

Synthesis and Characterization of Date Palm Fiber-Based Bio-Char and Activated Carbon and its Utilization for Environmental Remediation

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Abstract

Natural agricultural products have the potential to be a substitute for synthetic polymers absorbents in environmental remediation and spill cleanup and recovery. One of these natural by-products which is available in many regions of the world is Date Palm Fibers (DPF). DPF (also known as surface fibers) is a natural sorbent that exhibits good sorption characteristics. In an attempt to valorize this abundant, low cost byproduct, we have investigated the micro porosity of this raw material by carrying out two procedures. First, hydrothermal pyrolysis using a scalable reactor with only water as solvent at high temperature (480 °C) under dry nitrogen atmosphere. Biochar yield was found varying from 30.5 to 33.6% (32%), with little bio-oil.

Then the resultant biochar was physically activated with KOH and the mixture heated to 750°C (0.5°C/min) and kept at 750°C for 2 hours under Argon flow in a tubular furnace to produce activated carbon. Second, the resulting biochar and activated carbons were then characterized by Particle Analyzer to measure their surface area, pore volume and pore diameter.

For biochar, surface area : 1.1374 m²/g, pore volume 0.001883 cm³/g, pore diameter 1355.874 Å and for activated biochar (activated carbon), surface area : 1220.2755 m²/g, pore volume: 0.062068 cm³/g, pore diameter: 19.0116 Å were determined by particle characterization analyzer. Scanning Electron Microscopy (SEM), and X-ray Diffraction (XRD) was also used for other analysis .

The oil sorption behavior of the raw DPF, biochar and activated biochar were examined with motor oil. The Oil Sorption Capacity (OSC) of raw DPF, biochar and activated biochar were tested with the oil as oil alone and an oil over water surface and the value calculated gravimetrically. The maximum OSC values of the sorbents were in the order of: DPF<biochar< activated Biochar with OSC values of 8.952, 11.712 and 33.402 g*oil/g*sorbent, respectively at a sorption time of 30 min. That indicate the OSC of the sorbents above are proportional with its surface areas.

The sorbent materials can be utilized in oil spill remediation as well as waste water treatment for removal of hydrocarbons pollutants from water. In addition biochar can be used as a soil amendment

for enhancing soil fertility.

Key words Date Palm Fiber, Biochar, Active Carbone, Sorption, Remediation.

Introduction

Petroleum products escape to the environments will have negative affect on water and land resources [1-3]. The negative impact on marine life, birds, and others are reported by many authors [4, 5]. For oil spill remediation there are many processes have been used among them chemical process such as the use of dispersants, mechanical such as “skimming” and solidification, biological like treatment with bacteria or enzymes as well as the in site burning and the use of sorbents [6-11]. Some researchers thinks many of these processes are not cheap and do not completely remove the spill materials [12]. During planning for environmental remediation one should consider a solution that will not add new problem to the environment. Natural sorbents are environmental friendly, inexpensive abundant and very efficient in oil spill clean up and so it is very promising to be used environmental remediation. Many authors have reported the effectiveness and environmental friendly of natural sorbents use for oil spill remediation. The scientific literatures are having many excellent reviews on the effective of natural sorbents for oil spill remediation among them Adebajo et al. [13], Karan et al. [14], She et al. [15] and others. Some of these sorbents which studied are peat moss, rice straws, and raw cotton, Kenaf, kapok, milkweed sugarcane bagasse, corn cob, rice hulls and others [1, 10, 15-21].

The literatures estimated about more than 100 million date palm trees worldwide and Iraq have around 18 million [3]. There are about 1,130,000 tons of date palm trees by-products are produced annually [22]. However, in Arab countries Date Palm (*Phoenix dactylifera-L*) trees by-products such as Date Palm Fibers (DPF) are in great abundance, but it have not thoroughly explored. DPF, as described by Zaid and Wet [23], “the base of the frond is a sheath encircling the palm. This sheath consists of white connective tissue ramified by vascular bundles. As the frond grows upwards, the connective tissue largely disappears leaving the dried, and now brown, vascular bundles as a band of tough, rough fiber attached to the lateral edges of the lower part of the midribs of the fronds and unsheathing the trunk. The palm date tree stem (trunk) is covered with a mash made of single fibers. Usually, these fibers create a natural woven mat of crossed fibers of different diameters”. Trimming of the date palm trees is carried out annually and with it a huge amount of date palm fibers are produced as by product [23].

In the last 20 years, much attention have given to natural fibers as substitutes to the artificial fibers. DPF (some call it Date Palm surface fibers) application have been growing in many sectors [24-26].

