Adsorbents from *Jatropha curcas* Shells Production, Utilisation Equilibrium, Kinetics and Thermodynamic Studies of their Adsorption: A Review

***Okeola F. O., Odebunmi E. O, Adebayo G.B. and Ameen O. M.**

Chemistry Department, University of Ilorin, Ilorin, Nigeria

***** Email: okeolaf@yahoo.com; okeola.of@unilorin.edu.ng
Phone Number: +2348058749768, +2348038626501

ABSTRACT

Jatrophas curcas is a multipurpose non-edible oil bearing and draught hardy perennial shrub. It is becoming popular for its eco-restoration of degraded land and production of biodiesel. Although much has been reported about the use of the oil, the cake and the glycerine from the seed of this plant, maximum benefit can still be obtained if the waste by-products are fully exploited. The seed cake is used as organic manure. The shells which constitute 39 % of the seed and fruit pericarp, has been reported being used as raw-materials for the production of biosorbent utilized for adsorption. Adsorption is widely used as an effective physical method of eliminating or lowering the concentration of wide range of dissolved pollutants (organic, inorganic) in an effluent. In the review *Jatropha curcas* shells have either been used directly in powder form as adsorbent or converted through carbonization and activation to activated carbon a typical adsorbent. Equilibrium, kinetics and thermodynamic studies on these adsorbents, as well as their characterization were reported. Powdered *jatrophas curcas* seed coats have also been reported to be used as adsorbent to remove metal ions from waste water. Activated carbons prepared from the shells were also use to remove anions, heavy metals, organics and dyes from water by adsorption Thus *Jatropha curcas* shells adsorbents were found to serve as low cost, locally available, highly efficient and eco-friendly adsorbents.

Key words: Adsorption, adsorbent, *Jatropha curcas*, kinetics, thermodynamics equilibrium, carbonization, activation, activated carbon

Introduction

Adsorption Adsorbent, and Adsorption capacity

Adsorption is a process in which a substance is attached to the surface of solid or liquid. It is a property of an interface between two immiscible phases (Bahl, 2008). The substance adsorbed (i.e. adsorbate at a solid) (i.e. the adsorbent may be gases, liquids or other solids).

The quantity of material (adsorbate) adsorbed by a given of quantity adsorbent depends on the physical condition of the adsorbent. Most material are being adsorbed when the solid (the adsorbent) is porous or finely divided (Ramamurthi and Jaikumar,2009).

Adsorption is widely used as an effective physical method of separation in order to eliminate or lower the concentration of wide range of dissolved pollutants (organic, inorganic) in an effluent Ansari, 2009;Kipling 1956).

Adsorption may be physical or chemical process, although in some cases, both process occur simultaneously. While physi-sorption is due fundamentally to weak van-der Waal force and generally reversible, chemisorptions is adsorption involving stronger binding forces comparable with those leading to formation of compounds.

Adsorption finds numerous applications both in the laboratory and industry. Some of these include removal of poisonous gas in gas mask, gasoline vapour reclamation in gasoline vapour emission control canisters, removal of materials from solution such as heavy metal ions and colour matters in industrial effluents and as catalyst of catalytically active substances. These and other adsorption applications make the agents of adsorption, the adsorbent very important (Ansari, 2009 Okeola, 1999).

Adsorbent

Adsorptive material (adsorbent) is usually porous solids which is able to adsorb considerable amount of gas or liquid. Adsorption occurs mainly on the pore walls inside particles. An adsorbent may occur naturally or may be prepared synthetically. The most important natural adsorbent are bleaching earth and zeolites and the most important synthetic adsorbents are activated carbon, activated alumna, silica gel and ion exchange resins (Ansari 2009, Koch 1989. Almost all solids are able to adsorb a liquid or gas, but the adsorptive power of a typical adsorbent is much higher than that of the other solids because of their porous nature. Their adsorptive capacities are essentially determined by the specific surface areas which in some cases are directly related to the porosity. Table1 contain the list of primary adsorbents and their physical properties.

Among the adsorbents, activated carbon is well known and more efficient in the removal of broad spectrum of pollutant from air liquids and soil. Any carboneous materials (animal, plant and mineral origin) with high concentration of carbon can be simply changed into activated carbon.(Odebunmi and Okeola, 2001).
Several varieties of low-cost activated carbons from agricultural solid wastes can be produced, characterized and used to remove organic and inorganic pollutants from waters and effluents by adsorption process.

