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Removal of Nickel from Industrial Wastewater by an Agro-based Composite Adsorbent

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ABSTRACT

For many years, especially in emerging nations like India, the environment has been threatened by the increased output of industrial wastes and heavy metal toxicity. The usage of inexpensive adsorbents has recently attracted a lot of attention in studies on the removal of heavy metals like nickel from industrial wastewater. The use of agro-based adsorbent is an alternative to conventionally used activated charcoal. In this research, adsorption experiments were carried out using agro-based adsorbent prepared from rice husk, wheat husk, and soybean husk to reduce nickel from industrial wastewater. The adsorption process is simple, economical, and effective is the most preferred method used for the removal of toxic metals like nickel from industrial wastewater. Adsorbents prepared from these husks can be effectively used for adsorption due to low cost & high availability. Characterization of agricultural material by various tests like XRF, proximate analysis, bulk density, and iodine number was conducted on agro-based adsorbents to know the co-relation between removal efficiency and adsorption capacity. The effect of turbidity and pH parameters on Ni removal efficiency is also studied. Results indicated that wheat husk adsorbent appeared to be the most effective for the adsorption of Ni from wastewater as compared to soybean husk and rice husk adsorbent. Wheat husk, soybean husk, and rice husk have removal efficiency in the range of 62.50 to 73.33. Composite absorbents CA-2 with the proportion of 50% wheat husk, 33% soybean husk, and 17% rice husk have 82.50% efficiency, and CA-3 has 80.83% efficiency in removing Nickel. Wheat husk adsorbent, CA-2, and CA-3 are more effectively and sustainably used for the treatment of industrial wastewater to remove heavy metals.

INTRODUCTION

In response to global urbanization and increased industry, toxic effluents are routinely released, and many industrial processes produce wastes that contain metals, including Cu, Ni, Cr, and Pb (Perumal et al. 2014). They enter the biological cycle through the consumption of food, water, and air. Because they bioaccumulate, heavy metals are hazardous (Febrianto et al. 2009, Ngah & Hanafiah 2008). Industries like metal plating, mining, battery production, PCB manufacture, etc., discharge heavy metals into the environment. Wastewater contains the hazardous heavy metal ion nickel, which is non-biodegradable. Mammals are teratogenic and carcinogenic to metallic nickel. There is an urgent need for a method that can effectively remove heavy metals from aqueous effluents without incurring high costs due to drawbacks such as incomplete removal, high reagent costs and energy requirements, and the production of toxic sludge that needs to be disposed of carefully. Adsorption is a process in which specific adsorptives are transferred arbitrarily from the fluid phase to the surface of suspended particles. Adsorption has advantages over other technologies due to its straightforward design, sludge-free environment, and potential for inexpensive initial investment, as well as land requirements. One of the many readily available lignocellulose materials in the globe is rice straw. The third-largest grain crop in the world by overall production is rice. The cellulose, hemicelluloses, and lignin are found in rice straws. Both natural and wheat husk fibers are three-dimensional polymeric composites that are predominantly composed of cellulose, hemicelluloses, and lignin, with a very tiny amount of protein, starch, fat, and ash. Soybean husk has several qualities that make it a possible adsorbent with binding sites capable of extracting metals from an aqueous solution. The wheat husk is easily accessible in large quantities, and the treatment method of bio-sorbent appears to be cost-effective (Said et al. 2018). Using agricultural leftovers, such as rice straw, has several benefits, including ease of regeneration, low cost, little processing needed, strong adsorption capacity, and simple technique (Asif & Chen 2017). A study was carried out on the removal of nickel from wastewater and found that the removal of nickel from wastewater is effectively possible by different technologies. Different technologies to remove nickel are studied with their effectiveness, and the findings are discussed (Bhagat & Khandeshwar 2019). With a percentage removal of up to 99%, adsorption employing a variety of inexpensive adsorbents was found to be a very effective method (Dhokpande et al. 2013). Biological methods were also effective and produced encouraging results. The injection of nickel at a higher percentage through water results in serious health issues such as damage to the lungs, kidneys, and gastrointestinal tract. It has also been linked to cancer. Therefore, it is essential to create efficient and affordable technologies to extract and/or recover nickel (Arunkumar et al. 2015). Using an agricultural adsorbent to remove the nickel from wastewater is possible to treat chemical wastes by turning agricultural solid wastes into adsorption media—a practice known as waste-to-waste treatment. By treating wastewater containing nickel and other

A. Procurement and drying of husk.

B. Chemical treatment of husk.

C. Husk before thermal treatment.

D. Husk after thermal treatment.

E. Husk before thermal treatment.

F. Husk after thermal treatment.

Fig. 1: Preparation of adsorbents from agricultural husk.

