Adsorption Capability of Activated Carbon Prepared from Silica Extracted Rice Husk by Chemical Activation

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Abstract: This research is done to optimize the rice husk residue usage with the production of activated carbon by using the chemical activation method consecutively after the silica extraction. The silica extracted rice husk was impregnated with different concentration of KOH solution and then burned to activation at the predetermined activation temperature to obtain the activated carbon. The optimum condition to produce the activated carbon is impregnation with 30 % concentration of KOH solution at 750 $^{\circ}$C activation temperature. The maximum methylene blue uptake of the activated carbon produced are tested to be 322.36 mg g^{-1}. The adsorption isotherm model study was done using Langmuir isotherm. The Langmuir isotherm adsorption capacity, Q_e was calculated to be 416.67 mg g^{-1} while the rate of adsorption, b value was 4.8 mg^{-1} with R^2 0.9995. The results obtained from this study show that the silica extracted rice husk is a suitable precursor for preparing an activated carbon and the activated carbon have good absorption capacity.

1 INTRODUCTION

Rice is the world's second most important crop in the world after wheat and is a primary source of food for more than half of the world's population mainly for Asian. More than 90 % of the world's rice is grown in Asia making it as both the largest producer and consumer of rice (Rajamoorthy et al, 2015).

Among major rice producing countries in Asia are China, India, Indonesia, Bangladesh, Vietnam and Thailand with rice production rate around 145 million to 16.4 million metric tonne per year. Still, it is estimated that about 70 % increase in rice production yearly is needed to cater the demand of Asian population growth in the future. Malaysia, being a part of Asia has also been a country where rice is the staple food of Malaysian. The food consumption pattern of Malaysian adult population shows that rice is at top of the list of 10 most consumed food daily where majority of Malaysian consume rice twice a day and on average, two and a half plates of rice per day (Noor Shuhadah et al, 2012). The importance of rice as Malaysian staple food has made The Ministry of Agriculture and Agro-based Industry (MOA) implemented

DasarAgromakanan Negara (DAN) to ensure the stability of the country's rice stock by increasing the production of Malaysia local rice (Norimah et al, 2008). However, the increase in rice production also means the increase of waste income due to the milling process of rice. Rice grains are generally coated by a protective covering layer known as rice husk. The husk is indigestible to human as it is made of hard materials including silica and lignin in order to protect the seed during the growing season. The rice husk are removed from the grain during the milling process to create the brown rice which then milled further to produce the white rice. There is roughly 0.28 kg of rice husk by-product produced for each kg of milled white rice. The worldwide annual rice husk output is about 80 million tonnes and according to statistic by Malaysian Ministry of Agriculture, 408,000 metric tonnes of rice husk are produced in Malaysia each year. In the past, rice husk was considered and treated as useless agricultural waste by farmers hence it was usually burned. This leads to the release of carbon dioxide (CO_2) gas to the atmosphere which is not only harmful to the environment but also gives negative impact to human health as it can affect the respiratory function (Noor Syuhadah et al, 2012). Thus, many major rice producing countries

including Malaysia have made finding on the usage of rice husk and ways to utilize it commercially as their research priority. Findings have led to a few implementations of rice husk as fuel in power plant, due to its high calorific power (Noushad et al, 2012), as insulation or building materials since it is highly resistant to 3 penetration of fungal and moisture decomposition, as fertilizer, capacitors (Le Van et al, 2014), pollutant and odour removal, gas separation and catalysis (Liu et al. 2011). The high silica content of silica in rice husk has attracted interest on how to use it commercially as a viable raw material for the production of silicates and silica. The rice husk ash especially obtained from the combustion step of rice husk contains silica of over 60 %. Apart from silica, other major component of rice husk and rice husk ash are about 10-40 % carbon and minor other mineral compositions (An et al, 2011) Production of activated carbon from rice husk has also attracted the interest of various parties. Activated carbon is a versatile adsorbent due to its good adsorption property from its high surface area, fast adsorption kinetic and large adsorption capacity (Cheenmatchaya et al. 2014). It can be produced from variety of raw materials such as fruit shell, straw, residual waste and from agricultural by-products (An et al. 2011). In earlier researches, many researchers' focuses were on the production of either an activated carbon or a silica from rice husk ash. The usage of rice husk ash is preferred because of its higher content of silica and carbon compared to rice husk. The procedure was usually complex hence the carbon source and silicone source could not be sufficiently utilized. Only a few researches found the production of silica and activated carbon done consecutively (An et al. 2011).

