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Utilization of Wheat and Pigeon Pea Husk Adsorbents to Remove Copper and Nickel from Industrial Wastewater

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ABSTRACT

The importance of the removal of heavy metals from industrial wastewater has received immense attention due to the increase of industrialization and heavy metals potential to harm human health. Different conventional methods are used for removing heavy metal contamination, but these methods have several disadvantages. Adsorption process is a simple, economical, and effective method to remove toxic heavy metals from industrial wastewater, but conventional adsorbents are neither readily available nearby nor economic therefore agro-based adsorbents are developed with chemical and thermal treatment. Developed adsorbents from the wheat husk and pigeon pea husk are used in filter units as filter media in combination with sand and aggregate. Activated wheat husk and pigeon pea husk adsorbents significantly removed Cu with 75.42% and 77% efficiency respectively and Ni with 72.50% and 74.17% respectively from industrial wastewater. This paper also studied the removal efficiency by varying pH and turbidity in synthetic solutions. The extent of removal of heavy metals by developed agro-based adsorbents is also correlated by proximate analysis, fixed carbon content 30 to 32%, bulk density 0.42 to 0.61 g/cc, iodine number 75.34 to 852.31, and chemical composition of adsorbents containing SiO_2. Characterization of prepared adsorbents also supported that developed adsorbents have great potential to carry the adsorption process to remove heavy metals from water and wastewater.

Key words: *Adsorption, Agro-based adsorbents, Heavy metals, Proximate analysis, Column experimentation.*

Introduction

Around the world, environmental contamination brought on by the buildup of heavy metals in water and wastewater has become a severe issue. Urbanization and industrialization have significantly increased heavy metal pollution, which has a negative impact on living things. Significant environmental and health issues arise when large concentrations of heavy metals are released into water bodies. The most common heavy metals in wastewater include copper, nickel, silver, chromium, etc. These metals affect the central nervous system of the human body and are known to cause many health issues such as gastrointestinal disorders, vomiting, convulsion depression, etc. Uses for the dangerous heavy metal copper include electroplating, plating, smelting, and other processes. Although copper is necessary for living things at low concentrations, in greater concentrations it can be poisonous. Basically, the concentration of these heavy metals is treated by means of conventional methods such as ultra-filtration, re-

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verse osmosis, ion exchange, and chemical precipitation. Due to its simplicity, viability, and affordability, the adsorption process has been identified as one of these technologies that shows promise. Adsorption occurs when a gas or liquid solute builds up on a liquid or solid surface known as the adsorbent, creating a molecular film known as the adsorbate. Additionally, this method may eliminate various pollutants, making it a more effective tool for reducing water pollution. The elimination of heavy metals and metalloids now has a potential alternative in the form of bio-sorbents. Many adsorbents have been employed in the removal of heavy metals from effluents in recent years, but efficiency and economy must be attained. Agricultural wastes like wheat husk and pigeon pea husk are abundantly available in nearby areas and have good potential to be activated carbon and adsorbents.

