Preparation of the Adsorbents from agricultural wastes for Industrial Waste Water and Effluents Treatments

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***Abstract*- 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. Modification of agricultural by-product could enhance their natural capacity and add value to the by-product. A wide range of low-cost agricultural product and by-product sorbent enhance their natural capacity and add value to the by-product. A wide range of low-cost agricultural product and by-product sorbent and their modification for removing heavy metals from water and wastewater.**

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***Index Terms*- Waste Water and Effluent Treatment, Natural Adsorbents, Agricultural Waste Materials.**

I. INTRODUCTION

Adsorption is an ecofriendly technique used now a days to treat the agricultural waste water. The sewage water is firstly filtered through suitable low cost and ecofriendly filter medias. Then the filtered water is subjected to adsorption using low cost and easily available natural adsorbents. The combination of both these adsorbents was used for treatment of waste water with different proportion of height and contact time. Water pollution due to organic contaminants is a serious issue because of acute toxicities and carcinogenic nature of the pollutants. Among various water treatment methods, adsorption is supposed as the best one due to its inexpensiveness, universal nature and ease of operation.

Many waste materials used include fruit wastes, coconut shell, scrap tyres, bark and other tannin-rich materials, sawdust and other wood type materials, rice husk, petroleum wastes, fertilizer wastes, fly ash, sugar industry wastes blast furnace slag, chitosan and seafood processing wastes, seaweed and algae, peat moss, clays, red mud, zeolites, sediment and soil, ore minerals etc. These adsorbents have been found to remove various organic pollutants ranging from 80 to 99.9%. The present article describes the conversion of waste products into effective adsorbents and their application for water treatment.[1].

1.1 Followings are the number of materials

1. Leaf mound
2. Rice husk
3. Groundnut husk
4. Coconut husk
5. Palm pressed fibers
6. Coconut shell
7. Coconut jute
8. Coconut tree sawdust
9. Cactus
10. Wool and pine needles
11. Sugarcane Bagasse
12. Saw dust
13. Corn cob
14. Corn hulls

15. Almond shells

Natural adsorbent mention above are capable to removal of color and COD from distillery spent wash. The efficiency of decolorization and COD removal is very high by using sugar cane bagasse as natural adsorbent. [5].

Activated carbon produce from the natural material is an invaluable adsorbent used extensively in industries such as food processing, pharmaceuticals, chemical, petroleum, mining, nuclear, automobile and vacuum manufacturing to purify, decolorize, deodorize, dechlorinate, detoxicate, filter, recover salts and used as catalysts and catalysts supports.[2].

1.2 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 in the industrial environment.

1.3 Advantages of Adsorption Process

1. There is complete mineralization of oranic 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

1.4 Application of Adsorption (Adsorbent) in wastewater Treatment [5].

1. Chemical Industry - Due to the increase in various rules and regulation regarding waste treatment have enforced the application of adsorbent removal of COD, Color and organic component from waste water.

2. Pharmaceutical Industry - From these industrial wastewaters has always been troublesome owing to the wide variety of chemicals used in drug manufacturing, which leads to wastewaters of variable composition and fluctuations in pollutant concentrations. The pharmaceutical industry is in most complex organic chemicals that are resistant to biological degradation.

Adsorption are suitable pretreatment for an extremely polluted pharmaceutical wastewater, mostly due to recalcitrant compounds.

3. Pulp and Paper Industry - Number of effluents resulting from the different stages of paper making and some of these pollutants are naturally occurring wood extractives (tannins, resin acids, lignin, etc.), others are xenobiotic compounds that are formed mostly in pulp manufacture (chlorinated lignin, phenols, dioxins and furans, among others). Paper and pulp waste water / effluents are highly colored and contain high organic loads and Adsorption is effective for the treatment of pulp bleaching effluents.

4. Textile Industry - This sector known for its high-water consumption as well as the amount and variety of chemicals used throughout the different operations. Textile effluents are in a great part due to color associate large environmental problem. Nature of textile wastewaters from the dyeing and finishing stages is mainly attributable to the extensive use of various dyestuffs and chemical additives. Textile effluent characterized by high organic matter content (COD), color. By the various studies adsorption process is effective in removing COD, dyes and color.

5. Dye-Process Industrial Waste - Development of on-site wastewater treatment technologies suitable for dye process industries such as the wood-floor sector and nature of their activities these industries generate lower volumes of highly polluted wastewaters after cleaning activities. Adsorption process is potentially feasible options for treatment of these wastewaters.

6. Pretreatment to wastewater, sludge or contaminated soil

Organic pollutant destruction, Toxicity reduction and Biodegradability improvement.

7. Other Application

1. Removal of Heavy Metals from waste water
2. Removal of various dyes from waste water
3. Removal of BOD, COD and Color from the waste water
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 such as ammonia and nitrates.
8. Removal of TS, Tot-P and Total-N from wastewater

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.[3].

Azhar 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. [3].

Theivarasu 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. [3].

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. [5].

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].

