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Synthesis, Modification And Characterization Of Activated Carbon And Their Evaluation For The Removal Of Heavy Metal Ions From Waste Water

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Abstract: There are two major sources of water i.e. surface water and ground water. The sources of surface are like ponds, river, sea etc. which is available for agricultural, domestic and industrial purposes. While ground water generally includes water from hand pumps, wells and tube wells. Surface water pollution is a serious problem in India at the present time. We largely depend on surface water for domestic, irrigational and industrial requirements; therefore it should be of good quality along with its aesthetic value in the scenic environment. Surface water pollution is easier to follow whereas there seems to be lack of proper understanding about the pollution of ground waters. The general impression being that after filtering or percolating through the soil cover, the waters are generally purified. While this in a way is true, the soil does not have an unlimited capacity to detain pollutants.

Key Words: Water pollution, ground water, agricultural, domestic & industrial pollution.

In U.P the crops gram, pulses, maize, cotton wheat and bajra are harvested. Agricultural wastes from these crops are available in ample amount. These wastes can be exploited for getting useful adsorbents. Although manufacturing of activated carbon from agricultural by products by conventional heating has been investigated, yet, preparation of activated carbon from agricultural and forest waste by microwave irradiation has been scarcely reported. We prepared activated carbon from 10 kinds of agricultural and forest waste such as sawdust, sugarcane bagasse, corn cob, bamboo, wheat straw, stems of beans, bean shell, rice straw, coconut coir, jute coir by microwave irradiation. The results are shown in Table-19. According to the experimental data, four stages, preheating, drying, carbonization and activation in conventional processing can be completed only 6-15 minutes by microwave irradiation. The microwave processing is 23-50 times as fast as the conventional processing.

Though activated carbon is an ideal adsorbent for organic matter due to its organophilic character, it is not economical for waste water and soil treatment owing to its high production and regeneration costs, and about 10-15% loss during regeneration by chemical or thermal treatment. High cost of activated carbon and synthetic resins has prompted search for substituents that are abundant, cheap renewable and ecofriendly. So the use of agricultural residues such as saw dust or industrial by-products like baggase have received considerable attention (Achari and Anirudhan, 1995; Gaghate et al., 1990; Haribabu, 1992; Rai and Surendra, 1999; Rangaraj et al., 1999). Most of these materials contain functional groups associated with proteins, polysaccharides like lignin, cellulose and hemi cellulose. The pollutant uptake is believed to occur through coulombic attraction and ion exchange processes involving these groups. Though these materials generally do not have high sorption capacity as compared to activated carbon, they are plentiful, inexpensive and renewable. This offers an attractive approach to the removal of metal cations in solution. The sorptive capacity of these materials could be enhanced by various physicochemical modifications to improve their physical and structural properties making them more suitable for full scale filter applications. The goal of this research is to evaluate the ability of sawdust, sugarcane bagasse, corn cob, bamboo, wheat straw, steam of beans, bean

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shell, rice straw, coconut coir, and jute coir to remove heavy metals from the glassindustry effluent as effective, low cost, biomass sorption media.

OBJECTIVES- Evaluation of sorptive capacity of modified sorbent under different conditions for individual toxic metals. Preparation of Sorbent by agrowaste by microwave irradiation

Sagaun saw dust used in this study was collected from the saw machine at Panchkuian, Agra. Coconut coir, rice husk and wheat stem were obtained from the local market.