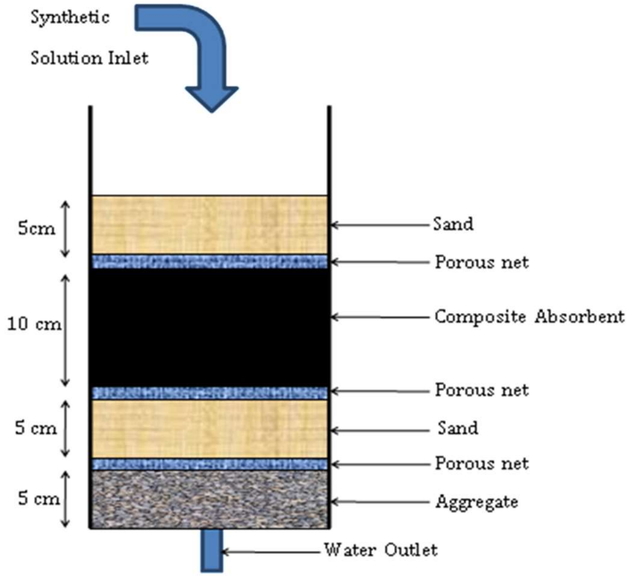

Fig. 2: Filter model with adsorbent, net, sand, and aggregates.

heavy metal ions, pine sawdust's ability to adsorb nickel and other heavy metal ions was examined in this work. To ascertain the interactive effects of the various components on the adsorption capacity, the results were analyzed using response surface methods and a factorial design. The utilization of pine sawdust may offer a potential method for removing nickel ions from multi-component aqueous solutions, according to the study's findings (Moodley et al. 2011). Removal of heavy metals utilizing various fruit peels, vegetable peels, and organic waste is effectively used. Researchers discovered that it is feasible to remove and recover Ni from electroplating wastewater using orange peel. The recovery of Ni by column operation is found to be higher than by batch method. This study demonstrated the fundamental effects of several parameters over the banana peel, including pH agitation, speed, and contact time, with the best results at pH 7,100 rpm and 90 minutes of contact time. The study investigated the carbon bio-sorbent made from pomegranate peels for removing iron from aqueous solutions (Abdulfatai et al. 2013, Darge & Mane 2015). Studies concluded that the sustainable methods for converting waste materials into valuable low-cost adsorbents utilized are quite successful at either treating or removing heavy metals from the environment (Khalili et al. 2000, Jain 2015)

MATERIALS AND METHODS

Materials and reagents are required for physical, chemical treatment, and thermal treatment to obtain adsorbent: rice husk, wheat husk, soybean husk, phosphoric acid, distilled water, and nickel powder.

Instrumentation required: Muffle furnace, oven, weighing machine, pH meter, UV spectro-photometer, sieve shaker, XRF machine, SEM, and XRD.

Every year, a large amount of rice husk (RH), Wheat Husk (WH), and Soybean Husk (SH) are generated in agricultural areas as bi-products and considered agricultural waste. To get rid of dirt and other soluble contaminants, these full husks were repeatedly washed in double-distilled water. Then, they were dried for 24 h at 105°C. The desired adsorbent size of 1 mm was obtained by sieving it through meshes, and it was then kept in an airtight container. To ensure that the reagents were completely adsorbed onto the raw material, these 500 g of husks were combined and steeped in a 1 M H_3PO_4 solution for 24 h at room temperature in 500 mL with 150 rpm of stirring. Following this procedure, the chemically treated husks were repeatedly washed in distilled water to maintain a steady pH level. This adsorbent was later oven-dried for 4 h at 110°C. After this, thermal treatment in a muffle furnace is given in the absence of oxygen at a temperature of 320^{o}C to obtain the agro-based adsorbent (Fig. 1).

Similarly, all husks (WH, SH & RH) are treated, and adsorbents are prepared to carry out the adsorption process. A synthetic solution containing nickel is prepared concentration of 6 mg.L^{-1} from standard nickel solution (1000 mg.L^{-1} Ni in diluted HNO_3) with distilled water (Zhang et al. 2014). A filter unit was prepared for the treatment of water. It is a circular acrylic pipe consisting of coarse aggregate, fine aggregate, adsorbent material, and sand placed bottom to top in order of decreasing grain size (Fig. 2, Table 1). Each layer was separated by a net so that the materials did not get

Table 1: Filter components with dimension.

S.N.	Component	Dimension
1.	Filter unit	Diameter = 10 cm & Height = 60 cm
2.	Aggregate	Size = 20 mm & Height = 05 cm
3.	Porous Net	Micro-net 600 micron
4.	Sand	Size = 150-600 micron & Height = 5 cm
5.	Adsorbent material	Height = 10 cm

mixed, and there was continuous passage of water through the filter (Ratnoji & Singh 2014).

