^{0}C as well as the different times respectively shows that the higher the temperature with respect to time the yield decreases. Hence to obtain larger quantity of the activated carbon high temperature can be use with short time.

3.2.2. Moisture

Activated carbon is generally towards moisture free basis, although occasionally reported in some studies with some moisture content of 3, 8 or 10 % [3]. This may be due to the humid conditions that the activated carbon adsorbs considerable moisture over period of time. They may adsorb as much as 30 % to 38 % of moisture and still appear dry. Moisture content of activated carbon does not affect the adsorptive power, but obviously it dilutes the carbon [7]. Moisture content increases with higher activator concentration from the results of the proximate analysis of millet waste (spike, stalk, leaf and husk) from table 3.1, 3.2, 3.3, and 3.4 respectively. This is due to the dissolving process of residual pyrolysis and organic minerals on the surface of activated carbon which will get better as the activator concentration increases. Increased concentration of activator will increase the amount and volume of pores and surface area, which cause an increase in the performance of activated carbon to absorb water from air [19]. The increase in pyrolysis temperature will cause the C and H bonds in charcoal to be released completely.

3.2.3. Ash content

The results of the ash content analysis of millet waste (spike, stalk, leaf and husk) from table 3.1, 3.2, 3.3, and 3.4 respectively. The lower the ash content, the better the activated carbon because ash content can lead to increase hydrophilicity and can have catalytic effects, causing restructuring process during regeneration of used activated carbon[27]. The presence of excessive ash can lead to clogging of the pores on the activated carbon so that the surface area of activated carbon becomes reduced [20]. Ash content increases with increasing temperature so that organic compound of activated carbon decreases, however the content of inorganic compound is relatively fixed. However the ash content is influenced by the silica content of the raw material. This would mean that the higher the silica content, ash content is also higher [26].

3.2.4. Volatile matter

The results of the volatile content analysis of millet waste (spike, stalk, leaf and husk) from table 3.1, 3.2, 3.3, and 3.4 respectively. The high volatile content carbon makes this materials good precursor for production of activated carbon. The levels of volatile substances decrease as temperature increases. This is because the addition of activators causes changes in the structure and properties of activated carbon absorption [21]. KOH activators cause degradation of organic material that weakens the surface structure of activated carbon. In addition, this activator also releases volatile substances and develops an active carbon micro pore structure. At higher temperatures, volatile materials will be released into more and cause less carbon to be formed [22]. This is due to higher pyrolysis temperatures, so that the decomposition of components contained in raw materials such as water, tar, and volatile materials is also higher [23].

3.2.5. Fixed carbon

The results of the fixed carbon analysis of millet waste (spike, stalk, leaf and husk) from table 3.1, 3.2, 3.3, and 3.4 respectively. Which was physically activated under the optimum preparation conditions. According to [21], the high fixed carbon makes this materials good precursor for production of activated carbon. The value of the resulting fixed carbon content fluctuates as the pyrolysis temperature increases from 300, 400 and 500^{0}C at different time. Fixed carbon content is also affected by cellulose and lignin content that can be converted to carbon atoms. At a higher temperature there is a decrease in fixed carbon caused by the breakdown of activated carbon structures with high temperatures or burning of carbon. Decrease in carbon content can also be caused by the reaction between the carbon and the activator at high concentrations, which can damage the micropore on the carbon surface [24].

3.2.6. Bulk density

The observed bulk density, of the results of the proximate analysis of millet waste (spike, stalk, leaf and husk) activated carbon from table 3.1, 3.2, 3.3, and 3.4 respectively. The relatively low density of the activated is attributed to the random arrangement of micro-crystallites and with strong cross-linking which produce porous structure [25].

3.2.7. Percentage removal of chromium from Atomic adsorption Spectroscopy (AAS)

It can be seen that from Figure 3.1 that the % removal of chromium using the derived activated carbon from millet waste (Spike, Stalk, Leaf and Husk). The best performance is at the temperature 300ºC with %removal of 85, 88, 80 and 98% respectively. However the lowest performance was found to be at temperature of 500ºC with % removal of 50, 82, 50 and 78. For (Spike, Stalk, Leaf and Husk) respectively. Though they have low performance at 500ºC but the removal is good. And also similar result was obtained by [28].

4. Conclusion

It can be said from this study that among the two activating agents used in the preparation of the activated carbon from millet waste, potassium hydroxide was the best activating agent. The production conditions of 300ºC carbonization temperature for 30 min and activation temperature of 85ºC, for 30min with impregnation ratio of 3:2 favored the preparation. The characterization of activated carbon from millet waste in terms of bulk density, ash content, moisture content, fixed carbon, yield, volatile matter, bulk density, respectively of the millet husk and leaf using KOH activating agent are better when compared with the stalk and spike using H_2SO_4 activating agent. Therefore it can be concluded that the leaf and husk derived activated carbon can be said be the best carbon produce because of their high fixed carbon and volatile content. But from the adsorption studies of the (AAS) it shows that all the waste are good precursor for activated carbon production. Hence millet waste is a good potential source of activated carbon on the other hand and the use of it to produce activated carbon helps in solving environmental pollution problems.

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