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Preparation, Characterization and Utilization of Coconut Adsorbents as a Filter Media for Wastewater Treatment

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Authors' contributions

This work was carried out in collaboration among all authors. Author SK designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors RMJ and MM managed the analyses of the study. Author SS managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The present study investigates the characterization of different coconut based low cost adsorbents like coconut shell biochar, zinc chloride impregnated coconut shell activated carbon, coir fibre and coir geotextile and their suitability characteristics as a filter bed in different wastewater treatment process. The characterization study helps to investigate their physical, chemical and morphological properties like proximate and ultimate analysis, iodine number, decolorizing power, SEM, Surface area using BET, Particle size and Zeta potential. The experiment results showed that among the different adsorbents activated carbon has high fixed carbon content (82.99 percent), more surface area (590.8 m^2 g^{-1}), low ash content (1.31 percent) with a decolorizing power of 240-300 mg g^{-1}. The coir fibre and coir geotextile having neutral pH with negative surface charge easily adsorbs the positive cations from aqueous solutions at highest apparent density. The experimental findings suggest that the activated adsorbent which shows better results as an effective filter media for adsorption of organic compounds and pollutants from wastewater.

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Keywords: Cocos nucifera; biochar; activated carbon; coir geotextile; characterization.

1. INTRODUCTION

Agricultural products that deliver various materials such as coconut shell, fibre, husk, palm kernel, walnut shell and groundnut shell are excellent sources for carbon production. These source materials are very cheap, easily available and renewable in nature [1]. The use of this products for carbon production does not affect the human food consumption [2]. In the year 2018-19, India has 2178.74 hectares under coconut cultivation with 21,384.33 million tonnes nuts production and 9815 nuts per hectare productivity [3]. The major coconut growing areas in India are Kerala, Tamil Nadu, Karnataka, Andhra Pradesh, Andaman and Nicobar, Lakshadweep and Pondicherry. Among the states, Tamil Nadu has cultivation area of 437.34 hectares with 5311.05 Million nuts production and 12,144 tonnes nuts per hectare which suppresses the India's average nut productivity [3]. In Tamil Nadu, Coimbatore (Pollachi), Kanyakumari, Thanjavur, Erode and Nagercovil were the leading coconut producing districts due to their favourable climatic conditions for nut production. From the total weight of coconut fruit, 15% of the fruit occupied by coconut shell and 20-30% of husk occupied by fibre which was globally discarded every year. Among different carbon sources, coconut based materials like shell, fibre etc. was rich in cellulose, hemicellulose and lignin which were not easily degraded [4].

Coconut shell which is the hardest part of coconut present on the outer side of coconut husk by protecting the inner flesh. The coconut shell material was converted to char by thermochemical conversion process in absence of oxygen at 600-800°C leaves liquids and gases as other products. The final product obtained by this process as fixed carbon char (Biochar). During this process, pores were formed at low rate which filled with tar like pyrolytic residues and need activation process to improve pore diameter and inner surface area of char [5]. In the activation process the carbon products were treated to release the hydrocarbons, tar and other organic compounds attached to carbonized char. By this process, the pores were formed which are still closed and size and number of pores were increased with enhanced surface area and adsorption properties which was called as activated carbon [6].

Activated carbon which is a solid, porous, black carbonaceous materials which exhibits greater adsorption capacity due to their extended surface area, high porosity and variable surface chemistry. In developing countries due to high cost and non-renewability of commercial activated carbon, they limit the usage as an adsorbent material. To reduce this, researchers studied the activated carbon production from different agricultural residues such as fruit stones and seeds, shells of walnut, almond, peanut and coconut, coir pith and husk, sugarcane baggase, cotton stalks etc. Among the precursors, coconut shell used as an effective raw material for production of activated carbon due to its high carbon content and low ash content which was widely used as an adsorbent for many applications such as liquid and gas purification, waste water treatment, industrial pollution control, removal of odour, solvent recovery, electrode for capacitors etc [7,8,9,10].

