Biointerface Research in Applied Chemistry
Platinum Open Access Journal (ISSN: 2069-5837)

Review
Volume 10, Issue 6, 2020, 7243 - 7256
https://doi.org/10.33263/BRIAC106.72437256

Synthesis, Characterization and Performance of Low-Cost Unconventional Adsorbents Derived from Waste Materials

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Scopus Author ID 57209247314
Received: 11.04.2020; Revised: 30.05.2020; Accepted: 2.06.2020; Published: 7.06.2020

Abstract: Adsorption of the solute on solid is an equilibrium operation. The nature of adsorbent affects the efficiency of the operation. High surface area per unit volume is the essential attribute of a good adsorbent. Isotherm and kinetic studies are carried out to understand the adsorption mechanism. Many investigators have carried out batch experiments to study the effect of various parameters like initial adsorbate concentration, pH, particle size, and adsorbent dose. The use of adsorbents derived from low-cost materials makes this operation acceptable in industries. Regeneration of adsorbent can reduce the problem of disposal of used adsorbent material. Optimum values of affecting parameters can be determined from batch experiments. The isotherm and kinetic constants can be determined from batch experiments. These investigations give us an idea about the nature of adsorbent, maximum adsorption capacity, the order of adsorption, and optimum pH. This review sheds light on investigations on adsorbents derived from unconventional adsorbents and their characterization and performance evaluation.

Keywords: Kinetic; isotherm; pH; concentration; active sites; surface area; size.

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1. Introduction

Adsorption involves mass transfer from the fluid to solid. Certain solids have the ability to concentrate certain substances on their surfaces [1].

Removal of impurities from liquid and gaseous effluent is one of the significant applications of adsorption. The solid must possess certain physical and chemical properties if it is to be used as an adsorbent. Large surface area per unit weight is the most important criterion in the selection of adsorbent. Silica gel, activated carbon, Fuller's earth, Alumina, clays are examples of conventional adsorbents. Adsorption equilibria is a relation between the adsorbed solute concentration and concentration in the fluid at equilibrium.

Freundlich and Langmuir's isotherms are used to explain the equilibrium at isothermal conditions. First and second-order kinetic models are to describe adsorption kinetics.

2. Isotherms and Equilibria

2.1. Isotherms.

Freundlich isotherm equation is represented as [1],

$$C^* = k\,[v\,(C_0 - C^*)]^n \quad [1]$$

v ($C_0 - C^*$) is apparent adsorption per unit mass of adsorbent, k, and n are constants. C_o and C are initial and, at any time, t, concentrations of the solution. C^* is the concentration at equilibrium.

Alternatively [2-5],

$$X/M = k\, C^{1/n} \quad [2]$$

X/M is apparent adsorption per unit mass of adsorbent, k, and n are constants.

Langmuir isotherm is expressed as [4-6]

$$q_e = \frac{q0\, b\, Ce}{(1+bCe)} \quad [3]$$

where

q_e =equilibrium adsorbent-phase concentration of adsorbate (mg/ L)

C_e =equilibrium aqueous-phase concentration of adsorbate (mg /L)

Q^0 is the monolayer adsorption capacity (mg/ g)

b is the constant related to the free adsorption energy and the reciprocal of the concentration at which half-saturation of the adsorbent is reached.

These equations are linear in their logarithmic forms. These isotherm equations can be plotted on the logarithmic scale or taking their logarithms to examine their linearity. If the graph is linear with R^2 values near 0.99, the isotherm is validated.

2.2. Kinetics.

Adsorption kinetics of the fluid-solid system must be understood before its application. The kinetics models explain the mechanism of adsorption. Pseudo first order and pseudo-second-order kinetic models are used to explain the kinetics of adsorption. These models are expressed as the following equations [7-11].

$$ln\,(q_e - q_t) = ln\, q_e - k_1\, t \quad (5)$$

Where $q_{e\,(X/M)}$ is the mass of metal ion adsorbed at equilibrium (mg/g), q_t is the mass of metal adsorbed at time t (mg/g), k_1 is the first-order reaction rate constant.

The pseudo-second-order model is represented as follows [7-11].

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{t}{q_e} \qquad (6)$$

where k_2 is the second-order reaction rate constant.

3. Factors Affecting Adsorption

For a given adsorbent material and adsorbate, the following are the factors affecting batch adsorption.

3.1. Initial concentration of adsorbate.

If the initial concentration of adsorbate is high, then the driving force for adsorption is more, and adsorption is fast. This effect is expected up to a certain maximum concentration of adsorbate corresponding to the adsorption ability of the adsorbent. There is a certain maximum concentration that can be treated for a given adsorbent-adsorbate combination [12-25].

