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Synthesis and characterization of plant based carbon obtained from deoiled cakes of jatropha curcus and pongamia pinnata

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Abstract
Deoiled cakes of Jatropha curcus (Jatropha) and Pongamia pinnata (Karanja) were used as raw material for producing porous carbon and their efficiency were studied in this work. Jatropha and karanja residues after oil extraction were extracted with various solvents and also pyrolyzed at 300–550 °C for 2 hours followed by soaking these in concentrated HCl, $ZnCl_2$, KOH, and 50% H_3PO_4. Surface analysis of pyrolyzed and solvent extracted char using FTIR spectroscopy and SEM indicated the functional groups, which are considerably different when compared with raw materials that lead to greater adsorption potential. Based on the results obtained, this activated carbon derived from solvent extraction and chemical activation could be used as a low-cost adsorbent. This work focused on the conversion of jatropha and karanja deoiled cakes into to activated carbon (AC). The AC produced were characterized in terms of pH, ash content, moisture content and adsorption capacity.

Keywords: jatropha curcus, pongamia pinnata, activated carbon, deoiled cakes

Introduction

Jatropha curcas, also called physic nut or purging nut, is an important industrial crop and its seeds are used as a major source of biodiesel fuel currently being used in India and other countries [1]. The plant seed consists of 60% kernel and 40% shell in which he kernel has 40–50% of oil [2]. (Singh *et al*., 2008) [2]. Biodiesel production from Jatropha generate large quantum of residual deoiled seed cake [3] and its utilization or safe disposal needs to be addressed [4] along with the value addition for bio-absorbents production was to be investigated [5, 6].
Pongamia pinnata is a drought resistant, semi-deciduous, nitrogen fixing leguminous tree [7] and contains a high oil content (31–33% by weight; wt.%) [8, 9]. The karanja seeds after tranesterification shows good calorific value as well as iodine number ans so on. However, the oil extraction process for biodiesel generates huge amount of waste residue which cannot be used as a feed for animals because of its toxicity [10, 11].
The formation of the porous structure is achieved by elimination of a large amount of internal carbon mass. High porosity carbons can be obtained only at a high degree of char burn off. For the chemical method, pyrolysis char would be impregnated with some chemical reagents, such as ZnCl2, H3PO4, NaOH, and KOH [12-16]. Preparation of activated carbon using chemical and physical activation has been widely studied using various low-cost biomass materials [17, 20].
Numerous natural materials used for the sorption of heavy metals ions from the environment, such as plant parts, algae, fungi, bacteria, fruit peels, fruits seeds, activated charcoal, rice husk, rice straw [21-24].
The most-widely used adsorbents are the activated carbons, derived from agricultural low value byproducts such as agro-wastes & residues which have the advantage of exhibiting a high adsorption capacity for organic pollutants due to their high surface area or porous structure. Besides these physical characteristics, the adsorption capacity of a given carbon produced from different sources is strongly influenced by the chemical nature of the surface and functional groups. Due to these functional groups such as carboxyl's, phenols, lactones, aldehydes, and anhydrides, the carbons have an acid–base character, it is now known that the acid or base character of carbon depends on its preparation and treatment conditions where it was oxidized [25]. Present work explores the scope of two agro-wastes, the deoiled cakes of two major oil-seeds known to yield biodiesel (*viz*. jatropha and karanja) as adsorbent, which otherwise would be a menace-because of its toxicity (rendering it unsuitable as animal feed) and magnitude (resulting from increased production of biodiesel). Therefore, this work focused on the production of AC from jatropha and karanja as a value addition strategy and its application in wastewater treatment to develop a low-cost, effective alternatives as adsorbents. Then the adsorbents developed were subjected to different activation with different solvents and activating agents, to investigate the evolution of the surface characteristic and pore structure of these developed adsorbents

Materials and methods

Plant materials Collection

Deoiled Cakes of Jatropha and Karanja were procured from Bannari amman sugar mills, Sathyamanagalam, Erode District, Tamil Nadu, India.

Pretreatment of the Raw-Adsorbents

The raw adsorbents (obtained as per 3.2.1 above) were used to carry out solvent extraction using soxhlet apparatus (at the boiling points of the solvents, at ambient pressure condition) separately using analytic grade methanol, toluene, hexane and water (using 100g/1700ml proportion of adsorbent / solvent). The study was carried out for 24 hours, followed by drying with oven at 110ºC for overnight. Besides, the raw samples were also used for acid and alkali-leaching (by keeping in dil. HCl & NaOH respectively).

