Utilizing Agricultural-based Adsorbents to Remove Copper and Nickel from Industrial Wastewater

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ABSTRACT

Due to rapid industrialization, industrial wastewater quantity and heavy metals are increasing day by day and thereby new alternative methods for treatment of water and waste water are need of hour. Heavy metals can be removed from wastewater more effectively through adsorption, however this method requires a good adsorbent that is widely accessible nearby. The heavy metals copper and nickel are removed from industrial effluent using adsorbents made from agricultural waste in this research work. Adsorbents prepared from coconut shell and rice husk, which are considered as waste materials, can be effectively used for adsorption due to low cost and high availability. Chemical and thermal treatments are given to develop the effective adsorbents from coconut shell and rice husk. Potential of removal of heavy metals by agro-based adsorbents are also evaluated by various tests like XRD, XRF, proximate analysis, bulk density, iodine number. In characterization, behaviors of material are studied and developed adsorbents are found effective with good adsorption capacity for the removal of heavy metals. In this work, industrial wastewater with pH variations and treated. Adsorbent from coconut shell are 70% effective in removal of nickel and adsorbents from rice husk are 65% effective. Copper removal is 75-76% which is more than nickel by both the adsorbents. Both adsorbents bring down the metal ion concentrations in the industrial wastewater below permissible limits 3 mg/l. For the effective and inexpensive removal of various heavy metals, a variety of agro-based adsorbents made from locally abundant waste are helpful.

Keywords— Adsorbents, Coconut Shell, Heavy Metals, Industrial Wastewater, Rice Husk

I. INTRODUCTION

The removal of heavy metals and other pollutants from industrial waste water can be accomplished using a variety of methods. Chemical precipitation, ion-exchange, electro-floatation, membrane separation, reverse osmosis, electro-dialysis, and solvent extraction are a few examples of these techniques. Scientists are currently paying attention to the development of less expensive treatment methods that utilize adsorption phenomena, especially in developing nations like India, due to the high operating costs of these sorts of treatment approaches [1]. The development of inexpensive adsorbents for the treatment of wastewater containing heavy metals is being given attention in light of the aforementioned issues. In comparison to other approaches, adsorption is currently widely used in environmental remediation applications worldwide. Activated carbon adsorption has been recognized as a prominent and effective technology among all types of adsorption materials, however the procedure is expensive. In order to generate low-cost adsorbents with good sorption capacity to remove heavy metal ions from wastewater, more methods have been researched. The research of using agricultural products as adsorbents has received a lot of attention recently. Due to their low cost and great adsorption capacity for the removal of different aquatic contaminants, agricultural wastes made from coconuts have drawn a lot of attention as efficient bio-sorbents. Coconut shells make an ideal raw material source for the production of activated carbon due to their high carbon content and hardness. The least dusty and most suited for organic chemical adsorption are activated carbons made from coconut shells. They are primarily micro-porous. When compared to other types of activated carbon, coconut shell-based carbon has the maximum hardness, which makes it the best carbon for purifying water. A significant amount of rice husk production results from the processing of rice. A type of fibrous material called rice husk has a significant quantity of silicon, with a content of about 96.34%. The primary organic components of rice husk include cellulose, hemicellulose, and lignin. It is conceivable to think of rice husk as an adsorbent because of its composition. According to earlier studies, ionic dyes have been removed from aqueous solutions using rice husk, and cadmium has been sorbeted from effluents using rice husk that has been pretreated. In our preliminary research, using untreated rice husk, we investigated the adsorption of heavy metals. One of the important elements for humans is copper. In nature, it is abundantly distributed as metal. The three types of copper that can occur in an aquatic environment are soluble, colloidal, and particulate. It is a less common element that