Adsorbents used for the removal of heavy metals (Copper and Nickel): Zeolites are natural or synthetic crystalline aluminous silicates also used as an adsorbent. High-activation adsorbents include silica gel. Silica gel is an amorphous, stable, non-toxic, and chemically inert form of silicon dioxide (SiO_2). Many conventional techniques, including chemical precipitation, chemical oxidation, reverse osmosis, ion exchange, and electrolysis, can effectively remove heavy metals from a substance. One of the potential methods for removing heavy metals is the adsorption of industrial and agricultural waste materials. As bio-sorbents, algae, fungi, and bacteria are also employed (Lakherwal, 2014). The advantages of this technology are assessed for the heavy metal removal from wastewater using the available adsorbent materials. Here, natural adsorbents like clay and bio-sorbents like a banana peel, astragalus, and chestnut shell as adsorbent materials for removing various heavy metal ions from water and wastewater, artificial materials such as metal oxides, zeolites, and carbon nano-materials are also being considered. Results showed that the majority of heavy metal ions could be successfully removed by these adsorbents. The removal of heavy metals from industrial wastewater can be done in several ways, including adsorption, precipitation, ion exchange, reverse osmosis evaporation, and bio-sorption. However, it has been noted that magnetic nano-materials functionalized with biopolymers, including chitosan and cellulose, have been used to remove hazardous metals from aqueous solution instead of heavy metals using diverse agricultural wastes, such as rice, straw, and coconut shells (Jane, 2015). The recovery of Ni by column operation is higher than the batch procedure when using orange peel to remove and recover Ni from electroplating effluent. Basic impacts of different parameters, including pH agitation, speed, and contact duration, were observed over the banana peel. The best results were obtained at pH 7,100 rpm and 90 minutes of contact time. For removing Fe from an aqueous solution, use pomegranate peel carbon bio-sorbent. When utilized for treatment or removal, the inexpensive adsorbents are quite effective (Jain, 2015). A batch experiment is superior for determining the effects of different adsorption settings on the removal of lead metal ions by agricultural wastes. Maximum adsorption requires two hours of contact time, and the type and quantity of the bio-sorbent determine how well metals are removed. The removal of heavy metals from wastewater has been done using agricultural and industrial waste byproducts like rice husk and fly ash. The removal of heavy metals can be done using a variety of adsorbents, which can be employed as inexpensive adsorbents. To remove heavy metals from water and wastewater, agricultural waste can be used as a low-cost natural adsorbent. Optimization factors such as contact time, pH, temperature, particle size, and starting ion concentration are crucial for assessing capacity, efficiency, and viability. Research and validation are required for the Freundlich, Langmuir, and Temkin adsorption isotherms. Use of Wheat Husk as an Adsorbent: Heavy metal ions can be removed from wastewater by using wheat husk as an adsorbent. There are significant quantities of wheat husk everywhere, but notably in rural areas. Phosphoric acid is used to make wheat husk-activated carbon, which is useful for the efficient adsorption of Copper and Nickel ions. Time, pH, adsorbent dosage, and beginning concentration all have a role in the adsorption of copper and nickel. Adsorbent made from pigeon pea husk: The adsorbent made from pigeon pea husk seems to be successful in removing Cu and Ni from the synthetic solution. The preheated adsorbent created by chemical activation with phosphoric acid is evidently a successful adsorbent for the removal of heavy metals. Pigeon pea husks contain between 50 and 60 percent carbohydrates, which have the potential to be employed as metal adsorbents. Pigeon Pea husk can be used to make an efficient metal adsorbent by simply carbonating them. Cu was thought to have been adsorbed by the adsorbent in an endothermic manner (Wan and Hanafiah, 2008). Due to its superior chemical and physical qualities, activated carbon can be used to cleanse waste gases as well as adsorb organic compounds, heavy metals, and other contaminants. Dehydration and carbonization of the raw materials are followed by activation, which is effectively a two-phase process, to produce activated carbon. The typical ingredients used to make the activated carbon utilized in wastewater treatment are peat, coconut shell, sawdust, wood char, lignin, banana peel, etc. In the region of Vidharbha, Maharashtra, India wheat husk and pigeon pea husk are abundantly available and possess good potential to be good adsorbents. Activated carbon prepared from the waste wheat husk and pigeon pea husk is used to remove Cu and Ni from synthetic solution by adsorption. At a temperature of 280°C - 330 °C most of the adsorbents will be activated after chemical treatment which helps to increase the efficiency of removal of heavy metals (Bhagat and Khandeshwar, 2019).

Materials and Methods

Preparation of adsorbents from the wheat husk and pigeon pea husk: The abundantly available agriculture waste materials such as pigeon pea husk and wheat husk were collected from agricultural fields. The collected materials were washed first with distilled water to remove soluble impurities and other particulate matter.

The materials were dried for 24 hours in an open environment. After drying, 500g material is allowed to soak in 500 ml of 1% HCl. At the end of 24 hours, the collected materials were again washed with distilled water and dried in the open air. The resultant dried materials were kept in a muffle furnace at a temperature of 250-320 ºC for 4 hours. It was then later powdered and sieved through a 4.75 mm IS sieve to acquire agro-based adsorbent.

Preparation of synthetic solution: The concentration of copper and nickel in industrial wastewater is generally 12 mg/l and 6 mg/l respectively therefore synthetic solution prepared containing copper 12mg/l and nickel 6 mg/lwere prepared simultaneously to study the adsorption removal efficiency of adsorbents. Copper standard solution and Nickel Standard solution is used to make stock solution.

