Natural Adsorbent from Agricultural Products for Heavy Metal Removal

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ABSTRACT

Adsorption is an ecofriendly technique used now a days to treat the various type of waste water. Then the filtered water is subjected to adsorption using low cost and easily available natural adsorbents. Agricultural by-products are currently being extensively used to prepare activated carbon because of their availability in large quantities at low cost. Recently activated carbons have been commonly produced from waste products such as wheat husk, almond shells, coconut shell, rice husk and sugarcane bagasse etc. Adsorption process capable to remove impurities, heavy metals, COD and color from the waste water at high efficiency with low cost. Heavy metals having specific gravity more than 5 and are toxic in nature. Heavy metals cause of water as well as soil pollution. So, removal of heavy metals is necessary for protection of environment and human as well as animal and wild life. The percentage of each metal adsorbed increases with the pH. The percentage of each metal adsorbed as function of the pH. The maximum adsorption of each metal ion occurs at pH range between 5 and 6. As per observation the efficiency of metal ion adsorption is related with the contact time as contact time increase percentage of metal adsorption increase. The maximum adsorption is noticed between time of 3-3.5 hours for heavy metal at concentration of 5-10 ppm with pH value 5-6. After these optimum value removal or adsorption of heavy metals are constant.As concentration increases % removal decrease at particular stage after that rate of adsorption constant. As concentration increases % removal decrease at particular stage after that rate of adsorption constant. The optimum value of concentration of metal between 5-10 ppm.

Keywords – Natural Adsorbents, Heavy metal Removal, Adsorption Process, Sugarcane Bagasse and Rice Husk.

1. INTRODUCTION

The development of low-cost adsorbents prepared from cheaper and readily available materials. Solid substance with large surface area, micro porous character and chemical nature of their surface have made them potential adsorbents for the removal of heavy metals from industrial waste water. The use of agricultural products and by-products has been widely investigated as a replacement for current costly methods of removing heavy metals from water and wastewater. Some of the agricultural materials can be effectively used as a low-cost sorbent. Activating agents have a great influence on the influence the pore development and surface characteristics of the activated carbon produced. Activating agents such as phosphoric acid (H_3PO_4), Sulphuric acid (H_2SO_4) potassium hydroxide (KOH), sodium hydroxide (NaOH), calcium chloride ($CaCl_2$), zinc chloride ($ZnCl_2$) use for the chemical activation process.

1.1 Activated Carbon Applications

1. Activated carbon is an excellent and versatile adsorbent.

2. Treatment of industrial waste water.

3. Air purification in food processing and chemical industries

4. Purification of many chemical

5. Purification Food

6. Purification pharmaceutical products

5. In respirators for work in hostile environments.

6. In a variety of other gas-phase applications.

7. Removal of color, taste and inorganic impurities from drinking waters

8. Removal of heavy metal ions from waste water

9. Main applications include the adsorptive removal of color, odor, taste and other undesirable organic.

10. AC can act as a filter material in air cleaning filters for removal of gases and vapors.

11. AC most effectively adsorbs soluble organic compounds from aqueous solutions.

12. It is widely used to remove color and other impurities such as those causing foaming.

1.2 Advantages of Adsorption Process

1. There is complete mineralization of organic matter.

2. There is no need for any processing units on the surface.

3. This process reduces organic loading in terms of chemical oxygen demand and done the removal of recalcitrant and toxic pollutants.

4. Adsorption process is a relatively economical method since it requires no additional energy when compared to many other Process.

5. Adsorption process very effective at removing resistant organic compounds

6.Adsorption process capable of complete mineralization of organic contaminant

7. Less susceptible to the presence of toxic chemicals

8. Produce less harmful by-products

9. Less maintenance required

10. Low production of residual sludge

1.3 Application of adsorbent in wastewater

1. Removal of Heavy Metals from waste water

2. Removal of various dyes from waste water

3. Removal of BOD, COD and Color.

4. Removal of Toxic chemicals from waste water

5. Extraction of impurities presents in waste water

6. Removal of organic pollutants from wastewater

7. Removal of pollutants like ammonia and nitrates.

8. Removal of TS, Tot-P and Total-N.

1.4Properties of Heavy Metals

1. Heavy metals occur bottom of the periodic table.

2. Heavy metal has high densities.

3. They are toxic in nature.

4. Heavy metals are non-degradable.

5. Density > 5 gm/cc.

6.They have harmful effect on biological systems.