The specifications of the filter unit are as follows:

A synthetic solution of Ni concentration 6 $mg.L^{-1}$ is prepared and passed through the designed filter, where an adsorbent layer is sandwiched between sand layers. The aggregate layer also supports the bottom sand layer. A porous net separates every layer to avoid disturbance of layers in the filter unit.

Various tests are performed on husk to co-relate the adsorption process and the Ni removal efficiency:

1) Proximate analysis: Proximate analysis is the process of using established techniques to estimate the contents of moisture, volatile matter, fixed carbon, and ash.
   a. A sample's moisture content is determined by dividing the mass of its solids by the mass of its water, represented as a percentage.
   b. Ash content: The amount of any organic material that is left over after being burned at extremely high temperatures is known as the ash or mineral content.
   c. The amount of volatile matter in a sample is calculated as the mass loss following a 7-minute heating process at 900 degrees Celsius, with less moisture loss.
   d. Fixed carbon is the solid, combustible residue that is left behind when a particle is heated, and the volatile stuff is evacuated.
2) Bulk Density: Bulk density is the weight of a material per unit volume. It is typically used for material powders.

Table 2: Proximate analysis of agricultural husk.

S.N.	Proximate Analysis Parameter	Wheat Husk	Soybean Husk	Rice Husk
1.	Moisture Content, %	8	10	6
2.	Volatile Matter, %	51	54	59
3.	Ash Content, %	15	15	16
4.	Fixed Carbon, %	26	21	19

3) Iodine number: The amount of iodine that one gram of activated carbon may adsorb from a typical 0.1N iodine solution when the equilibrium iodine concentration is precisely 0.02N is known as the iodine number. The iodine number determines the amount of micro-pores in the activated carbon.
4) X-ray fluorescence (XRF) is a non-destructive analytical method for figuring out the elemental makeup of materials. An approach for gauging coating thickness and for studying materials is X-ray fluorescence Analysis (ED-XRFA).

Effects of pH and turbidity are also studied. pH is a measure of hydrogen ion concentration, a way to quantify a solution's acidity or alkalinity. Like smoke in the air, turbidity is the cloudiness or haziness that results from a vast number of tiny particles that are often imperceptible to the unaided eye. By measuring the intensity of light as a beam of light travels through a sample solution, a technique called spectrophotometry can determine how much light a chemical component absorbs. Every substance either absorbs or transmits light across a specific spectrum of wavelengths, according to the fundamental tenet. The spectrophotometer measures Ni removal. The study of characterization provides valuable insights into the effects of modifications on the adsorption behavior of activated carbon, which is the main cause of adsorption processes (Ahmadpour 1996).

RESULTS AND DISCUSSION

Characterization of agricultural waste material is done in various ways and starts with proximate analysis, where fixed carbon content is measured by following the formula.

Table 3: Developed Composite Adsorbent (CA) and their combination.

SN	Combinations			Composite Adsorbent	
1.	Rice Husk (17%)	Wheat Husk (33%)	Soybean Husk (50%)	CA-1	RH:WH:SH (17:33:50)
2.	Rice Husk (17%)	Wheat Husk (50%)	Soybean Husk (33%)	CA-2	RH:WH:SH (17:50:33)
3.	Rice Husk (33%)	Wheat Husk (50%)	Soybean Husk (17%)	CA-3	RH:WH:SH (33:50:17)
4.	Rice Husk (33%)	Wheat Husk (17%)	Soybean Husk (50%)	CA-4	RH:WH:SH (33:17:50)
5.	Rice Husk (50%)	Wheat Husk (33%)	Soybean Husk (17%)	CA-5	RH:WH:SH (50:33:17)
6.	Rice Husk (50%)	Wheat Husk (17%)	Soybean Husk (33%)	CA-6	RH:WH:SH (50:17:33)

Table 4: Proximate analysis of composite adsorbents.

S.N.	Proximate Analysis Parameter	CA-1	CA-2	CA-3	CA-4	CA-5	CA-6
1.	% Moisture Content	8	10	9	7	7	6
2.	% Volatile Matter	52	48	49	55	54	56
3.	% Ash Content	17	13	15	17	17	18
4.	% Fixed Carbon	23	29	27	21	22	20

Fixed carbon FC = 100 – (% Moisture content + % Volatile matter content + %Ash content)

The fixed carbon content for wheat husk is 26%, soybean husk is 21%, and rice husk is 19%, which is good for the adsorption process (Table 2). Proximate analysis indicates that all three waste materials have good potential to be good adsorbent. Proximate analysis of composite adsorbent is also carried out for the same above-mentioned reason.