Two filter units are fabricated to carry out column experimentation work where in transparent acrylic pipe, sheet and iron stand used. Layers of aggregate, adsorbents are placed as shown in figure.

A. Procurement and drying of pigeon pea husk and wheat husk

B. Washing and soaking of agricultural material PPH and WH

C. Developed adsorbent after chemical and thermal treatment

E. *Standard nickel and copper solution are used for the preparation synthetic solution*

Fig. 1. Development of adsorbent and preparation of synthetic solution

Prepared synthetic solution similar to industrial waste are filter through filter nut to remove the heavy metals.

Results and Discussion

Characterization of Material is necessary to know the potential of the material to carry out the adsorption process. It is also necessary to co-relate the re-

Table 1. The specifications of the filter unit used in the present study

Sr. No.	Component of filter unit and media layer	Depth
1.	Fine Aggregate	2 cm
2.	Activated Carbon/Adsorbent	10 cm
3.	Fine Aggregate	4 cm
4.	Coarse Aggregate	4 cm

moval with properties of material. The first proximate analysis is carried out and the results are shown in Table 2.

The fixed carbon content in both the agricultural

Fig. 2. Filter unit containing adsorbent as filter media

waste material is found 30 to 32% which is good. Iodine number is 756.34 mg/g and 852.31mg/g for PPH and WH respectively. The above characteristics of the material help in identifying the potential of the material to get efficient adsorbents used in adsorption experiments.

The residual carbon present in the sample aids us in determining the quantity of moisture and ash content. It demonstrates that while the ash level of activated carbon made from the wheat husk is low, the moisture content is considerable. The adsorbent's ability to float is provided by its bulk density. Basically, bulk density has an impact on how well heavy metals may be absorbed. In terms of the adsorbent's floatability feature, it offers a bird's-eye view. According to this theory, adding activated carbon to water will cause it to sink, improving its contact with the adsorbate and facilitating an efficient adsorption process. A substance's adsorption capacity is directly inversely related to its bulk density. The better the adsorption process will be, the higher the bulk density. Unwanted has a high ash content. The adsorptive capacity of activated carbon can be assessed quickly and easily using the iodine number. Pigeon peas and wheat husks have sufficient iodine number and that indicates good ability if adsorbents are developed from it. High fixed carbon content in both materials is also advantageous from an adsorption point of view. Filter unit con-

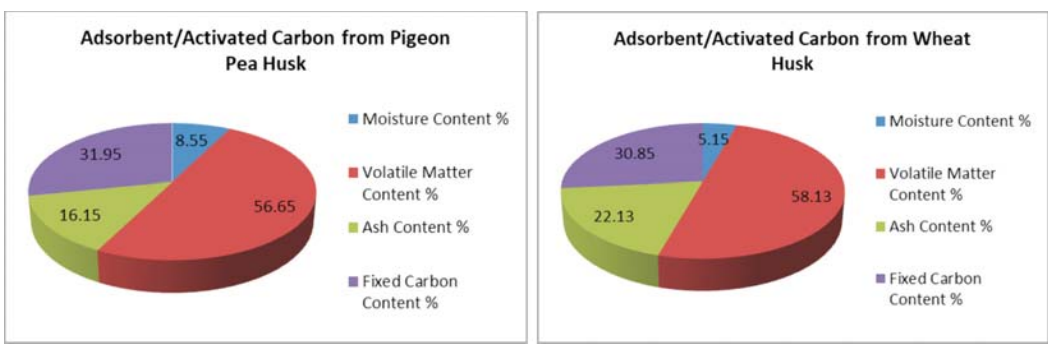

Fig. 3. Proximate analysis of pigeon pea and wheat husk

Table 2. Proximate analysis of the agricultural material and its characteristics

Sr. No.	Characteristics	Adsorbent/Activated Carbon from Pigeon Pea Husk	Adsorbent/Activated Carbon from Wheat Husk
1.	Moisture Content %	8.55	5.15
2.	Volatile Matter Content %	56.65	58.13
3.	Ash Content %	16.15	22.13
4.	Fixed Carbon Content %	31.95	30.85
5.	Bulk Density (g/cm^3)	0.61	0.42
6.	Iodine No. (mg/g)	756.34	852.31

taining adsorbent as filter media: The filter unit consists of an acrylic circular pipe of 10 cm diameter. It consists of coarse aggregate, fine aggregate, activated carbon, and again a small layer of fine aggregate i.e., sand. Each layer in the filter unit was separated by a net so that the materials of different layers do not get mixed and there is the continuous passage of water through the filter.