3.2. pH.

Adsorption is pH sensitive process. Depending on the positive or negative charges on adsorbate, optimum pH is basic, acidic, or neutral. Heavy metals adsorb efficiently at pH value

6 to 8. At very low pH, metal ions compete with H^+ ions. At a very high pH, the precipitate can be formed. These kinds of reactions and interactions affect the optimum pH value [12-20].

3.3. Adsorbent dose.

With an increase in adsorbent, the surface available for adsorption increases. The adsorption increases up to certain adsorbent dose, beyond which, the effect of the increase in adsorbent amount becomes insignificant. This is due to the inability of adsorbate to reach to the adsorbent surface [20-25].

3.4. Particle size.

Smaller particle size corresponds to more surface area for adsorption. However, very small particles can affect the adsorption adversely by forming a slurry. There is an obstacle to the adsorbate particles due to highly dense slurry. So, there is certain optimum particle size, below which the adsorption is adversely affected by a further decrease in the particle size [15-25].

4. Adsorbent Preparation and Activation

The preparation of adsorbent from low-cost adsorbent has given a boost to research on adsorption preparation from low-cost adsorbents [26-31]. Domestic and agricultural waste can be used for adsorbent preparation [31-35].

Thermal and chemical activation methods can be used for adsorption preparation. General steps in adsorbent preparation are Washing, cutting/size reduction, atmospheric drying, thermal drying, chemical and/or thermal activation [36-40]. Various low-cost materials are used for the preparation of adsorbent for the removal of various pollutants from wastewater [41-45].

Applicability of the adsorbent in industries depends on the adsorption efficiency, regeneration, recovery, and disposal cost of the adsorbent. Desorption studies of the adsorbent are also required before using the adsorbents in pilot plants or industries [46-51]. This review contains insight into investigations carried out to study adsorbent preparation and its characterization for the removal of adsorbates from the solution.

5. Investigations on Adsorbent Preparation and Characterization

5.1. Coconut husk, banana trunk, sago humppas, rice husk, and oil palm.

Samsuddin used agricultural wastes, namely coconut husk, banana trunk, sago humppas, rice husk, and oil palm for characterization and preparation of adsorbent in his investigation [52]. Pre-treatment using alkali, acid, and oxidizing chemicals disrupt the structure. In the pre-treatment, the properties of the biomass are altered to increase the number of sites. In the case of alkali, pre-treatment, Solvation, and saponification takes place [51]. This results in a decrease in the degree of polymerization. Acid treatment increases porosity and surface area. Low pH after acid treatment is not favorable for adsorption [52].

Okafor *et al*. used a coconut shell as a starting material for the preparation of bio-sorbent [53]. The adsorption was second order and followed intraparticle diffusion models closely. The data also followed the Freundlich isotherm.

Ju and Ezuma used Palm Kernel fiber and Coconut husks for chromium removal from synthetic wastewater [54]. They cut the material into small pieces and fibers, washed with hot water to remove moisture. They kept this material in an oven at a temperature of 80^{o}Cfor 24 hours. Coconut husk adsorbent was better adsorbent with more than 90 percent removal against 84 percent removal of Kernal fiber adsorbent. Coconut husk adsorbent followed Freundlich isotherm indicating multilayer adsorption on heterogeneous sites. Kernal fiber adsorption was a single layer and followed Langmuir adsorption.

Many similar investigations are reported on the application of coconut and groundnut waste for removal of organic pollutants like phenol and inorganic pollutants like heavy metals from wastewater [55-64]

5.2. Hard shells of apricot stones, bagasse, almond, walnut, hazelnut shells, and almond.

Soleimani and Kaghazchi used five different adsorbents in their investigation, namely hard shells of apricot stones, bagasse, almond, walnut, and hazelnut shells [65]. The chemicals used in chemical activation are dehydrating agents. These chemicals influence pyrolytic decomposition and inhibit the formation of tar[65].In the preparation of adsorbents, they studied the effect of various parameters like activation time, activation temperature, impregnation ratio on the adsorbent. The study indicated that there is a certain optimum time above which the pores enlarge and even collapse. An increase in temperature decreases the iodine number and carbon yield. Based on these studies, final parameters have to be selected for bulk synthesis of the adsorbent. The values of optimum parameters depend on the starting raw material. Activated carbon prepared from hard shells of apricot stones yielded the best properties.

5.3. Potato peels.

Kyzas and Mitropoulos carried out an investigation on the preparation of adsorbent from potato peels by the hydrothermal method [66]. They used potassium hydroxide as an impregnation agent supplemented by the thermal activation method. A pH value of 6 was found to be optimum for lead removal. Further increase in pH may result in the formation of the precipitate.