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is nevertheless necessary for organisms. A dangerous level of copper in the human body results in pathological alterations in the brain tissues as well as hypertension, anaemia, liver and kidney damage, stomach irritability, and other conditions. Specific bone diseases are brought on by excessive copper consumption. The permitted copper levels in drinking water are 0.5 mg/L, 0.05 mg/L, and 1.3 mg/L, respectively, according to the WHO, BIS, and USEPA. According to both the USEPA and the CPCB in India, the highest permissible concentration level in industrial wastewater is 3.0 mg/L. Natural water contains nickel as a divalent cation with a pH range of 5 to 9. It is thought that plants and some domestic animals require nickel, the metal that makes up the enzyme urease. Nickel is a fairly hazardous element when compared to other transition metals [2]. Nevertheless, it is widely recognized that inhaling nickel and its compounds can cause major issues, such as lung cancer. Additionally, nickel can result in nickel-eczema, a skin condition. Concern over the quantity of heavy metals in natural streams has grown in recent years. The USEPA and WHO both set safe levels of
nickel in drinking water at 100 g/L and 70 g/L, respectively. Nickel in drinking water is permitted up to 0.02 mg/L according to Indian guidelines established by BIS 1998. By USEPA and CPCB, India, the maximum allowed content of nickel in industrial wastewater is 3.0 mg/L [2].

The Need of Water Treatment by Adsorption

Today, a variety of industrial sectors generate a large amount of hazardous waste. Heavy metals are used in water pollution and constitute a severe threat to the environment and public health because of their toxicity, accumulation in the food chain, and persistence in nature. Because of the ongoing changes in water quality brought on by growing industrial activity, there has been discussion about how to increase the current water treatment system's capacity to remove heavy metals. Among the methods used to treat water and wastewater advanced treatment. The high expense of operating these kinds of treatment technologies has, however, sparked interest in developing less expensive treatment procedures that make use of adsorption processes. Moreover, considering the huge quantity of agricultural waste produced in our country, it is necessary to reuse or dispose them off carefully and use of agricultural wastes as adsorbents is an efficient option. In this situation, activated carbon can be utilised to filter out harmful substances specifically and create an effluent that is typically biodegradable. Heavy metals are typically eliminated by this procedure since they are present in industrial effluent in large quantities [25].

Aim of Work

In the age of industrialization and technological breakthroughs, it is extremely important to treat wastewater using cost-effective and efficient methods. The different organic and inorganic contaminants present in the effluent necessitate a specialized and efficient treatment method. One such technique is adsorption using inexpensive adsorbent. Many researchers have utilised adsorption with success to remove organic waste, phenol, and heavy metals. Heavy metal concentrations in water have substantially increased as a result of industrial activities such electroplating, metal finishing, metallurgical manufacture, chemical manufacturing, and mining. The ecosystem is significantly impacted by the ions of heavy metals as mercury, lead, chromium, cadmium, nickel, copper, and zinc. They must be removed since they are extremely harmful when they are compounds or ions. To get undesired heavy metals out of the industrial wastewater is the main goal of the job. The process employed for this is adsorption, and the adsorbents used were made from coconut shell and rice husk [25].

Literature Review

In single, binary, and tertiary systems, Zn, Cd, and Hg ion adsorption on rice husk ash (RHA) has been studied. To examine the competitive adsorption features, batch tests for mono- and multi-component systems with different metal ions concentrations (mg/l) were also conducted. For its ability to adsorb mono component solutions of metal ions, rice husk ash (RHA) has been investigated. Testing has also been done on the applicability of the isotherm models Freundlich and Langmuir to forecast the equilibrium uptake of Zn, Cd, and Hg in mono, binary, and tertiary systems. [9]. The study looks into how uncooked rice husk might be used as an adsorbent to remove arsenic from drinking water. Models of Freundlich and Langmuir isotherms were used to analyse the adsorption behaviour. In order to determine the saturation concentration, fixed bed adsorption capacity, and time needed for 50% adsorbate breakthrough, three kinetic models—Adam-Bohart, Thomas, and Yoon-Nelson—were used. Finally, solidification was carried out to dispose of the rice husk [10]. Carbonized rice husk was also used in water and wastewater purification. Banana peels and M. thilapia fish scales are two natural adsorbents that are inexpensive and widely accessible that are used to absorb water. Waste water was treated using a combination of both of these adsorbents with varying height and contact duration ratios. Compared to the basic features of agro wastewater, iron content, potassium, TSS, turbidity, phosphorous, etc. This approach is very economical, economical, and environmentally beneficial. Using inexpensive adsorbents does this, aiding in the reduction of the water shortage [22]. Ni2+ was eliminated from aqueous solution using granular activated carbon. Additionally, the effects of the acid-modified carbon's Ni2+ adsorption capabilities were examined. In batch studies, the metal sorption properties of unmodified and modified granular activated carbon were determined. When compared to raw, granular activated carbon, the modified carbon had a higher capacity for adsorbing nickel ions [17]. By using finer activated carbon, or Ac-iii, there is a maximum reduction in the chemical oxygen demand, biological oxygen demand, and iron content of water. The decrease in cod and bod indicates that coconut shell activated carbon is effective for removing organic pollutants. In contrast, there was hardly any iron, bod, or cod decrease in the case of sand filters. Both types of filtration materials reduced turbidity in a nearly identical manner. In order to remove suspended particles, iron, and total organic carbon from water in the future, treatment plants may consider using coconut shell activated carbon instead of sand filtration. A trash that would otherwise be simply disposed will be used thanks to this technology, which is very useful, affordable, and cost-effective [11].

