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THE ADSORPTION CAPACITY OF METHYLENE BLUE BY ACTIVATED CARBON IMMOBILIZED YARN

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ABSTRACT

This study investigates the adsorption capacity of activated charcoal immobilized yarn against the methylene blue. Activated charcoal from refillable water filtration was used as a comparison. Activated charcoal was prepared by open and closed pyrolysis using a muffle furnace. The characterization of activated charcoal includes water content, adsorption of methylene blue, observations using a stereo microscope and Scanning Electrone Microscope. The results revieled that a 100 mesh activated charcoal powder with 75% Polyvinyl Acetate showed better properties in terms of thickness of the yarn layer, stability, and the surface morphology. The adsorption capacity test for the effect of the optimum time shows that activated charcoal from refilled water was optimum at 12 minutes, while activated charcoal regeneration, closed pyrolysis, and open pyrolysis took 9 minutes. The concentration of 1, 2, 3, and 4 ppm showed the absorbance results directly proportional to the increase in concentration. The greater the concentration, the higher the percentage of adsorption capacity. The comparative test for the type of activated charcoal with the best capacity is activated charcoal refill, regeneration, open pyrolysis, and closed pyrolysis, respectively. Based on the study results, it can be concluded that the immobilized activated charcoal yarn with 75% adhesive and 100 mesh activated charcoal powder can be effectively used to adsorb methylene blue, and open pyrolysis activated charcoal immobilized yarn can be regenerated.

Keywords: Carbon, Fiber, Adsorbent, Waste, Polyvinyl, Methylene blue.

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INTRODUCTION

Indonesia is a country with the largest textile industry in Southeast Asia. Besides having a positive impact, the development of the textile industry also causes an increase in pollutants from the textile industry waste. Textile industrial waste is usually produced from rinsing or washing in the dyeing process for cotton, cloth, tannins, and leather. Thus, waste is produced from liquids such as synthetic dyes. Methylene blue (MB) is a common dye utilized due to its cost-effectiveness.[1,2] The threshold value for MB in water is (5-10) mg L^{-1}. Suppose it passes this threshold and is immediately discharged into the water without the initial handling. In that case, it can cause severe environmental problems because the environment does not readily degrade MB. Although the mud in the water can absorb MB, it will be safer for the environment if the waste is treated before being discharged into the water using the adsorption method. Activated carbon (AC) is an abundant alternative adsorbent commonly used as a liquid molecular adsorbent and is very suitable for removing odors, colors, heavy metals, and other pollutants from industrial wastes.[3] The use of AC as an adsorbent continues to increase. However, there are remaining problems often encountered, such as quality, properties, and limited variations because AC is primarily available in powder and granular form.[4,5] Moreover, using AC to adsorb the MB is commonly carried out due to simplicity but is less effective due to contamination.[5] Therefore, innovation is necessary to change the form of AC, which is easy to use but affordable to the broader community. Research on AC continues to be developed by researchers starting from the processing stage, activation process, characterization, and variety of forms and uses. The absorption capacity of AC is strongly influenced by its surface and porosity.[6] Studies on activating carbon fabric (CF) as an adsorbent of chemical protective clothing have been reported.[7] However, the manufacture of this product requires sophisticated equipment and is relatively expensive because it requires polyacrylonitrile (PAN) polymers to produce the CF. It has never been reported that the powder and granular form of AC was modified into activated charcoal immobilized yarn (ACIY). In this research, powdered and granular AC was modified and immobilized on the yarn to promote applicability and practicality at an affordable price.

EXPERIMENTAL

Material and Equipment

The equipment used in this research includes a digital balance (OHAUS), a series of yarn-making tools, rulers, scissors, rotary evaporator (Buchi, Switzerland), stirring rod, spatula, beaker, measuring cup, pestle, mortar, magnetic stirrer, top stirrer, volume pipette, measuring flask, UV -Vis (Shimadzu 1800, Japan), oven (Memmert, Germany), desiccator, kiln, pyrolysis apparatus and sieve. The chemicals used in this research were purchased from Merck, Germany, including ethanol (C_2H_5OH), hydrochloric acid (HCl), sodium hydroxide (NaOH) 98%, methylene blue ($C_{16}H_{18}$ ClN_3S), distilled water (H_2O), and universal indicators, Polyvinyl Acetate (PVAc), palm oil shells, and hemp yarn.