7. Specific gravity of heavy metal is more than 5.

8. They have tendency to accumulate in organisms.

9. They have high relative atomic weight.

1.5 Forms of Heavy Metals

1. Salts
2. Oxides or Hydroxides
3. Organic form i.e., alkyl lead or methyl mercury.
4. Inorganic metals to be toxic
5. Dissolved organic and inorganic ligands
6. Colloids
7. Particulate matter

2. LITERATURE REVIEWS

The presence of incorporated phosphoric acid brings about a shift in the decomposition temperature (300 ºC) that prevents the burnout of charcoal and raises its yield. It is thus demonstrated that the temperature (800 ºC) is suitable for the production of activated carbon. Activated carbon has been prepared by using 10 % $ZnCl_2$ and 20 % H_3PO_4 as activated agents. The removal of metal ions, phenols, which are industrial wastes it was thought appropriate to study the adsorption kinetics for acids such as oxalic acid and acetic acid.The activated charcoal was dipped in a 10 % ZnCl2 solution and kept for 24 hrs., filtered through Whatman paper No. 41 and washed with 2 N HCl to remove zinc and washed with water till washing shows pH = 7. **Aznar et al. 2005 studied the removal of Methyl** red dye using treated sugarcane bagasse and compared the results with those obtained using powered activated carbon. As per the study one portion of ground bagasse was treated with 1% formaldehyde in ration of 1:5 at 50 °C for 4 hours followed by activation at 80 °C for 24 hours. The other portion of the bagasse was treated with sulfuric acid and heated in a muffle furnace for 24 hours at 150 °C. **Thieveries and Mylsamy 2010 conducted** an adsorption study of Rhodamine-B dye on char prepared by treating the coconut shell with concentrated sulfuric acid at ratio of 1:1. The activation was performed by heating in a muffle furnace at 550 °C for 7 hours, followed by washing and drying. **Agricultural wastes being porous and lightweight** due to fibrous nature, are non-conventional low-cost adsorbents for metal adsorption. Carboxylic and hydroxyl functional groups on surface of agricultural wastes have high affinity for heavy metal ions. Physicochemical modifications of wastes can enlarge surface area, type of adsorbing sites, porosity etc., thus improving sportive capacity, which may compensate for the cost of additional processing. Regeneration of spent adsorbent has become a cost effective and sound environmental option. [3]. **The sugar producing industry generates tons of bagasse** as a waste product has the potential to be converted to bio char. In this study, sugarcane bagasse was converted to bio char at 600 ◦C for 3 hours and a heating rate of 10 ◦C/min using the pyrolysis technology. A bio char yield of 75% was achieved with a surface area of 500 m2 /g and a particle size of < 2 mm. The bio char was used as an adsorbent media in sugarcane wastewater and the changes in the pH, biological oxygen demand (BOD), chemical oxygen demand (COD), total nitrogen (Tot-N), total phosphate (Tot-P) and the total solids (TS) were monitored using standard methods. **Application of the bagasse based bio char in sugarcane wastewater treatment** resulted in the significant reduction of BOD, COD, Tot-N, Tot-P and TS by 85.1%, 84.8%, 74.8%, 76.5% and 84.7% resp. Experimental analysis shows the bio char (Adsorbent) in sugar cane wastewater treatment resulted in significant decrease in BOD, COD, TS, Tot-P and Tot-N by more than 70%. The bagasse-based bio char had good adsorption properties essential in sugar wastewater treatment. [5]. **The mass of Bagasse** varied from 2 to 20 g/L; the pH of the solution being maintained to 5 with a contact time of two hours. With a Bagasse mass of 10 g/L, the percentage of removal metal was 77.5% for nickel ion and 89.95% for cupric ion. **With a Bagasse mass of 10 g/L** the percentage of removal metal was 77.5% for nickel ion and 89.95% for cupric ion. This increase of percentage of adsorption can be explained by the presence of high number of adsorption sites on the area of adsorbent. **Study influence of contact time** on the adsorption of copper and nickel ions on the Sugarcane Bagasse was carried out at pH 5 with a Bagasse mass of 20 g/L while varying this parameter from 1 to 5 hours. [6].**Patil Kishor P. and Patil Vilas S. has investigated** that the efficiency of removing copper ions and Zinc ions from Copper Chloride and Zinc Chloride, using naturally based adsorbents like Sugarcane Bagasse. The acid modified sugarcane bagasse and Cu (II) solution were kept in contact for various time periods 10, 20, 30, 60 min. The % removal of Cu was obtained 85 - 90%.**At an adsorbent dose of 0.8 g / 50 ml** is sufficient to remove 80 – 100% Cr (VI) from aqueous solution having an initial metal concentration of 20mg/l at a pH value of 1. The maximum removal obtained was 99.8% at pH2.**Dr. P. Akhila Swathanthra, Dr. B. Sarath Babu, M. Srinivasa Rao, Dr. V. V. Rao has studied** that Adsorption behavior of copper from waste water has been investigated in this paper using Bagasse. The maximum removal of Copper is above 93% was observed at pH of 5 for Bagasse in 100 ppm Copper solution. [7].**By experimental study methods** adsorption material bagasse, rice husk, wheat husk sawdust is more popular due to efficiency as well as cost of treatment. In present study bagasse from sugarcane industry were used to remove the toxic metal from waste. **Experiment study to** evaluate the adsorption process, and it was found that the bagasse was found to adsorb 96.4% of Cadmium (II) and 93.8% of iron (II). [10].**By experimental study methods** adsorption material bagasse, rice husk, wheat husk sawdust is more popular due to efficiency as well as cost of treatment. In present study bagasse from sugarcane industry were used to remove the toxic metal from waste. Experiment study to evaluate the adsorption process, and it was found that the bagasse was found to adsorb 96.4% of Cadmium (II) and 93.8% of iron (II). [8].**The activated carbon of rice husk, which activated by H_3PO_4** at temperature of 450 °C, has the highest adsorption capacity. According to gasoline adsorption study, the optimum conditions were 0.1 g of activated carbon 70 °C of adsorption temperature and 30 minutes of adsorption time. Physical characterization of the activated carbon obtained was performed by scanning electron microscopy (SEM).[11].**Activated carbon (AC) from sugarcane bagasse by the physical activation process** (CO2) and subsequently evaluate the physicochemical characteristics of the AC and of the bio oil produced. In a physical activation process, the biomass is pyrolyzed, under an inert atmosphere (N_2). During the pyrolysis were evaluated the effect of different temperatures (400, 500, 700 and 800 °C) on the physicochemical characteristics of the AC.