Activated carbon prepared from different carbonaceous materials by either physical or chemical activation methods. Chemical activation involves both carbonization and activation process by which the raw material was impregnated with different activating agents like HCl, H_2SO_4, KOH, $ZnCl_2$, NaOH and pyrolyzed between temperature at 400-800 °C in the absence of oxygen [2]. Chemical activation having several advantages over physical activation performed at lower temperature with minimum treatment time which develops good porous structured activated material [11].

Coir fibre is a 100 percent organic and versatile natural fibre extracted from husk part of coconut fruit, generally golden in colour after cleaned from coconut husk called as golden fibre. It is coarse, reddish brown fibre made up of small threads at a length of 0.03 to 0.1 cm and diameter of 12 to 24 micrometer which is high in lignin content and less cellulose resistant to biodegradation [12]. Coir fibre which is rich in organic matter with high specific area and wetting ability suitable for microorganism's adhesion and biofilm formation. Coir geotextile is made from coconut fibre drawn from the coconut fruit husk. It is available in various mesh matting with international trade name, depending on the size of the mesh and the place of origin. Coir geotextile is a durable and strong organic fibre which is rich in lignin and cellulose having more specific area and wet ability factors important for bacterial adhesion in fixed film process [13]. Geotextiles are planar sheets like structure that are fairly dense and can be in form of woven, unwoven, or knitted. They are able to transmit fluids across or in plane but it may hold the suspended particles and organic compounds [14]. Coir geotextile are divided mainly into two categories as woven and non-woven geotextile in different weight of 400, 700 and 900 gsm in woven quality and 300, 450 and 550 gsm of non-woven quality. The width of coir geotextile will be 1 metre to 4 metre and length will be 20 to 70 metre. Woven type coir geotextile are generally produced at right angles by interlacing fibres whereas nonwoven geotextile were created through mechanical, heat and chemical bonding of direct or randomly oriented fibres [15]. It can easily dissipate the energy of flowing water and absorbs more solar radiation. Mainly coir geotextiles are highly used in construction activities like road pavement, soil stabilization and to prevent soil erosion in hilly areas effectively serve as reinforcement, separation, filtration and drainage functions.

This study paper investigates the comparative characteristics of coconut based adsorbent materials, their physical, chemical and morphological properties and suitability characteristics as an effective filter media in waste water filtration process.

2. MATERIALS AND METHODS

2.1 Adsorbent Collection and Preparation

Coconut shell (*Cocos nucifera*) and Coir fibre was collected coconut processing industries located at Pollachi, Tamil Nadu, India and coir geotextile sample at 700 gsm was collected from regional coir board office, Pollachi. The adsorbent sample was washed with distilled water to remove impurities and sun dried for 12-24 hours for complete removal of moisture content. The quantity of sample was cut into small pieces and powdered by using mixer and sieved through 0.2 mm sieve and stored in air tight container for further analysis purpose.

2.2 Chemical Activation

Chemical activation of the pyrolysed sample was done with zinc chloride of 99.9% purity to make an impregnation ratio of 10% (w/v). The slurry was properly mixed and kept 24 hours for proper soaking of zinc chloride on its surface. After that, the excess zinc chloride solution was drained out and the slurry was kept in hot air oven at 110°C for 6 hours. Then the impregnated material was placed in muffle furnace at 550°C for one hour. The impregnated sample was treated with 0.1 M HCl for 24 hours to remove cations and repeatedly washed with distilled water to remove excess acid and dried in hot air oven at 110°C for six hours [16]. In this process, zinc chloride chemical was used for activation purpose because it acts as a dehydrating agent which lowers the carbonization temperature during chemical activation process and restricts the tar formation and promotes the charring of carbon [17].