All the agrowaste was dried in the sun, crushed, washed thrice with distilled water and rinsed with 1% HCl to remove water soluble impurities particularly metal ions and surface adhered particles. Then it was kept in 0.1 N NaOH solution overnight to remove lignin and in 0.1 N CH3COOH to remove alkalinity developed due to NaOH. Thereafter, it was washed well with distilled water till the wash water became colourless. Now it was dried at 110º C in an microwave for 10 minutes to get rid of moisture and other volatile impurities. Its carbon was prepared by keeping 4 parts of the above agrowastes with 3 parts by weight of conc. H2SO4 in microwave for 10 minutes. The carbonized agrowastes were washed with distilled water to remove free acid (SO42- ions). Then it was soaked in 1% w/v sodium carbonate solution overnight to remove any residual acid. Again it was washed with distilled water and dried in microwave for 10 minutes. The carbons so obtained were ground in a mortar with a pestle and sieved through standard sieves to get the different particle sizes less than 600 microns through the study. The carbon so prepared as above was called was pseudoactivated carbon (PAC) because actual activated carbon (AC, active carbon or activated charcoal) is obtained by the destructive distillation of wood, nut shells, animals bones or other carbonaceous material and is activated by heating upto 800-900º C with steam or CO2, which results in a porous honey comb like internal structure. The internal surface area of an actual AC averages about 1000 m2/g, and specific gravity ranges from 0.08 to 0.5. It has adsorptivity for many gases, vapours and solids, but it is much expensive than PAC.

Batch tests were conducted under different steady state and transient rate conditions such as initial toxic concentration, sorbent dose, contact time, pH and temperature. The initial and equilibrium concentrations of heavy metals in solutions were determined using a Perkin - Elmer Analyst 100 AAS. Experiments were conducted at room temperature. Desorption studies were also carried out using various strengths of acids and bases.

Characterization of the Carbons Or analysis of sorbent for their physico-chemical analysis

The carbons were characterized for 18 parameters (Strelko et al., 2002; Singh et al., 2004; Trung et al., 2003)-

A. Composition (in %); Moisture, ash, carbon, silica, alumina, iron, sodium, potassium, calcium, magnesium and phosphorous

B. Properties: pH, conductivity (µS/m), specific gravity (g/l), porosity(mg/l) surface area(m2/g).

C. Fourier transform infrared spectra were collected on a Perkin- Elmer RX1 FTIR spectrometer, using KBr pellets of the samples.

RESULTS AND DISCUSSION- The pH values were determined by mixing 1 g of material with 50 ml of deionized water and recording pH with the help of pH meter at 1h intervals for 12 h or till almost constant pH. Conductivity of the sorbents, saturation extracts were determined with a conductivity meter. The bulk or apparent density (g/ml) of the sorbent was obtained by dividing its mass by volume. The true or particles density (g/cc) is the average density of the particles of dried sorbent. It was obtained by the RD method in which the mass of sorbent is divided by the mass of water displaced. Porosity of sorbent is the fraction of sorbent not occupied by sorbent particles. The bulk density is less than true density because porosity is included in the bulk. Moisture content (%) of sorbent samples was determined by weighing about 1 g of the sorbent in silica dish before and after drying for 2 h in an oven at 110º C. After determining the percentage of

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moisture, the content in the dish was ignited at a bright red heat at 1000º C for 1 h to calculate the loss of ignition (%) or ash content. Surface area (m2/g) was determined by the glycol retention method and results were compared with BET surface area. Or it can be determined by surface area analyzer. Porosity of sorbent is the fraction of sorbent not occupied by sorbent particles. Porosity/the total pore surface area was determined by mercury porosimetry (Poresizer 9320, Micromeritics). To find the total mineral constituents of the sorbent, it was digested with HNO_3+H_2SO_4+$HClO_4$ called triple acid. Al_2O_3 (%) and Fe_2O_3 (%) were determined using 2% precipitating calcium oxalate from the HCl extract and then titrating it with N/20 $KMnO_4$ solution. MgO percentage was estimated by precipitating Mg as $MgNH_4PO_4$ from from the HCl extract and then igniting it to get MgP_2O_7 (pyrophosphate). SiO_2 percentage was determined by fusing the sorbent with sodium carbonate, decomposing the fused mass with HCl and $SiO_2.yH_2O$.

The residue was extracted with dil. HCl. filtered and the residue was ignited in a Pt crucible at about 1050º C to get SiO_2. CaO percentage was found by precipitating calcium oxalate from the HCl extract and then titrating it with N/20 $KMNO_4$ solution. The % of elements was calculated from the % of their oxides.