Pyrolysis

10 kg of palm kernel was washed thoroughly with water to remove dirt and dried. 8 kg of palm kernel were put into a pyrolysis tool. After the charcoal was obtained, the% yield was calculated, and the charcoal was then activated using 25% NaOH for 24 hours. The activated charcoal obtained was filtered and washed with distilled water until a neutral pH was obtained, the pH was adjusted with 3 M HCl, dried, and mashed to the size of granular activated charcoal.[8]

AC Immobilized Yarn Preparation

The role of PVAc as an adhesive dramatically affects the results of immobilization of activated carbon to the yarn therefore, to obtain the optimum percent of the adhesive, various compositions of PVAc and ethanol were prepared in the amount of 65, 70, 75, 80, 85, and 90%.

Preparation of AC Powder

100 grams of Activated Carbon (AC), finely ground and sieved with a 60-mesh sieve. ± 80 grams of activated charcoal that pass through the 60-mesh sieve is then put into a container of activated charcoal powder in a series of tools. Then, the same treatment was repeated for 80 and 100 mesh smoothness. Hemp yarn marked with a knot every 30 cm is passed through a series of containers containing PVAc 65, 70, 75, 80, 85, and 90% and passed through 100 mesh of activated charcoal powder. The optimal dosage of PVAc was used to immobilize AC in yarns with variations of 60, 80, and 100 mesh. There were four different carbon prepared and studied in this work, including Close Pyrolysis Activated Carbon (CPAC), Open Pyrolysis Activated Carbon (OPAC), Recovered Activated Carbon (RAC), and Refilled Water Activated Carbon (RWAC).

AC Stability Test

The stability test of the AC immobilized yarn can be observed from the amount of activated charcoal immobilized and released from the yarn after turning with a rotary evaporator. Five strands of AC immobilized thread were taken from each dose of adhesive, and a variety of activated charcoal powder was put into a rotary evaporator at a speed of 40 rpm for 5 minutes.

Layers' Stability and AC Homogeneity

A yarn layer thickness test was carried out to observe thickness differences. Thickness is measured using a digital screw micrometer. AC homogeneity test aims to observe the surface of the yarn, which is covered by activated charcoal powder. Yarns that AC has immobilized. The homogeneity was observed using a stereo microscope. The homogeneity level of the AC immobilization on the yarn surface was indicated by the invisible white surface of the yarn.

Adsorption Experiment

Contact Time

0.52 gram of immobilized CA was mixed with 4 ppm methylene blue. Each mixture was stirred at a speed of 200 rpm.[9] For various contact times, respectively 3, 6, 9, 12, and 15 minutes. The absorbance of each mixture was measured using a UV-Vis spectrophotometer (Shimadzu, Japan) at a wavelength of 664 nm.

Langmuir Isotherm

250 ml of methylene blue with various concentrations of 1, 2, 3, and 4 ppm each hung 0.52 grams of refilled activated charcoal immobilized on the thread. Then each solution was stirred using an upper stirrer

according to the optimum contact time. The filtrate was analyzed using a UV-Vis spectrometer at a maximum wavelength of 664 nm to determine the adsorbed MB levels.[10]

Adsorption Capacity Comparison Test

Testing of each thread of activated charcoal immobilized was carried out using 0.52 grams of activated charcoal which was immobilized in the yarn with a variation of the optimum concentration of Fox glue adhesive (PVAc) in the range of 65-90%, optimum fineness of activated charcoal powder in the range of 60-100 mesh, and the concentration. 10 ppm methylene blue solution. Then a control test was also carried out on the absorption of the methylene blue solution by hemp yarn covered with Fox glue (PVAc) with the same test as the adsorption capacity comparison test.

Yarn Regeneration

Regeneration of threads immobilized by open pyrolysis activated charcoal is carried out by cleaning first using refill water and rinsing last using distilled water, then drying for ± 2 hours. Activated charcoal from refilled water that is free from impurities is regenerated using acid-base with the addition of 150 ml 6 M HCl, which is stirred by a magnetic stirrer at 60˚C for 1 hour and left at room temperature for 24 hours. Activated charcoal soaked for 24 hours is filtered, washed with refill water, then neutralized using 150 mL 0.1 M NaOH for 1 hour, washed with refilled water, and rinsed using distilled water until the pH is neutral. In the final step, activated charcoal that has been neutral is dried in an oven at 70˚C for 24 hours.[11]

