Application of Agricultural Wastes Activated Carbon for Dye Removal – An Overview

Amirza, M. A. R.[1], *Adib*, M. M. R.[1,*], and *Hamdan*, R.[2]

[1]Department of Water and Environmental Engineering, Faculty of Civil and Environmental Engineering, Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia.
[2]Department of Chemistry Engineering Technology, Faculty of Chemistry Engineering Technology, Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia.

Abstract. Dyes are an important class of pollutants and can even be identified by the human eyes. Disposal of dyes in precious water resources must be avoided especially those that are not easily biodegradable, however, and for that various treatment technologies are in use. Among various methods adsorption occupies a prominent place in dye removal. The growing demand for efficient and low-cost treatment methods and the importance of adsorption has given rise to agricultural waste. This review highlights and provides an overview of these activated carbons prepared by non-woody and woody materials and their application for dyes removal. From a comprehensive literature review, it was found that many researchers used non-woody material as activated carbons to removal dye contaminants.

1 Introduction

Saving water to rescue the planet from water polluted situation and to make the future of mankind being safe is what we need now. With the growth of mankind, society, science and technology our world is reaching to new high horizons but the cost which we are paying or will pay in near future is surely going to be too high. Among the consequences of this rapid growth is environmental disorder with a big pollution problem. Besides other needs the demand for water consumption ("Water for People Water for Life" United Nations World Water Development Report UNESCO) has increased tremendously with agricultural, industrial and domestic sectors consuming 70, 22 and 8% of the available fresh water, respectively and this has resulted in the generation of large amounts of wastewater [1] containing a number of 'pollutants'.

In recent years, properties of dyes solution as their visibility and recalcitrance generate problem for various industries such as manufacturing of fabrics, paper, silk, cotton, cosmetics and textile [2-16] in order to color their products [4, 6, 10, 17-28]. Wastewater contaminated by synthetic azo-dyes have complex aromatic molecular structure which make them inert and biodegradable [12, 14-15, 29-31] difficult when discharged into the environment [3, 5, 8, 10-11, 16, 26] if not being removed properly [13, 27, 32-34]. The

* Corresponding author: adib@uthm.edu.my

worldwide production of synthetic dyes is estimated at 7×10^5 to 1×10^6 tons dyestuff [28] produced annually with 10%-60% of dye-containing effluents are emitted into the environment without proper treatment [5, 9, 12, 22, 28, 30, 35]. Dyes contaminants from industrial disposal cause severe problems associated, as dyes given toxicity [12, 14-15, 27, 30-31] to the aquatic flora and fauna life that are present in the ecosystem [2-4, 8-11, 20-23, 25, 36]. The effluent of dyes may damaging the aesthetic nature of the environment [2, 4, 18, 23, 25, 27, 32, 35, 37-38] includes visible pollution, reduce photosynthetic activities and not readily biodegradable by the indigenous [2, 4-6, 8, 22, 25, 28, 31, 36, 38], even at low concentrations [15, 17, 19, 35]. As well as the potential mutagenic and carcinogenic effects of cancer [2, 3, 5, 9, 11, 15, 19-23, 28, 31, 36] after transformations or degradation yield compounds such as benzidine, toluidine and other aromatic amines [8, 25, 32, 35, 39-40] are indiscriminately. Where certain dyes were determined to have a direct effect on the newly formed micronuclei, chromosomal breakage and aneuploidy in human cells [19]. The dyes also can cause severe damage to human beings such as allergy dermatitis [3, 9, 36], skin irritation [3, 9, 26, 36], dysfunction of kidney [25], reproductive system, liver, brain and central nervous system [2, 20, 25].

Conventional techniques available for dye removal such as photo-catalytic degradation [3, 7-8, 15, 21, 25, 36], Fenton degradation, electrochemical degradation [3-4, 8, 14, 21-22, 26, 33, 36, 41], electrocoagulation [8, 11, 15, 26], liquid-liquid extraction [21], irradiation, biodegradation [6], photochemical [11], coagulation-flocculation [12, 16, 20, 27, 37], membrane filtration [6, 16, 21, 27, 29], ultrafiltration membrane [3, 15], ion exchange [29, 32] and persulfate oxidation [2, 16-18, 27, 40] is efficient and economically feasible but it also create secondary pollution notably spent catalyst and fouled membrane that requires further disposal [19]. However, these conventional methods have proven to display low effectiveness of dye removal inherent constraints due to their high cost [13, 29], high energy requirements and generation of hazardous by-products [2, 6, 32, 33, 37, 38] caused by their complex aromatic compounds [25].