Soxhelt Extraction Method

The deoiled cakes were grinded and 100 grams of powder was taken in the soxhelt apparatus with 300 ml of respective solvents used for extraction for a time of 24 hours.

Preparation of activated carbon

Deoiled Cakes of Jatropha and Karanja were dried for overnight, at 105 ^{0}C in an electric oven, followed by crushing and sieving to obtain the particles having an average diameter of 125 µm. The sample, thus obtained, was dried, desiccated and preserved for subsequent analysis and kept in air-tight containers. The activation method used involves use of 100 g of adsorbent (oven-dried) in respective reagents (H_3PO_4, HCl, KOH & $ZnCl_2$, in three-levels of dilution reagent: water being 1:1, 1:2 & 1:3) in 1: 17 v/v (sample/adsorbate), heated at 105º C in oven for 1h, followed by thorough washing of the adsorbent with distilled water, and thereafter heated in N_2-atmosphere at 300-550ºC for 2h. The samples so generated were washed with distilled water, dried and sieved for use in adsorption studies.

Fig 1: Deoiled Cakes of Jatropha and Karanja (Before and after activations)

Characterization of activated carbon

SEM-EDX Analysis

Topographic information attainable using the SEM allows that surface features such as pore characteristics, the description of which has been a significant preoccupation of AC chemicals studied and measured. EDX (Energy-Dispersive X-ray analysis) is used to figure out the elements present in the sample of specimen.

FTIR Analysis

The chemical structure and functional groups were analyzed using the infrared spectrum. The FTIR analysis of the raw and activated carbon were performed and compared.

Results and Discussion

1. Proximate Analysis of Jatropha and Karanja

The samples were analyzed for their proximate characteristics (moisture content, volatile matter, ash content and fixed carbon): The combustion characteristics (esp. proximate analysis) of the spent adsorbents were also studied and compared.

2. Moisture content

About 1g of each of the finely powdered adsorbent samples was weighed in separate crucibles. The crucible was placed inside an electric hot air oven, maintained at 105º- 110ºC. The crucible was allowed to remain in oven for 4 hours and then taken out, cooled in a desssicator and weighed. Loss in weight is reported as moisture (on percentage basis) (APHA, 1999)

$$Percentage\ of\ moisture = \left(\frac{Loss\ in\ weight\ of\ adsorbent}{Original\ weight\ of\ adsorbent}\right) X100$$

3. Volatile matter

The dried adsorbent samples left in the crucibles from above experiment were then covered with a lid and placed in an electric furnace (muffle furnace) maintained at 925º±20 ºC.

The crucible was taken out of the oven after 7 minutes of heating. The crucible was cooled first in air, then inside a desiccator and weighed again. Loss in weight in volatile matter was calculated on percentage basis (APHA, 1999).

$$Percentage\ of\ volatile\ matter = \left(\frac{Loss\ in\ weight\ of\ adsorbent}{Original\ weight\ of\ adsorbent}\right) X \quad 100$$

4. Ash Content

The residual of adsorbents of the crucibles from above experiment was again heated (without lid) in a muffle furnace at 1000 ºC for 30 minutes. The crucible was then taken out, cooled first in air, then in a desiccator and weighed. Heating, cooling & weighing was repeated till a constant weight. The residual deposits on the crucible were reported as ash on percentage basis (APHA, 1999).

$$Percentage\ of\ ash = \left(\frac{Weight\ of\ ash}{Weight\ of\ adsorbent}\right) X \quad 100$$

Table 1: Proximate analysis of raw adsorbents

Proximate analysis	Raw Jatropha	Raw Karanja	Activated Jatropha	Activated Karanja
Moisture content	4.2	9.0	1.3	3.74
Volatile Matter	65.3	77	31.69	42.6
Fixed carbon	13.5	18.9	10.2	12.5
Ash content	20.0	3.6	12.7	1.9

5. Characterization of adsorbents

Studies were carried out to evaluate the characteristics of the adsorbents used, both chemically (elemental analysis) as well as physically (micro-topographically). Using SEM-EDX, the composition of the raw adsorbents are estimated as given in table 2. Here we find that activated samples has maximum carbon content compared to other adsorbents and the raw samples has least carbon content. Jatropha has 0.5% of Sulphur, which is highest of all. Rest of the parameters are less, indicating the adsorbents are safe for combustion, but need supplements for composting due to less N, P and