RESULTS AND DISCUSSION

The charcoal from the oil palm shell is activated using a chemical process, chemical activation is chosen because it has many advantages such as single-step activation, low temperature & activation time, better yields, and a larger pore structure.[12] In this study, the activation process was carried out by soaking palm shell charcoal for 24 hours in a solution of caustic soda flake 98% (NaOH) dye, this soaking process is called impregnation. The process of activating using a chemical method is divided into two: impregnating the raw material first, then combustion (open pyrolysis) or the raw material being burned first, and then the charcoal from pyrolysis (closed pyrolysis) is impregnated and obtained from closed and open pyrolysis palm shell charcoal. Based on the data, the percentage of closed pyrolysis was 37.50%, and open pyrolysis was 42.86%.

Fig.-1: Activated Carbon (A) RAC, (B) CPAC, (C) OPAC

Effect of PVAc Dosage and Powder Fineness

PVAc is commonly used as an adhesive for porous materials, so activated charcoal immobilized threads remain flexible and not stiff, PVAc must be diluted. Alcohol is an excellent solvent for diluting PVAc because it can increase the resistance and thermal and mechanical properties of PVAc.[13,14] In this study, ethanol was used because of its superiority of colorless and volatile properties so that it does not affect the properties of PVAc. The pores' state very much influences the adsorption rate of the AC, the pore structure is related to the surface area of the substance. The Activated Carbon Powder (ACP) has a broader surface area and more porous ratio, so the adsorption speed increases compared to granular AC[15,16] indicates that the yarn immobilized by 100 mesh of ACP has more pores and a wider surface area than the 60 and 80 mesh powders.

Yarn Coating Thickness

The thickness of the yarn layers immobilized by ACs was different due to the effect of the dosage of PVAc. Measurement of the thickness of the thread using a digital screw micrometer three times to obtain more accurate results, as shown in Table-1.

Table-1: Diameter Measurement Results Using a Digital Screw Micrometer

Concentration (%)	Yarn (mm)	Yarn + PVAc (mm)	Yarn+PVAc+AC (mm)
65	1.166	1.987	1.332
70	1.166	1.201	1.598
75	1.166	1.210	1.604
80	1.166	1.316	1.790
85	1.166	1.607	1.859
90	1.166	1.869	1.956

Based on the data for measuring the thickness of the layer using a digital screw micrometer, the results were insignificant in several variations of the dosage of PVAc. However, they were linear from a concentration of 65% to 90% due to the higher dosage of PVAc, the more (clumpy) the thread is covered with PVAc, the thicker the resulting layer of thread will be. The thickness of the activated charcoal immobilized yarn layer based on the variation of the fineness was obtained, namely, the smoother the activated charcoal immobilized in the yarn, the smaller the thickness of the yarn layer due to the influence of the activated charcoal powder size and vice versa. Diameter measurements are repeated three times to obtain more accurate results, the average results of the measurement data can be observed in Table-2.

Table-2: Yarn Diameters

Particle Size (mesh)	Yarn (mm)	Yarn + *PVAc* (mm)	Yarn+*PVAc*+AC (mm)
60	1.166	1.210	1.604
80	1.166	1.210	1.602
100	1.166	1.210	1.601

Stability of the ACIY

The stability of ACIY can be measured by using the rotation of the rotary evaporator round bottom flask, the rotation speed given was 40 rpm for 5 minutes. The amount of activated charcoal powder attached or released can be seen from the difference in the active yarn scales before and after the rotary evaporator using a digital balance as in Table-3. Table-3 shows that the quality of activated charcoal immobilized yarn is best if using Fox glue adhesive (PVAc) 75% because with this formula; activated charcoal threads are resistant to friction (rotary evaporator), so the amount of activated charcoal immobilized in the yarn is still outstanding, which is equal to 94, 7% and the activated charcoal released after turning at a speed of 40 rpm for 5 minutes was only 5.28%.

