AGRICULTURAL WASTE ACTIVATED CARBON CHARACTERIZATION AND PARAMETER FACTOR EFFECT ON TEXTILE DYE REMOVAL: A REVIEW

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ABSTRACT

Activated carbon are widely used in industrial wastewater treatment operation because of the effectiveness in removing dye. Removal of dye wastewater are importance as with small amount of dye causing undesirability and contain pollutant that can influent the quality of water. This review focus on the characterization and effectiveness of agricultural waste activated carbon use to remove dye in textile wastewater. This review provided information on activated carbon proximate and ultimate analysis including activated carbon characteristic such as BET surface, bulk density, SEM, FTIR. The operational factor effecting the removal of dye is also reviewed in term of adsorbent dosage, contact time, pH solution, dye concentration. This review approach agricultural activated carbon to be applicable in large scale used and as replacement of high cost conventional activated carbon.

Keywords: Activated carbon, adsorption, dye.

INTRODUCTION

Generally, nearly 100,000 types of dyes are available in colour index nowadays and the worldwide annually produce over 7.0x105 tonnes per year [1-3]. The synthetic dyes gained popularity in textile industries due to their variety in colour, plus their stability to light and detergent [4]. Dye is a coloured substances that widely used in textile process to produce bright and lasting coloured when binding with fabrics [5]. The textile industries discharged more than 100 tones dyes per year into stream worldwide [6]. Thus without a proper treatment, the disposal of textile dye can contribute to major environmental and ecosystem problem as dyes in textile wastewater are carcinogenic, mutagenic and toxic [7]. Therefore, many different technique has been studied by researchers to treat dyes in wastewater [8-10].

Physical treatment using adsorption method gain popularities in removing pollutant in water due to their efficiency [11, 12]. Adsorption is an efficient technique which can be applied for the removal of soluble and insoluble contaminant and pollutant [13-15]. There are several type of material can be as an adsorbent which is sea material (i.e: chitosan, yeast), natural material (i.e: clay, zeolite), miscellaneous material (i.e: starch), agricultural waste (i.e: kenaf, bagasse) and industrial waste (i.e: tyre) which include in the term of activated carbon (AC) [16-18]. Activated carbon was reported having the best efficiency in removing water contaminant [19-21]. The wide range of activated carbon use in many countries will develop the continuing increment in activated carbon demand. Activated carbon is known in black, solid substance and have a large surface area with strongest physical adsorption forces [22,23]. There are three physical form of activated carbon can be produce depending on their application which is granular, powdered and pellet activated carbon.

Agricultural waste has become one of the main sources in production of activated carbon [13, 20, 24]. By the conversion of agricultural waste into activated carbon, another environmental friendly alternative can be provide to dispose the waste and reducing the disposal costing. On the contrary, the low cost material for activated carbon can be used as replacement for the expensive conventional activated carbon. Most of the activated carbon are produce by two stage process which is carbonization then followed by activation [25]. As reported by several author, the activation process of activated carbon give effect to the characterization and efficiency of activated carbon. Yusufu et al. [26] mention that activation method and the physical and chemical properties of material is important to characterize the activated carbon. Therefore, this review concern on the characterization of activated carbon including the factor effect during removal of dye.

ACTIVATED CARBON CHARACTERIZATION

The adsorption capacity of an activated carbon for a specific adsorbate is dependent on its physico-chemical properties. The physical and chemical properties of activated carbon are important to be determine the suitable form to be used in specific application. In addition, the mechanism and the procedure of activation process also giving effect to the porosity development [27]. The proximate analysis was performing according to the American Standard Test Method (ASTM) and the analyses of carbon samples in terms of moisture content, ash residues content and volatile content based on the complete combustion of the sample. The fixed carbon content was calculated by mass difference. The ultimate analysis of activated carbons was performed using an elemental analyser. The surface morphologies of activated carbon carried out by Scanning electron microscopy (SEM) while the surface functional groups study is done using Fourier transform infrared spectroscopy (FTIR).