The maximum theoretical adsorption capacity was about 215-250 mg/g at the temperature range of 20 to 40 ^{o}C [66]. The adsorption data fitted in the Langmuir isotherm equation with R^2 value above 0.99.

The use of Banana peels and potato peels for the removal of pollutants from wastewater is the most sought-after research area. Taralgatti investigated adsorbent preparation and removal of copper from wastewater by using these adsorbents [67]. Copper removal above 90 percent was obtained with potato peels by her during the investigation. Adsorbent preparation steps were drying, washing, and again drying in the furnace for two days.

5.4 Mustered cake.

The mustered cake was used for the preparation of adsorbent by Singh *et al*. [68]. This adsorbent was used by them for phenol removal from wastewater. According to them, adsorption characteristics of these adsorbents are due to oxides of various elements and also the presence of silica. They used hydrogen peroxide to oxidize adhered organic matter. Scanning electron microscopy indicated homogeneous structure pores with cylindrical shapes.

The specific surface area was above 25 m^2/g [68]. The adsorption followed Langmuir isotherm, indicating monolater adsorption on the finite number of homogeneous sites. The adsorption was a pseudo-second-order.

5.5. Palm trees, waste cartons, and plastics.

Activated carbon can be prepared from mixed waste by using carbonization and zinc chloride activation [68]. Alothaman *et al*. used waste material containing palm trees, waste cartons, and plastics [69]. A three-step preparation process included carbonization, impregnation, and then activation for a specific period. The yield of activated carbon was a function of the amount of zinc chloride and activation temperature [69]. Zinc chloride inhibits the formation of volatile matter, which increases the bulk density. With an increase in temperature, the bulk density decreases due to an increase in the evolution of volatile matter [69].

5.6. Apple waste.

Ramdane *et al*. used the microwave activation method with nitrogen activation of activated carbon. They investigated the activated carbon prepared from apple waste [70]. They observed that microwave radiation temperature and time had a considerable effect on the activation of carbon. An increase in these parameters had an adverse effect on the yield. 800^{o}C and 10 minutes were the optimum value of these two parameters. Ultimate and proximate analysis endorsed apple waste as adsorbent material with high lignocellulosic material. The cadmium uptake followed Langmuir isotherm indicating monolayer and homogeneous adsorption. The microwave-assisted phosphoric acid activation method can be used for the preparation of adsorbent from apple waste for adsorption of methylene blue [71]. They carried out a separate analysis of adsorbent obtained from pulp and peel-based waste. For pulp-based adsorbent, there was a 20 percent increase in adsorption for an increase in time from 10 to 15 minutes at lower (550^{o}C) activation temperature. For higher activation temperature (700^{o}C) after 12-13 minutes, the adsorption decreases, indicating inactivation of activated sites after 13 minutes [71]. It shows that optimum time decreases with increasing temperature.

5.7. Dry fruits of acacia.

Dry fruits of acacia were used for the preparation of activated carbon for fluoride removal from wastewater by Hanumantharao *et al*. [72]. They used washing and thermal activation. Dilute nitric acid was used for washing. Before washing, the material was washed with distilled water and carbonized at 700^{o}C in the furnace. The BET surface area of the adsorbent was more than 700 m^2/g [72]. They observed that fluoride ion adsorption favored acidic conditions. The adsorbent surface was highly heterogeneous and followed Freundlich isotherm.

5.8. Banana peels.

Banana peels were used for the preparation of activated carbon by Chafidz *et al*. [73]. They used zinc chloride impregnation with 3:1 proportion. The resulting adsorbent was able to remove carbon monoxide above 97 volume percent. Banana peels were used for the preparation of biosorbent by Hossain *et al*. In this process, the banana peels were washed with distilled water and then dried at 105 ^{o}C in an oven for 24 hours [74]. According to their studies, 28 mg copper per gram of banana bio sorbent was adsorbed. The banana peel sorbent followed second-order kinetics. It followed both isotherm models, namely Langmuir and Freundlich. The advantage of this process is that it removes the need for high-temperature carbonization and hence saves much energy.

Parlayici and Pehlivan investigated the use of biowaste derived adsorbent for chromium removal [75]. The adsorbents were derived from cranberry kernel shell, rosehip seed shell, and banana peel. In the adsorbent preparation procedure, they isolated and washed cranberry kernel shells, rosehip seed shell with water, and then soaked in water. Another material banana peel was washed with water. After that, all three adsorbent materials were kept in the furnace for one day. FT-IR analysis confirmed the existence of many functional groups on the adsorbent surface [75]. Adsorption was high at lower pH values. At lower pH values, a large number of H^+ ions are formed. These ions neutralize the hydroxyl group present on the biomass. This causes hindrance to diffusion of positively charged dichromate ions [71-76]. Highly protonated adsorbent surfaces coordinate with the oxyanion present in the solution through electrostatic interaction. At pH value below 2, the removal decreases due to the formation of more polymerized chromium oxide species [73].