Table-3: Mass of Immobilized and Discharged Activated Charcoal

No	Experiment	AC mass before evaporation (g)		Quantity of AC after evaporation	
	PAVc's Dosage (%)	Powder (*mesh*)	Sticked (g)	Immobilized (%)	Discharged (%)
1	65	100	0.1367	81.4	18.6
2	70	100	0.1984	88.2	11.8
3	75	100	0.2837	94.7	5.28
4	80	100	0.2380	91.6	8.40
5	85	100	0.1040	87.5	12.5
6	90	100	0.1279	75.8	24.2

The quality of the activated charcoal threads decreased in the adhesive by 65% and 70% due to the effect of the low dosage of PVAc. Whereas in the adhesive treatment, 80, 85, and 90% of the effect of the adhesive dose were excessive, causing Fox glue (PVAc) to clot on the thread and not evenly. The stability of activated charcoal based on the fineness immobilized in the yarn is tested using a rotary evaporator at a speed of 40 rpm for 5 minutes. Yarn immobilized by activated charcoal with a fineness level of 60 mesh has a higher level of stability, which is 96.98%. However, yarn immobilized by 100 mesh of activated charcoal is best because it shows a more even or immobilized yarn surface compared to 60 mesh of activated charcoal; this is also influenced by the degree of fineness of activated charcoal. Based on the results of the observations, the percentage data were obtained, as shown in Table 4.

Table-4: Observation Data on the Amount of Activated Charcoal Immobilized and Released

No	Experiment		AC mass before evaporation (g)	Quantity of AC after evaporation	
	PAVc's Dosage (%)	Powder (mesh)	Sticked (g)	Immobilized (%)	Discharged (%)
1	75	60	0.3255	96.98	03.01
2	75	80	0.3087	94.62	05.37
3	75	100	0.1907	85.10	14.89

Observation of Yarn Surface Using Stereo Microscope

Observation of the yarn surface using a stereo microscope is carried out to ensure that all yarn surfaces are covered by activated charcoal powder. The stereo microscope was chosen because this microscope can be used to view solid specimen surfaces with realistic three-dimensional images of the entire surface of the object with a depth of perspective. From the observation in Fig.-2, it can be seen that the thread surface of the 65% and 70% adhesive is uneven, and the thread surface is still white.

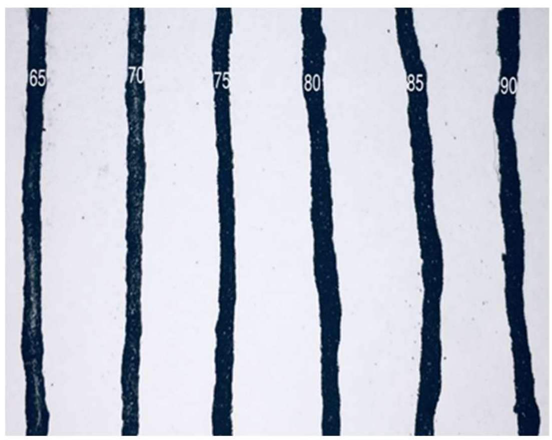


Fig.-2: The surface of the ACIY with different dosages of PAVc

Meanwhile, the yarn surface of the 75, 80, 85, and 90% adhesives coated with activated charcoal powder is evenly marked by the invisible white surface of the thread. However, the thicker the adhesive concentration makes the surface of the thread lumpy, and the thickness is not the same size, so it is best to dose 75%. The surface of the yarn covered with ACP can be observed using a stereo microscope. From the results of the observation in Fig.-2, it can be seen that the surface of the yarn covered with 100 mesh of ACP is more even compared to 80 and 60 mesh. This result happens because the more delicate the ACP, the easier it is for the ACP grains to flatten the surface of the yarn, while the immobilized yarn of the coarser ACP looks uneven due to the varying size of the ACP.

AC Adsorption Capacity Test for Methylene Blue

Effect of Contact Time

The effect of contact time on the adsorption of methylene blue needs to be done to determine the enormous absorption time needed by refilling water-activated charcoal immobilized yarn, closed pyrolysis, open pyrolysis, and regeneration of methylene blue maximally. The contact times used in the study were 3, 6, 9, 12, and 15 minutes using 0.52 grams of activated charcoal immobilized thread in 4 ppm methylene blue. The methylene blue solution was analyzed by UV-Vis spectrophotometer at a maximum wavelength of 664 nm. Observations after the optimum contact time show that the adsorption capacity of methylene blue has decreased and an insignificant increase because the optimum adsorption capacity occurs at the optimum time of each activated charcoal adsorbent.[8] The optimum contact time between activated charcoal refill water, closed pyrolysis, open pyrolysis, and regeneration can be observed in the graph of the relationship between absorbance and optimum contact time, as shown in Fig.-3.