Table 2: Composition of the adsorbents

Element	Weight %	Atomic %
C	57.78	66.09
O	36.42	31.27
Mg	0.11	0.06
Al	0.18	0.09
Si	3.96	1.94
P	0.20	0.09
S	0.11	0.05
Cl	0.13	0.05
K	0.21	0.07
Ca	0.68	0.23
Fe	0.21	0.05
Totals	100.00	

Karanja Raw

Element	Weight %	Atomic %
C	63.58	70.22
O	35.50	29.43
Mg	0.08	0.04
P	0.17	0.07
S	0.08	0.03
K	0.41	0.14
Ca	0.19	0.06
Totals	100.00	

Activated Karanja

Element	Weight %	Atomic %
C	59.67	67.03
O	37.81	31.88
Na	0.09	0.05
Mg	0.48	0.26
Al	0.05	0.03
Si	0.15	0.07
P	0.50	0.22
S	0.20	0.09
Cl	0.11	0.04
K	0.55	0.19
Ca	0.39	0.13
Totals	100.00	

Jatropha Raw

Element	Weight %	Atomic %
C	71.69	80.39
O	24.21	17.18
Mg	0.7	0.39
Al	0.31	0.23
Si	0.04	0.01
P	0.97	0.26
S	0.25	0.15
Cl	0.26	0.11
K	0.52	0.46
Ca	0.98	0.81
Fe	0.07	0.01
Totals	100.00	

Activated Jatropha

Estimation of Surface Morphology

Using SEM- EDX, the surface morphology of the adsorbents were studied. It can be seen from the micrographs that the external surface of the chemically acidulated adsorbent is full of alterations and striations with cavities and also can also be seen that some scatterings are found on the surface of the adsorbents (as well as, to some extent, some blockage of the entry of pores). In some adsorbents the surface was also found to be covered with several globular silica bodies that contain sharp, conical agglomerations. It was also noted that there was a thickening of the basal cell forming a surrounding brim with attendant depressions allowing the adsorbent surface to accommodate the silica bodies.

Moreover, the silica globules seemed to be more exposed due to the leveling off or shrinkage of the thick basal cell brim. The solid surface was found to be transformed to uneven textures and layers. The presence of small pores on most of the adsorbed-adsorbents' surface showed the possible development of rudimentary pore networks. The images are presented below (Figure 2 and 3).