DPF have been used as reinforced material in Epoxy composites [27-29] and fillers in polymeric composites [30-32]. In addition DPF were utilized as sorbent for removal of copper, lead and crystal violet dye from aqueous solutions [33-35]. The empty palm fruit bunch was studied as oil sorbent and factors affecting its absorption capacity have been reported [36].

While DPF as lignocellulosic natural fiber, its utilization as sorbent for oil spill remediation from sea surface are promising. To our best knowledge there is no work have been published on the application of DPF as sorbent for oil spill remediation outside this group. The effects of temperatures and weather on OSC of DPF were studied [37].

Bansal and Goyal considered activated carbon” as universal adsorbent for effluent treatment and is commonly used for the remediation of various pollutants samples” [38].

Scientific data shows that several works have been done on the synthesis of activated carbons from date palm biomass [39-41]. Very recently, Joardder et al. [39] produced bio-oil and activated carbon from date palm seeds in a fixed bed reactor using pyrolysis technique. Date seeds and leaf was studied as potential feedstock for bio-fuel and bio-char production by Pyrolysis process [40]. Al-Zubaidy studies the impact of activated carbon from date palm kernel powder for the remediation of water polluted with oil [41]. Ahmad et.al. reviewed in his paper the adsorption capacities of different adsorbents from date palm-based for the remediation of different pollutants (dyes, heavy metals, pesticides, oils, organic compounds, etc.) [42]. Jibril et al. [43] investigated the effect of different activated agents such as H_3PO_4 and KOH in carbonization of date palm trunk. Date stones were used by many researchers as basic sources for the production of activated carbon [44-46].

While as the authors know, there is no one have attempted to produce Biochar or active carbon from date palm fibers (surface fibers). To improve the performance of date palm fibers in oil spill cleanup and pollution remediation we produced Biochar from DPF and then active Biochar. All the materials were evaluated in oil sorption process. In addition, the research other objective is to develop a process to convert the by-product fibers into a usable value-added end product.

Material and Experimental Procedure

Materials

The Date Palm Fibers employed in this research were obtained from date Palm trees planted in Basrah region south of Iraq. Before cutting it to small pieces, the mesh mats palm date fibers (PDF) was intensively cleaned with distilled water to remove unwanted dusts and then air dried and then cut in to small pieces (6-8 mm). The dried cut (PDF) pieces (6-8 mm) were crushed in Willey mill crusher and sieved to be used. The one which passed the 1.0 mm hole sieve but did not pass 0.1 mm hole, (0.1-1.0

mm) was selected for this work. Then it was sieved again to remove all the small and fine particles. Specifications of Date Palm fibers studied are; its light brown Colour with size between 0.1-1.0 mm diameter and density each one gm. occupied 10.2 mL.

Oil Samples: The oil used through all experiments was a motor oil (SAE-30, AMSOIL, Toronto, Canada) having kinematic viscosity around 9.3-12.4 (cst) at 100 °C, ash content is < 0.5% (company information) and Specific Gravity of 0.8687 (calculated from density/ ASTM D-1298 (API Gravity)).

X-Ray Diffraction: The XRD analysis was done according to manufacturer. A Bruker (UK) D2 PHASER instrument with scattering angle, 2 θ , scanned from 7° to 70° was used to analyse the crystalline structure of the Biochar and active carbon samples (30 kV, 10 mA using Cu K α radiation).

Particle Characterization Analysis / Analyzer: The biochar and activated biochar were analyzed for surface area, pore volume and pore diameter using Tristar II 3020 analyzer (Micromeritics Instrument Corporation, Norcross, GA, U.S.A) with N₂ gas adsorption/ desorption isotherm.

Scanning Electron Microscope (SEM): The SEM images of DPF and active carbon were taken on a Hitachi SU3500 Variable Pressure Scanning Electron Microscope combined with an Oxford Aztec X-Max50 SDD energy dispersive detector at the Surface Science Center, University of Western Ontario, London, ON, Canada. The images show at Figure (1).