2.3 Characterization of Adsorbent

pH and Electrical conductivity was determined using ELICO made pH meter and EC meter, respectively [18]. Proximate analysis like moisture content (% oven dry basis), ash content, volatile matter and fixed carbon were analyzed using standard procedure and ultimate analysis like Carbon, Hydrogen, Nitrogen and Sulphur were measured using CHNS analyzer [19]. The physical properties like bulk density, particle density and porosity was determined by cylinder method [20]. Total organic carbon (%) was measured by muffle furnace method [21]. Iodine number was determined to know the performance of adsorbent material [22]. Decolorizing power of adsorbents was analyzed to gain knowledge about amount of material for treating coloured solutions [23]. Morphological features of the sample were determined using Scanning Electron Microscope (Quanta 250, FEI, Netherlands). Then the particle size and zeta potential was measured using particle size analyzer (Horiba Scientific Nanopartica SZ-100, Japan). The surface area was determined using SMART SORB 92/93 surface area analyzer [24].

3. RESULTS AND DISCUSSION

3.1 Physico-chemical Characterization of Adsorbent

3.1.1 pH and EC

The pH and Electrical conductivity of the coconut adsorbent sample was represented in the Table 1. The activated carbon had lower pH compared to biochar, due to activation process, functional groups present in activated carbon which decreases the pH value [25]. The Electrical Conductivity of biochar was 1.11 dS m^{-1} and activated carbon value was less than 1 (0.52 dS

m^{-1}) which reduce the salt content during activation and washing process due to which EC was reduced. The adsorbents like coir fibre and coir geotextile having neutral pH value and salt concentration were little high due to improper leaching process compared to other adsorbents.

3.1.2 Density and porosity

The density is an important parameter when an adsorbent material is removed by filtration process. An adsorbent material with adequate density helps to improve the filtration rate even after cake formation on their filter surface. The bulk density and particle density of adsorbents was presented in the Table 1. The difference between the coconut adsorbents attributes to variation in chemical composition of their particle and sizes. The density of coconut adsorbents was higher than *Ceiba petandra* shell activated carbon (0.48 g cm^{-3}) [26] and *Conocarpus sp.* pruning waste activated carbon (0.49 g cm^{-3}) [27]. The porosity of the carbon char was 16.67 percent and after activation process it increased as 35 percent because the activating agent which improve the porosity by dehydration and degradation of carbon material. The activated carbon showing more pore space indicates that adsorptive properties was higher due to high porous nature compared to all other adsorbents which heavily adsorb all the organic compounds and pollutants present in waste water. The porous nature of coir fibre and geotextile was less when compared to other adsorbent materials due to their extreme fibre length and fibre density.

3.1.3 Zero point charge

The zero point charge (pHzpc) of an adsorbents helps to indicate the net surface charge of ion present in the solution. The pHzpc is the point where the curve of pH_{final} vs $pH_{initial}$ intersects the line $pH_{initial} = pH_{final}$. The zero point charge was determined by pH drift method and the results were represented in the Fig. 1. pH value change in adsorption system leads to the chemical characteristic transformation on sample surface and form of adsorbate plays a major role in adsorption process. When pH of the solution is lower than pHzpc shows net positive charge, while at higher than pHzpc shows net negative charge [28]. From the results it was observed that the material like activated carbon having free surface charge nearly neutral pH suitable for waste water treatment even under neutral conditions. But the coir fibre and geotextile having net negative charge on the surface which effectively adsorbs the positively charged ions present in the waste water solution.