Adsorbents were analysed using standard methods and their properties are presented in Table 1 and 2. Such carbons are expected to be more than four times more effective than raw agrowastes.

Table: 1

Characteristics of activated carbon or sorbent prepared by microwave irradiation

SNO	Green material	Microwave irradiation (Min)	Yield of activated carbon	pH	Ash(%)
1.	Sawdust	15	75.82	7.00	4.56
2.	Sugarcane bagasse	12	76.10	7.62	8.44
3.	Corncob	15	80.13	7.21	5.55
4.	Bamboo	15	76.20	7.52	4.54
5.	Wheat straw	10	80.78	7.33	6.54
6.	Stem of beans	15	78.80	6.24	6.49
7.	Beans shell	10	78.58	6.88	8.54
8.	Rice straw	13	79.77	7.01	6.56
9.	Coconut coir	15	77.65	6.99	6.78
10	Jute coir	15	78.84	7.54	7.57

Table - 2

Parameters	Saw dust	Sugarcane bagasse	Corn Cob	Bamboo	Wheat Straw	Stem of beans	Beans shell	Rice straw	Coconut coir	Jute Coir
Compositions(in%)										
Moisture	4.23	7.66	4.35	2.45	5.54	4.56	5.68	5.55	3.27	4.87
Silica(SiO_2)	6.54	5.67	3.32	5.67	3.48	4.66	6.76	3.56	6.87	6.23
Alumina(Al_2O_3)	2.32	1.08	1.04	1.53	1.20	2.21	2.22	1.41	1.23	1.34
Iron(Fe_2O_3)	0.27	0.19	0.32	0.12	0.23	0.34	0.14	0.54	0.32	0.21
Sodium	0.11	0.15	0.08	0.09	0.14	0.15	0.10	0.06	0.13	0.10
Potassium	0.23	0.27	0.25	0.27	0.24	0.26	0.31	0.30	0.31	0.28
Calcium(CaO)	0.38	0.36	0.41	0.38	0.37	0.39	0.40	0.34	0.36	0.33
Magnesium(MgO)	0.01	0.03	0.07	0.04	0.02	0.05	0.06	0.08	0.09	0.08
Phosphorus	0.03	0.04	0.05	0.04	0.02	0.07	0.06	0.03	0.05	0.08
Properties										
Conductivity(µS/m)	0.92	0.80	0.75	0.62	0.55	0.67	0.54	0.56	0.45	0.78
Porosity(ml/g)	1.01	0.92	0.83	0.72	0.64	0.45	0.21	0.34	0.45	0.13
Surface area(m^2/g)	456	397	328	298	320	412	122	223	332	153

The physicochemical properties of carbonized agrowastes vary widely from plant to plant and method to method of carbonization. They depend on variety of plant and area in which it is cultivated, temperature of carbonization, operational features and efficiency of the heating equipment like microwave, oven or furnace. Their scanning electron micrographs can depict their morphology particularly linear or curved type fibers with holes in fibers and at other places in skeletal structure. The number and size of pores of a carbon can also be determined. The greater the number and larger the size of pores but the smaller particle size make a better sorbent. The bands of their FTIR spectra can indicate the presence of carboxy, hydroxyl, sulphonic etc. groups responsible for cation exchange.

REMOVAL OF HEAVY METALS BY SORBENTS- Four sorbents were selected for four heavy metals removal from waste water. Biological residues such as coconut coir (CC), sagaun sawdust (SS), Wheat stem (WS) and rice husk (RH) contain functional groups associated with polysaccharides, lignin, cellulose and proteins as major constituents. Many researchers have found that anion adsorption sites on such minerals like alumina and clay are aquo groups (-M-OH2) and hydroxo groups (-M-OH). The surface chemistry of an oxide in contact with an aqueous solution is determined to a large extent by deprotonation or a hydroxyl ion association reaction. There are several biological by- products in addition to CC, SS, WS and RH that potentially could be modified with physicochemical treatments. Grinding increases surface area. Carbonizing increases

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sorptive capacity. Pelletizing enhances density making the sorbent more suitable for use in packed column. Al2(SO4)3, Al2O3, SiO2, alum, lime, polyaluminium chloride, silica gel, TiO2, CeO2, polycarboxylic acids and surfactants may introduce additional hydroxo groups on CC, SS, WH, and RH to be exchanged with contaminant ions. Surfactants of opposite charge can enhance the sorptive capacity of carbon by getting adsorb it. For example, sodium dodecyl sulphate molecules may sorb onto the carbon with their anionic head groups exposed to the aqueous phase so that the PAC acts more like a cation exchange resin.