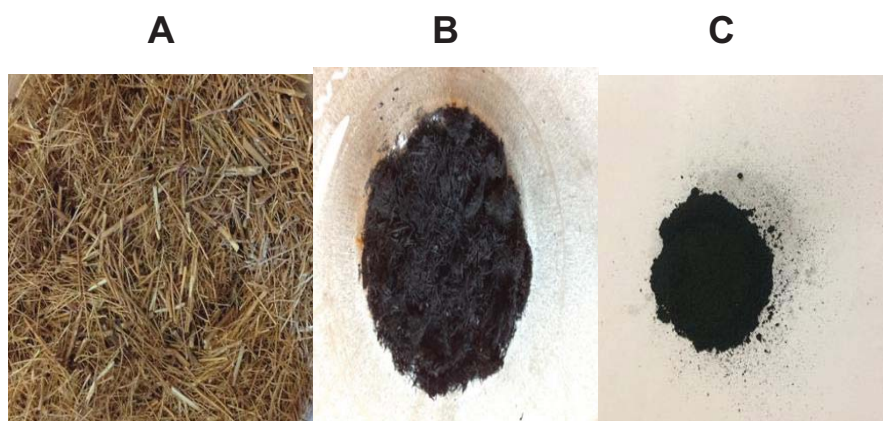


Fig. (1) Photos of (A) DPF, (B) biochar, and (C) activated carbon.

Biochar preparation procedure: The pyrolysis experiments were performed on 10 gm. of dried date palm fiber along with two hundred mL of deionized (DI) water was sealed in stainless steel of 600 mL Hastelloy C-276 reactor (Autoclave Engineers, Erie, Penn, U.S.A), with a sweep Nitrogen gas connection. The reactor was heated externally by an electrical vertical furnace with a programmable control up to 480 °C for two hours. A K-type thermocouple was used to control the

temperature inside the reactor. While pressure reached up to 3870 psi at the highest temperature during the pyrolysis. The resultant biochar was recovered by vacuum filtration and washed with DI water multiple times then vacuum dried overnight at 120°C. Figure (1), shows photos of DPF, biochar and activated carbon.

Activated Biochar Preparation: The dry biochar was thoroughly mixed with KOH/NaOH using a mortar and pestle, then heated to 750°C (0.5°C/min) and kept at 750°C for 1h under Argon flow in a tubular furnace(21100 Tube Furnace, Barnstead Thermolyne, USA). The resulting product was thoroughly washed with diluted HCl followed by deionized water until the pH reached 7.0. Finally the products were vacuum dried overnight at 120°C.

Sorption Process: To investigate the behaviors oil sorption of the sorbent materials, oil spill has been simulated in a 250 mL glass beaker by adding about 18 g of motor oil to about 200 ml of fresh water flowing the procedure of Hussein *et. al.* (47) with little modification. For the sorption of the oil spill 0.200 g of DPF sorbent material was spread gently over the surface of the oil in 250 mL. The beaker was little gaited by hand gently for particular times (5- 60 min). The thickness of the spilled layer is about 7.5 mm. All the experiments were run at temperature of 25±2 ° C. In case of biochar 0.100 g Sorbent were moved up vertically with wire net and the sorbent was let to drain by hanging the net over the beaker for 4 min. The sample weight was determined and recorded. Sorption behaviors of DPF with motor oil only was studied also. About 50 g of oil was transferred into 250 mL beaker and the same weight of sorbents were spread gently over the surface of oil in the 250 mL beaker. Other steps close to that steps above. Sorption capacity of activated biochar was determined by placing a 0.050 g of the powder in small filter paper beg and transferred it into 250 mL beaker contained about 50 g motor. The wet oil bag removed, let to drain for 4 min and weighed. The same process was done with blank filter paper only to determine the amount of oil adsorbed by the paper.

The sorption process test setting for the research study is shown in Figure (2).

The DPF and biochar tests were run in triplicate at least and the average was taken. The Relative Standard Deviation (RSD) was less than 7%.

The OSC of the sorbent were obtained with the formula as amount of motor oil sorbed divided by the weight of sorbent used:

$$\text{OSC} = \frac{\text{New weight gain}}{\text{Initial weight of sorbent}}$$

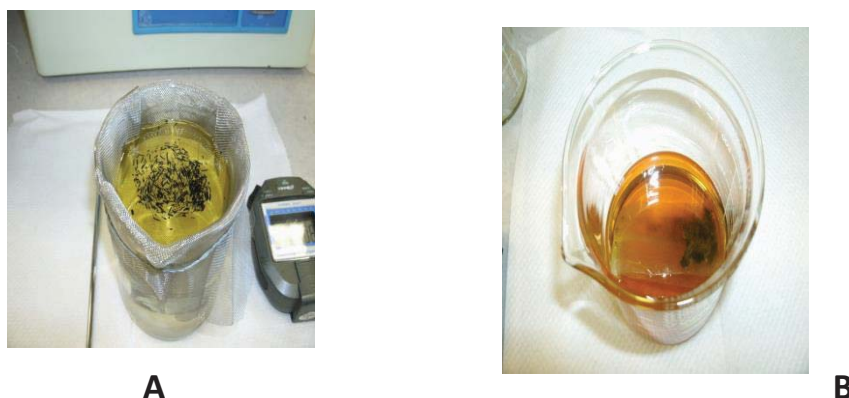


Fig. (2) Photo of the, A. Biochar sorption set for oil over water, B. the activated Biochar sorption set for oil only