3.1.4 Proximate and Ultimate analysis

The proximate and ultimate analytical results of coconut adsorbents under optimum conditions were presented in the Table 2. From this analysis, the activated carbon's fixed carbon content was found to be very high compared to pyrolysed char material resulting as a better adsorbent for adsorption studies. The results shown that high volatile matter, low ash and sulphur content of the precursor makes it an excellent material for activated carbon preparation [17]. Due to the activation process, the fixed carbon content was increased from 52 to 82.99 percent and total organic carbon content increases from 68.3 to 80.6 percent respectively. The moisture content of all the carbon adsorbents was less which shows no effect on its adsorptive potential, it dilutes the carbon which necessitates the use of additional weight of carbon during activation process. During carbonization and activation process, the elements like hydrogen, nitrogen and sulphur present in activated carbon has been reduced because the volatile compounds (H,N,S) were removed from the starting material due to enriched carbon present in coconut shell. The coconut shell biochar was chemically activated with zinc chloride helps to remove hydrogen and oxygen as H_2O and H_2 instead of CO, CO_2 and hydrocarbons were removed [29]. The experiment observation investigates that the carbon content arose from 70.14 to 75.74 percent and reduction of hydrogen, nitrogen from 11.16 to 2.01 percent and 3.53 to 0.31 percent, respectively. Due to the highest carbon content it significantly increases the active sites of activated carbon material. Compared to other adsorbents, coir based fibre and geotextile having low ash content, high volatile matter and medium carbon content which also used as a filtration layer media for growing of microorganisms for degradation of organic compounds present in waste water.

3.2 Morphological Characterization of Adsorbent

3.2.1 Scanning Electron Micrograph

The scanning electron micrographs helps to observe the surface morphology of coconut based adsorbents and were represented in the

Fig. 2 at 20 µm at 10 kV with 5000 x magnification. In this study, the surface morphology of adsorbents varies significantly due to little pore development but the activated carbon image showed that number of pores was increased which looks as net like structure. During the process of activation, zinc chloride reacts with char materials which oxidizes and erode the hydrocarbons and tar compounds attached on the char surface, then the blocked pores were exposed and numerous new pores formed and pore diameter and volume of pores were increased on the activated carbon surface [30]. The SEM image of coir adsorbents surface appears to be smooth due to their waxy coating on their surface and the pore size was very less. The results showed that pore size of coconut shell biochar ranges from 659.4 to 840.6 nm and the coconut shell activated carbon ranges from 641.1 to 990.8 nm and coir fibre and geotextile ranges from 605.3 to 715 nm and 625.3 to 745.5 nm respectively. The SEM images depict that the activated carbon had more number of pores and increased pore size compared to other adsorbents due to their higher activation temperature and alkali chemical agent. The pores of the prepared activated carbon were high compared to physic nut plant activated carbon (<2 µm), which would facilitate higher adsorption [31].