Activated carbon also known as activated charcoal is the oldest yet versatile adsorbent that is derived from charcoal. It is an excellent adsorption property as it can adsorb various substances from gas and liquid streams. For adsorption of gases and vapours, a granular types of activated carbon is usually used while for 5 purification of liquid, a powdered activated carbon is more desired. Activated carbon has been reportedly used as adsorbent for a wide varieties of inorganic and organic pollutants dissolved in aqueous media or from gaseous environment (Gupta et al. 2008), such as for different types of dyes (Ahiduzzaman et al. 2016) and heavy metals (Bishnoi et al. 2004), as electrode materials for batteries and capacitors (Le Van et al. 2014), as odour removal, gas separation and catalysis (Chen et al. 2011). The effectiveness of activated carbon to react as adsorbent is because of

its highly developed pore structure and large internal specific surfaces area (Le Van et al. 2014). Activated carbon is deem as a material of major industrial and has been produced extensively on a commercial scale. The commonly used materials to prepare commercial activated carbon are coal (El Qada et al. 2006), wood (Sahu et al. 2010), petroleum (Niasar et al. 2018), pitch (Gao et al. 2017), etc. However, this non-renewable starting materials are relatively expensive and sometimes even low in availability hence this has increased the production cost of activated carbon with limited usage. This has led to a growing research interest mainly by developing nations in the production of activated carbon from a natural, renewable and low cost materials especially for application regarding waste water treatment (Tan et al. 2010), remediation and decontamination process (Chen et al. 2011). Focus is given especially in the production of activated carbon from waste materials such as agricultural by-products as it will not only solve the problem of waste disposal but also convert a potential waste to a valuable product (Thomas et al. 2017). Among the agricultural by-product studied by researchers for the production of activated carbon are almond shells (Thitame et al. 2015) , date pits (Girgis et al. 2002), coconut shells (Huang et al. 2015), sugarcane waste and rice husk (Kalderis et al. 2008). Activated carbon made from this agricultural by-product are found to be effective in the removal of pollutants from water (Thomas et al. 2017). However, the properties of the activated carbon 6 produced is governed not only by the raw materials used, but also by the method of the activation used About 571 million tonnes of rice resulting in approximately 140 million tonnes or rice husk are produced annually in the world (Kalderis et al. 2008), 96 % of which are generated in developing country. Rice husk major constituents are cellulose, hemicellulose, lignin and mineral components though the content of each depends on the rice variety, climate conditions and geographic localization of the culture (Chen et al. 2011). On early days, rice husk is considered as a low energy resource, thus is always discarded or burned on the field which are unfavourable to environment (Noor Syuhadah et al. 2012). The production of activated carbon from rice husk does not only produce an activated carbon with good adsorption properties but will also alleviate the problem of disposal and management of this waste by-product. Production of activated carbon from rice husk generally can be achieved through 2 routes, physical activation also known as thermal activation and chemical activation

(An et al. 2011, Chen et al. 2011 and Thomas et al. 2017). In physical activation, rice husk is firstly carbonized into rice husk char at high temperatures (between 600-900 ºC) followed by activation at an elevated temperature (between 600-1100 ºC) in the presence of a suitable oxidizing gases such as CO_2, steam, air or their mixture (Chen et al. 2011 and Thomas et al. 2017).

In chemical activation, rice husk is mixed with a chemical agent such as potassium hydroxide (KOH) (An et al. 2011), sodium hydroxide (NaOH) (Le Van et al. 2014), phosphoric acid (H_3PO_4) (Cheenmatchaya et al. 2014), zinc chloride ($ZnCl_2$) (Chen et al. 2011). Then the carbonization and activation are performed simultaneously at activated temperature (between 400-900 ºC). During activation, disorganized carbon is removed by exposing the crystallites to the action of activating agent which leads to the development of porous structure. The efficiency of the activated carbon is also strongly influenced by a relatively 7 small amount of chemically bonded heteroatoms (mainly oxygen (O) and hydrogen (H)) (Chen et al. 2011 and Thomas et al. 2017).

Chemical activation is usually a much more preferred method to produced activated carbon as it provides two important advantages in comparison to physical activation and the process can be performed at a lower temperature and the global yield of chemical activation tends to be greater because burning off charcoal is not required (Mohanty et al. 2006). However, an admixed method of physical and chemical process can also be applied (Le Van et al. 2014).