II. METHODOLOGY

Agricultural waste procurement: Copper was sorbent from aqueous solutions using rice husk, a surplus agricultural byproduct. Rice husk is a biomass material made of wood-like polymers, and its abrasive hardness is a common property of materials with a greater lignin content. Approximately 15-20% of the bulk of this substance is made up of inorganic material, of which SiO2 accounts for 95% or more. The rice husk has a strong potential for removing heavy metals such as Cu, Ni, and many more under experimental settings due to its high adsorption capacity, binding processes, and pretreatment techniques. Due to its easy availability, low cost, reusability, high efficiency, easy processing, application, and recovery without any negative environmental effects, the use of rice husk as a possible bio-adsorbent for metal remediation and environment management technologies has increased in recent years. The high adsorptions of rice husk even at low metal concentrations, lack of need for extra nutrients, ease of operation, and strong affinity and high selectivity towards heavy metals due to the presence of binding groups on its surface are a few additional benefits. In comparison to other standard bio-adsorbents, rice husk has an adsorption capacity that is at least as great, according to a number of research organizations. In addition, its use is creative and environmentally benign, making it a fantastic illustration of long-term trash management.

Preparation of Rice Husk Adsorbent

Retained rice husk was washed with distilled water to get rid of dirt and other impurities after sieving to eliminate particles smaller than or equal to one millimeter, and it was then dried for 24 hours as shown in Figure 1. In order to fully adsorb the chemicals onto the raw material, 500 g of this rice husk was combined with 500ml of H_3PO_4 solution and left to stand at room temperature for 24 hours. The modified rice husk was then filtered and repeatedly rinsed in distilled water after this process. This adsorbent was later dried in an oven at 110°C for four hours. The rice husk that had been treated with H_3PO_4 was then obtained, as shown in Figure 2.

Figure 1: Rice husk washed by distilled water

Figure 2: Rice husk activated carbon

Coconut Shell as Adsorbent

In countries where coconuts are harvested, like India, activated carbons are frequently supplied. These activated carbons are made utilizing coconut shells as the raw material. The pore structure of activated carbons made from coconut shells is often tighter and more microscopic than that of carbons made from coal. This occurs as a result of the coconut shell's natural pore structure, which is different from coal's natural pore structure. This activated carbon's micro-porosity makes it suitable for some applications. Additionally, compared to similar grades of coal-based carbons, coconut shell-based carbons are frequently tougher, more abrasion-resistant, and lower in ash. The primary benefit of using coconut shell activated carbon is that it excels in applications requiring the removal of taste, odor, and dissolved organic chemicals from water that contains suspended particles. Its balanced pore structure also gives the filtered water a high polish and a more effective adsorption range. As indicated in figure 3, the coconut shells were cleaned with distilled water and dried for 24 hours at 110 °C to minimize the moisture content. The dried samples were then broken into smaller pieces and sieved to a size of 1-2 mm. Coconut shells were then carbonized for two hours at a temperature of 320 °C without the presence of oxygen. Following carbonization, these samples were combined with water and KOH in a stainless steel beaker with a weight ratio of 1:2 for the KOH to the sample. Four hours of water evaporation at 130°C

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were followed by heating these dried mixes to 800°C and holding them there for an hour. After being cooled to room temperature, the items were cleaned with HCl and deionized water. Thus, the activated carbon made from coconut shells depicted in figure 3 was produced.