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 produced activated carbon has low content of inorganic constituents compared with the precursor. The adsorption isotherm data were fitted to three adsorption isotherm models and found to closely fit the BET model with R2 equal 0.948 at pH 3. The maximum loading capacity of the produced activated carbon was 110 mg pesticides/g adsorbent [6].

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). . . . [8].

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. [9].
The activation step was realized under flow of 150 ml min-1 of carbon dioxide (CO2) 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%) . . . [9].

ADSORPTION PROCESS

3.1 Introduction

Process which leads to the equilibrium distribution between the adsorbent and the solution is nothing but adsorption. Based on the interpretation of the adsorption isotherms which translate the relation between the concentration of pollutant in solution and its adsorbed quantity. COD and color concentration and quantity of adsorbent can be calculated by using this adsorption isotherms.
Accumulation of molecules from a material dissolved in a solvent on to the surface of an adsorbent particle which is carried on adsorption process. This process can be used to take away soluble organic from wastewaters and drinking waters which is the most application of adsorption process.
For pollution control usually deals with adsorption process can control of organic compounds. Adsorption technology used in applications like wastewater purification, recovery of volatile organic compounds (VOCs), air-separation, drying of air, removal of bitter ingredients in fruit juices.
Adsorption takes place on the surface of substance is known as the adsorbent and the substance which is adsorbed is known as adsorbate. This is a surface phenomenon in which the solutes are concentrated at the surface of adsorbent. Adsorbents one which effective that have a highly porous composition so that their surface area to volume ratio is very high.
Particle of the solute component contact with the adsorbent by a combination of physical, ionic and chemical forces. As adsorbent is in contact with a solution the quantity of adsorbed solute increases on the surface of the adsorbent and decreases in the solvent. Number of molecules of solute is equal in the solvent and on the adsorbent, it represents the adsorption equilibrium.

3.2 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.

3.3 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.

Depending on the type of bonding involved adsorption can be classified as follows:

1. Physisorption

Adsorption occurs as result of energy differences and/or electrical attractive weak forces such as the Van der Waals forces is called as physical adsorption or physisorption. In this type of adsorption adsorbate molecules are physically attached to the adsorbent molecules (solid surface). Physisorption is dependent on the attractive forces between adsorbent and adsorbate.
The reversibility of physisorption is dependent on the attractive forces between adsorbent and adsorbate. If these forces are weak, desorption is readily affected. The heat of adsorption for physisorption is at most a few Kcal/mole and therefore this type of adsorption is stable only at temperature below 150°C.

2. Chemisorption
Adsorption occurs when a chemical compound is produced by the reaction between the adsorbent and the adsorbed molecule type of adsorption called as chemical adsorption or chemisorption. In chemical adsorption process procedure is one molecule thick and irreversible because energy is released to form the new chemical compound at the surface of the adsorbent and energy would be necessary to reverse the process.
Adsorption occurs when the molecules in the liquid phase are attached to the surface of solid as a result of the attractive forces at the adsorbent and overcoming the kinetic energy of the adsorbate molecules. Unlike physisorption this procedure is one molecule thick and irreversible because energy is released to form the new chemical compound at the surface of the adsorbent and energy would be necessary to reverse the process.
Both processes take place when the molecules in the liquid phase are attached to the surface of solid as a result of the attractive forces at the adsorbent, overcoming the kinetic energy of the adsorbate molecules. The substance that is being removed from liquid phase at the interface is call adsorbate or sorbent.

3.4 Classification of Activated Carbon
Classification is made for general purpose based on their physical characteristics. There are four types of activated charcoals are classified as follows.
1. Powdered activated carbon (PAC)
It made in particulate form as powders or fine granules less than 1.0 mm in size with an average diameter between 0.15 and 0.25 mm. It has large surface to volume ratio with a small diffusion distance. This is made up of crushed or ground carbon particles, 95–100% of which will pass through a designated mesh sieve or sieve.
2. Granular activated carbon (GAC)
It has relatively larger particle size compared to powdered activated carbon and consequently and presents a smaller external surface. GAC are therefore preferred for all adsorption of gases and vapors as their rate of diffusion are faster. It widely used for water treatment, deodorization and separation of components of flow system.
Granular Activated Carbon (GAC) from sugarcane Bagasse by activating at different temperatures, using molasses as a binder. Since the commercial application of activated carbon is affected by their physical and chemical properties. The objective of this study was to produce activated carbons from sugarcane bagasse, characterize them i.e. Adsorption capacity in terms of iodine and decolorizing power.
3. Extruded activated carbon (EAC)
This is combines powdered activated carbon with a binder which are fused together and extruded into a cylindrical shaped activated carbon block with diameters from 0.8 to130 mm. EAC are used for gas phase applications because of their low pressure drop, high mechanical strength and low dust content.
4. Impregnated carbon
Impregnated carbon is porous carbons containing several types of inorganic impregnant such as iodine, silver, cations have also been prepared for specific application in air pollution control especially in museums and galleries. This can be used for drinking water can be obtained from natural water by treating the natural water with a mixture of activated carbon and Al (OH)3, a flocculating agent.