Result and Discussion

The date palm lignocellulosic fibers are composing mainly from cellulose, hemicellulose, waxes and lignin. While cellulose and hemicellulose are hydrophilic, waxes and lignin is hydrophobic. The outer layer of the fiber is expected to be lignin with a central lumen (void) [28,30]. Biochar yield from DPF was found to be about 32%, with little bio-oil.

Scanning Electron Microscope; Across section SEM image of a DPF (Fig. 3 A) reveals a large number of hollows (lumens) at a single fiber collected and bonded by a layer with larger lumen at the center. The same observations were also reported in the literature on DPF (28, 30). Fig. 3 B shows the SEM image of DPF surface. Rough surface and large cavities are very clear. The large number of lumens as well as rough surface and cavities are contributing to high sorption capacity of DPF. Fig. 3 C is image of active carbon from DPF biochar. The presence of large quantity of nano-sized flakes could contribute to the high surface area of the active carbon and ultimately to high oil sorption.

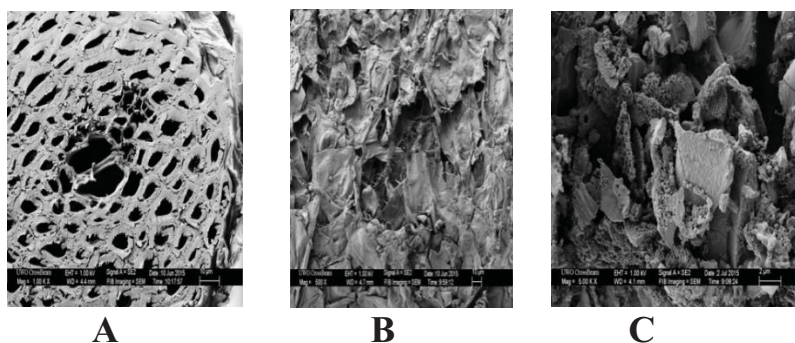


Fig. (3) Scanning electron micrographs (SEM) images of Date Palm fiber; A (cross section), B (surface) and C for active carbon of DPF.

XRD Analysis: The XRD pattern of the biochar and activated biochar of DPF are shown in Figure (4) It is observed that two broad peaks appear at 24° and 40° of biochar and 26.6° and 39.6° of activated biochar in the crystalline patterns which typify α -cellulose XRD pattern [36].

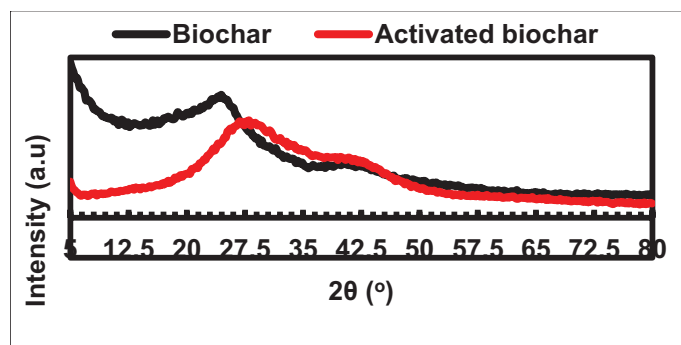


Fig. (4) XRD patterns of biochar and activated biochar of date palm fiber

The diffraction patterns at 2θ positions, 14.63° , 18.84° and 22.24° of biochar show the presence of microcrystalline cellulose I and lignin as reported in [36]. Moreover, this shift in the peaks between biochar and activated carbon are an indication of increase in the crystallinity index of activated carbon over the biochar. This increases due to the removal of amorphous material during carbon activation at high temperature [28].