Adsorption capacity

Equilibrium studies of adsorption process

In the study of adsorption, the quantity adsorbed at equilibrium depends on various factors including the nature of the surface and the adsorbate, the temperature and the pressure(or concentration).If for a given system one keeps the temperature constant and studies the amount of adsorption as a function of pressure (or concentration),the resulting relationship is known as adsorption isotherm. Thus an adsorption isotherm shows how the amount of adsorbed materials depends on the equilibrium pressure of a gas or concentration of their solution at constant temperature (Alfin and Suryadi, 2011;Sharma and Sharma, 1980). It is a mean of examining an adsorbent and adsorption process. Figure 1 shows a Langmuir isotherm of adsorption of methylene blue on activated carbon from *jatropha curcas* shell (Okeola *et al* 2012)

Kinetics modeling of adsorption process

The process of adsorption is time dependence, thus the kinetics parameters which are helpful for the production of adsorption rate gives important information for designing and modeling the adsorption processes. The kinetics of an adsorbate onto an adsorbent can be carried out. Batch adsorption data can thus be analysed using pseudo-first order and second kinetic models (Ho and McKay 2000).

Thermodynamics of adsorption process

Adsorption can be studied in a range of temperature. Thermodynamic parameters can be obtained from these various temperatures by keeping other variable constant. The data obtained are the changes that occur during the adsorption in free energy, (ΔG), heat content, (ΔH) and entropy (ΔS).Thermodynamic study will show if a process will be spontaneous or not.(Babarinde *et. al.* 2008)

***Jatropha curcas* plant**

Jatropha curcas is a non-edible, oil bearing and draught hardy shrub with ecological advantages. It belongs to the Euphorbiaceae family. The *Jatropha* plants start yielding from the second year of planting, but in limited quantity. If managed properly, it starts giving 4–5 kg per tree production from the fifth year onwards and seed yield can be obtained up to 40–50 years from the day of plantation and on average the seed yield is up to 5 tons/hectare (Cano-Asseleih *et al* 1989)

. Freshly harvested *Jatropha* dried fruit contains about 35–40% shell and 60–65% seed (by weight). The fruits are 2.5 cm long, ovoid, black and 2–3 halved. The plant has nearly 422 fruits per kg available after decortications of *Jatropha* seed for oil extraction (Kumar *et al*, 2003) .Jatropha can be of maximum beneficial to man if main product(biodiesel) and by-products(seed shell, seed coat, seed cake and glycerine) are fully exploited. Seed coat is used as mulch and can be used for the removal of heavy metals from wastewater. The coats contain cellulose and lignin, and the surface of cellulose in contact with water is negatively charged that can attract cations such as Cu(II) ions. Lignin has various polar groups (alcohols,
carbonyls, acids, amino, phenol,hydroxyls a nd ethers) as potential chemical bonding agents for chemisorptions (Jain *et al*, 2008).

Jatropha curcas has been observed to grow well with more than 600mm rainfall per year and it can withstand long period of drought. The plant sheds its leaves during a prolonged dry season. *Jatropha curcas* prefers temperatures averaging 20-28 degrees Celsius (68-85 degrees Fahrenheit). It can, however, withstand a very light frost which causes it to lose all its leaves and may produce a sharp decline in seed yield. One tonne of *Jatropha curcas* seeds will produce up to 600 litres of biodiesel with proper management. Recommended planting rates of *Jatropha curcas* are 2,000 / 2,500 plants per hectare (2.5 acres). One person can professionally plant, manage and harvest 5-8 hectares of *Jatropha curcas*, 30kg of *Jatropha curcas* fruit can be harvested per person, per hour (http://www.jatrophacurcasplantations.com).

Adsorbents Produced from *Jatropha curcas*

Jatropha curcas shells have been used directly as adsorbent or converted to activated carbon a typical adsorbent. (Sinthupinyo *et al*, 2008, Jain *et al*, 2008, Ameen *et al* 2012, Okeola *et al* 2012)

Sinthupinyo *et al*, 2008, produced high surface area activated carbon from jatropha shell by either the thermal treatment under steam atmosphere or potassium hydroxide impregnation followed by nitrogen atmospheric thermal treatment. The carbon produced was successfully utilized to purify glycerol in the biodiesel production process and was said to be a better substitute for the activated carbon derived from natural coal in general.