The activation step was realized under flow of 150 ml min-1 of carbon dioxide (CO_2) at 800 °C for 2 hours, using a heating ramp of 10 °C min-1. Pyrolysis temperature does not significantly affect the characteristics of activated carbons obtained. The total yield for the activated carbon production ranged between 24 and 54%, decreasing with the increase of the pyrolysis temperature. In this way, the process can be conducted at 400 ºC, achieving, thus, energy saving and higher total yields (54%). [12].**As per experimental analysis the adsorption of** the metal ions was adsorbent dosage, adsorbate concentration, contact time and pH dependent. The optimum contact time, adsorbent dosage, and pH were found to be at 105 min, 8 g and pH 8 for Fe^{2+} and at 40 min. The results revealed also a good removal of Fe^{2+} and Pb^{2+}using **rice husk** under optimum conditions with removal percentage 91% and 94% resp. [13].**Chemical activation of the sewage** sludge with $ZnCl_2$ and H_2SO_4 produced activated carbon of high adsorption capacity comparable with that of commercial activated carbon. produced activated carbon has a highly porous structure and a specific surface area of 580 m^2 /g. The adsorption isotherm data were fitted to three adsorption isotherm models. The maximum loading capacity of the produced activated carbon was 110 mg pesticides/g adsorbent. [15].

3.1 Theory of Adsorption

In this process molecules of a gas or liquid contact and adhere to a solid surface. It occurs at an interface between any two phases. Rate of adsorption of various substances on solids is due to the increased free surface energy of solids to their extensive surface. For high surface area there is need of porous size of adsorbent. Rate of adsorption on powder form adsorbent is greater than the normal due to presence of active area. The most widely used methods for potable and wastewater treatment is the adsorption method and most of the heavy metals are efficiently removed. The liquid-solid interface is carried out in water and wastewater treatment process.