Effect of pH: The pH scale monitors the concentration of hydrogen ions, which reveals whether a solution is acidic or alkaline and has an impact on metal adsorption. The pH scale typically ranges from 0 to 14. At 25 ^{o}C, basic or alkaline aqueous solutions have a pH above 7, while acidic aqueous solutions have a pH below 7. Neutrality is defined as a pH of 7.0 at 25 ^{o}C. A very strong acid may have a pH below 14, while strong bases may have a pH beyond 14.

Initially pH in synthetic solution was less which is acidic but after passing through filter unit pH of solution is increased upto 7. The following changes in pH were observed while the removal of metals from the synthetic solution similar to water and wastewater by adsorption.

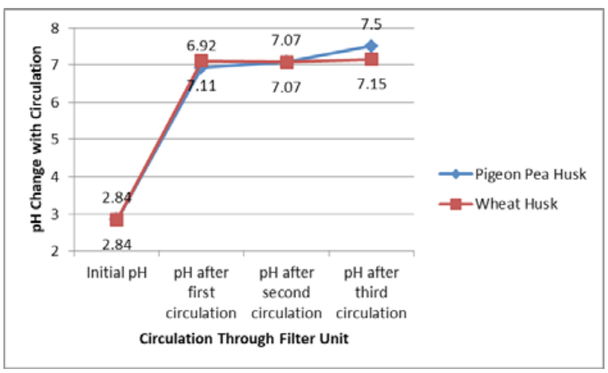

Fig. 4. Changes in pH during the column experiment study

The results of the pH test shows that after passing the wastewater from the filter unit, the pH of the wastewater tends to become neutral pH with each circulation.

Effect of turbidity on metal adsorption: Turbidity is a term used to describe the cloudiness or haziness that a fluid exhibits due to numerous tiny particles that are normally invisible to the unaided eye. The turbidity of the water and wastewater is a key factor in the evaluation of its quality. Nephelometric turbidity units are the most popular unit of turbidity measurement (NTU). There are several methods for determining the turbidity of the water, with the most straightforward being a measurement of the attenuation, or weakening, of a light source as it passes through a water sample. The following changes in the turbidity while removal of metal ions was observed.

There is a significant increase in turbidity of the wastewater after each circulation from the filter unit. This is because of the use of activated carbon. However, the turbidity can be easily removed by various techniques such as filtration and reverse osmosis.

Using the X-ray fluorescence (XRF) analytical technique, which is non-destructive, it is possible to determine the elemental makeup of materials. The elemental composition of a material sample can be determined qualitatively and quantitatively using X-ray fluorescence analysis. It can also be used to measure coatings and coating systems.

When primary X-rays excite the atoms in a material sample, electrons from the innermost shells are liberated, and the resulting vacancies are then filled by electrons from the outer shells. This occurrence forms the basis for XRF analysis. Constituent SiO_2content is more inwheat husk (70.10%) than pigeon pea husk (38.60). Inmany study it was a

Table 3. Changes in pH during the column experiment study

Sr. No.	Adsorbent Material	Initial pH	pH after first circulation	pH after second circulation	pH after third circulation
1.	Pigeon Pea Husk	2.84	6.92	7.07	7.50
2.	Wheat Husk	2.84	7.11	7.07	7.15

Table 4. Changes in turbidity (NTU)

Sr. No	Adsorbent Material	Initial Turbidity	Turbidity after 1^{st} circulation	Turbidity after 2^{nd} circulation	Turbidity after 3^{rd} circulation
1.	Pigeon Pea Husk	0	0	1	1.2
2.	Wheat Husk	0	0	1	1.3

Table 5. Elemental analysis by XRF

Sr. No.	Constituents	Pigeon pea husk (% wt.)	Wheathusk (% wt.)
1.	SiO_2	38.60	70.10
2.	CaO	8.01	0.15
3.	MgO	0.30	0.016
4.	Al_2O_3	25.879	1.46
5.	Fe_2O_3	11.30	0.19
6.	TiO_2	1.46	0.024
7.	S	9.02	1.30
8.	P	2.38	19.60

concludedthat more SiO_2 means good for adsorption.