In additional to already mentioned methods, the adsorption process has been widely being used for dyes removal. Adsorption techniques particularly by carbon adsorbents has emerged to be the best one of the most promising alternatives [3, 5-6, 9-12, 22-23, 27, 30] efficient technology, which finds diverse applications in several disciplines of purification and decontamination processes [34] for the removal of organic pollutants from wastewaters. Factors that affect the adsorption technique in terms of initial cost, simplicity of design, ease of operation, high removal efficiently, regeneration capacity and insensitivity to toxic substances [5, 18-19, 21-22, 25-26, 29-30, 40] have been found to be among the most effective and proven treatments of dye-loaded wastewater. The adsorptive properties of activated carbon depends on the surface area, good internal porous structure, high thermal stability, low acid/base reactivity and chemical structure of the surface [35, 38, 43].

The most important characteristics of an adsorbent are the quantity of adsorbate it can accumulate which is usually calculated from the adsorption isotherms. Dyes that are difficult to biological breakdown can often be removed by using the adsorbents. A good adsorbent should generally possess a porous structure (resulting in high surface area) and the time taken for adsorption equilibrium to be established should be as small as possible so that it can be used to remove dye wastes in lesser time.

2 Production of Activated Carbon from Agricultural Waste

Generally, activated carbons (ACs) is considered as the most commonly used adsorbent in water and wastewater treatment worldwide [16, 42]. The properties of ACs are a tasteless, solid, microcrystalline, non-graphitic form of black carbonaceous material with a porous

structure [16]. ACs has been regarded as a unique and versatile adsorbent due to its extended surface area [26, 28-29, 31], micro porous structure [26], good chemical and mechanical stability, high adsorption capacity [26, 29]. The high degree of activated carbon surface reactivity are caused by heterogeneous porous structures [28-29] which make them perfect adsorbents [11, 15, 17-19, 21, 27, 32-33, 36, 43-46]. The ACs can be produced from a variety of natural and synthetic substances, where lignocelluloses materials being one of the most used precursors [42]. The composition of the lignocelluloses material regarding the cellulose, hemicellulose and lignin content determines, to some extent, the porosity development of the produced ACs [24, 28]. However, a major challenge of commercial available activated carbon (CAC) are still considered expensive materials for many countries due to the use of non-renewable and relatively expensive starting materials such as coal [2-7, 9, 12-13, 19, 22, 26-28, 30, 35-36, 38, 46] but poor regeneration limit its potential usage in the treatment system [2, 7, 11, 13, 27-28, 32, 37]. Commonly in market, commercial activated carbon (CAC) was sales in range of RM90.00/Kg, however for production of sugarcane bagasse activated carbon (SBAC) was in range of RM12.00/Kg. For that reason, production of SBAC using lower heating temperature (500°C) and using raw material from agricultural waste compared with CAC being used higher heating temperature (≥ 1000°C).

Therefore, ACs obtained from agricultural byproducts has the advantage of offering an effective [4, 27, 47,], low cost replacement for non-renewable coal-based granular activated carbon (GAC) [3, 8-11, 24, 27-29, 36] may provide that they have similar of better adsorption efficiency [25,43] such as solid pineapple waste [17], oil palm empty fruit bunches [44], *Thevetia peruviana* [18], cattail [46], peanut shell [40], rambutan (*Nephelium lappaceum*) [35], banana empty fruit bunch (BEFB) and *delonix regia* fruit pod (DRFP) [43], palm shell waste-based [37], coir pith [32], orange peel [38], sawdust and rice-husk [19]. The production of activated carbon prepared from agricultural waste can be revised based on non-woody materials (Table 1) and woody materials (Table 2).

Apparently, activated carbons are prepared by physical and chemical activation processes [15]. In physical activation also known as pyrolysis process, the precursor is being carbonized at high temperature and activated by passing CO_2 of steam under pressure to increase the porosity and surface area [8, 28]. Meanwhile, in chemical activation, both activation and carbonization process take place simultaneously in which the raw precursor is impregnated with activating agents and heated at desired temperature [5, 8, 25, 28, 48]. Chemical activation leads to both physical and chemical modifications on produce activated carbon. The agricultural wastes provide both woody and non-woody materials for adsorption methods replaced with commercial activated carbon. For woody materials composed lignocellulose only compared with non-woody materials composed sugar, starch, lignocellulose and oils [49]. Many authors used non-woody materials due to the composition of materials such as starch and lignocellulose which have high pore structure compared with woody materials.