Figure 3: Prepared of coconut shell activated carbon

Filter Unit

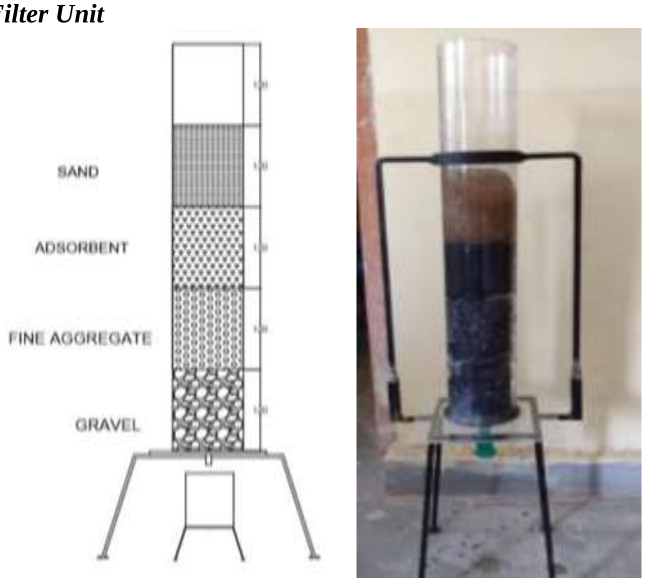

Figure 4: Filter unit consists of prepared adsorbent

A filter unit was prepared for the treatment of water. The filter unit, which was a circular acrylic pipe, consisted of coarse aggregate, fine aggregate, adsorbent material and sand placed bottom to top in order of decreasing grain size. 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 continuous passage of water through the filter. The specifications of the filter unit are as follows:

Table 1: Filter components with dimension

Filter unit	Diameter = 10cm Height = 60cm
Coarse aggregate	Size = 20mm Height = 12cm
Fine aggregate	Size = 12mm Height = 12cm
Sand	Size = 150-600 micron Height = 12cm
Adsorbent material	Height = 12cm

Proximate analysis: By using standard procedures, proximate analysis determines the contents of moisture, volatile matter, fixed carbon, and ash. Using the following method, the proximate analysis of the various types of activated carbon was completed.

Moisture Content: We then weighed a little amount of activated carbon made from coconut shells and put it in a petri dish. The spread of the dish was exquisitely even. Following that, it was baked for 1.5 hours at 105 to 110 C. The petri dish was either left open or unprotected during the heating process. After heating, the Petri dish was taken out and refrigerated in a desiccator. The dried sample's weight was determined after cooling [31].

M=100(B-F)/ Moisture content (B-G)

Where B = weight of petri dish plus original sample, F = weight of petri dish plus dried sample, and G = weight of petri dish The rice husk activated carbon's moisture content was also determined using the same process [30].

Ash content: A silica crucible was used to hold 1 g of the material. It was heated to 750 C for 1.5 hours in a muffle furnace. The crucible was left open as it heated up. The crucible was heated to the proper temperature, cooled in a desiccator, and then the weight of the ash was determined.

A=100(F-G)/ash content (B-G)

Where, G=Mass of a crucible that is empty, B = sample plus crucible mass, F = crucible mass plus a sample of ash. The rice husk activated carbon's moisture content was also determined using the same process.

Volatile matter content: In a cylindrical crucible with a lid on top, a known quantity of sample was taken. Following that, it was heated in a muffle furnace for precisely 7 minutes at 925 C. Following cooling in a desiccator, the crucible was weighted.

Fixed carbon content: It is calculated by Fixed carbon (FC) = 100 – (% moisture content+ % volatile matter + % ash content)

Bulk Density: Weight per volume of a substance is referred to as bulk density. Generally, it is utilized for material powders. A solid sample's flow uniformity and packaging quantity are represented by this bulk density test. A kilograms per cubic meter (kg/m3) is used to express it.