In preparation of of this activated carbon Sinthupinyo *et al*, 2008 ,after removing the seeds procure from Thailand washed the jatropha shell sample with deionised water, dried in the oven at 378 K for 24 hours, then crushed, and sieved to the desired size. In some runs the dried sample was impregnated in 10 wt % concentration of potassium hydroxide or phosphoric acid aqueous solution with mass ratio of solution/sample equal to 1.0 for 24 hours. The solution was decanted off, and the sample was dried in the oven for 24 hours, before thermal treatment (in an electrically heating furnace).The prepared activated carbon was boiled with deionised water at 373 K for two hours, it was dried in the oven at 378 K for 24 hours, powdered, and stored in a desiccator.

The result obtained from ultimate and proximate analyses carried out on the jatropha shells used for the carbon. is summarized in table 2 The results have been observed to give low yield of AC but superior in surface area due to relatively low fixed carbon and high volatile matters contents, and also high oxygen content would promote the formation of porous structure, hence good quality adsorbent. (Sinthupinyo *et al* 2008) The AC porosity in the case of steam tend to be superior to that of nitrogen because of the extent of steam reforming. The chemical impregnation showed that the yield could be improved by phosphoric acid and potassium hydroxide respectively. Potassium hydroxide showed improved surface area (A_{BET}) which reached maximum of 520 m^2/g at 923K (Sinthupinyo *et al* 2008)

In another development in the same year, powdered jatropha seed coat was reported to be used as an adsorbent. This direct seed coat adsorbent was used effectively for removal of Cu (II) ions from waste water. (Jain *et al*, 2008)

The Jatropha seed coat procured from Vikas Sansthan, New Delhi, India was dried at 100^oC in an oven for 24 h. ground, sieved (70 mm mesh) and stored. The seed coat adsorption gave useful results as indicated in table 3 The adsorbent was subjected to various experimental studies, including kinetic equilibrium and isothermal studies. The result shows that Jatropha seed coat can be used as an effective adsorbent for removal of metal ions from waste water. It provide a reasonable adsorption capacity for Cu (II) .Adsorption was found to be influenced by solution pH, metal ion concentration and agitation time. Adsorption equilibrium was reached in 80mins and 82-

89% of Cu (II) was removed by jatropha seed coat of initial Cu (II) concentrations (20-50 mgl^{-1}). Elovich rate equation has been used to describe the kinetics of copper-seed coat sorption system at a pH of 4 and sensitivity analysis has been used to identify a true kinetic model. Metal/seed coat isotherms have been developed and analysed according to three isotherm models(equations). Freundlich and Langmuir models were found significantly better than Redlich Peterson model.
The most plausible mechanism of adsorption was found to be electrostatic attraction of Cu (II) towards lignocelluloses polar groups in powdered jatropha seed coat. The coat according to the finding contains cellulose and lignin, and the surface of cellulose in contact with water is negatively charged that can attract cations such as Cu(II). Lignin has various polar groups (alcohols, carbonyls, acids, amino, phenolic hydroxyls and ethers) as potential chemical bonding agent for chemisorptions (Jain *et al*, 2008)

In a recent development Okeola *et al* 2012, prepared and studied activated carbons from fruit pericarp and seed coat of *Jatropha curcas* using KOH and NaCl as activating agents leading to the production of four samples of activated carbons JPS, JPP, JCS and JCP (Table 4) The adsorption of the dye, methylene blue, cationic in nature was used for comparative study of the samples of activated carbons produced, under the same conditions of amount of adsorbent, contact time, temperature and pH. The results presented in Table 4 show that the activated carbon from *Jatropha* fruit pericarp activated with NaCl (JPS) has the highest adsorption capacity of 92.4 %, while *Jatropha* seed coat KOH activated sample (JCP) with adsorption capacity of 74.3 % was the least in the set. Others include *Jatropha* fruit pericarp activated with KOH (JPP) 88.5 % and *Jatropha* fruit coat activated with NaCl (JCS) 88.9 %.