2 EXPERIMENTAL

2.1 Acid Pre-treatment of Rice Husk

300.0 g of the rice husk sample was washed thoroughly 2 times with normal water and 5 times with distilled water respectively. The rice husk was then dried in a universal oven (Memmert, UFE 600, Germany) at 110 ºC for 24 h. 40.0 g of the dried rice husk was weighted into a 1000 mL beaker and treated with 1 M solution of HCl at 75 ºC for 90 minutes in a water bath (Julabo, tw20, Germany). The suspension was filtered to extract the solid residue of rice husk which was then dried again in a universal oven (Memmert, UFE 600, Germany) at 110 ºC for 24 h.

2.2 Extraction of Silica

40.0 g of the acid pre-treated rice husk was immersed in 600 mL of 10 % of NaOH solution in a beaker and heated at 90 ºC for 60 minutes in a water bath (Julabo, tw20, Germany). The suspension was let cool for 2 h and then filtered to extract the sodium silicate solution from the rice husk.

2.3 Preparation of Activated Carbon

The silica extracted rice husk was washed with distilled water and dried in an universal oven (Memmert, UFE 600, Germany) at 110 ºC for 24 h. 10 g of the silica extracted rice husk was then impregnated with 100 mL of 10 %, 20 % and 30 % of KOH solution in a beaker and heated at 90 ºC for 60 minutes in a water bath (Julabo, tw20, Germany).

After that, the mixtures were let cool to room temperature before being filtered to remove the excess KOH solution. The impregnated rice husk was then transferred into a 100 mL porcelain crucible and dried overnight at 80 ºC in a universal oven (Memmert, UFE 600, Germany). The dried impregnated rice husk was then burned in a burn out furnace (UginDentaire, Programix 100, Freance) at heating rate of 10 ºC/min from room temperature to the final activated temperature of 750 ºC, and 800ºC and the final temperature was maintained for 60 minutes. The samples were then let cool to room temperature and then washed with distilled water repeatedly by using vacuum filtering setup to remove the activating agent. The product obtained was then dried in the universal oven (Memmert, UFE 600, Germany) at 110 ºC for 24 h. Finally, the activated carbon obtained was grinded and stored in adesiccators.

2.4 Adsorption Study of Activated Carbon

Firstly, the activated carbon obtained was evaluated with Methylene Blue (MB) dye adsorption test. The test was done to find out the best combination of the activation factors used (temperatures and rice husk: KOH % concentration ratio) to obtain the best product of activated carbon from the silica extracted rice husk. A commercial activated carbon was also used as control.

A standard calibration graph was plotted by finding the absorbance value for a series of Methylene Blue (MB) solution concentration from 0.5, 1.0, 1.5, 2.0 and 3.0 mg L^{-1} that were prepared in 5 different volumetric flasks. The maximum

absorbance of the solution at 664 nm of wavelength (λ) was measured by using a spectrophotometer (Thermo scientific, Genesys 20, USA). Distilled water was used as blank.

A series of Methylene Blue (MB) solution concentration 50, 100, 150, 250 and 300 mg L^{-1} was prepared in 5 different volumetric flasks. 0.01 g of the each activated carbon sample obtained was then weighted (Sartorius, BSA224S CW analytical balance, Germany) and mixed with 15 mL of each MB solution prepared in a beaker. The mixture was stirred and kept for 24 h at room temperature. Next, the dye solution was transferred into a falcon mask and centrifuged (Hettich, Universal 32 R, Germany) for 20 min at 1500 rpm to settle down the carbon particle at the bottom of the tube. Clean solution obtained was then filtered using a pore size 0.45 μm filter paper to remove the remaining carbon particle from the solution. The maximum absorbance of the solution at 664 nm of wavelength was then measured by using a spectrophotometer (Thermo scientific, Genesys 20, U.S.A). Distilled water was used as blank.

The coefficient of extinction was calculated by plotting a calibration chart of absorbance with respect to the MB concentration. The concentration of MB after adsorption was then determined by using equation:

$$C_e = \frac{A}{E} \quad (1)$$

where C_e is the concentration of MB solution after adsorption, mg L^{-1}; A is the absorbance ; E is the coefficient of extinction, L mg^{-1}.

The amount of MB absorbed was than calculated by using the following equations:

$$q_e = \left(\frac{(C_o - C_e) \, V}{m} \right) \quad (2)$$

where q_e is the uptake of dye adsorbent, mg g^{-1}; C_o is the initial concentration of dye, mg L^{-1}; C_e is the final concentration of dye, mg L^{-1}; V is the volume of dye solution, L; m is the weight of activated carbon, g.

3 RESULT AND DISCUSSION

The effect of different activated conditions variable on physical and chemical characteristics of the activated carbon products will be discussed in this section. The influencing factors on the methylene blue adsorption capacity of activated carbon

products are investigated and compared with the commercially bought activated carbon.