Six different types of agro-based composite adsorbents (CA-1, CA-2, CA-3, CA-4, CA-5 & CA-6) are developed from agricultural waste (Table 3). These six developed composite adsorbents are used in the filter unit as one of the layers for heavy metal removal, and the results of individually used adsorbents with composite adsorbents are compared.

Fixed carbon content for CA-1 is 23%, CA-2 is 29%, CA-3 is 27%, CA-4 is 21% CA-5 is 22% and CA-6 is 20%. CA-2 and CA-3 have a little bit more fixed carbon content, and the remaining CA has a fixed carbon content in the range of 20% to 23%, which is good for the adsorption process. Proximate analysis indicates that all six CA have good potential to be good adsorbents (Table 4, Fig. 3).

The bulk density of a material is directly proportional to the adsorption capacity of that material. The more bulk density, the better the adsorption will be carried out. As per calculation, the bulk density of WH, SH, CA-2, and CA-3 were found to be maximum as compared to others (Table 5). A graphical representation of bulk density is shown in Fig. 4.

Iodine adsorption is a simple and quick technique to determine the adsorptive capacity of adsorbent, also known as iodine number. As per the results, the Iodine number of WH, CA-2, and CA-3 are found to be maximum (Table 6). Overall, all the adsorbents have a good iodine number. It suggests that WH adsorbent and composite adsorbents like CA-2 and CA-3 have good adsorption capacity (Fig. 5).

The XRF analysis reveals the SiO_2 content of the rice husk, wheat husk, and soybean husk (Table 7,

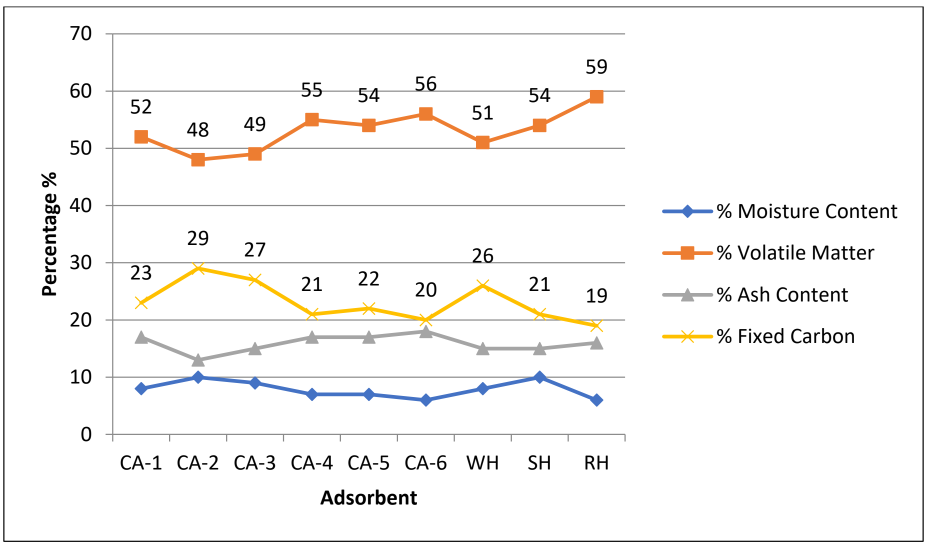

Fig. 3: Fixed carbon content by proximate analysis.

Table 5: Bulk density of husk and composite.

Adsorbent Material	RH	WH	SH	CA-1	CA-2	CA-3	CA-4	CA-5	CA-6
Bulk Density, $g.cc^{-1}$	0.65	0.90	0.78	0.80	0.89	0.88	0.70	0.75	0.69

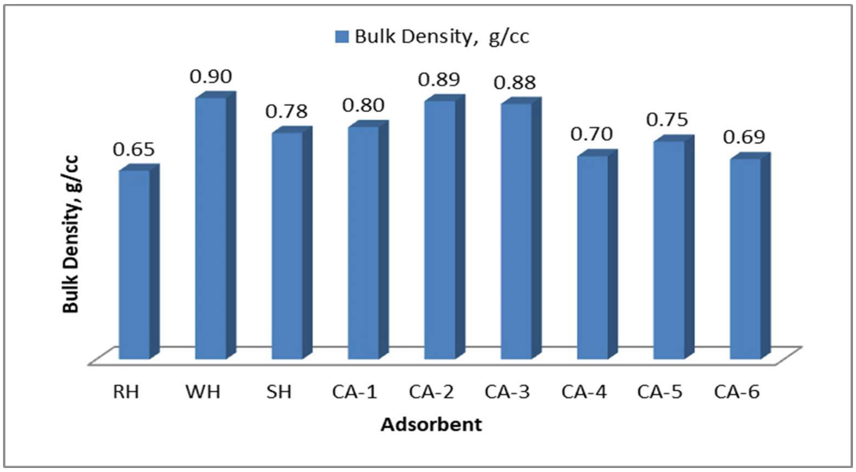

Fig. 4: Bulk density of husk and composite.