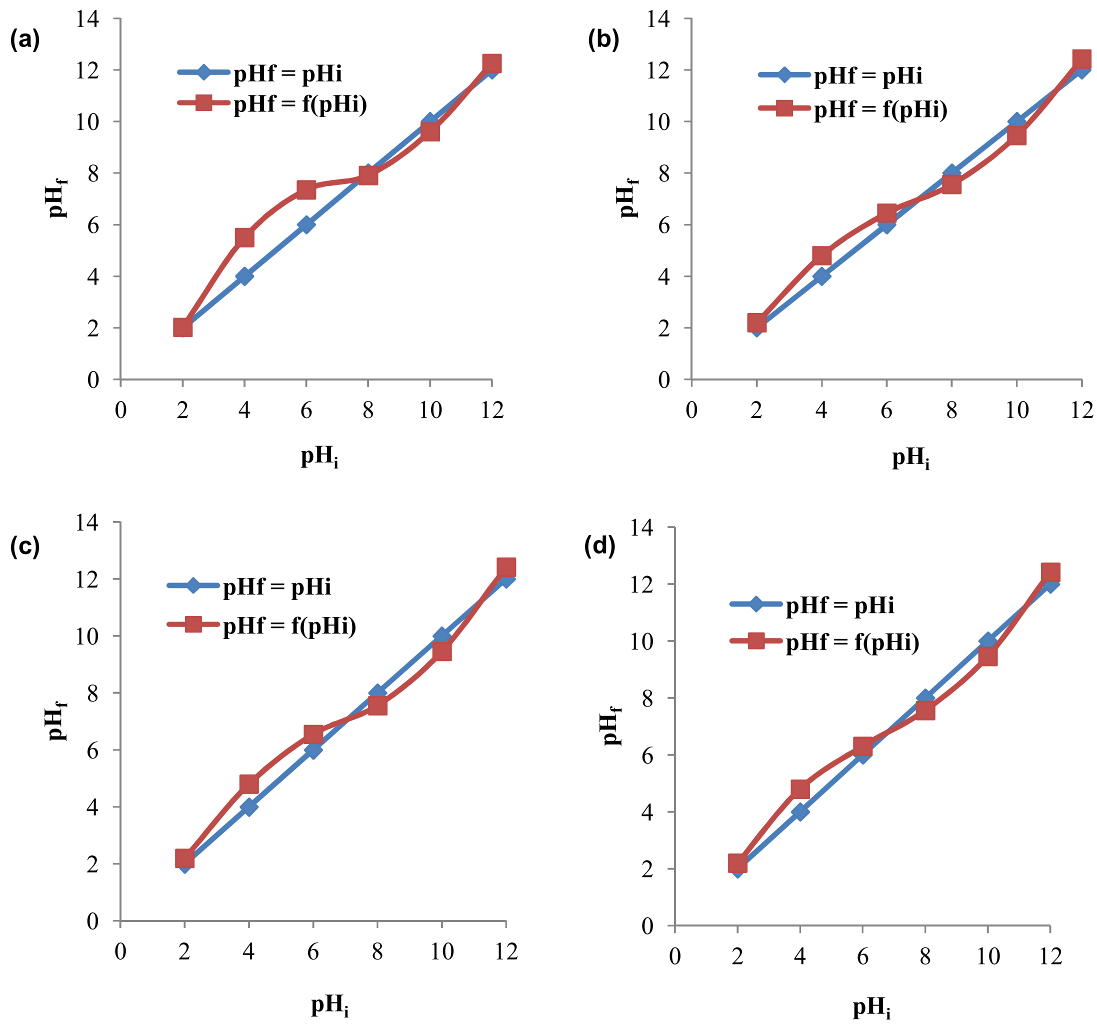


Fig. 1. Zero point charge of coconut adsorbents (a) CSB (b) CSAC (c) Coir fibre (d) Coir geotextile

Table 1. Physical properties of coconut adsorbents

Properties	CSB	CSAC	Coir fibre	Coir geotextile
pH	8.53	7.0	7.2	7.5
Electrical Conductivity (dS m^{-1})	1.11	0.52	1.81	1.79
Bulk density (g cm^{-3})	0.5	0.78	1.40	1.46
Particle density (g cm^{-3})	0.6	1.2	1.62	1.65
Porosity (%)	16.67	35	13.6	11.6
pHzpc	7.9	6.8	6.5	6.4
Total Organic carbon (%)	68.3	80.6	75.6	76.9

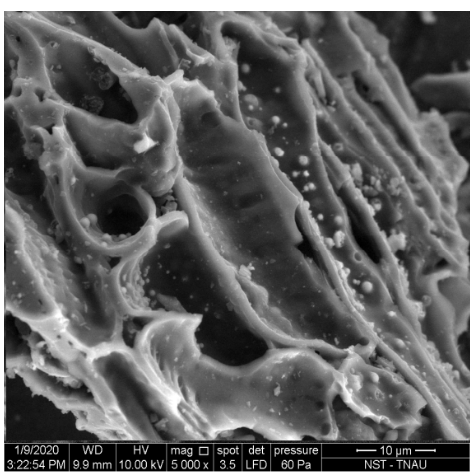
Table 2. Proximate and Ultimate analysis of coconut adsorbents

Sample	Proximate analysis (%)				Ultimate analysis (%)			
	Moisture	Ash	Volatile	Fixed carbon	C	H	N	S
CSB	3.6	4.5	39.90	52	70.14	11.16	3.53	0.01
CSAC	1.5	1.31	14.20	82.99	75.74	2.01	0.31	0.05
Coir fibre	5.1	2.9	70.3	21.7	46.75	5.41	0.91	0.13
Coir geotextile	5.2	2.5	71.8	20.6	47.74	6.81	1.01	0.15