Proximate and ultimate analysis

Table 1 shown the result obtained from proximate and ultimate analysis done by previous researches. Previous studies mention that suitable activated carbon should be low in ash but rich in carbon and volatiles [28,29]. Proximate analysis is useful to investigate the amount of carbon and ash content in the activated carbon. The ash consists of mainly minerals such as silica, alumina, iron, magnesium and calcium. Ash in activated carbon is undesirable and taken as impurity. Ash may interfare with carbon adsorption through competitive adsorption and catalysis of adverse reactions [30]. The most active products are usually produce from materials with lowest ash content as it reduces reactivation efficiency and overall activated carbon activity [31]. During carbonization and activation process, the sample fixed carbon content changes after exposed to high temperatures. The fixed carbon content increased steadily while volatiles decreased significantly with temperature [30, 32]. At higher temperatures, organic substances become unstable as the heat provides energy to the molecules to break their bonding and linkages and once these are broken, the substances will be discharged both as gas and liquid products [28].

The ultimate analysis gives the composition of the biomass as well as the prepared activated carbon in wt% of major component which is carbon, hydrogen and nitrogen and other like oxygen and sulphur. The analysis is very useful to investigate the purity of total carbon in activated carbon [33]. The results of ultimate analysis can be effected by the impregnation ratio and activation temperature during producing the activated carbon. Activated carbon are considered as carbonaceous material as it contain higher carbon content. High carbon content also indicates that aromatic structure becomes dominant when much amount of activating agent is used [29].

Table 1. Proximate analysis of agricultural activated carbon based on previous research

Activated Carbon	Proximate Analysis				Ultimate Analysis					Ref.
	Moisture (%)	Ash (%)	Volatile (%)	Fixed Carbon (%)	Carbon, C (%)	Hydrogen, H (%)	Nitrogen, N (%)	Oxygen, O (%)	Sulphur, S (%)	
Kenaf core fiber	5.60	6.90	77.30	11.00	44.02	7.82	0.46	47.42	-	[34]
Corncobs	4.36	3.20	21.14	71.30	58.12	<1	<1	41.25	-	[35]
Bagasse	-	2.91	-	-	22.36	-	30.69	30.52	-	[36]
Orange peel	-	3.40	14.90	81.70	82.5	2.50	0.90	14.10	-	[33]
Tomato	9.22	25.56	11.30	53.92	79.38	1.76	1.70	17.06	0.10	[37]
Grape waste	8.74	2.14	71.16	17.96	49.46	5.56	-	43.53	-	[38]
Coconut husk	9.50	27.84	27.53	35.13	-	-	-	-	-	[39]
Palm fiber	6.01	20.98	14.26	58.75	-	-	-	-	-	[39]
Palm shell	6.21	6.94	15.37	71.48	-	-	-	-	-	[39]
Pumpkin seed shell	7.60	3.90	70.97	17.53	48.79	7.52	3.97	39.72	-	[29]
Apple waste (pulp)	15.11	15.35	62.96	6.56	74.30	2.77	1.55	21.37	-	[40]
Apple waste (peel)	12.27	7.43	72.89	7.40	72.52	2.75	0.97	23.75	-	[40]

Surface characterization

The surface characterization of activated carbon is important to be carried out to investigate the limit amount of material and size of molecules can be adsorbed by the adsorbent for suitability in applications. Generally, larger surface area give better performance in adsorption [41]. In determining the surface area and pore structure, adsorption of N2 gas at –196 °C is used and the isothermal equilibrium data is fitted using either Langmuir or BET equations [27].Temperature has been reported to play an important role in producing optimum surface area of activated carbon. One of the foremost important properties of activated carbon is that they are highly microporous and have large surface areas [14,21]. These properties could be affected by varying the amount of activation reagents and other reaction parameters such as temperature and reaction time [27]. Several result reported in previous research are tabulated in Table 2. The micropores are responsible for the large surface area of activated carbon particles and are created during the activation process [31].