Batch experiments- All chemicals used were of analytical grade unless otherwise specified. Distilled deionised water (DDW) was used throughout the experiment. Five standard solutions of 1, 5, 10, 15 and 20 mg/l concentrations of Cr, Ni, Cu and Zn for instrument calibration and sorptive study were prepared by diluting their stock solution of 1 g/l, i.e., 1 ml 1 mg metal. To prepare the Cr (VI) stock, 2.828 g anhydrous K2Cr2O7 was dissolved in about 200 ml DDW, 1.5 ml conc. HNO3 and diluted to 1 litre with DDW. The stock solution of Ni, Cu and Zn were prepared by dissolving 1.000 g of 99.5% AR 325 mesh metal powder from CDH, New Delhi in a minimum volume of 1:1 acid (HNO3 for Ni, Cu and HCl for Zn) and diluting to 1 litre with 1% (v/v) acid.

The capacity studies were carried out by transient batch tests. In 25 numbers (Five concentrations of four metals each +5 controls one for each) of 100-ml capacity PVC bottles with screw caps, 0.05 g of sorbent was added to each bottle of five sets, each set having 6 bottles 5 for different concentrations of a metal and one for its control. 50 ml of the metal solution or effluent was added to each bottle. The solution was buffered with 0.07M sodium acetate-0.03 M acetic acid to pH 4.8. Each bottle was shaken in a reciprocating shaker at 180 rpm for 5 h at room temperature, and the metal concentration was measured per hour until the solution reached equilibrium. The controls without sorbent are to be demonstrate metal uptake due to the sorbent, not from the other sources such as the wall of the container, centrifuge tube etc. The content was centrifuged at 2000 rpm for 10 minutes and the supernatant liquid was filtered using a 0.45-µm membrane filter. The filtrate was analysed for metal ions. The initial and final concentrations of the metal solutions were determined using a Perkin-Elmer Analyst 100AAS by Sandard Methods (APHA et al., 1998). The concentration of the metal was determined by the standard calibration curve prepared under similar condition. Experiments were triplicated and results averaged.

Kinetic Studies- Kinetic studies were conducted in continuously stirred tank reactors (CSTRs) having laboratory scale baffled batch sorbers. Each unit consisted of a 2-litre plastic vessel (beaker) of internal diameter 0.13 m holding a volume of 1.80 litres toxic solution. Mixture was done by a six-bladed, flat plastic impeller of 0.065 m diameter and blade height of 0.013 mm. A variable speed motor was used to drive the impeller using a 0.005-m diameter shaft. All the six plastic baffles were evenly spaced around the circumference of the vessel, positioned at 60º intervals and held securely in place on top of the vessel that was used to obtain kinetic data and the variables namely effect of initial toxic ions concentration and effect of sorbent mass. The impeller speed was set at 350 rev. /min. All components of the batch system design: impellers, baffles and vessels, were constructed from PVC to minimize metal ion sorptions by fixtures. In all contact-time investigations, 10 g of media was added to a stirred tank that contained 1.8 litres of toxic ions solution and the timer was started. 4single-component solution of 10 mg/l toxic concentration was used. At increasing time intervals of 1 h, aliquots of 5 ml sample were drawn with a 10- ml plastic syringe upto a maximum of 10 h. The solution was immediately filtered into tubes, preserved with nitric acid and analysed on the ASS. Normalized toxic ions removal (C0?Ct)/ (C0?Ce) was compared with the overall percentage removal (C0?Ct)/C0 for various times throughout the run in the single and multi toxic

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systems. The experimental data were analysed using four sorption kinetic models: the pseudofirst order, the Ritchie second order, the modified second order and the Elowich equations.