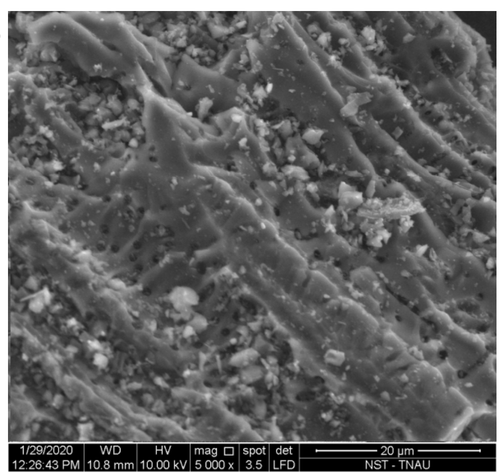
3.2.2 Surface area analysis

The surface area and pore volume of coconut adsorbent samples analytical data were represented in the Table 3. The surface area of coconut shell biochar, coconut shell activated carbon, coir fibre and geotextile found to be 45.3 m^2 g^{-1}, 590.8 m^2 g^{-1}, 28.5 m^2 g^{-1} and 32.8 m^2 g^{-1} respectively. From this analysis, the activated carbon impregnated with zinc chloride showing higher surface area and pore volume compared to that of inactivated char. Because the activation process, removes the impurities present on the char surface which helps to increase the surface area and pore volume in the activated carbon surface. Also the coir fibre and coir geotextile which has moderate surface area and pore volume compared to other adsorbents because of its lower porosity. Greater the adsorbent's specific surface area, better its adsorption performance and filtration ability of wastewater [32]. Similarly, the oilpalm empty fruit bunch were used to produce activated carbon activated using steam, which has the surface area upto 700 m^2 g^{-1}, when the carbonization temperature was 765°C [33].

(a)



(b)



(c) (d)

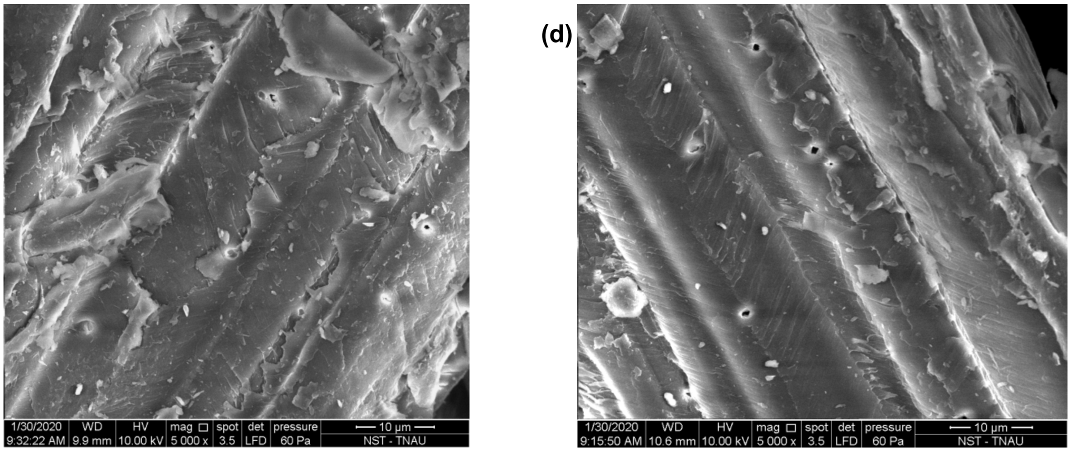


Fig. 2. SEM images of coconut adsorbents (a) CSB (b) CSAC (c) Coir fibre (d) Coir geotextile