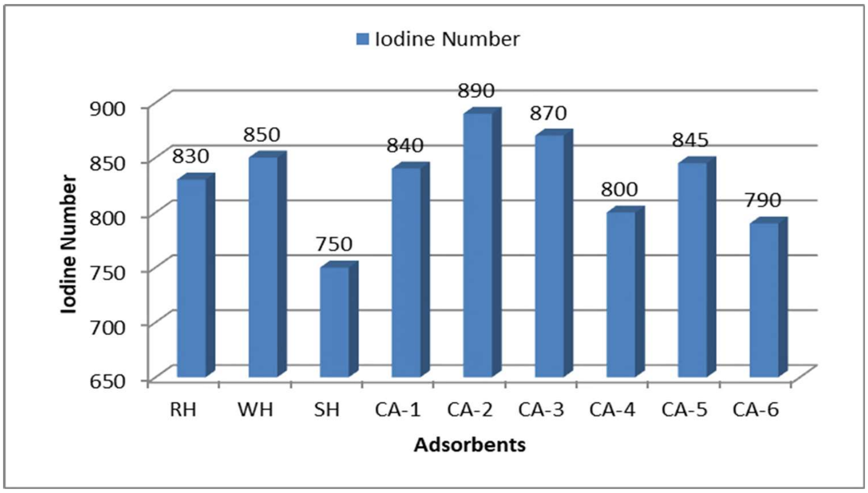

Fig. 5: Iodine number of adsorbents.

Table 6: Iodine number of adsorbent.

Adsorbent	RH	WH	SH	CA-1	CA-2	CA-3	CA-4	CA-5	CA-6
Iodine Number, $mg.g^{-1}$	830	850	750	840	890	870	800	845	790

Table 7: Elemental composition by XRF.

S.N.	Constituents	Wheat Husk [%wt.]	Rice Husk [%wt.]	Soybean Husk [% wt.]
1.	SiO_2	70.8	38.6	44.4
2.	CaO	0.21	8.01	7.44
3.	MgO	0.097	0.3	0.25
4.	Al_2O_3	6.01	25.879	24.7
5.	Fe_2O_3	3.105	11.3	10.7
6.	TiO_2	0.03	1.46	1.35
7.	S	1.24	9.02	8.5
8.	P	18.5	2.38	1.84

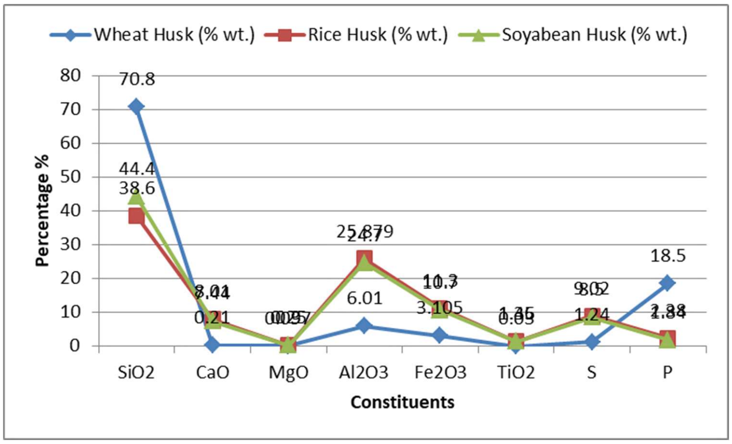

Fig. 6: Constituents (SiO_2) by XRF.

Table 8: Changes in pH after circulation through filter unit.

S.N.	Adsorbent	Initial pH	pH after 1st circulation	pH after 2nd circulation	pH after 3rd circulation	pH after 4th circulation	pH after 5th circulation
1.	Wheat Husk	2.60	5.45	5.80	6.55	6.80	6.75
2.	Soybean Husk	2.60	5.05	5.55	6.10	6.05	6.10
3.	Rice Husk	2.60	5.00	5.40	5.85	6.10	6.05
4.	CA-1 R17:W33:S50	2.60	5.40	5.75	6.10	6.40	6.45
5.	CA-2 R17:W50:S33	2.60	5.50	5.95	6.55	6.85	6.95
6.	CA-3 R33:W50:S17	2.60	5.45	5.90	6.50	6.80	6.75
7.	CA-4 R33:W17:S50	2.60	5.40	6.05	6.30	6.40	6.50
8.	CA-5 R50:W33:S17	2.60	5.35	5.60	6.00	6.10	6.10
9.	CA-6 R50:W17:S33	2.60	5.00	5.25	5.90	6.00	6.05