The XRF analysis reveals the SiO_2 content of the pigeon pea husk and wheat husk. Since the SiO_2 content is more, the capacities to absorb the adsorbates are more.

Initial concentration of Cu in synthetic solution was 12 mg/l, after third circulation through filter containing wheat husk adsorbent reduced the Cu concentration to 2.95 mg/l which is 75.42 % removal. Pigeon pea husk adsorbent reduced the Cu

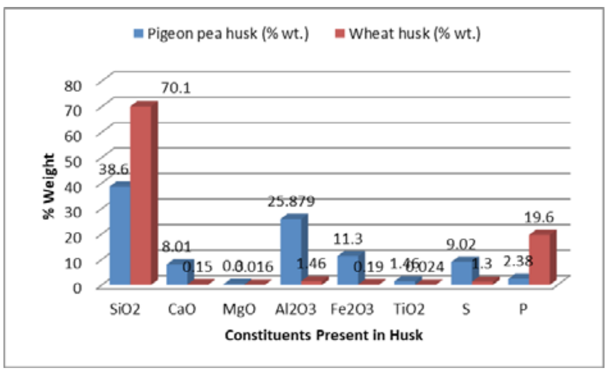

Fig. 5. Constituents present in husk by XRF analysis

level to 2.74 mg/l which is 77.17%.

Initial concentration of Ni in synthetic solution was 6 mg/l, after third circulation through filter containing wheat husk adsorbent reduced the Ni concentration to 1.65 mg/l which is 72.50% removal. Pigeon pea husk adsorbent reduced the Ni level to 1.55 mg/l which is 74.17%.

As per WHO industrial effluent standards Cu level must be less than 3 mg/l and in above results indicate that after third circulation Cu level is 2.95mg/l and 2.74 mg/l therefore above developed adsorbents results satisfy the norms

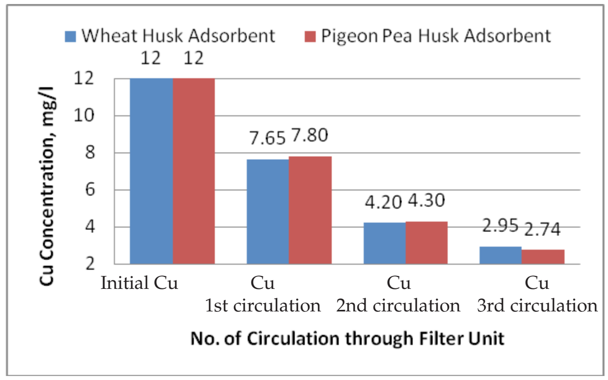

Fig. 6. Removal of Copper by wheat husk and pigeon pea husk

As per WHO industrial effluent standards Ni level must be less than 3 mg/l and in above results indicate that after third circulation Ni level is 1.65 mg/l and 1.55 mg/l therefore above developed adsorbents results satisfy the norms.

Heavy metal removal by pigeon pea husk for Cu is 77.17% and for Ni is 74.17% which slightly more than wheat husk adsorbents 75.45% in case of Cu

Table 6. Removal of Copper by wheat husk and pigeon pea husk

Sr. No.	Agro-based adsorbents	Initial Cu, mg/l	Cu after 1st circulation, mg/l	Cu after 2nd circulation, mg/l	Cu after 3rd circulation, mg/l	Cu removal efficiency, %
1	Wheat Husk Adsorbent	12	7.65	4.20	2.95	75.42
2	Pigeon Pea Husk Adsorbent	12	7.80	4.30	2.74	77.17

Table 7. Removal of Nickel by wheat husk and pigeon pea husk

Sr. No.	Agro-based adsorbents	Initial Ni, mg/l	Ni after 1st circulation, mg/l	Ni after 2nd circulation, mg/l	Ni after 3rd circulation, mg/l	Ni removal efficiency, %
1	Wheat Husk Adsorbent	6	3.14	2.36	1.65	72.50
2	Pigeon Pea Husk Adsorbent	6	3.05	2.25	1.55	74.17