Table 2. Surface characterization by other researchers

Activated Carbon	Surface Area	Micropore Area	Micropore vol.	Particle size	Bulk density	Ref.
Untreat kenaf	13.68 cm^2/g	7.16 m^2/g	0.004 cm^3/g	-	-	[34]
Kenaf core fiber	299.02 cm^2/g	229.20 m^2/g	0.12 cm^3/g	-	-	[34]
Rice husk	949.32 cm^2/g	-	0.3316 cm^3/g	0.125-0.150 mm	756.27 g/l	[42]
Sugarcane bagasse	771.24 cm^2/g	-	0.2746 cm^3/g	0.125-0.150 mm	822.51 g/l	[42]
Bamboo waste	988.24 m^2/g	-	0.69 cm^3/g	200-300 µm	-	[43]
Rambut peel	908.35 m^2/g	-	0.52 cm^3/g	1.0-2.0 mm	-	[12]

Prosopos juliflora plant	616 m^2/g	-	-	-	0.603 g/cc	[44]
Cassava peel	1605 m^2/g	1100 m^2/g	0.513 cm^3/g	-	-	[45]

SEM analysis

Scanning electron microscope (SEM) analysis is useful to study the particle size, surface texture and the development of porosity in activated carbon [39]. SEM also can be used to observe the morphology surface of activated carbon before and after the batch analysis of adsorption study been done [38, 46]. SEM is suitable to track the transformations happen in the precursor material during activated carbon formation. Figure 1 shown the different morphology surface of non-woody agricultural waste activated carbon and precursor. Figure 2 below provide the different morphology surface of woody agricultural waste activated carbon and precursor. The SEM analysis prove that activated carbon provide larger surface area than a raw material. As shown in Figure 1 and 2, the precursor has no hole or cavity on their external surface, while the external surface of activated carbon has homogeneous hole and cavities. It seems that the cavities resulted from the evaporation of the activating agent, thus leaving the previously space occupied by the activating agent compounds [47, 48].

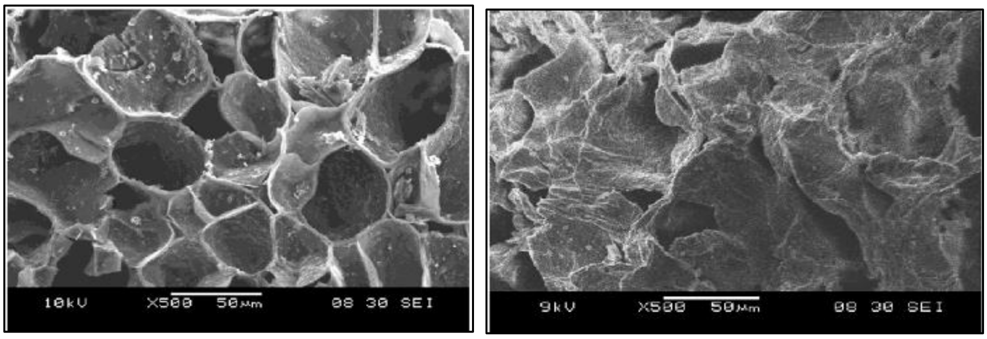

Figure 1. SEM picture of non-woody agricultural adsorbent a) mangosteen peel AC [32] b) mangosteen peel [32]

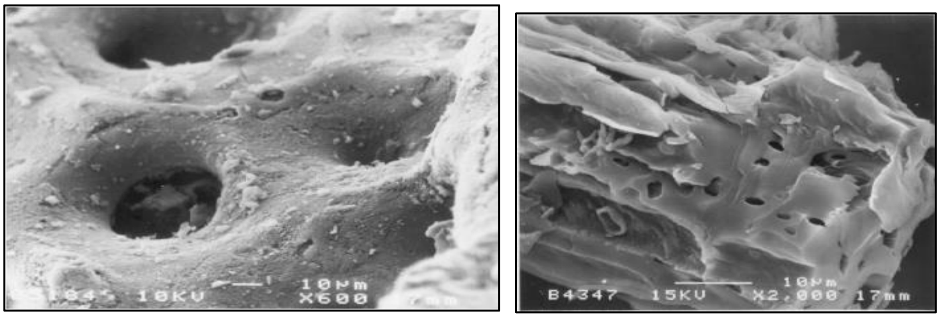

Figure 2. SEM picture of woody agricultural adsorbent a) gelam wood bark AC [27] b) gelam wood bark [27]