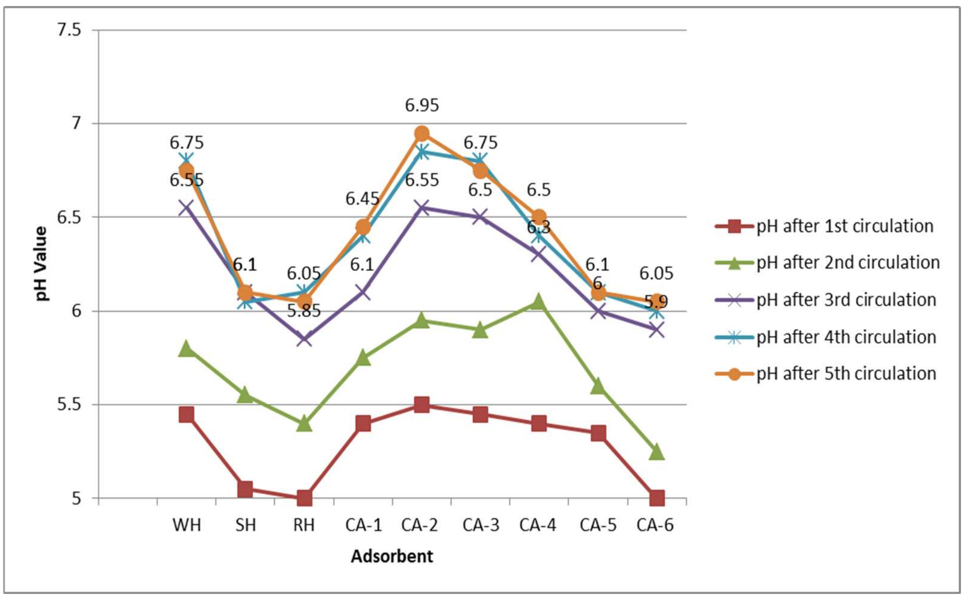

Fig. 7: Changes in pH after passing through filter unit.

Table 9: Changes in turbidity

S.N.	Adsorbent	Initial turbidity (NTU)	Turbidity after 1st circulation	Turbidity after 2nd circulation	Turbidity after 3rd circulation	Turbidity after 4th circulation	Turbidity after 5th circulation
1.	Wheat Husk	0	0.30	0.35	0.40	0.50	0.70
2.	Soybean Husk	0	0.10	0.20	0.20	0.35	0.55
3.	Rice Husk	0	0.20	0.25	0.30	0.35	0.45
4.	CA-1 R17:W33:S50	0	0.05	0.15	0.25	0.40	0.55
5.	CA-2 R17:W50:S33	0	0.25	0.30	0.30	0.45	0.65
6.	CA-3 R33:W50:S17	0	0.15	0.20	0.25	0.30	0.50
7.	CA-4 R33:W17:S50	0	0.05	0.05	0.15	0.20	0.40
8.	CA-5 R50:W33:S17	0	0.05	0.10	0.15	0.35	0.55
9.	CA-6 R50:W17:S33	0	0.10	0.15	0.20	0.30	0.40

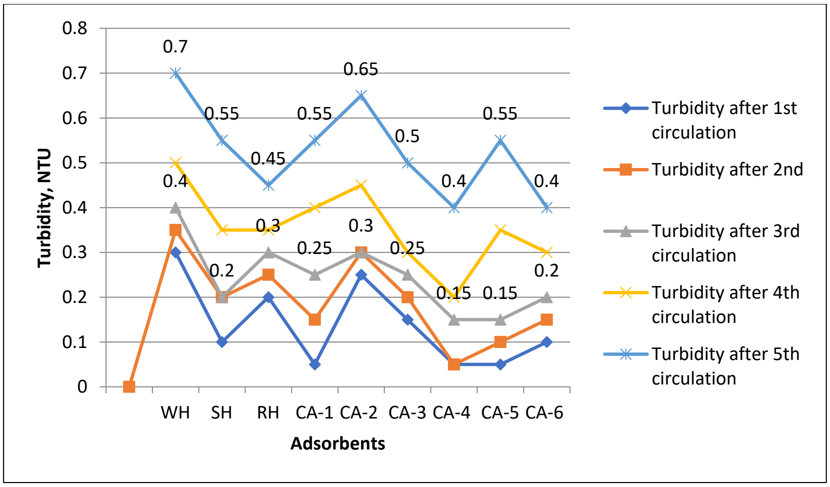

Fig. 8: Changes in turbidity after circulations.

Table 10: Nickel removal by developed adsorbent.