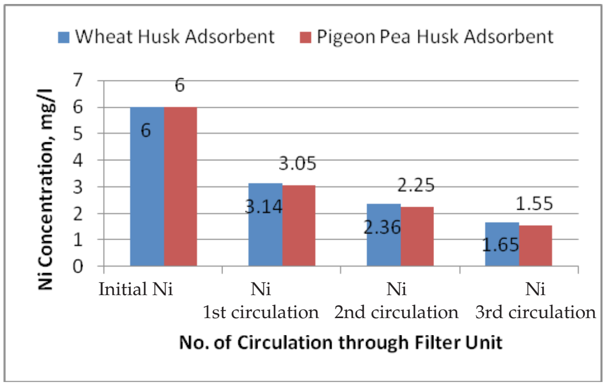

Fig. 7. Removal of Nickel by wheat husk and pigeon pea husk

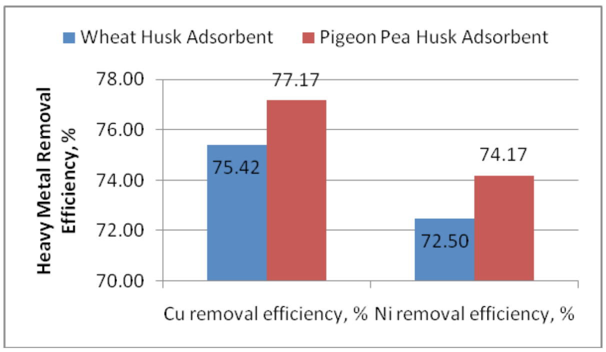

Fig. 8. Removal of Copper and Nickel in % by wheat husk and pigeon pea husk adsorbent

and 72.50% in case of Ni.

Both the developed adsorbents are efficient to remove the heavy metals and can make industrial wastewater safe for disposal in sewer, water body and on low laying area by reducing the heavy metals as per required by effluent standards..

Conclusion

The concentration of metal ions affects the removal of heavy metal. This study have been conducted to determine which adsorbents are most successful at removing hazardous metals like Cu and Ni from wastewater. Adsorption offers a lot of potential for removing heavy metals from water and wastewater especially industrial wastewater according to an assessment of several heavy metal removal techniques and adsorbents. Many characterization procedures, including proximate analysis, bulk density, pH, and iodine number test, are performed as per standard procure and found that fixed carbon in percentage will play role in adsorption and which is directly proportional to removal efficiency.A close investigation of activated carbon gives a solid sense of the sample's physical characteristics.In this study fixed carbon is 31.95% and 30.85% respectively for PPH and WH adsorbents. Iodine number must be more than 750 mg/ to have good adsorbents and in our test both material have the iodine number 756.37 mg/g and 852.31 mg/g for PPH and WH respectively. The adsorption column experiment which consist of filter unit containing adsorbent layer one of the major component for the removal heavy metals and after experiments found that pigeon pea husk adsorbent are 74.17% and 77% removal efficiency for Ni and Cu respectively. For what husk adsorbent, Ni and Cu removal efficiency are 72.50% and 75.42% respectively. Disposal of the spent adsorbents should be done in an environmentally friendly way. The cost factor is also an important parameter that should be considered before selecting such developed adsorbents for water pollution control. It was discovered that the adsorbent type, depth layer, initial concentration, pH, and turbidity all had an impact on how much of the heavy metals were removed. The sustainable adsorbents are easily accessible, regenerate when exposed to alkali, and must be burned off to completely remove heavy metals. From the adsorption test described above, it can be inferred that both materials-wheat husk and pigeon pea husk may be utilized successfully to remove heavy metals from industrial effluent or any wastewater.

Future Scope

Similarly in future many agricultural waste materials may be used from nearby area for development of adsorbents and their results are compared with

Table 8. Cu and Ni removal in % by developed agro-based adsorbents

Sr. No.	Agro-based adsorbents	Initial Cu, mg/l	Cu after 3rd circulation, mg/l	Cu removal efficiency, %	Initial Ni, mg/l	Ni after 3rd circulation, mg/l	Ni removal efficiency, %
1	Wheat Husk Adsorbent	12	1.14	75.42	6	1.17	72.50
2	Pigeon Pea Husk Adsorbent	12	2.10	77.17	6	1.75	74.17

economic feasibility and technical viability at large level for industrial waste water treatment specially for heavy metal removal. Need to increase the efficiency and adsorbent development at low cost with sustainability.

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Conflict of Interest: None.

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