3.5 Biosorption
In biosorption method microbial biomass can passively bind large amounts of metals which providing cost-effective solution for wastewater management. Biosorption method does not consume cellular energy, positively charged metal ions are sequestered, primarily through the adsorption of metals to the negative ionic groups on cell surfaces.
For the biosorption method polysaccharide coating found on most of bacterial cell-walls or other extracellular structures such as capsules and the longed contact with the metal-bearing solution. Living biomass may also be able to sequester the metal intracellularly by an active process known as bioaccumulation.
Biosorption method are more suited over the conventional methods such as precipitation, oxidation/reduction, ion exchange, filtration, membranes or reverse osmosis and evaporation have been used for the treatment of metal pollution. To describe experimental results for the sorption of metal ions by microorganism’s adsorption isotherms have been commonly used.
Adsorption isotherms are the Langmuir and Freundlich equilibrium models are most commonly used. Biosorption have been used in the last twenty years and are based on the metal sorption potential of certain natural and cheap biomasses like algae, fungi, bacteria and waste plant materials.

3.5 Biological adsorption processes

1. Algal adsorption

In this main organism that have been used in adsorption and it occurs due to the rugged nature of algal biomass. They can be cultivated easily and cheaply. Algal biomass has large surface area chelating potential are attractive for removal impurities.

2. Bacterial adsorption

It occurs due to presents of several metal-binding components contribute to the adsorption process. The bacteria like Streptomyces elongates and Pseudomonas aeruginosa are capable to removal of uranium from seawater.

3. Fungal and yeast adsorption

By the experimental study Fungal and yeast like Chitin (Polymer of N-acetyl glucosamine) which is an effective metal adsorbent and is the main chemical constituent in fungal cell wall. It has a significant adsorptive capability and it can be enhanced by chemical treatment.

4. MATERIAL AND METHODOLOGY

4.1 Preparation of (Adsorbents) Activated Carbon

Generally activated carbon can be prepared from various raw materials including agricultural and forestry residues. Generally, 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.

4.1.1 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.

4.1.2 Chemical Activation

Preparation of activated carbon by chemical activation 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 (< 800 oC) compared to the physical activation temperature (800 – 1100 oC)
2. Single activation step
3. Higher yields
4. Better porous characteristics
5. Shorter activation times.

4.2 Activating agent and Material

1. ZnCl2

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 straw, 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.

4.3 Sample preparation

Locally sourced agricultural wastes used include **Sugarcane bagasse, Coconut husk** (CH), Banana trunk (BT), Sago hampers (SW), **Rice husk (RH)** and oil palm empty fruit bunch (EFB) will be used. The agricultural wastes were cut into smaller pieces and washed extensively with running tap water to remove dirt and other particulate matter. The washed materials were dried in an oven at 105 °C for 24 hours. The products were then ground and stored in airtight containers.

4.4 Various treatment of Adsorbent

1. Acid Pretreatment: Concentrated minerals acids (H_2SO_4, hydrochloric acid or HCI), ammonia-based solvents (ammonia or NH3 and hydrazine), aprotic solvents (dimethyl sulfoxide or DMSO), metal complexes (ferric sodium titrate) and wet oxidation can be used for cellulose crystallization and disruption of lignin.

The accessibility of cellulose to enzymatic hydrolysis is increased by hemicelluloses solubilization. The pretreatment process with dilute acids is often conducted under high temperature and pressure. This process requires shorter time compared to the concentrated acid. In addition, it requires lower temperature and pressure for degradation of cellulose. The concentrated acids can be powerful and strong agents for hydrolysis but they are highly reactive, toxic and corrosive. The reactor resistant to corrosion is needed that renders the process costly. Recovery of concentrated acid is necessary to make the process economical and effective. The cost for acid recovery system is usually high.

2. Alkali Pretreatment

Oxidizing agents such as hydrogen peroxide (H_2O_2) and ozone are effective for removal of lignin. Other alkaline solutions such as NaOH, calcium hydroxide (Ca (OH), NaOH-urea, and sodium carbonate (Na_2CO_3) have also been investigated for hydrolysis of agricultural wastes.

When the pretreatments are done using 0.5-2 M alkali at a temperature between 120 and 200 °C the enzymatic hydrolysis of lignocellulosic wastes is improved and the saccharification is facilitated substantially. Pretreatment using NaOH, H_2O_2 and sodium hypochlorite ($NaHClO_3$) could enhance the crystallinity of the cellulosic biomass with alkali pretreatment processes required lower temperature and pressure compared to other pretreatment methods.

3. Chemical Pretreatment

Sustainability and cost are important criteria to be considered in pretreatment of agricultural wastes. Various chemicals have been utilized to treat the agricultural biomass for example alkali pretreatment, acid pretreatment, ozonolysis, organic solvents pretreatment and oxidative pretreatment. The pretreatment processes could result in increase in pore size and surface.

Sulphuric acid (H_2SO_4) and sodium hydroxide (NaOH) are most commonly used. The delignification and recalcitrant of lignin structure require high temperatures, combination of chemicals for a period of time and other harsh pretreatment conditions.

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