The Jatropha fruits were collected in different locations in Ilorin metropolis, Nigeria. The fruit was dried and the pericarp removed to free the seed, the seed was further dried and dehulled, *i.e*. the shells were removed. The fruit pericarp and the seed coat were converted to activated carbon by chemical activation method. This was carried out by washing the *Jatropha* seed pericarp and coat with distilled water, sun dried and oven dried at 105 ^{0}C for 1 hr. The process of converting to carbon was carried out by burning in a limited air supply in a burning chamber which allowed thin cloud of gases and volatile products to ooze out. The charred product was allowed to cool to room temperature, ground to workable particle size and sieved with a laboratory sieve (75 mm mesh sieve). The sample was then purified with 0.5 M – 1.0 M HCl solution, rinsed with water and dried in an oven at 100 ^{o}C for 45 min.(okeola *et al* 2012)
Equilibrium study on adsorption was carried out and the adsorption data were analyzed using the Langmuir Isotherm. The results reported indicate that activated carbons from the fruit pericarp and the seed coat of *Jatropha curcas* can be used as high performance adsorbents with the fruit pericarp activated carbon showing the higher adsorption capacity. The adsorption data fitted well to the Langmuir model and adsorptive area of 824 – 910 m^2/g was obtained for the activated carbon.

Also. Physic nut (*Jatropha curcas* L.) shell residue from the seed used for oil extraction for biodiesel production was pyrolyzed at 400–800 °C with periods of 15, 120, and 240 min to obtain char precursors. .(Viboon S. 2008)Activated carbon, with favorable Brunauer–Emmett–Teller surface area in a narrow range, was prepared by soaking these chars in concentrated KOH, H_3PO_4, as well as a pure CO_2 gas flash activator. It was also reported that the maximum specific surface area of 532.30 m^2 g^{-1} was developed for the alkaline-treated sample. The carbon fraction of activated materials was as much as 90 wt %, significantly higher than the char precursor. Mesopore of 2–50 nm and total pore volumes of the materials were also significantly enhanced by these activations. Nitrogen adsorption isotherms of physic-nut-waste-activated carbons indicated that they were mainly mesopores. Pores of char activated by KOH and H_3PO_4 are irregular, of different shapes and sizes. According to the data obtained, physic nut residue pyrolyzed at 800 °C and followed by KOH activation confirmed to be a low-cost adsorbent with favorable surface properties.

In another related work, Namasivayam *et al* 2007) used Jatropha husk, the agricultural solid waste generated from bio-diesel industries, as a starting material to produce activated carbon. In their studies, the jatropha husk activated carbon (JHC), the feasibility of removal of toxic anions, dyes, heavy metals and organic compounds from water was investigated. Sorption of inorganic anions such as nitrate, selenite, chromate, vanadate and phosphate and heavy metal such as nickel (II) was also studied. Removal of organics such as bisphenol, 2-chlorophenol was included in the investigation. Adsorption of acid dyes such as acid brilliant blue, acid violet, basic dyes such as methylene blue, direct dyes such as direct red-12B, congo red, reactive dye like procion red were investigated as well to assess the possible use of the adsorbent. Results show that jatropha husk activated carbon can be used as an adsorbent for the removal of toxic

Activated carbon was also processed from *Jatropha curcas* shell with KOH as an activating agent. The pollutants from water, effects of reaction parameters such as activation temperature activation time and mass ratio of KOH to *Jatropha curcas* shell on activation of *Jatropha curcas* shell were studied. **(Zhao et *al*.2007)**

The structure and adsorption of the activated carbon were characterised by means of nitrogen adsorption, BET, etc.The results accordingly showed the optimum reaction conditions as follows:850℃ for activation temperature,240 min for activation time and 4:1(mass ratio of KOH to *Jatropha curcas* shell) for the ratio. Under the optimum condition, the activated carbon had iodine adsorption and specific surface of 2218.44 mg/g and 1890 m^2/g respectively, which belongs to sorbent of micro-porous class. The results also indicate a good quality adsorbent. (Shang *et al* 1999)

Preparation of activated carbon has also been carried out using steam as the activating agent by microwave heating from Jatropha hull. The response surface methodology (RSM) technique is utilized to optimize the process conditions. The influences of the three major parameters, activation temperature, activation time and steam flow rate on the properties of activated carbon were investigated to identify the significant parameters. The optimum conditions for the preparation of activated carbon were found to be an activation temperature of 900 °C, activation time of 19 min and steam flow rate of 5 g/min. The optimum conditions resulted in an activated carbon with an iodine number of 988 mg/g and a yield of 16.56% respectively, while the BET surface area evaluated using nitrogen adsorption isotherm correspond to 1350 m^2/g, with the pore volume of 1.07 cm^3/g. The activated carbon is hetero porous with the micro pore volume contributing to 40.8%. (Duan. X. *et al* 2011),