S.N.	Adsorbent	Initial Ni concentration [mg.L^{-1}]	Ni after 1st circulation [mg.L^{-1}]	Ni after 2nd circulation [mg.L^{-1}]	Ni after 3rd circulation [mg.L^{-1}]	Ni after 4th circulation [mg.L^{-1}]	Ni after 5th circulation [mg.L^{-1}]	Ni Removal %
1.	Wheat Husk	6	4.7	3.35	1.6	1.55	1.45	73.33
2.	Soybean Husk	6	4.8	3.75	1.7	1.6	1.55	71.67
3.	Rice Husk	6	4.85	3.85	2.25	2.1	1.8	62.50
4.	CA-1 R17:W33:S50	6	4.75	3.55	2.2	2.1	2.05	63.33
5.	CA-2 R17:W50:S33	6	4.35	2.95	1.05	1	0.95	82.50
6.	CA-3 R33:W50:S17	6	4.55	3.2	1.15	1.05	1	80.83
7.	CA-4 R33:W17:S50	6	4.65	3.7	1.55	1.45	1.4	74.17
8.	CA-5 R50:W33:S17	6	4.65	3.3	1.65	1.55	1.5	72.50
9.	CA-6 R50:W17:S33	6	4.8	3.75	1.95	1.9	1.8	67.50

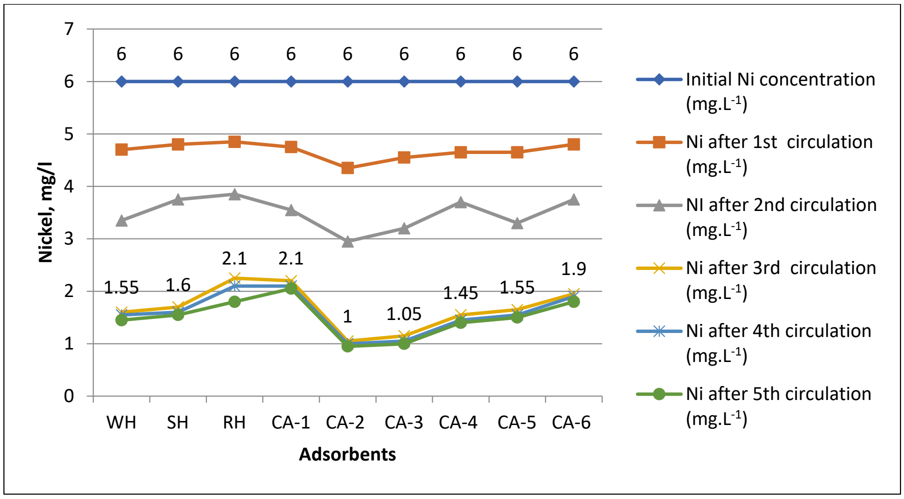

Fig. 9: Changes in Nickel concentration after passing through filter unit.

Fig. 6). Since the SiO_2 content is higher in the wheat husk, the capacity to absorb the adsorbates will be greater.

Variation in pH Value

Initially, the pH of the synthetic solution was the same as we have only one type of solution. On passing the synthetic solution through the filter, it did not give much changes in pH, but it had a change in pH as the number of circulations increased. Wheat husk adsorbent has the maximum impact on pH value and gives a pH value of 6.20. CA-2 and CA-3 are good in bringing pH value to neutral. The changes in pH were recorded in the removal of nickel from the industrial wastewater by adsorption (Table 8, Fig. 7).

WH adsorbent, CA-2, and CA-3 bring pH greater than 6.5 after the third circulation, and that shows great potential to neutralize the acidic wastewater.

Turbidity Variation

A synthetic solution prepared from distilled water and nickel consists of no turbidity initially. However, as a synthetic solution comes in contact with an adsorbent little bite, turbidity increases, which is negligible, and it can be removed by other options in combination.

There was a general increase in turbidity after each circulation from the filter unit (Fig. 8). This is because of the use of adsorbent. From Table 9, it is evident that the turbidity of CA-4 and CA-6 was found to be minimal.

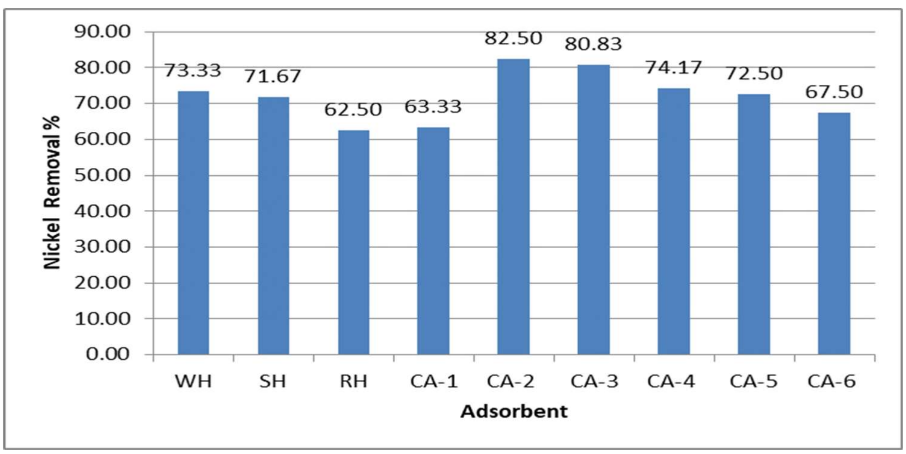

Fig. 10: Nickel removal % after 3rd circulation.