1. Liquid-liquid
2. Gas-liquid
3. Gas-solid
4. Liquid-solid interfaces.

3.2 Factors that Affect an Adsorption

1. Physical characteristics of the adsorbate that is molecular size and molecular polarity,

2. Chemical characteristics adsorbate such as concentration of the adsorbate in the liquid phase (solution) and chemical composition.

3. Physical and chemical characteristics of the adsorbent, that is pore size, surface area and chemical composition.

4. Liquid phase pH and temperature.

5. The residence time of the system.

3.3 Preparation of Adsorbents

Activated carbon can be prepared from various raw materials including agricultural and forestry residues. The most of the precursors used for the preparation of activated carbon are rich in carbo. Production of AC was achieved typically through two routes, physical activation and chemical activation. Typical preparation of activated carbon involves carbonization of the raw material in the absence of oxygen and activation of the carbonized product.

1. Physical Activation

Physical activation is a two-step process. It involves carbonization of raw material followed by activation at elevated temperatures in the presence of suitable oxidizing gases such as carbon dioxide, steam, air or their mixtures. Carbonization temperature ranges between 400 oC to 800 oC and activation temperature ranges between 800 oC to 1100 oC Physical activation of various raw materials.

2. Chemical Activation

Preparation of activated carbon by chemical activation (Temperature < 800 oC) is a single step process in which carbonization and activation is carried out simultaneously. Initially the precursor is mixed with chemical activating agent, which acts as dehydrating agent and oxidant. The most commonly used chemical activating agents are H_3PO_4, $ZnCl_2$ and KOH.

Chemical activation offers several advantages over physical activation

1. Lower activation temperature compared to the physical activation temperature

2. Single activation step

3. Higher yields

4. Better porous characteristics

5. Shorter activation times.

3.4 Activating Agent and Material

1. $ZnCl_2$

Corn cob, coconut shells, macadamia nutshells, peanut hulls, almond shells, hazelnut shells, apricot stones, rice husk, tamarind wood, cattle-manure, pistachio-nut shells, bagasse, sunflower seed hulls.

2. KOH

Rice straw, corn cob, macadamia nutshells, peanut hulls, olive seed, rice husk, cassava peel, petroleum coke, coal, cotton stalk, pine apple peel.

3. H_3PO_4

Hemp, Peanut hulls, almond shells, pecan shells, corn cob, bagasse, sunflower seed hulls, lignin, grain sorghum, rice straw, oak, birch, sewage sludge, chestnut wood, eucalyptus bark, rice hull, cotton stalk, jackfruit peel.

4. K_2CO_3

Pine apple peel, corn cob, cotton stalk, almond shell, coconut shell, oil palm shell, pistachio shell, walnut shell, bamboo. During this stage, the oil was allowed to settled in the beaker for 24 hours and was decanted into another beaker, while the residue at the bottom of the beaker was discarded.

4.EXPERIMENTAL ANALYSIS

4.1 Preparation of Synthetic Wastewater

1. Take 5 mg metal powder in crucible then add 5 ml of concentrated nitric acid in to it.

2. Heat it tills all brown fumes removed.

3. The remaining blue solution in crucible will dilute in 1000 ml of distilled water.

4. If we take 5 mg solution in 1000 ml water is 5 ppm solution.

5. Similarly, we can make 10 L synthetic water of various concentrations solution.

6. For make various ppm solution follows the process steps from 1 – 4 as above mentioned.

4.2 Adsorbent from Sugarcane Bagasse

The adsorbent was selected for removal of Copper by sugarcane bagasse. It is a waste product from sugar mill mainly composed of glucose, cellulose, pentose, and lignin. Adsorbent (Sugarcane bagasse) collected from Sugar industry.

1. Crush the sugar cane bagasse and make powder.

2. The adsorbent was washed with distilled water.

3. Dried it at room temperature to avoid the release of color by adsorbent into the aqueous solution.

4. The activation of adsorbent is carried out by treating it with concentrated Sulphuric acid (0.1N) and is kept in an oven maintained at a temperature range of 150º C for 24 hr.