CONCLUSIONS

The use of *Jatropha curcas, shells* (i.e. fruit pericarp and seed coat)as raw material for

preparation of activated carbon has been reviewed and the findings shows that adsorbents resulting from these shells can be used effectively for adsorption requirement. The pericarp activated carbon sample was found to be an effective adsorbent for organic and inorganic
solutes from aqueous solution. The unconverted seed coat which was used directly as adsorbent has a suitable adsorption capacity to remove metal ions The specific surface areas estimated for these activated carbons from *Jatropha curcas* ,shells was found to be comparable related to the values obtained for activated carbon from similar raw materials . The review also found that the adsorption of *Jatropha curcas* adsorbent depend on pH, adsorbate dosage, initial dye concentration and contact time. The adsorption equilibrium is practically achieved in a reasonably short time. The adsorption process follows mostly Freundlich and Langmuir isotherm. The finding on kinetics data fitted mostly with pseudo- second order kinetics. Generally adsorbents from *Jatropha curcas* especially the activated carbon are found to
be highly effective for the removal
inorganic and especially organic substances from water and effluents by adsorption process. They provide an alternative means in the use of conventional and expensive methods in the treatment of industrial effluents. The use of the prepared activated carbon in the purification of glycerol in the biodiesel production would increase the profit of the biodiesel and also replaces adsorbent obtained from natural coal.

Table1 Major type of Adsorbents (Okeola 1999)

Adsorbent	Internal porosity	External porosity	Bulk density Ib/ft^3)	Surface area(m^{2}g)
Activated Alumina &	30-40	40-50	45-55	200-300
Bauxite				
Bone Char	50-55	18-20	40	~100
Silical gel	~70	~40	~25	320
Carbon	55-75	35-40	10-30	600-1400
Acid Treated Clay	~30	~40	35-55	100-300

Table 2 Ultimate and proximate analyses of jatropha shell (sinthupinyo *et al* 2008)

Ultimate analysis (wt%)	Proximate analysis (wt%)
C H N O	Volatile matter Fixed carbon Ash
43 6 1 50	90 0 10

Table 3.

Characterisation of powdered jatrophas seed coat(Jain *et al* 2008)

Character	Particle density Partcle size pH moisture loss on ignition cellulose
Value	2.99g/cm^3 55µ 7.95 8.77% 92.95%, 34.78%
Character	Hemicellulose lignin N P K
Value	24.27% 1.79 % 0.95% 0.16% 9.15%

Table 4. **Adsorption of methylene blue, acetic acid and potassium permanganate by active carbon produced from *Jatropha* fruit pericarp and seed coat oat (Okeola *et al* 2012**

Active carbon source	Activating agent	Sample code	Adsorption capacity (%)	Yield of carbonized product	Ash content (%)	Fixed carbon (%)
Fruit pericarp	NaCl	JPS	92.4	39.8	34.8	65.7
	KOH	JPP	88.5	39.8	ND	ND
Seed coat	NaCl	JCS	88.9	35.7	25.6	74.4
	KOH	JCP	74.3	35.7	ND	ND

ND = not determined

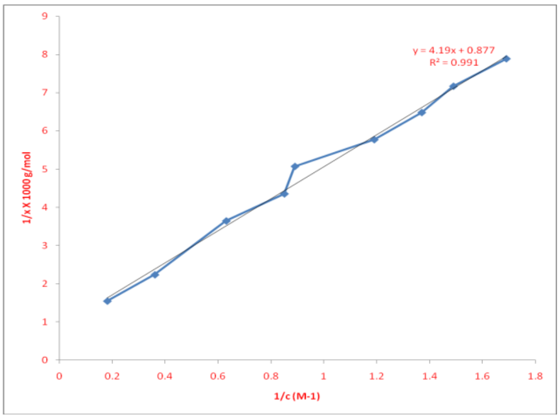

Figure 1: Langmuir isotherm of plot of adsorption of acetic acid on activated carbon from *Jathropha curca* shell

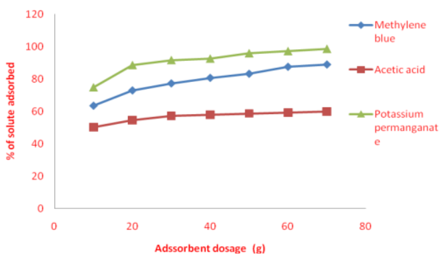

Figure 2. Effect of adsorbent (*Jatropha curcas* AC) dosage on different solute (Okeola *et al* 2012).

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