The optimum time of several yarn adsorbents immobilized by activated charcoal in adsorbing methylene blue varies. The optimum time for the optimal refill of activated charcoal is 12 minutes, and the adsorption capacity was 87.05%. The optimum time of activated charcoal regeneration both closed pyrolysis and open pyrolysis were optimal at 9 minutes, and the results of the adsorption percentage are 85.11; 78.24; 80.64. respectively. The optimum contact time between the activated charcoal immobilized yarn adsorbent, and methylene blue adsorbate is longer than the pre-optimum contact time so that more dye is absorbed.

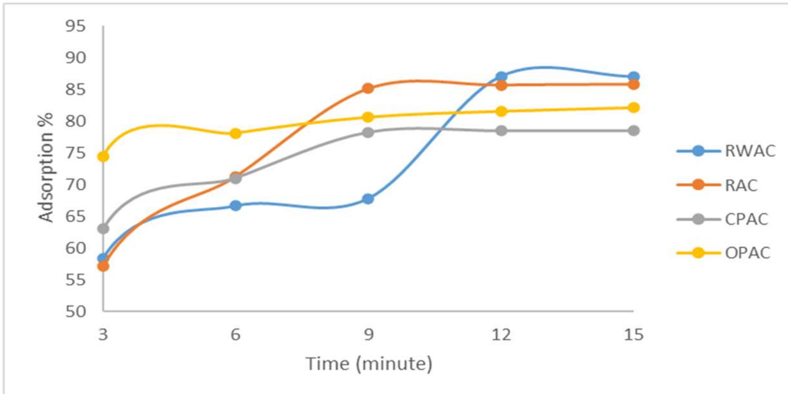


Fig.-3: Optimum Contact Time of Samples

Effect of Concentration

For various concentrations of 1, 2, 3, and 4 ppm, the absorbance obtained is directly proportional to the increase in concentration. The greater the concentration, the higher the percentage capacity. Therefore, a concentration of 4 ppm is the best variation between 1, 2, 3, and 4 ppm. The adsorption of methylene blue by activated charcoal highly depends on the pore structure and surface chemical properties of the activated charcoal used as an adsorbent.[17] Refillable water-activated charcoal and refill water regeneration are made from coconut shells. Closed and open pyrolysis-activated charcoal made from oil palm shells. Activated charcoal from coconut and oil palm shells functions as an adsorbent because it contains cellulose. Coconut shell contains 34% cellulose[18], and palm kernel shells contain 29.7% cellulose.[19] Cellulose can interact with methylene blue's cationic substance because it contains hydroxyl and carboxylic acid functional groups. These functional groups can be ionized into anionic compounds negatively charged. The negative charge on the surface of the activated charcoal will bind to the positive charge of the methylene blue dye. Therefore, the cationic dye methylene blue adsorption process using activated charcoal adsorbent can take place.[17] Based on the observations in Fig.-4, variations in the concentration of methylene blue from several types of activated charcoal obtained different adsorption capacities with the best order of activated charcoal refill, regeneration, open pyrolysis, and closed pyrolysis. This difference can be caused by the ingredients for making activated charcoal and the process of activating activated charcoal. Activated charcoal made from coconut shells looks superior to charcoal made from oil palm shells, this is because the cellulose content in coconut shells is higher than in palm shells, and another factor is the process of activating different activated charcoal.

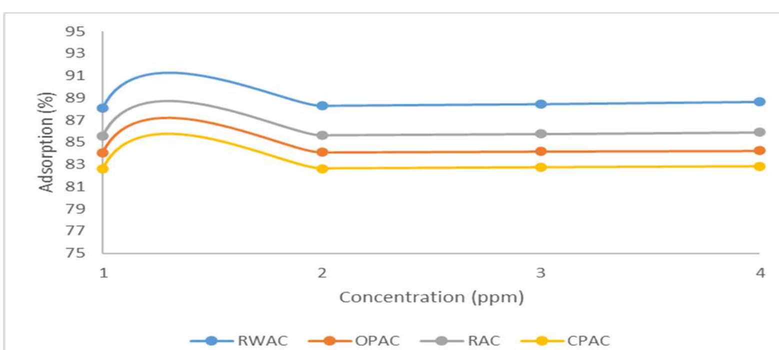


Fig.-4: Effect of Methylene Blue Concentration on the Adsorption Capacity

Langmuir Isotherm

The methylene blue adsorption process using activated charcoal adsorbent immobilized by activated charcoal is assumed to follow the Langmuir isotherm pattern because, based on the results of previous studies, the Langmuir adsorption isotherm has been successfully used to explain the adsorption of basic dyes.[20-22] Therefore, this study determined the adsorption capacity using the Langmuir adsorption isotherm equation.