Column experiments- Column studies were conducted with the most suitable four modified sorbent. For large-scale treatment of wastewater containing heavy metals, continuous flow absorbers are preferred. The batch reactors predict only the carbon effectiveness whereas column experiments make the study more representative.

The operating flow rate and depth of carbon are the two important process variables and these variables must be established in order to determine the dimensions and number of columns necessary for the best performance.

Column experiments were preferred in a glass column of 5 cm diameter. The activated carbon of known depth was packed into the column with glass wool at the bottom, the effluent was stored in a container fitted with a glass socket at the bottom and rubber tube with adjustable lock was connected to it.

The feed tank was placed above the column in order to facilitate the gravity flow. The effluent was allowed to pass through the carbon bed and it was collected periodically. The collected effluents were analyzed to determine the residual heavy metals concentration using atomic adsorption spectrophotometer.

Batch mode desorption studies- After adsorption experiments, the metal ion loaded carbon were separated and slightly washed with distilled water to remove unadsorbed metal ions on the activated carbon surface. They were stirred with 250 mL of HCl of various concentrations ranging from 0.00325 to 0.1 M for 12 hr. Metal ion concentrations were analysed as before. All the chemicals are used of analytical reagent grade.

Table-3

Effect of Cr, Ni, Cu and Zn concentration on their % removal at agitation time 4 h, rpm 240 and sorbent dose 1g/l, pH 4.8 and temperature 250 C

Metal	Concentration(mg/l)	RHC	CCC	WSC	SSC
Cr(VI)	1	921	900	882	850
	5	894	881	844	800
	10	841	823	767	708
	15	756	71.0	668	583
	20	680	622	560	484
Ni(II)	1	976	957	942	927
	5	970	947	926	903
	10	954	91.8	883	844
	15	91.2	860	795	71.1
	20	841	765	700	628
Cu(II)	1	942	935	91.8	880
	5	926	91.6	890	835
	10	891	868	838	760
	15	81.1	788	733	624
	20	724	687	633	540
Zn(II)	1	954	946	922	907
	5	941	93.1	905	820
	10	91.4	893	844	801
	15	851	800	751	680
	20	784	720	641	564
Cr(VI)	20	61.0	445	660	772
Ni(II)	49	730	352	885	663
Cu(II)	36	880	665	664	455
Zn(II)	56	71.0	664	783	767

* The second set of data is for metal ions present in glass industry effluents.

The uptake of metal ions at 1, 5, 10, 15 and 20 mg/l concentration by different sorbents was thus calculated and results are listed in table -3.

The data were analysed using the Freundlich and the Langmuir equations:

In a =In k +l/n In c Freundlich equation

c/a =1/qb+c/Q Langmuir equation

Where a (mg/g) is the metal sorbed per unit mass of sorbent (a=x/m where x mg of metal is sorbed on m grams of sorbent), c (mg/l) is equilibrium concentration in aqueous phase, k (mg/g) and n are Freundlich constants related to adsorption capacity and adsorption intensity respectively, Q (mg/g) and b (1/g) are Langmuir constants related to adsorption capacity of sorbent and adsorption maximum (energy of adsorption) respectively. The Langmuir constants were calculated at five initial concentrations under optimal conditions (Table 4).