Table 3. Morphological analysis of coconut adsorbents

Properties	CSB	CSAC	Coir fibre	Coir geotextile
BET surface area ($m^2 g^{-1}$)	45.3	590.8	28.5	32.8
Pore volume ($g\ cc^{-1}$)	0.0145	0.0187	0.0059	0.0081
Pore diameter (Å)	12.57	18.89	7.09	7.25
Pore size (nm)	659.4-840.6	641.1 – 990.8	605.3 to 715	625.3 to 745.5
Particle size (nm)	680.5	245.6	801.5	811.9
Zeta potential (mV)	-29.5	-36.5	-12.1	-13.8
Iodine number	451	985	215	254
Decolorizing power ($mg\ g^{-1}$)	120	240-300	75-80	90

3.2.3 Particle size analysis and zeta potential

Zeta potential determines the characteristics of coconut adsorbents to estimate the surface charge that can be used to understand the physical stability of suspensions. The zeta potential and particle size analysis of coconut adsorbents was presented in the Table 3. The zeta potential measurement of all the adsorbents was negatively charged. The activated carbon had high dense negative surface charge (-36.5 mV) compared to char material (- 29.5 mV). Hence the activated carbon effectively adsorbs more positive charged ions on their surface from various dyes and pollutants present in waste water. The particle size was recorded lowest in activated carbon (245.6 nm) compared to biochar (680.5 nm). The decreasing particle size increases the surface area of activated product that increases the adsorption capacity [34]. The particle size of coir fibre and coir geotextile was found to be high and zeta potential was low negative surface charge compared to other adsorbents shows little adsorption capacity.

3.2.4 Iodine Number

Iodine number helps to estimate the surface area of different adsorbent material and acts as an indicator to measure the micro porosity and adsorptive capacity of coconut adsorbents. The higher iodine number of adsorbent carbon attributes the presence of large micropore structure and greater possibility of adsorbent having more surface area due to their enlarged pore structure. The results on iodine number of adsorbent sample were represented in the Table 3. The iodine number of activated carbon was high compared to other reported adsorbent materials like KOH impregnated activated carbon produced from bean husk (894.65 $mg\ g^{-1}$) and oilpalm empty fruit bunch activated carbon (356.46 $mg\ g^{-1}$) [33,35]. This exists because of the presence of large micropore structure due to chemisorption (using zinc chloride) that takes place in pores of carbon material during carbonization and activation process [35].

3.2.5 Decolorizing power

Decolorizing power of the adsorbent material is expressed in terms of milligrams for methylene blue adsorbed by 1 gram of adsorbent material. The result of decolorizing power of coconut adsorbents was represented in the Table 3. Decolorizing power is calculated by using methylene blue test. The decolorizing power was high in zinc chloride impregnated activated carbon which is in range of 240 – 300 mg g^{-1} and low in coir fibre. The Methylene blue number indicates the ability of a carbon material to adsorb high molecular weight substances like dye molecules [36]. The decolorizing power of coconut adsorbents was high compared to *Melaleuca cajuputi* wood bark based activated carbon (51 mg g^{-1}), which shows high colour removal efficiency in wastewater [37].

4. CONCLUSION

From the study, physical, chemical and morphological characteristics of coconut based adsorbents were obtained. The pore volume and BET surface area found to be very high in coconut shell activated carbon (CSAC) which is microporous in nature. Iodine number and decolorizing power of activated carbon shows significant results which effectively adsorb the pollutants and decolorize dye based organic compounds present in wastewater. But, the coir fibre and geotextile have least pore development and surface area has little adsorption capacity. In future, further studies to improve the porosity and surface area of the coir fibre and geotextile by activation process which highly adsorbs the pollutants present in wastewater should be made. The characterization results showed that the coconut based activated carbon were effectively used as a promising filter media for waste water treatment. The conversion of coconut waste into valuable adsorbent materials add additional value to the produce and generates employment opportunities, minimize the waste in coconut processing industry and reduce the environmental impacts. So the coconut waste products suit to cheap and best natural alternative to commercial carbon sources for wastewater treatment process and pollution control studies.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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