Fruit peels of banana, orange, citrus, lemon, and jackfruit, were used the removal of lead from wastewater by Pathak *et al*. [76]. They studied surface, physical and chemical characteristics. Analysis indicated high (85 %) volatile content in fruit peeks. The moisture content after washing and drying was about 5-8 %. Porosity was in the range of 40 % to 60% [76].

5.9. Ram horn, plantain peels, sugar cane chaff, car tire, and ram hair.

Nwufo *et al*. investigated various waste materials, namely Ram horn, plantain peels, Sugar cane chaff, Car tire, and Ram hair for preparation of bio sorbent [77]. These materials were carbonized at 200^o to 350^o C and then activated with 1 N hydrochloric acid. According to this study, the adsorption was monolayer, and the results were in agreement with Langmuir isotherm.

5.10. Datestone.

Ahmida *et al*., in their work, used date stone as a starting material for adsorbent preparation [78]. They prepared adsorbent from the date stones at three different temperatures, 300, 500, and 700^0 C. They studied the effect on nickel removal by the adsorbent. According to these studies, the adsorbent activated with sodium hydroxide was more effective than the adsorbents activated by sodium chloride and nitric acid. This research also indicated that the chemical activation method was a better option than thermal activation for these types of the adsorbent. This method exhibited changes in the surface functional groups. Also, good development of the porous structure and low activation temperature were other advantages of the chemical activation [78].

5.11. An eggshell-anthill composite.

An eggshell-anthill composite (EAC) was used for adsorbent preparation by Yusuff [79]. In his work, he investigated EAC preparation conditions, namely an activation temperature, an activation time, and an eggshell/anthill mass ratio) on the nickel ions uptake.

They carried out equilibrium studies for this solid, liquid adsorption process. Of all the affecting parameters, activation temperature had a significant effect on the equilibrium [79].

5.12. Olive waste.

Physical and chemical activation methods were used for the preparation of adsorbent from Olive waste by Enaime *et al*. [80]. Olive mill solid waste was impregnated into olive mill wastewater. They used potassium hydroxide and nitric acid as activating agents. This investigation indicated that samples activated by potassium hydroxide show a higher surface area followed by the adsorbent prepared by physical activation. Almost all the adsorbents prepared by physical and chemical activation followed Freundlich isotherm with R^2 value above 0.93. Also, adsorption of Indigo carmine on the adsorbents followed second-order kinetics.

5.13. Orange juice waste.

Cerium and lanthanum metal ion loaded phosphorylated orange juice waste was found to be very effective for fluoride removal by Ghimire [81]. He dried the waste at 30^{o}C for three days. Then they phosphorylated the waste to convert the carboxyl group to phosphate groups.

5.14. Palm date pits.

Palm date pits were used as a source of low-cost adsorbents by Esmael *et al*. [82]. They used this material for adsorption of three heavy metals, copper, hexavalent chromium, and iron. They found that particle size of 0.5- 0.8 mm was optimum for adsorption. Optimum pH was 4-7. They obtained a removal efficiency of 90 percent for these adsorbates.

5.15. Paper industry waste.

Paper industry waste contains organic matter and calcium compounds. Mensez *et al*. investigated de-inking paper sludge from recycled paper-press manufacturing (HP) and other organic sludge from eucalyptus virgin pulp mill (RT) [83]. Inorganic content was more in de-inking paper sludge adsorbent. HP leads to mesoporous material, whereas RT leads to highly microporous adsorbents. Higher copper removal was obtained by HP. Cation exchange leads to heavy metal removal.

Tanaka prepared adsorbent for cadmium removal from wastewater from solid waste of paper industry [84]. They dried and treated the sludge at 950 ^{o}C to form sludge carbon. They washed this carbon with distilled water and then again dried it at 100 ^{o}C for 24 hours to remove moisture. Their study indicates that cadmium ions precipitate at pH 8. Hence pH values higher than 8 are not favorable for cadmium removal. At very low pH, the hydrogen ions compete with metal ions. Hence pH value less than 2 affects the removal adversely. pH value near 6 favors adsorptions. Langmuir equation explained the equilibrium.