Table 4

Comparison of Langmuir and Freundlich constants and correlation coefficients (R2 values) for the sorption of heavy metals by RHC, CCC, WSC

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and SSC

System	Langmuir constants and R^2			Freundlich constants and R^2		
	Q, mg/g	b, l/g	R^2	k, mg/g	1/n	R^2
CuRHC	15.9636	0.7730	0.9974	7.421	0.5026	0.9664
CuCCC	14.3752	0.7580	0.9985	6.984	0.4685	0.9528
CuWSC	13.1133	0.6130	0.9997	6.198	0.4604	0.9758
CuSSC	11.4381	0.5976	0.9998	5.101	0.4310	0.9830
NiRHC	18.7712	2.3150	0.9992	16.985	0.4822	0.9801
NiCCC	17.1762	1.4425	0.9997	11.883	0.4752	0.9483
NiWSC	15.8783	1.1180	0.9983	9.430	0.4781	0.9802
NiWSC	14.0015	0.9785	0.9964	7.412	0.4550	0.9265
CuRHC	17.3082	1.0062	0.9990	9.696	0.4838	0.9511
CuCCC	15.6277	0.9954	0.9997	8.935	0.4717	0.9822
CuWSC	14.2538	0.8526	0.9994	7.510	0.4500	0.9830
CuSSC	12.2522	0.6489	0.9976	5.538	0.4504	0.9533
ZnRHC	18.3008	1.1851	0.9978	11.150	0.5087	0.9883
ZnCCC	16.3208	1.1633	0.9986	9.921	0.4743	0.9487
ZnWSC	14.9821	0.8556	0.9997	8.268	0.8022	0.9644
ZnSSC	12.5571	0.8491	0.9138	6.876	0.4146	0.9467

Sorption capacity is found to decrease with increase in metal concentration. The higher uptake at lower initial concentration can be attributed to the availability of more isolated metal ions. Sorption rate is very rapid during initial period of contact due to the availability of more sites for sorption and more than 60 per cent of sorption is reached within 1 hour. However, equilibrium was attained after 2 h for Cr, 3 h for Zn, 3.5 h for Cu and 4.0 h for Ni. RHC could remove greater amount of these metals than any other carbon. It was found to remove 92.1% Cr(VI), 97.6% Ni, 94.2% Cu and 95.4 % Zn at concentration 1 mg/l, sorbent dose 1 g/l, rpm 240, agitation time 4 h, pH 4.8 and temperature 250 C. The order of metal removal capacities for these chemical adsorbents was RHC>CCC>WSC>SSC. The order of removal / sorption of metals was Ni>Zn>Cu>Cr. The effect of various parameters can affect the adsorption such as initial metal concentration, adsorbent dose, contact time and pH. Adsorption decreases with rise in metal concentration but increases with increase in adsorbent dose.

The rate of adsorption is high in the beginning as sites are available and unimolecular layer increases. Adsorption and desorption occur together and rates become equal at a stage called adsorption equilibrium when isotherms are applied. That is why there is little increase in % removal on increasing contact time from 4 to 5 h.

The pseudoactivated carbons may consist of oxides of silicon, calcium, magnesium, iron etc. They may have anion adsorption sites similar to minerals like alumina and clay. Such sites are aquo groups and hydroxo groups (-M-OH). The surface chemistry of an oxide in contact with an aqueous solution is determined to a large extent by deprotonation or a hydroxyl ion association reaction. However, some evidences suggest that an anion like Cr2O7- can be adsorbed ion exchange mechanism even though the surface is neutral. Cr (VI) had not been in dichromate form, it would have been sorbed to the maximum extent based on its ionic size and potential.

CONCLUSION- In the present study, the emphasis was laid down to know the efficiency of activated carbon in removing the heavy metals from glass industries waste water. This investigation was an endeavour in the direction of the treatment of waste water from glass industry before discharge into the river.

On the basis of population of agricultural wastes we selected ten agrowaste for the preparation of activated carbon. After the preparation of activated carbon they were analysed for their physico-chemical characteristics. The physic-chemical properties of carbonized agrowastes vary widely from plant to plant and method to method of carbonization.

They depend on variety of plant and area in which it is cultivated, temperature of carbonization, operational features and efficiency of the heating equipment like microwave, oven or furnace. The goal of this research is to evaluate the ability of sawdust, sugarcane bagasse, corn cob, bamboo, wheat straw, steam of beans, bean shell, rice straw, coconut coir, and jute coir to remove heavy metals from the glassindustry effluent as effective, low cost, biomass sorption media.

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