3.1 Methylene Blue Dye Adsorption Test

Table 1: The uptake value of methylene blue by activated carbon, q_e for activation temperature 750 ºC.

Initial MB Conc (mgL^{-1})	q_e (mg g^{-1})			
	AC750 -10	AC750 -20	AC750 -30	CAC
50.0	73.121	73.313	74.981	74.814
100.0	139.78	142.95	149.97	147.53
150.0	177.91	212.31	224.59	183.43
200.0	220.73	268.52	298.01	202.77
300.0	251.90	331.51	418.88	219.96

Table 2: The uptake value of methylene blue by activated carbon, q_e for activation temperature 800 ºC.

Initial MB Conc (mg L^{-1})	q_e (mg g^{-1})			
	AC800 -10	AC800 -20	AC800 -30	CAC
50.0	71.946	72.768	74.257	74.814
100.0	138.25	140.95	147.86	147.53
150.0	176.97	181.43	219.65	183.43
200.0	210.40	216.97	262.42	202.77
300.0	245.44	276.67	322.35	219.96

Initial MB Conc (mgL-1)	qe (mg g-1)			
	AC750 -10	AC750 -20	AC750 -30	CAC
50.0	73.121	73.313	74.981	74.814
100.0	139.78	142.95	149.97	147.53
150.0	177.91	212.31	224.59	183.43
200.0	220.73	268.52	298.01	202.77
300.0	251.90	331.51	418.88	219.96

and table 2 contain the uptake value of methylene blue by all activated carbon prepared in the 750 ºC and 800 ºC respectively and the commercial activated carbon bought. From the experimental data obtained it is seen that activated carbon with good adsorption properties can be made from a silica extracted rice husk.

This meansthat the porosity of the activated carbon structure can be created not only just by

leaching out silica and removing the chemical activating agents in the carbonized samples by washing. In the case of low or absence of silica content in the solid residue of rice husk, the activating agent KOH will reacted with carbon to produce an activated carbon (An et al 2011).

The used of KOH as an activating agent in the preparation activated carbon has been known to produce activated carbon with good pore development, greater specific surface area though at the cause of a typically low percent yield. The intercalation of metallic potassium ions into the carbon network during the development of pores of the activated carbon accelerates the carbon loss. During the activation process, the following reactions take place (Hui et al, 2015).

$$C + 2KOH \rightarrow 2K + H_2 + CO_2 \quad (3)$$

$$C + 2KOH \rightarrow 2K + H_2 + CO \quad (4)$$

$$CO_2 + 2KOH \rightarrow K_2CO_3 + H_2O \quad (5)$$
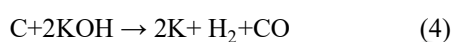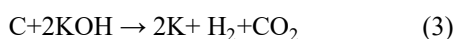
The potassium carbonate decomposed during activation and CO_2 gas was released. The reaction between the activating agent and the carbon precursor lead to the decomposition of the volatile organic compounds and in return formed the porous surface of the activated carbon samples (Muniandy et al. 2014).

3.1.1 The Effect of Initial Concentration of Methylene Blue on Uptake Value of Methylene Blue by Activated Carbon
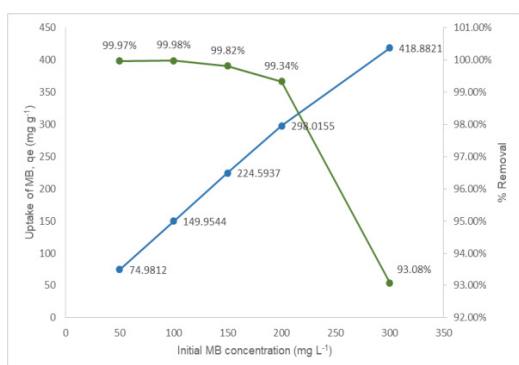

Figure 1: The effect of initial concentration of methylene blue on uptake value of methylene blue by activated carbon.

Figure 1 shows the effect of initial concentration of methylene blue on the uptake value and % removal of methylene blue by activated carbon prepared at

activation temperature of 750 °C and 30 % concentration of KOH solution. The figure shows that though the uptake of methylene blue by activated carbon used increased from the low to high concentration of methylene blue, the % removal of methylene blue were actually decreased.

At low concentration of methylene blue, sufficient adsorption sites of activated carbon are available for the adsorption of methylene blue. At higher concentration, relatively less available sites of activated carbon caused the reduction in the % of adsorption. Hence, increasing the initial methylene blue concentration decreased the % removal of methylene blue from the solution due to the saturation of adsorbent or activated carbon with methylene blue solution.