Based on the review of Table 1 and Table 2 for materials woody and non-woody, it can be concluded that specific surface area of activated carbon produced depends significantly on the type of materials and activating agents used in the adsorption processes. Specific surface area was important parameters used in evaluating many capabilities of powders and porous materials such as activity, adsorptive performance and catalytic performance [50]. The factors affecting on the measured value of specific surface area of activated carbon such as degassing temperature, degassing time and adsorption point number.

Certain agricultural waste products have been tested and proposed for dye removal. Which agricultural waste adsorbent is better to being used? There is no direct answer to this question due to their each agricultural waste has its specific physical and chemical characteristics such as porosity, surface area and physical strength, as well as inherent

advantages and disadvantages in wastewater treatment. However, it is clear from the present literature survey that agricultural waste adsorbents may have potential as readily available, inexpensive and effective sorbents. They also possess several other advantages that make them excellent materials for environmental purposes such as high surface area (Table 1 and 2).

Table 1. Non-woody materials of agricultural waste activated carbon.

Raw precursor	Chemical used in impregnation method	Pollutants in dye removal	Specific surface area analysis (BET) (m^2/g)	Ref.
Pomegranate peel	$ZnCl_2$, nitric acid solution	Dye direct blue 106	-	[2]
Orange peel	H_2SO_4	Dye direct navy blue 106	-	[3]
Coir pith	H_2SO_4	Dyes reactive orange 12, reactive red 2 and reactive blue 4	Coir pith carbon (598) and Granular coir pith carbon (557)	[12]
Rice husk	H_2SO_4	Dyes crystal violet, direct orange and magenta	98.27	[13]
Rice husk	-	Dyes congo red and magenta	-	[14]
Sugarcane bagasse	$ZnCl_2$	Basic dye	-	[15]
Hazelnut bagasse	$ZnCl_2$	Dye acid blue 350	1489	[16]
Solid pineapple waste biomass, SPWB (crown, leaves, stem)	$ZnCl_2$	Dye methylene blue (MB)	1002 (leaf), 955 (stem), 794 (crown)	[17]
Thevetia peruviana	Na_2SO_4, H_3PO_4, $ZnCl_2$, KOH, HCl, H_2SO_4, direct pyrolysis, dolomite, H_2SO_4 + H_2O_2	Dyes methylene blue (MB), Basic green 4, Acid violet 49, Reactive orange 4 and Direct blue 71	329.27 (pyrolysis), 205.63 (Na_2SO_4), 862.39 (H_3PO_4), 503.77 ($ZnCl_2$), 561.1 (KOH), 156.24 (HCl), 390.69 (H_2SO_4), 85.93 (Dolomite), 299.85 (H_2SO_4 + H_2O_2)	[18]
Sawdust and rice-husk	-	Dye acid yellow 36	Sawdust carbon was 516.3 while rice-husk was 272.5	[19]
Silk cotton hull, coconut tree sawdust, sago waste, maize cob, banana pith	H_2SO_4	Dyes were rhodamine-B, congo red, methylene blue, methyl violet, malachite green	-	[20]
Flamboyant pods	NaOH	Dyes acid yellow 6, acid yellow 23 and acid red 18	2854	[23]
Esparto grass	-	-	1122	[24]
Avocado peel	-	Dyes naphthol blue black and reactive black 5 and basic blue 41	53 (raw material) and 452 (after carbonization)	[25]

Table 1. Non-woody materials of agricultural waste activated carbon (*Cont...)*

Raw precursor	Chemical used in impregnation method	Pollutants in dye removal	Specific surface area analysis (BET) (m^2/g)	Ref.
Sugarcane bagasse pith	H_3PO_4 and $ZnCl_2$	Dye reactive orange	-	[27]
Grape processing waste	$ZnCl_2$	Dyes methylene blue and metanil yellow	1455	[28]
Apple pulp and peel	H_3PO_4	Dye methylene blue	1552 (apple peel) and 1103 (apple pulp)	[29]
Orange peel	H_3PO_4	Dyes methylene blue and rhodamine B	1090	[30]
Prickly pear peels, broccoli stems and white sapote seeds	H_3PO_4	Dyes acid blue 74, direct blue 80, basic blue 9 and basic violet 3	1025 (prickly pear peels), 1177 (broccoli stems) and 1043 (white sapote seeds)	[31]
Coir pith	$ZnCl_2$	Dyes acid brilliant blue, acid violet, methylene blue, rhodamine B, direct red 12B, congo red, procion red, procion orange	Precursor presence of $ZnCl_2$ was 910 and 167 for absence of $ZnCl_2$	[32]
Jerusalem artichoke stalk-based	$ZnCl_2$	Dyes methy orange and methylene blue	Jerusalem artichoke AC was 1632 compared to PAC was 1410 and 582 for GAC	[33]
Pomela skin	NaOH	Dyes methylene blue and acid blue 15	52 (Char) and 1335 (PSAC)	[34]
Rambutan (*Nephelium lappaceum*)	KOH	Dye acid yellow 17	971.54	[35]
Orange peel	H_2SO_4 + $NaHCO_3$	Dye methylene blue	-	[38]
Tomato processing solid waste	$ZnCl_2$	Dyes methylene blue and metanil yellow	1093	[41]
Date palm leaflets	KOH	Dye methylene blue	823	[42]
Banana empty fruit bunch (BEFB) and *Deloxia regia* fruit pod (DRFP)	H_3PO_4, KOH	Dye methylene blue (MB)	BEFB for 0.32 (untreated), 15.38 (H_3PO_4) and 1.04 (KOH) DRFP for 17.55 (untreated), 22.29 (H_3PO_4) and 0.01 (KOH)	[43]
Cattail	H_3PO_4	Dyes Neutral red and Malachite green	1279	[46]
Corncob	H_3PO_4	Dye methylene blue	1809	[47]