5. The pH measurements with a pH meter.

6. Then neutralize them with help of 0.1N NaOH or 0.1N H_2SO_4

7. Again, is washed with distilled water to remove the free acid and put in to oven for removal of moisture and then adsorbent is passed from 500-micron mesh size and collected for experiment.

4.3 From Rice Husk

1. Crush the rice husk and make powder.

2.Rice husks were washed carefully first with distilled water and then deionized water to remove particulate material from their surface.

3.Dried it at room temperature to avoid the release of color by adsorbent into the aqueous solution.

4. The activation of adsorbent is carried out by treating it with concentrated Sulphuric acid (0.1N) and is kept in an oven maintained at a temperature range of 150º C for 24 hr.

5. The pH measurements with a pH meter.

6. Then neutralize them with help of 0.1N NaOH or 0.1N H_2SO_4.

7. Again, is washed with distilled water to remove the free acid and put in to oven for removal of moisture and then adsorbent is passed from 500-micron mesh size and collected for experiment.

4.4 Experimental Procedure

1.Take 5-10 L known concentration solution.

2.Weighed amount of the adsorbent with waste water aqueous metal solutions of known concentration and pH value.

3. The metal solutions were agitated by agitator for a desired time.

4. The samples were withdrawn from the stirrer at the pre-determined time intervals and adsorbent was separated by filtration.

5. Analysis the concentration by help of the spectrophotometer.

6. Take the readings at various time of interval 30, 60 ,90 up to 120 min.

7. Analysis the optimum time of adsorption time.

8. Take the reading for various pH and also for various concentrations.

9. Follow the same procedure for different materials (Adsorbents) SCB and RH take the readings.

10. Calculate the % of heavy metals removal or adsorbed for these adsorbent SCB and RH.

11. Compare the results obtained by experimental analysis.

The % removal or reduction is given by,

% Removal of Metal = $(C_i - C_f) \times 100 / C_i$

Where, $C_i - C_f$ are the initial and final copper concentrations of metal (mg/l) in solution resp.

The adsorbed amount of metal can be calculated by,

$qe = V (C_i - C_f) / W$

where, V is the volume of the solution (L) and W is the mass of dry adsorbent used (gm).

5. RESULTS AND DISCUSSION

Adsorption of heavy metals carried out at various pH, Contact Time and different concentrations of metal solution or waste water. The different adsorbents used for the adsorption of heavy metals which are made from the agricultural waste products SCB (Sugarcane Bagasse) and the RH (Rice Husk). Experiment for adsorption of heavy metal by using SCB and RH are carried out at pH 3 – 6, Contact time from 0.5-3 hrs. and concentration of 5-30 ppm. The calculating the % of heavy metal removal with the help of spectrophotometer by comparing the units on it.

5.1 Effect of pH on Rate of Adsorption

SR.NO.	pH	% Removal of Heavy Metals with SCB	% Removal of Heavy Metals with RH
01	3	56	60
02	3.5	60	64
03	4	64	68
04	4.5	70	74
05	5	82	86
06	6	90	92

Table Effect of pH

Table shows the % removal of heavy metals from pH value 3-6 by using the SCB and RH. Table shows the effect of pH on % reduction of metals. The % removal of metal increase with increase in pH. The optimum value of pH 5-6 in which higher % of metal removed. Both adsorbents have good efficiency for removal of heavy metals.

5.2 Effect of Contact Time on Rate of Adsorption

SR.NO.	Time in hrs.	% Removal of Heavy Metals with SCB	% Removal of Heavy Metals with RH
01	0.5	45	48
02	1	52	56
03	1.5	68	64
04	2	78	80
05	2.5	84	88
06	3	90	92

Table Effect of Contact Time

Table shows the % removal of heavy metals at contact time of 0.5-3 hrs. by using the SCB and RH. Table shows the effect of contact time on %

reduction of metals. As per observation it's clear that the % removal of metal increase with increase in contact time. The optimum value of contact time is 2.5-3 hrs. in which higher % of metal removed.