Removal of Nickel by Filter Unit Containing Adsorbent

As shown in Fig. 2, filter units containing adsorbents are used to remove nickel by passing the synthetic solution through a layer of adsorbent, and the results observed are presented in Table 10.

The initial nickel concentration in the synthetic solution was 6 $mg.L^{-1}$. After every circulation of the solution from the filter, the nickel concentration was found to be reduced. Nickel reduction by wheat husk is 1.60 $mg.L^{-1}$, which is more as compared to rice husk and soybean husk adsorbent (Fig. 9). Maximum reduction of nickel by CA-2 and CA-3 as compared to CA-1, CA-4, CA-5, and CA-6.

Though all the materials show a reduction of nickel in the range of 1.60 to 2.25 after 3rd circulation and 3 $mg.L^{-1}$ is the permissible value as per WHO standards for industrial wastewater effluent, therefore after 3rd circulation, all adsorbents reduce the concentration of nickel below 3 $mg.L^{-1}$. Hence, all developed agro-based adsorbents are efficient for treatment, and only three circulations through filter units are needed.

Wheat husk adsorbent has a 73.33% nickel removal efficiency, which is the highest in comparison with rice husk and soybean husk adsorbent. CA-2 has the 82.50% nickel removal efficiency which is the highest as compared to CA-1, CA-4, CA-5, and CA-6 (Fig. 10).

CONCLUSION

In this work, three agro-based adsorbents from WH, SH, and RH were developed, and six different combinations above three husks were used to develop six composite adsorbents. After testing samples for characterization of material by proximate analysis, bulk density, iodine number, and XRF, it is concluded from testing results that wheat husk has more potential to carry out the adsorption process and remove more heavy metals like nickel from wastewater. In proximate analysis, the fixed carbon content in the wheat husk is 26%, the soybean husk is 21%, and the rice husk is 19%. Bulk density for all husk material is found in the range of 0.65 to 0.90 $g.cc^{-1}$, which indicates good potential to carry out the adsorption. The iodine number indicate the adsorption potential, and all developed adsorbent has the iodine number in the range from 750 to 890 $mg.g^{-1}$. Wheat husk has the iodine number 850 $mg.g^{-1}$, and rice husk has the 750 $mg.g^{-1}$, which is the lowest as compared to other developed adsorbents. CA-2 has the highest adsorbent number, 890 $mg.g^{-1}$. Iodine number 890 $mg.g^{-1}$ indicates great potential to carry out the adsorption process. We also tested the material by XRF to know the various constituents present in the material. As per the XRF result, the SiO2 percentage is more than other constituents. In wheat husk, the SiO2 percentage is 70.80%, which is favorable for adsorption. WH adsorbent, CA-2, and CA-3 bring pH greater than 6.5 after the third circulation, and that shows great potential to neutralize the acidic wastewater. Nickel concentration in industrial wastewater is 6 $mg.L^{-1}$; therefore synthetic solution of the same concentration of 6 $mg.L^{-1}$ is prepared. With the help of a filter containing adsorbent nickel is removed. After every circulation of the solution from the filter, the nickel concentration was found to be reduced. Nickel reduction by wheat husk is 1.60 $mg.L^{-1}$, which is more as compared to rice husk and soybean husk adsorbent. Maximum reduction of nickel by CA-2 and CA-3 as compared to CA-1, CA-4, CA-5, and CA-6. All the used adsorbent materials show a reduction of nickel in the range of 1.60 to 2.25 after 3rd circulation, and 3 $mg.L^{-1}$ is the permissible value as per WHO standards for industrial wastewater effluent; therefore, after 3rd circulation, all adsorbents reduce the concentration of nickel bellow 3 $mg.L^{-1}$.

All developed agro-based adsorbents are efficient for treatment, and only three circulations through filter units are needed. Wheat husk adsorbent has a 73.33% nickel removal efficiency, which is the highest in comparison with rice husk and soybean husk adsorbent. CA-2 has the 82.50% nickel removal efficiency, which is the highest as compared to CA-1, CA-4, CA-5, and CA-6. Fixed carbon content, iodine number, bulk density, and SiO2 constituents were found in the required proportion, which supports our experimental work's results of nickel removal.

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