Lead removal by using paper waste-derived adsorbent was studied by Hong *et al*. [85]. They used waste from the newspaper industry and white paper industry waste. Their study indicated that Langmuir isotherm was followed by solute uptake. According to these studies, carbonate precipitation on the adsorbent surface due to the dissolution of $CaCO_3$ resulted in Pb (II) removal.

5.16. Pineapple waste adsorbent.

Yamuna and Kamraj investigated adsorptive properties of pineapple seed adsorbent with methylene blue as an adsorbate [86]. They treated dried pineapple shells with sulfuric acid and washed with distilled water. It was then dried in a Muffle furnace at 300°C for 3 hrs. The percentage of adsorption increased with an increase in pH. This may be because of the attraction between hydroxide and metal ions. Pineapple waste was acidic with pH of 5.7 [86].

5.17. Rice waste.

The use of rice as a starting material for adsorbent preparation has been done by a vast number of researchers for the removal of impurities from wastewater [66-96]. The adsorbent preparation by chemical activation method includes drying, crushing, and carbonization [97]. For chemical activation of rice husk adsorbent, nitric acid and hydrogen peroxide can be used for activation. Carbonization at around 1000°C can be done to obtain activated carbon from the rice husk. Rice husk can be combined with other agricultural waste material for obtaining better results [97, 98]. Nitrogen adsorption study confirms high surface area (above 500 g/m^2) of rice husk-based adsorbent. , The removal of methylene blue follows Langmuir isotherm [98].

5.18. Sewage sludge.

Treating waste using waste is the principle in the utilization of waste material for the synthesis of a low-cost adsorbent. Sewage sludge can be used for the preparation of adsorbent. This adsorbent was found to be effective for the adsorption of phenol from wastewater [99]. Steps involved in the preparation of adsorbent were Drying, chemical activation with hydrochloric acid, and thermal treatment at 650 °C. Freundlich isotherm and second-order model described the adsorption process.

5.19. Sunflower waste.

Waste biomass of sunflower can be used as a starting material for adsorbent preparation [100]. Sunflower head carbon and Sunflower stem carbon can be used for the removal of cadmium from water. As with many other investigations, the solute uptake followed Freundlich isotherm and second-order kinetics.

5.20. Tea waste.

Tea waste was used for the removal of phenol from wastewater by Girish *et al*. [101]. The surface area of 135 g/m^2, was comparable with other different agricultural adsorbents [102, 103]. Sulphuric acid activation is effective for improving the adsorbent properties [101-103]. Sahu *et al*. used tea waste from tea stalls for adsorbent preparation and used the adsorbent for heavy metal removal [103]. Adsorption efficiency was maximum for lead removal.

5.21. Watermelon, corn peel waste, stink bean peel powder.

Kanthasamy *et al.* used the powder of watermelon peel and corn peel for adsorbent preparation[104]. They used these adsorbents for the removal of dyes from wastewater. They observed that the maximum removal in batch studies occurred at a pH value of 3. The adsorption followed Temkin isotherm and second-order kinetics. The watermelon peel powder was found to be better adsorbent with 49 percent color removal against 19 percent color removal by corn peel. Similar experiments were carried out by Anuar *et al.* for the removal of dyes by using stink peel power[105]. They observed that the adsorption followed Freundlich isotherm and second-order kinetics.

6. Conclusions

Many investigations are reported on adsorbent preparation from waste material. Adsorbent preparation from agricultural waste is one of the most widely investigated research areas. Batch studies indicated that the optimum values of initial concentration, pH, adsorbent dosage, and particle size for a solute in a given solvent depend on the nature of adsorbent. Many investigations have been reported on the use of the novel adsorbents like ponkan mandarin, jackfruit, peanut peels, coir pitch, and cork waste [106-113]. The solute uptake by adsorption follows Langmuir or Freundlich isotherm depending on the nature of adsorption, homogeneity of the surface, and active sites. Novel adsorbents derived from sewage sludge, tea waste, an eggshell-anthill composite, ram horn, plantain peels, sugar cane chaff, car tire, and ram hair have shown promising results and can be used after detailed studies on their use in continuous contact equipment.

Optimization of the activation method can reduce time and energy. The use of chemical and thermal activation method depends on raw material composition and temperature required for activation. In most of the investigation, the temperature is optimized after conducting activation experiments at different temperatures. Various new technologies and materials are being explored to reduce treatment costs and increase the effectiveness of pollution control methods. The use of nanoparticles and nanomaterials is also a promising area of investigation in pollution control and environmental engineering[114].

Funding

There is no external funding involved in the preparation of this review article.

Acknowledgments

The author is thankful to the authorities of the affiliating institute for support and encouragement.

Conflicts of Interest

The authors declare no conflict of interest.

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