Table-5: Langmuir's Adsorption Isotherm

AC types	Ce (mg/l)	qe (mg/g)	1/Ce	1/qe
RWAC	0.1076	0.3815	9.2916	2.6207
	0.2139	0.8831	4.6750	1.2873
	0.3453	1.2719	2.8961	0.7861
	0.4439	1.6686	2.2525	0.4992
RAC	0.1300	0.3708	7.6897	2.6969
	0.2623	0.7535	3.8120	1.3272
	0.4256	1.2334	2.3498	0.8108
	0.5516	1.6169	1.8130	0.4185
CPAC	0.1570	0.3579	6.3714	2.1943
	0.3178	0.7268	3.1470	1.3758
	0.5166	1.1896	1.9357	0.8406
	0.6726	1.5587	1.4866	0.6415
OPAC	0.1435	0.3643	6.9688	2.7448
	0.2906	0.7399	3.4414	1.3515
	0.4726	1.2107	2.1157	0.5259
	0.6166	1.5857	1.6218	0.6306

The best adsorption isotherm of methylene blue by activated charcoal can be determined by comparing the linear regression equation and the coefficient of determination (R^2). The results of the research and calculations show that the coefficient of determination (R^2) is not so significant between several types of activated charcoal, and the R-value close to number 1 means that the results obtained are close to linear or good.[23] The value of the Langmuir isotherm determination coefficient can be seen in Table-6.

Tabel-6: Langmuir Isotherm Determination Coefficient Value

No	AC type	Y	R	Slope	Intercept
1	RWAC	$y = 0.2954x - 0.1130$	$R^2 = 0.9980$	3.3852	2.6072
2	RAC	$y = 0.3734x - 0.1489$	$R^2 = 0.9925$	2.6781	2.5077
3	CPAC	$y = 0.3116x - 0.2549$	$R^2 = 0.9807$	3.2092	1.2224
4	OPAC	$y = 0.4198x - 0.1716$	$R^2 = 0.9817$	2.3821	2.4464

The Langmuir isotherm equation obtained can determine the adsorption capacity (b) and the equilibrium constant (K) for methylene blue by each activated charcoal, as shown in Table 6. The adsorption capacity value obtained shows the adsorption capacity of the activated charcoal immobilized yarn in adsorbing methylene blue.

Open Pyrolysis Activated Charcoal Immobilized Yarn Regeneration

Regeneration of open pyrolysis activated charcoal immobilized yarn aims to restore its adsorption ability after reaching saturated conditions. Regeneration using chemical methods has the principle that the chemicals will bind the adsorbate to the pores of immobilized activated charcoal in the yarn. So that the adsorbate in the pores will be released from the surface of the open pyrolysis-activated charcoal immobilized in the yarn to compare the difference in the adsorption capacity of open pyrolysis and its regeneration can be observed in Fig.-5.

Based on the comparison picture of adsorption capacity of open pyrolysis and its regeneration, it shows that threads immobilized by open pyrolysis activated charcoal can be regenerated and reused. However, the ratio of adsorption capacity between open pyrolysis activated charcoal immobilized yarn and its regeneration is much different, which is about 30% compared to OPAC due to the amount of activated charcoal attached to each yarn; the amount of activated charcoal in the open pyrolysis activated charcoal immobilized yarn is 0.52, while the regeneration is ± 0.2. The reduced amount of activated charcoal in regeneration is due to the immobilized open pyrolysis activated charcoal on the yarn that is released during the washing process and activates when regenerating.