Table 1 represents some of the non-woody materials of agricultural waste of activated carbon reported. From the recent literature reviewed, adsorbents that stand out for high surface area are flamboyant pods (2854 m^2/g), corncob (1809 m^2/g), Jerusalem artichoke

(1632 m^2/g), apple peel (1552 m^2/g), hazelnut bagasse and grape waste with the surface area of (1489 m^2/g) and (1455 m^2/g). As usual, the solution pH is an important parameter in the adsorption process due to the interaction effect of the surface functional groups of the adsorbate and the adsorbent. Besides that, the properties of the activated carbon and of the dyes may indicate whether adsorption process is favorable in acid or base solutions.

[23] stated that at initial concentration of 500 mg/L, the maximum removal percentages of activated carbon from flamboyant pods were 99.88%, 99.87% and 98.97% for acid yellow 6, acid yellow 13 and acid red 18 dyes, respectively. Activated carbons prepared by Jerusalem artichoke stalk based mesoporous (MAC) were studied as adsorbents [33] for adsorption removal of anionic (methyl orange, MO) and cationic dyes (methylene blue, MB). It was observed that the amount of dyes adsorbed, q_t (mg/g) values increased from 170.2 mg/g to 223.7 mg/g with the pH value increased from 3.44 to 10.6 at initial 60 min for MO dye, but different condition for anionic dye (MO) stated that the q_t value decreased from 238.1 mg/g to 179.3 mg/g when pH was increased from 3.15 to 10.7 at initial 60 min. For instance, the MAC becomes negatively charged, which may produce a considerable high electrostatic attraction to simulate the MB moving toward the MAC surface, but when pH value was lower than pH_{pzc} (3.70) condition, the surface of MAC is positively charged by adsorbing H^+ ions which is attractive to the anionic dye due to electrostatic repulsion between MO and negative charges on the MAC surface. Therefore, the authors suggested that the electrostatic interaction attraction may enhance the adsorption rate but is not the primary adsorption mechanism between the cationic dye and the partially negative charge MAC surface.

Grape processing waste (GW) was studied by Saygili *et al*., (2015) for the adsorption of methylene blue (MB) and metanil yellow (MY) dyes. The authors investigated the impregnation ratio of $ZnCl_2$ increased from 1:1 to 1:6 ($GW/ZnCl_2$), the surface area values also increased from 911 m^2/g to 1361 m^2/g, respectively. It is obvious that impregnated with chemical as an activating agent is very efficient in order to produced ACs with high surface area and porosity. Due to the chemical activation, the equilibrium data for both dye adsorptions onto grape processing waste activated carbon (GWAC), showing the maximum monolayer adsorption capacity of 417 mg/g for MB and 386 mg/g for MY. This may attributed to the fact that $ZnCl_2$ selectively extracted H and O away from the GW rather than ACs. Hence, this causes eventually to an increase in the surface area and porosity [51].

Due to their low cost and local availability, agricultural wastes such as Fox nutshell and Holm oak acorn are classified as woody materials (Table 2) and can produced activated carbon used as adsorbents for dye removal [10, 21]. Recently, [21] studied the removal of methylene blue and phenol onto activated carbon from Fox nutshell (FNAC) by chemical activation ($ZnCl_2$). The maximum % removal of MB was observed at pH 11. Similar results are reported for the adsorption of methylene blue on Jute fiber and wheat shells [52-53]. When the pH value decreased at pH 3, the authors found that the low adsorption rate of MB occurred on FNAC due to the positive charged on the surface, causing H^+ ions to compete effectively with MB cations. The equilibrium adsorption of MB was increased from 249.88 tom968.74 mg/g which indicates that the MB adsorption process onto FNAC was exothermic in nature.