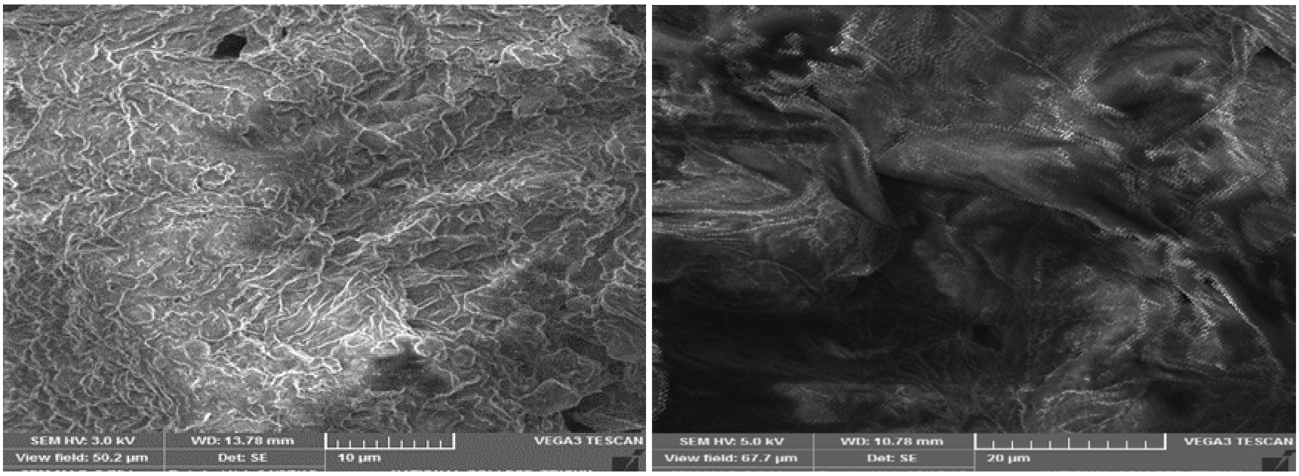

Fig 3: SEM Micrograph of Raw and activated Jatropha

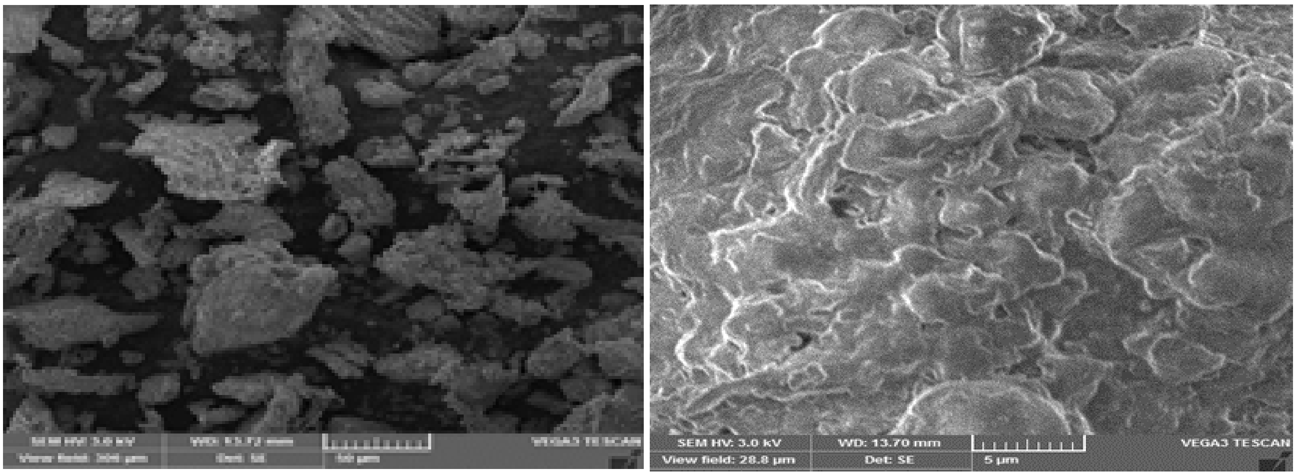

Fig 2: SEM Micrograph of Raw and activated Karanja

As we can observe in these micrographs, we find Karanja is mostly spheroidal layered, where as Jatropha are both mostly fibrous backed laminations, where the former has smaller granular matrices than the latter. Karanja on the other hand, granulated in a matrix of fine crushed particulates. All the adsorbents under study do have spaces sufficient inter-particular spaces to accommodate the dyes.

Application studies for the raw and pre-treated adsorbents

The studies indicate that 100-ppm blue dye gives 82% adsorption in all the dosages for Jatropha, at pH2. The adsorption, remains high in other concentrations, although relatively less. Effect of pH affected favorably for low pH, but not significantly. Hydro-extracted Jatropha follow the similar pattern, but higher than the raw. Methanol extraction and tolune extraction seemed to reduce the extraction with increasing pH and even dosage. With regard to karanja raw gives better result for blue dye at pH2, reducing with higher concentration. except hydro-extracted jatropha.

Based on the above results, the adsorbents can be used for further studies based on these selection are Jatropha (Jat-meth), hydro-extracted jatropha (Jat-hydro), toluene extracted jatropha (Jat-tol) and hydro-extracted Karanja (Kar-hydro) for treating different wastewater.

Conclusions

Jatropha and Karanja have been used to produce activated carbons. On this process, the solvent extraction followed by chemical activation method determines the porosity and surface area of the char. Physical activation using solvents and chemical activation using HCl, $ZnCl_2$, KOH, and 50% H_3PO_4 as the activating agent. By SEM and EDS analysis, it can be observed that amorphous and morphological characteristics of the carbon obtained by activation yields good porosity using chemical activation. The data obtained indicated that carbon contents of activated materials were 75.4–84.3%, depending upon the activation method, and notably higher than solvent treated adsorbents with higher surface area. FTIR analysis of the surface indicated main functional groups that are significantly enhances on the adsorption capability. Further studies on the adsorption efficiency of prepared activated carbon confirm the feasibility on the production of quality activated carbon from jatropha and karanja.

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