FTIR analysis

FTIR analysis used to determine the characterization of functional groups and surface chemistry in activated carbon, which are capable to adsorb dye ions. From Figure 3a, shown the disappearance of many absorption bands in the carbon spectrum after the carbonization and activation process. The decomposition of these groups probably due to high temperature chemical activation which release their by-product as volatile matter [24,38]. The peak of grape waste activated carbon at range of 1500-100 cm^{-1} correspond to carbonyl group, carboxylate ion, ash component and chelat bonded carboxylate structure [38]. For woody agricultural material, the similar pattern obtained and plotted in Figure 3b. The band located at about 1614 cm^{-1} which attribute to carbonyl group and 1201 cm^{-1} correspond to carboxylate ion groups. These functional groups formed possibly result from the extraction of H element and OH groups from aromatic rings during impregnation and activation process [24].

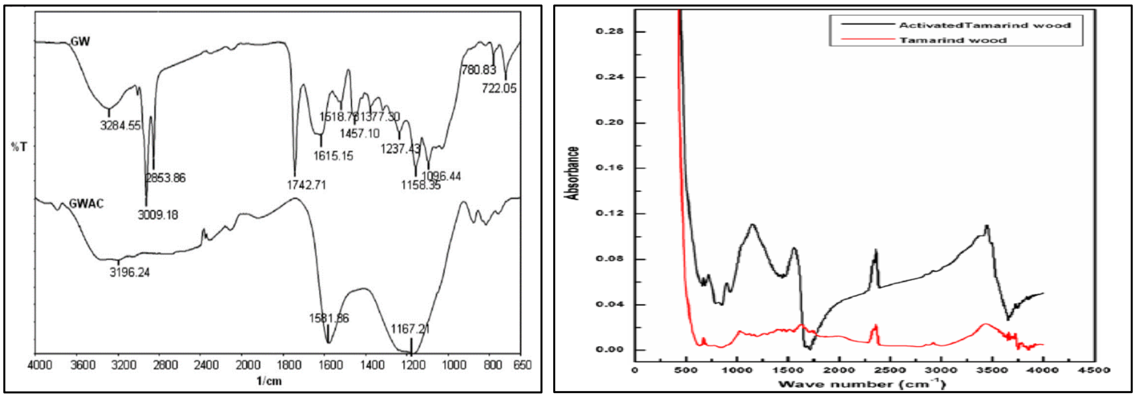

Figure 3. FT-IR pattern of agricultural adsorbent a) grape waste AC (non-woody) [38] b) Tamarind wood AC (woody) [24]

DYE ADSROPTION PARAMETER FACTOR

Table 3 shows several results of dye adsorption toward parameter factors conducted by previous researchers. In order to optimize the activated carbon produce in eliminating dye, various operational factors such as adsorbent dosage, contact time, pH and dye concentration is conduct. Thus, this parameter will be discussed in this review as these factors can be refer in process to remove dye using large scale treatment.