[10] studied that the highest micropore percentages was obtained using KOH as an activating agent for activated carbon from Holk oak acorn. Further, in order to know the effect of chemical treatment and to improve its efficiency the authors also tested the potential of the adsorbent by treating it with H_3PO_4 and using $ZnCl_2$, found that activated carbons obtained by H_3PO_4 had a mesopore structure while those obtained from $ZnCl_2$ had a heterogeneous pore distribution consisting of micropores and mesopores. Similar to the findings of [54] preparation of activated carbons from sewage sludge via chemical activation using H_3PO_4 may contribute large mesoporous and some microporous being

observed for all activated carbons. The increase in surface area of ACs due to a phosphoric acid reagent has been explained in the reviews as H_3PO_4 aiding the thermal decomposition of the lignocelluloses material.

Table 2. Woody materials of agricultural waste activated carbon.

Raw precursor	Chemical used in impregnation method	Pollutants in dye removal	Specific surface area analysis (BET) (m^2/g)	Ref.
Coconut shell	H_3PO_4	Dye yellow (Tartrazine E102)	-	[4]
Cocoa shell	Red mud + lime + KOH + $Al(NO_3)_3$ + Na_2SO_4	Dye reactive violet 5	-	[5]
Macore fruit	NaOH	Dyes methylene blue and methyl orange	229.51	[6]
Cashew nut shell	KOH + TiO_2, KOH, TiO_2	Dyes brilliant green and methylene blue	-	[7]
Walnut and poplar woods	H_3PO_4	Dye acid red 18	-	[8]
Coconut shell	H_3PO_4	Dye reactive blue 19	-	[9]
Holm oak acorn	KOH, $ZnCl_2$, H_3PO_4, H_2SO_4	Dye disperse orange 30	968 (H_3PO_4, 550°C), 1305 ($ZnCl_2$, 750°C), 582 (KOH, 750°C)	[10]
Apricot stones	H_3PO_4 + HNO_3	Dyes methyl orange and methylene blue	359.40 (H_3PO_4 + HNO_3) and commercial activated carbon (510.57)	[11]
Fox nutshell	$ZnCl_2$	Dye methylene blue (MB)	2869	[21]
Spent coffee grounds	KOH	Acid orange 7 and methylene blue	704.23	[22]
Peanut sticks wood	HCl and HNO_3	Dye methylene blue	218.89	[26]
Olive stone	-	Dye methylene blue	-	[36]
Palm shell-waste based	NaOH	Dye methylene blue (MB)	731.50	[37]
Peanut shell	Conventional pyrolysis (P sample) and microwave irradiation followed by pyrolysis (MW-P sample)	Dyes Direct black 38 and reactive red 141	370.10 (P sample) and 395.80 (MW-P sample)	[40]

3 CONCLUSIONS

The majority of the reviewed studies concluded that a wide range of activated carbons prepared from agricultural waste as adsorbents of dye removal. It is worthwhile noting that the removal of dyes can be done by various materials such as non-woody and woody waste. However, there exists no such methodology of which activated carbons can successfully remove all types of dyes at low cost adsorbents. The agricultural waste is converted into value-added materials as activated carbons, which are generated in huge amounts annually. The modification of the surface chemistry of carbons is also an important influenced,

although depending on the raw materials of activated carbon, both increases and decreases in surface area have been reported. It has been generally shown that many researchers using agricultural waste from non-woody materials compared to woody materials in term of removal dyes in water polluted. As a reason, non-woody materials is ease to be conducted and chemical composition of these agricultural waste have high ash content and fiber such as hemicellulose, cellulose and lignin which may reduce the mechanical strength and adsorption capacity of activated carbon.

As environmental regulations become stricter, the effectiveness and cost of treatment processes for dye becomes more significant. Instead, the dye removal data available in literature suggests that removal of dyes is possible by agricultural waste to a certain extent since some promising results are obtained in some of the studies. Therefore, it is understood that impregnation ratio of chemical activating agents significantly affect to porosity. With low impregnation ratio, the formation of tar is inhibited and the release of volatiles is promoted, producing more mesopores. Rather at higher impregnation ratio, the more swelling impregnated precursor materials and stronger release of volatiles in the activation process will be lead to the widening of pores: micropores formed are subsequently converted to mesopores [55]. The application of activated carbon in adsorption process was mainly depends on the surface chemistry and pore structure or porous carbons. If possible, develop modification of surface chemical activation method on activated carbon prepared by non-woody material should be provided. Various methods should be produced such as acidic treatment and alkaline treatment.

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