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Before anything else, find out how much the measuring cylinder that was employed in this experiment weighs. It was then reweighed after being filled with the provided sample of activated carbon. Then, it was put on the aluminum plate and dried in the oven for 60 minutes at 105°C. The dried sample's weight was measured following drying.

DB = (M2-M1)/v, where M1 is the mass of the measuring cylinder in g, M2 is the weight of the cylinder plus its contents, and V is the cylinder's capacity in liters.

III. RESULT AND DISCUSSION

Proximate analysis: It's critical to understand the characteristics of agricultural waste through proximate analysis.

Table 2: Proximate analysis of coconut shell and rice husk

Parameter	Coconut Shell Adsorbent	Rice Husk Adsorbent
% Moisture content	08.20	04.70
% VM	20.40	55.50
% Ash	12.50	16.20
% Fixed carbon	58.90	34.60

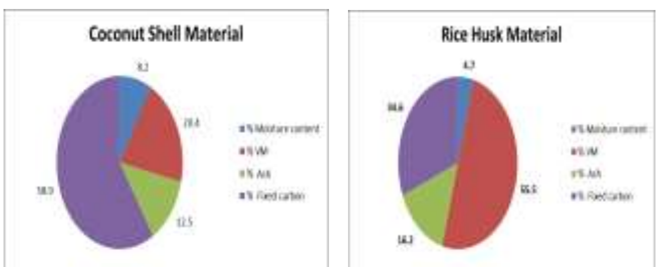

Figure 5: Proximate analysis of coconut shell & rice husk

With the remaining carbon in the sample, it facilitates our evaluation of the sample's moisture, volatile, and ash contents. It demonstrates that coconut shell-derived activated carbon has high moisture content while having a low volatile and ash content. Its high fixed carbon content makes it a desirable adsorbent.

Bulk density: The floatability attribute of the adsorbent is shown from a bird's-eye perspective. According to this theory, adding activated carbon to water will cause it to sink, putting it in better contact with the adsorbent and facilitating an efficient adsorption process.

Table 3: Bulk density and iodine number

Adsorbent	Bulk Density, (g/cc)	Iodine Number, (mg/g)
Coconut Shell	0.58	980
Rice husk	0.12	650

The bulk density of a material is directly proportional to the adsorption capacity of that material. More is the bulk density, better will be the adsorption carried out.

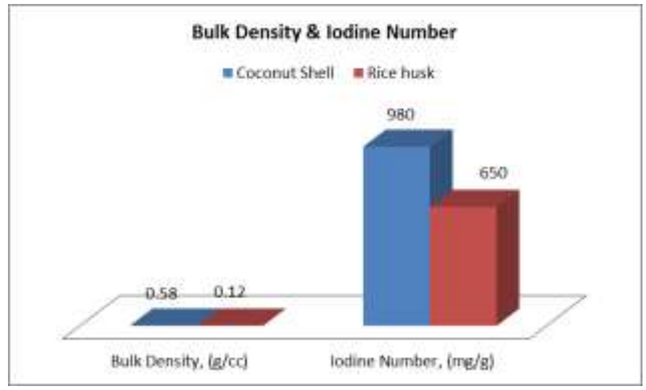

Figure 6: Bulk density and iodine number

Iodine number essentially serves as a measure for its micro-pore density. Since the iodine number and the sample's micro-porosity are directly inversely correlated, a higher iodine number denotes a greater sample's micro-porosity. The mechanical strength and adsorptive ability of activated carbon are both impacted by high ash content, which is undesirable. A quick and easy method to ascertain the iodine number, or adsorptive capacity, of activated carbon is iodine adsorption. It was noted that coconut shell activated carbon had the highest iodine number, which denotes the highest surface.

Table 4: Chemical composition by XRF analysis

Sr. No.	Constituents	Coconut Shell (%wt)	Rice Husk (%wt)
1	SiO_2	44.4	78.8
2	Cao	7.44	0.21
3	MgO	0.25	0.097
4	Al_2O_3	24.7	1.010
5	Fe_2O_3	10.7	0.105
6	TiO_2	1.35	0.03
7	S	8.50	1.24
8	P	1.84	18.50

The XRF analysis reveals about the SiO_2 content of the coconut shell and rice husk. Since the SiO_2 content is more, the capacities to adsorb the adsorbates are more.