5.3 Effect of Concentration on Rate of Adsorption

SR.NO.	Concentration in mg/l	% Removal of Heavy Metals with SCB	% Removal of Heavy Metals with RH
01	5	90	92
02	10	84	86
03	15	78	80
04	20	70	72
05	25	64	68
06	30	60	62

Table Effect of Concentration

Table shows the % removal of heavy metals from concentration of 5 -30 ppm by using the SCB and RH. Table shows the effect of concentration on % reduction of metals. As per observation it's clear that the % removal of metal increase with decrease in concentration. The optimum value of concentration is 5-10 ppm in which higher % of metal removed after this % reduction of metal is decrease.

5.4 Effect of Various Parameters on Rate of Adsorption

1. Effect of pH on the Metal Adsorption

Many research tasks on the adsorption of metal ion from aqueous solution showed that the pH of the solution has a great influence on the percentage of metal adsorbed. Low percentage of adsorption at low value of pH by a competition between the hydronium ions and the metal ions on adsorption site available on the adsorbent area. The percentage of each metal adsorbed increases with the pH. The percentage of each metal adsorbed as function of the pH. As per experimental study it observed that mass of adsorbent of 10 g/L and a time of contact of 3-3.5 hrs. the maximum adsorption of each metal ion occurs at pH range between 5 and 6.

2.Effect of Contact Time on Metal Adsorption

Contact time on the adsorption of copper and nickel ions on the Sugarcane Bagasse was carried out at pH 5-6 with a Bagasse mass of 5-10 g/L while varying this parameter from 0.5 to 3.5 hours. It is observed that the efficiency of metal ion adsorption is related with the increase with increase in contact time. The maximum adsorption is noticed between time of 3-3.5 hours for heavy metal.

3.Effect of Mass of Adsorbent on Adsorption

The percentage of adsorption or reduction of heavy metals depend on the of the mass of adsorbents which varied from 5 to 20 g/L the pH of the solution being maintained to 5-6 with a contact time of 3.5 hours. Adsorption increase with increase in mass of adsorbents but at particular stage its constant.

4. Effect of Concentration

The percentage of each metal adsorbed decreases with the increase in concentration. As concentration increases % removal decrease at particular stage after that rate of adsorption constant. In this experiment concentration vary between 5 – 20 ppm. As concentration increases % removal decrease at particular stage after that rate of adsorption constant.

The optimum value of concentration of metal between 5-10 ppm.

CONCLUSION

As per observation, it's clear that adsorbent produce from Coconut shell, rice husk sugarcane bagasse has good efficiency of heavy metal removal from the waste water. Adsorption is the efficient technique of removal of the heavy metal from the various types of waste water. Cost of this type of process is lowest than the convectional method.We study the removal of metal at various pH, Contact Time and the Concentration of metal shows the optimum values of all parameters. Heavy metal can easily adsorb from waste water by natural material with low cost and high efficiency.The percentage of each metal adsorbed increases with the pH. The percentage of each metal adsorbed as function of the pH. The maximum adsorption of each metal ion occurs at pH range between 5 and 6.As per observation the efficiency of metal ion adsorption is related with the contact time as contact time increase percentage of metal adsorption increase. The maximum adsorption is noticed between time of 3-3.5 hours for heavy metal at concentration of 5-10 ppm with pH value 5-6. After these optimum value removal or adsorption of heavy metals are constant.As concentration increases % removal decrease at particular stage after that rate of adsorption constant. As concentration increases % removal decrease at particular stage after that rate of adsorption constant. The optimum value of concentration of metal between 5-10 ppm.

FUTURE SCOPE AND DEVELOPMENTS

Future Scope

- Natural Adsorbent made from agricultural waste can be adopted to treat waste water.
- Adsorption Process improve the efficiency of conventional method.
- Natural Adsorbent can be used as an additional treatment to treat waste water.

Benefits

- Capital cost significantly less than convectional technologies.
- Operating cost significantly less than convectional technologies.
- Natural Adsorbent made from waste material hence cost of production is low.
- Natural Adsorbent manufacturing is easy and it can adopt in laboratory.
- Minimal operator attention.
- Consistent and reliable results.

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NOMENCLATURE

AC Activated Carbon

BOD – Biological Oxygen Demand

COD Chemical Oxygen Demand

CH - Coconut Husk

EAC - Extruded activated carbon.

GAC - Granular Activated Carbon

PAC- Powdered activated carbon

RH – Rice Husk

SCB – Sugarcane Bagasse

TSS – Total Suspended Solids

TDS – Total Dissolved Solids