Table 3. Reported operational parameter factors by other researchers

Activated Carbon	Dye removal	Operational parameter factors					Ref.
		Time	pH	Initial dye concentration	Dosage	Temperature	
Orange peel	Direct dye (navy blue 106)	-	2	150mg/l	6gL^{-1}	27±2°C	[49]
Ficus carica bast	Cationic dye (methylene blue)	120 min	2	20mg/l	60mg/l	55°C	[50]
Coconut shell	Reactive dye (blue 19)	20 min	12	10mg/l	1g/l	28°C	[51]
Olive stone	Cationic dye (methylene blue)	30 min	5	150mg/l	10gL^{-1}	25°C	[52]
Walnut	Acid dye (red 18)	120 min	5	100mg/l	1g	25°C	[53]
Poplar	Acid dye (red 18)	120 min	5	100mg/l	1g	25°C	[53]
Sugarcane bagasse	Cationic dye (methylene blue)	60 min 120 min	7	10 mg^{-1} 100 mg^{-1}	10gL^{-1}	20°C 50°C	[54]
Coconut shell	Cationic dye (methylene blue)	120 min	4.5	-	0.25g/25ml	28°C	[55]
Untreated coconut shell	Cationic dye (methylene blue)	180 min	4.5	-	0.25g/25ml	28°C	[55]

Effect of dosage

The amount of adsorbate uses for the operating condition give affect in order to determine the maximum adsorbent's capacity, therefore the dosage adsorbent parameter must be study. Figure 4 shown the example graph obtain from the dosage factor parameter. The effect of adsorbent dose for adsorption process can be carried out by preparing different number of adsorbents yet added to fixed initial dye concentration which then shaken until the equilibrium in time reached [56]. From Figure 4a, it was observed that reactive dye removal increased with increasing dose of activated carbon. Santhy and Selvaphaty [57] research prove that for 50 mg/L concentration of reactive dye (orange 12 and red 2), a complete removal was achieved with 3.0g/L dose of coir pith activated carbon while for reactive blue 4, 4.0g/L dose of activated carbon needed. A complete removal with only small amount of dose of adsorbent maybe due to the large availability of site in coir pith activated carbon surface for dye saturation. The increase in removal of dye with adsorbent dose due to the introduction of more binding site for adsorption [58]. The increment in uptake was observed in removing reactive dye (crystal violet) and basic dye (direct orange and magenta) by using non-woody agricultural waste which is rice husk activated carbon. The maximum adsorption capacity of 77.5%, 69% and 89% was reach at dosage of 1.5g for crystal violet, direct orange and magenta respectively [58]. The same pattern result also shown in Figure 4b for removal of cationic dye (Methylene blue) by using fox nutshell activated carbon, which fall in woody agricultural waste categories. At the equilibrium time, the percentage of dye removal rise from 79.82% to 99.96% which is nearly complete removal of dye with optimum dosage of 0.04g [23]. In Ghaedi et al. [59] study on peanut stick activated carbon from agricultural woody base material has been conducted toward removal of dye, where the maximum percentage of removal was found at adsorbent dosage of 1.8g. The amount of sorption site at the adsorbent surface will increase by rising the dosage which result on the increase of dye removal percentage along with increasing if adsorbent dose [60]. Based on the extension review, activated carbon from agricultural waste both from woody and non-woody basis showed the ability of adsorption toward dye using minimum dosage of adsorbent.

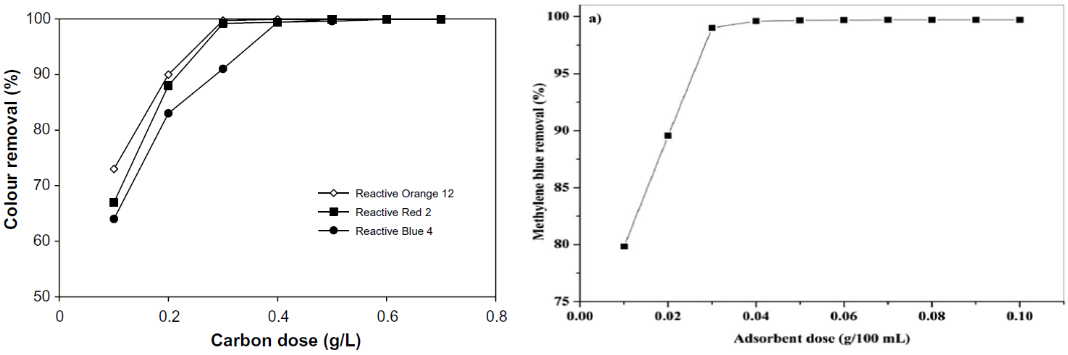

Figure 4. Effect of dosage on a) coir pith AC (non-woody) [57] b) fox nutshell AC (woody) [23]