X-Ray diffraction analysis for rice husk and coconut shell is also studied and results are as below.

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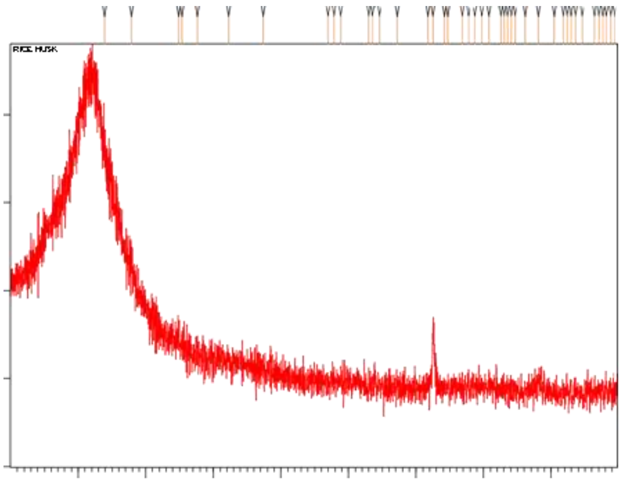

Figure 7: X-Ray diffraction analysis for rice husk

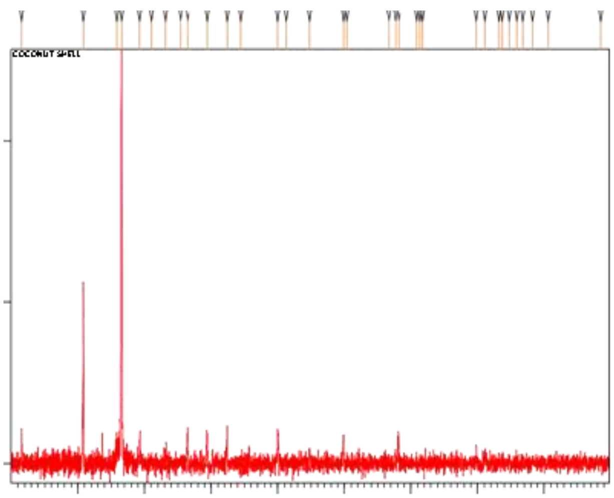

Figure 8: X-Ray diffraction analysis for coconut shell

XRD, XRF, iodine number, bulk density and proximate analysis shows that adsorbents prepared from coconut shell is better than rice husk.

Industrial wastewater collected and treated by passing through adsorbent filter and got following results.

Table 5: Removal Nickel and Copper from wastewater

Heavy Metals	**Initial Conc., mg/l**	**After treatment by coconut shell, mg/l**	**Removal by coconut shell, %**	**After treatment by rice husk, mg/l**	**Removal by rice husk , %**
Nickel	6	1.75	69.93	2.05	64.77
Copper	12	1.46	88.71	2.32	82.07

The following changes in pH were recorded while removal of nickel and copper from the industrial wastewater by passing through filter.