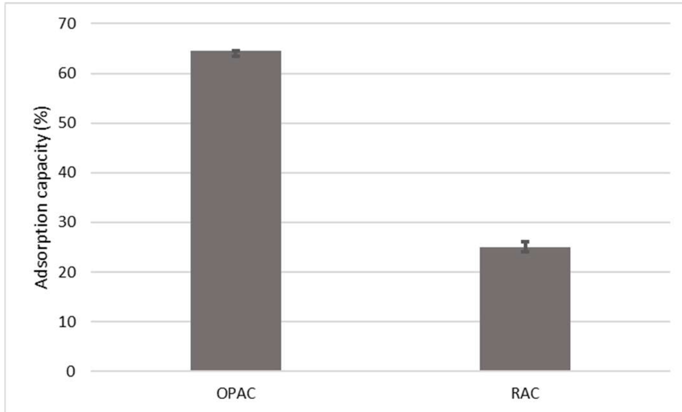
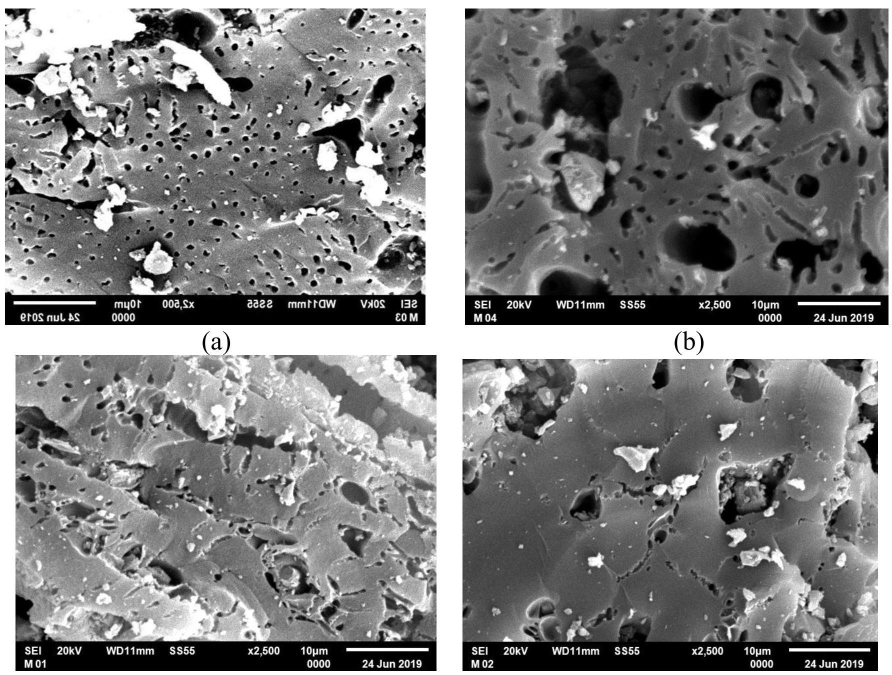


Fig.-5: Comparison between OPAC and RAC

Observation of Yarn Surface Using SEM

The yarn surface was observed using SEM for the surface morphology of the yarn that had been immobilized with activated charcoal powder. The results of SEM photos of immobilized threads can be seen in Fig.-6.



(a) (b)
(b) (d)

Fig.-6: SEM Images (magnification 2500 x) (a) OPAC, (b) CPAC, (c) RAC, and (d) RWAC.

Based on Fig.-6 (a), it can be seen that the morphology of the threads immobilized by activated charcoal from open pyrolysis has a porous surface with a relatively small size. In contrast, threads immobilized by activated charcoal from closed pyrolysis have pores that tend to be significant in size. As for the morphology of immobilized open pyrolysis activated charcoal, the regenerated yarn has a surface with a small number of pores.

ACIY Control Test

The control test on the activated charcoal immobilized yarn aims to prove the absorption process of the methylene blue solution without any external absorption factor and prove that the activated charcoal immobilized yarn is suitable for use as an adsorbent. The control test consisted of two treatments; the first was comparing the absorption of activated charcoal powder and activated charcoal immobilized yarn with a mass of 0.52 in 3 ppm for 9 minutes, and the second was the absorption test of threads that had been coated with PAVc.

Table-6: Absorbance Test of ACIY Control

Yarn + PAVc		AC		ACIY	
Before	After	Before	After	Before	After
0.6	0.6	0.6	0.072	0.6	0.086

The results showed that the absorbance between the immobilized activated charcoal and activated charcoal powder was insignificant, and the thread smeared with glue proved that it could not absorb the methylene blue solution without immobilized activated charcoal on the yarn.

CONCLUSION

Based on the research results, it can be concluded that the yarn immobilized by activated charcoal using 75% PVAc and a smoothness level of 100 mesh showed the most stable results. Water-activated charcoal refill, regeneration, closed pyrolysis, open pyrolysis, and Merck are suitable for adsorbents based on ASTM characterization. The optimum contact time of the test results for absorption of methylene blue activated charcoal refill is 12 minutes, while the regenerated activated charcoal, closed pyrolysis, and open pyrolysis is at 9 minutes. The adsorption isotherm for the absorption of methylene blue by activated charcoal follows the Langmuir isotherm pattern.

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