Effect of contact time

El-Sayed et al. [61] mention in his research that the performance of dye removal are significantly affect by the contact time between adsorbent and adsorbate. Based on literature information, the pattern for contact time factor parameter are basically shown as in Figure 5. Generally, the increase in contact time to a certain extent will increase the rate of dye removal. At some point, the amount of dye adsorbs remain in a state of dynamic equilibrium which the time required is called equilibrium time [62]. The amount of dye removal at the equilibrium time shown the maximum adsorption capacity of the adsorbent [6]. As shown in Figure 5a, the removal efficiency of reactive dye in different activation process of bagasse activated carbons was rapidly increase in the beginning but it gradually slow in time to reached equilibrium. The time taken to obtain maximum adsorption capacity for bagasse activated carbons are about 1 hours of shaking [63]. The plots also reveal that bagasse activated carbon by chemical activation of H_3PO_4 (AC1) and $ZnCl_2$ (AC2) nearly complete removing dye in compare with bagasse from physical activation (AC3). In Amin N.K. [64] research, the same method to produce has been conducted using pomegranate peel activated carbon, where PC1 produce using physical activation while PC2 and PC3 produce using chemical activation. As results, direct dye was rapidly adsorbed in the first 25 min and reach the equilibrium time at about 2 hours as plotted in Figure 5b [64]. The similar behaviour was obtain for corncob activated carbon in removal of methylene blue which the rapid increase happens in the first 10 min because of the strong attraction force between dye and the activated carbons [61].

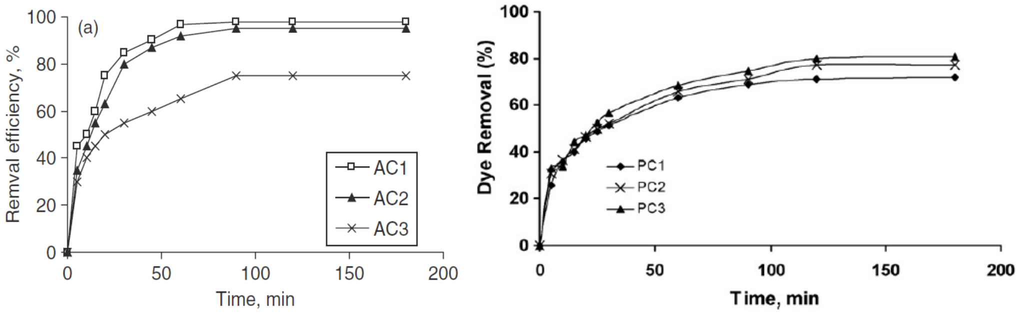

Figure 5. Effect of contact time on a) bagasse AC [63] b) pomegranate AC [64]

Effect of pH

The major important parameter in optimizing the dye adsorption process is the effect pH. The preference of high or low solution of pH solution are depend on the group of dye to be remove. The high pH solution provides the best efficiency in adsorption for cationic dye, while anionic dye has the higher removal of dye during low solution of pH [5,62]. Hazza and Hussein [52] conducted research on olive stone activated carbon toward removal of cationic dye and the maximum value adsorption of 9.5mg/g observe at pH 5. At lower pH, the surface charge may get positively charged, thus making H^+ ions compete effectively with dye cations causing a decrease in amount of dye adsorbed [65]. During higher solution of pH, the adsorbent surface may get negatively charged that enhances attraction through electrostatic force of the positive charge dye cations [66]. Malik et al. [67] research obtain different results in removing acid dye using activated carbon from sawdust and rice husk as the amount of adsorption decreases when the pH solution increased. Low pH (3 and below) was found to be favourable for maximum adsorption of acid yellow 36, which acid dye fall into anionic dye group. Malik et al. [67] also mention that lower adsorption of dye at alkaline pH due to the presence of excess OH- ions competing with the dye anions for adsorption sites. Anionic dye (Methyl orange) and cationic dye (Methylene blue) removal was investigated using adsorbent from apricot activated carbon [68]. Figure 6, give evidence where adsorption efficiency of activated carbon affected by the variation of pH. The rise in pH value from range of 3 to 10 decreasing the efficiency of dye removal from 99.6% to 59.74% for anionic dye (Figure 6a), while significantly increase the adsorption efficiency of cationic dye (Figure