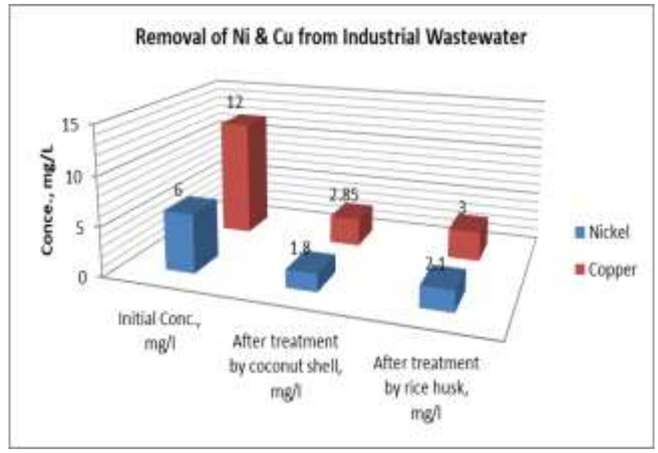

Figure 9: Removal of Cu & Ni from industrial wastewater

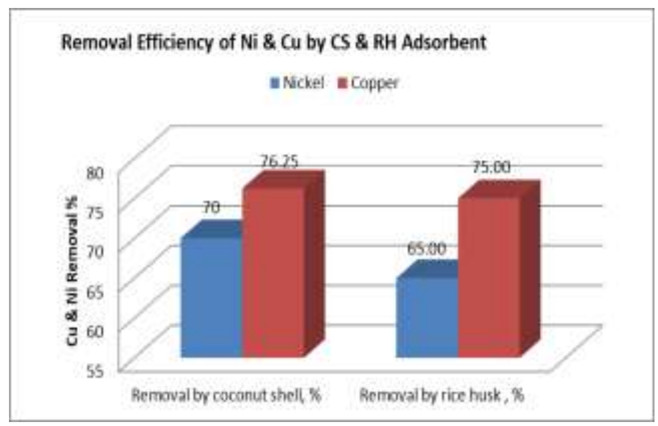

Figure 10: Removal efficiency of Cu & Ni

Table 6: Changes in pH while treatment

Adsorbent material	Initial pH	pH after 1st circulation	pH after 2nd circulation	pH after 3rd circulation
Coconut shell	2.3	3.45	4.2	5.8
Rice husk	2.3	3.9	4.9	6.1

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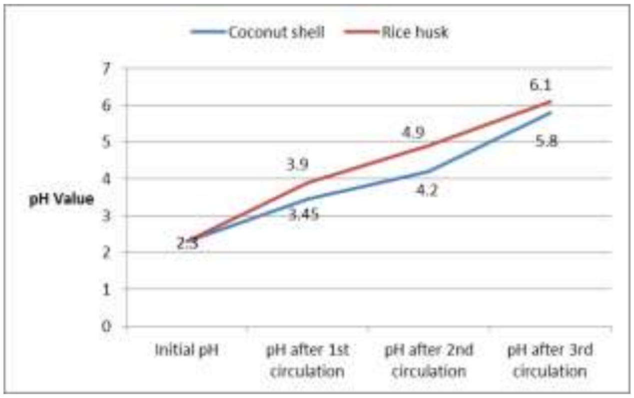

Figure 11: Changes in pH value of industrial wastewater

It has been found that the some characteristics of a material like fixed carbon content, porosity, etc. determine if the material is capable enough to be used for adsorption of heavy metals from the industrial wastewater. The higher value of fixed carbon content, iodine number and bulk density makes coconut shell and rice husk better adsorbents. Moreover, after treating the wastewater by adsorption using the filter unit, the pH of wastewater was almost neutralized. Also, the values of heavy metal concentration obtained by spectrophotometer are within the limits provided by CPCB.

IV. CONCLUSION

Numerous characterization techniques were used for the investigation, including proximate analysis, bulk density, pH, and iodine number test. An adsorption experiment follows this. Then a SEM test was run on the adsorbents to reveal their pore structure. An accurate picture of the sample's physical characteristics can be obtained from the proximate analysis of activated carbon. Based on the investigation, it was determined that rice husk has lower fixed carbon content than coconut shell activated carbon. It demonstrates that coconut shell-derived activated carbon has high moisture content while having a low volatile and ash content. According to our calculations and observations from the bulk density test, coconut shell has a larger bulk density than rice husk, indicating superior flow uniformity and solid fuel material packaging. We discovered via an iodine test that rice husk has an iodine number of 650 mg/g and coconut shell has an iodine number of 980 mg/g. Iodine number is essentially a gauge for the amount of micro-pores in the activated carbon. A higher iodine number denotes a higher micro-porosity of the sample since iodine number and micro-porosity are directly proportional. Rice husk activated carbon has bigger pores than coconut shell activated carbon. In comparison to rice husk, we discovered that activated carbon from coconut shells is better at adsorbing copper and nickel. Thus, we may draw the conclusion that both materials can be utilized successfully for the adsorption process to remove the heavy metals from the industrial waste water, even though coconut shell produces better results than rice husk.

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