Nitrogen adsorption analysis: Measuring pore structure of adsorbents by the using of inert gases is essential before liquid sorption experiments. The results of gas sorption analysis as, for biochar, surface area : $1.1374 \text{ m}^2/\text{g}$, pore volume $0.001883 \text{ cm}^3/\text{g}$, pore diameter 1355.874 \AA and for activated biochar (activated carbon), surface area : $1220.2755 \text{ m}^2/\text{g}$, pore volume: $0.062068 \text{ cm}^3/\text{g}$, pore diameter: 19.0116 \AA . The increases in the surface area are due to increases in porosity of active carbon and decrease in particle size of activated carbon. The increase in porosity can be attributed to the release of tars from cross-linked framework generated by the treatment with KOH at high temperature

Sorption Study: Motor oil sorption's behavior for DPF and biochar at oil only and oil over water are quite similar, with sorption at oil only very little higher than that of oil over water [18]. For DPF, OSC there are increases till 15 min and then remain nearly the same after that. While for biochar, OSC continue to be very closed throughout the tested times with small variations. In case of DPF the increase in OCS for the 5 and 15 min sorption time due to absorption of motor oil into hollows and cavities of the DPF [18, 19]. After, 15 min the lumens and pores would be filled or the large

molecules of motor oil subjected to size exclusion that prevent it from entering the hollows and could be both and so there are no farther increase in OSC [48]. In case of biochar, it seem the OSC remain same during all sorption periods tested. This indicated the sorption was very rapid due to phase transferred from biopolymer in DPF to an amorphous phase in biochar [48]. Since amorphous phase have larger pore sizes it accumulate more oil at short time, which what happened for biochar. The impact of the sorption time, expressed in minutes, on OSC behavior with DPF, biochar and activated biochar was shown at Table (1). The results show that sorption process is time dependent mostly. As expected, the amount of oil sorbed increased with sorption time as in DPF and activated biochar.

Table (1) The motor oil sorption capacity behavior of date palm fibers, biochar and activated biochar with effect of sorption time on that with flowing fixed factors: 1-draining time 4 min. 2- DPF and biochar tests for oil over water while activated biochar oil only 3- room temperature

S. No.	Sorption Time (min)	OSC of DPF	OSC of Biochar	OSC of Activated Biochar (Net)	OSC of Activated Biochar (total)
1	5	7.574	11.729	31.257	50.712
2	15	8.952	11.028		
3	30	8.803	10.955	33.402	52.632
4	45	8.869	11.800		
5	60	8.790	11.688	35.000	54.607

In addition the large molecules size and short range molecules of motor oil compounds from C20 to C34 contributed to short period for pore-filling and so there is little increase in OSC [48, 49]. The high values of OSC for activated biochar due to high surface area and increases in the pore volume and pore diameter as analysis showed (Surface area of activated biochar 1220.2755 m²/g, pore volume: 0.062068 cm³/g, pore diameter: 19.0116 Å) which can accommodate more large molecules of motor oil. There are continue increases in OSC which differ from DPF and biochar that may be due the fine powder of activated biochar. Motor oil will take longer time to penetrate all the fine particles.

The sorption-desorption taking place during sorption times and create small up-down in OSC values were probably due to change in the Surface tensions (surface free energy) of the solid and the oil. Although researchers did not explain why this phenomenon occurred [18].

The sorption processes of oil into DPF, biochar and activated biochar are in two ways, adsorption and absorption. Adsorption is the physical adherence or bonding of ions and molecules onto the

surface of sorbent e.g. DPF and others. The adsorption of oil onto DPF surface is dominant by the mechanism of van der Waals forces and hydrophobic (oleophilic) interactions between fiber surface (covered by lignin) and oil. The roughness of the fiber surface has an important role in oil sorption as it enhances the adsorption through increasing the surface area [17, 47, 50-52]. In addition the adhesive energy of the oil on the solid is contributing to adsorption of oil into the surface of sorbent [18]. The adsorption is rapid and takes place just when oil contacts the sorbent (DPF, biochar and activated biochar). While the absorption is the penetration of oil into the interior pores. In addition the DPF have amulti-hollows (lumens) in its center which gives more void volume for absorbed good amount of oil [18]. The absorption process is slower than adsorption. So, increase in sorption with increasing the contact times are mostly due to absorption. The mechanism of absorption is manage by diffusion process of oil as well as capillary pressure action [18]. In case of motor oil the absorption are very in DPF and biochar as molecular diameter are large compare to pore diameter (48).

Conclusions:

Date palm fibers as waste byproducts of date palm cultivation turn to value-added product with the pyrolysis process to produce biochar. The pyrolysis increase the porosity and crystallinity date palm derived biochar.

The same trend is with biochar derived activated carbon which has greater surface area. This characteristic give active carbon high sorption capacity flowed by biochar and then DPF. The high temperature of pyrolysis e.g. 480 °C and high temperature of activation of the biochar contributed to more porosity and higher surface areas for both biochar and activated carbin.

In addition active carbon has great potential to be used in waste water treatment as it have great affinity to sorb organic pollutants and also can be use in polluted soil remediation as well as a soil amendment for enhancing soil fertility.

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