6b) from 93.71% to 99% . Reduction of positively charge sites and increment of negatively charged sites create electrostatic repulsion between adsorbent and anionic molecules [68].

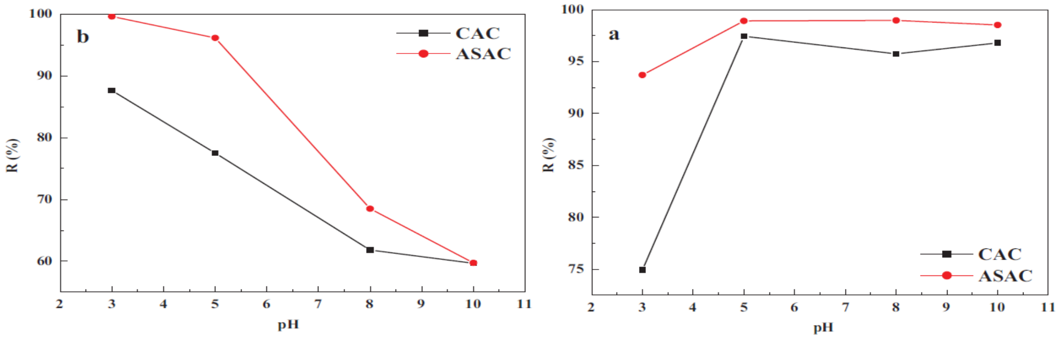

Figure 6. Effect of pH solution on a) anionic dye (MO) [68] b) cationic dye (MB) [68]

Effect of concentration

Adeyemo et al. [60] mention that the increment of the adsorbent loading capacity is affected by increasing of initial dye concentration. The high result of adsorption result from the increase in initial concentration which will larger the mass transfer driving force [69]. In general, the effect of dye initial concentration can be conducted using different dye initial concentration with fixed amount of dosage shaken together until equilibrium obtained [56]. In Peanut stick activated carbon research [59], dye removal increase at lower dye concentration due to the ratio of solute concentrations to adsorbent sites is low. Due to the increasing of repulsive force between bulk molecules and adsorbed dye, decrement of percentage removal happens [59]. The actual amount of dye adsorption increased by raising the initial concentration of dye. Figure 7 shown pattern observe for dye concentration effect in terms of removal percentage and amount of dye adsorbed using activated carbon from agricultural waste. Figure 7a provide graph on reactive dye removal percentage using activated carbon of coir pith from non-woody agricultural waste [57]. The reactive orange removal percentage increased with decreasing of dye concentration which the equilibrium attained at 4 hour for 90% to 60% removal on 20 mg/L to 100 mg/L concentration. The similar results obtained for removal of reactive red and reactive blue experiment [57]. For Hazza and Hussein [52] research, proved that the amount of dye adsorbed increased from 4.8 to 12.4 mg/g for increment in initial dye concentration from 50 to 150 mg/L as shown in Figure 7b.

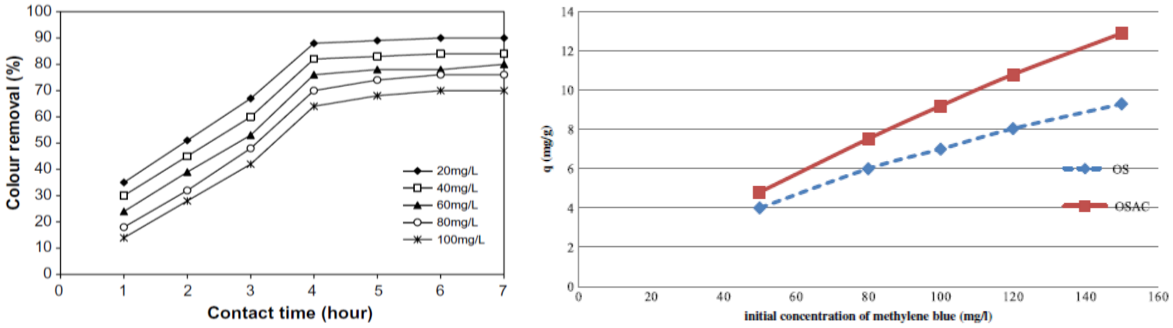

Figure 7. Effect of initial concentration by a) percentage removal [57] b) amount of dye absorbed [52]

DISCUSSION

Based on the literature information, activated carbon give evident as the most preferred adsorbent material to treat dye in wastewater treatment. Furthermore, agricultural waste like sugarcane bagasse, orange peel and grape waste prove to be promising basis material in producing activated carbon [5,41]. However, most of the study only focus one resources of adsorbent to remove dye from wastewater. It is difficult to compare the adsorbent as it may depend on the material performance as each agricultural waste material have its own feature for wastewater treatment and variable characteristic that can influence the properties of adsorbent. Based on existing literature, the proximate content of each activated carbon are dependent on the original structure of the material and the preparation condition. The high specific surface area of the agricultural waste activated carbon grant them a high adsorption capacity for textile dye in wastewater and other aqueous solution [70]. The decreasing of volatile matter and increment fixed carbon after undergoes activation and carbonization process was due to the pyrolytic effect [12, 71]. As shown in SEM analysis, the morphology of raw agricultural waste resource was uneven, rough and availability of little pores on the surface to be compared with agricultural waste after activation process [12]. It is difficult to optimize all of the agricultural waste in the same condition as the interest region of factors is different [32]. Not just that, the effectiveness of dye treatment major affect by the condition in adsorption process in term of contact time, adsorbent dosage, dye concentration, pH value and temperature. Extension studies in literature review show lot of research had been done on activated carbon, however those research focusing on removal contaminant other than dye were not mentioned in this review. Further research is necessary, especially concentrate in removing textile dye containing benzidine-based dye and o-tuildine as these dye is toxic and causing major damage to ecosystem and human body. Besides that, more investigation should be conducted using real industrial wastewater to predict the adsorption performance toward dye removal rather than using synthetic solution.

CONCLUSION

Various activated carbon from agricultural waste resource as adsorbent has been presented. The result from the reviews show that agricultural activated carbon is promising, efficient and economic that the conventional activated carbon [3, 38, 23, 32] as demonstrated by the higher BET surface area, pore volume and dye adsorption after significantly shorter adsorption contact time, optimize adsorbent dosage, pH value and also the concentration [40, 54, 63]. In addition, agricultural waste is easily available and renewable. Activated carbon agricultural waste turn to have greater adsorption capacity in compare with raw agricultural waste. The activation process help improves the morphology of the adsorbent and increase the availability of sorption site. It was also highlighted that the pH solution, contact time, dose of adsorbent and dye concentration has a major effect toward the efficiency of adsorption during eliminating dye contain in textile wastewater. The pH value is different based on the class of dye used while shorten time needed when strong attraction force applied. In term of dosage and concentration, the increments of each parameter will increase the adsorption capacity to adsorb dyes. According to the literature reviewed, agricultural waste activated carbon are often being test for other aqueous solution and pollutant yet the basis materials depends on the availability of local sources at a low cost. As to conclude, activated carbon from non-woody agricultural waste have a promising potential to be applied in large scale treatment, thus can be used as replacement of expensive conventional activated carbon.

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