3.1.2 The Effect of Activation Temperature of Activated Carbon on the Uptake Value of Methylene Blue
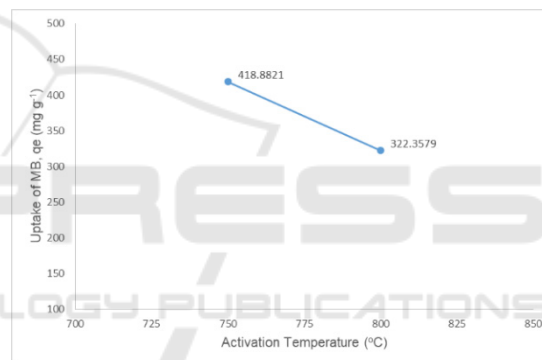

Figure 2: The effect of activated temperature of activated carbon on methylene blue uptake.

Figure 2 shows the uptake value of methylene blue with initial concentration of 300 mg L^{-1} by activated carbon produced with 30 % concentration of KOH solution. The figure shows that the uptake value of the methylene blue by activated carbon produced at activation temperature of 750 °C is higher compared to activated carbon produced at activation temperature of 800 °C. This shows that activation temperature influenced the adsorption capacity of an activated carbon. This is because the activation temperature used is among the parameters that highly influenced the formation of pore and its structure of an activated carbon.

The decrease of uptake value of methylene value from the activated carbon produced using activation temperature of 800 °C in comparison to 700 °C suggest that 700 °C is the optimum activation temperature for the preparation of the activated

carbon. Hence, the methylene blue adsorption capability of the activated carbon is actually increasing with the increase of activation temperature to a certain temperature and then decrease again if heat is still supplied (Rhaman et al. 2015)

The formation of pore is simultaneous with the destruction of pore. This suggest that at activating temperature of 750 ℃, the active reaction is at its maximum and increase the adsorption ability of the activated carbon. At this temperature, the active reaction led to a much more number of pore forming hence increased the specific surface area of the activated carbon. But, at activating temperature of 800 ℃, the destruction of pore is higher than the formation of pore hence resulting in a reduction of the specific surface area of the activated carbon (Guo et al. 2002).

This result suggested that to the activated carbon with optimum adsorption properties, the activation temperature used should be around 750 ℃.

3.1.3 The Effect of KOH Percentage Concentration of Activated Carbon on the Uptake Value of Methylene Blue
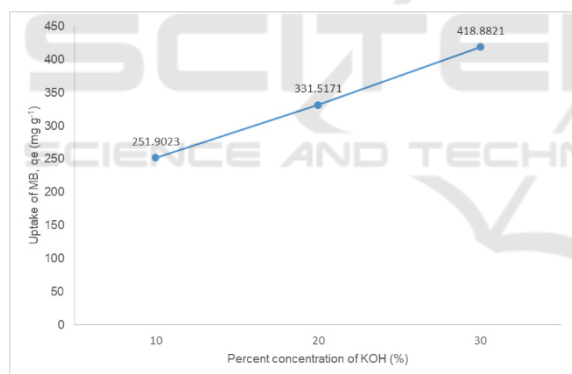

Figure 3: The effect of KOH % concentration of activated carbon on methylene blue uptake.

Figure 3 shows the effect of KOH % concentration of prepared activated carbon on the uptake value of methylene blue. The uptake value of methylene blue used are from the reaction between the activated carbon prepared at 750 ℃ with methylene blue initial concentration of 300.0 mg L^{-1}. The uptake value of methylene blue by activated carbon can be seen increases with the raise of KOH % concentration. This suggest that increase in activating agent promotes to a better production of activated carbon.

The increase of activating agent at higher % concentration of KOH solution promotes the contact

area between the rice husk and activating agent, hence increasing the adsorption ability of activated carbon. Also, since the adsorption ability of an activated carbon is related to its pore volume it can be said that the pore volume of activated carbon produced also increases with the increase of the KOH % concentration. Though further test has to be done to prove this suggestion (An et al. 2011, Rhaman et al. 2015 and Guo et al. 2002).

3.2 Adsorption Isotherm Model Study

The adsorption isotherm study is done to describe the interaction between solutes and adsorbents, and is critical in optimizing the use of adsorbents. We used the Langmuir model for this study.

3.2.1 Langmuir Isotherm

Langmuir isotherm assumes monolayer adsorption onto a surface containing infinite number of adsorption site of uniform strategies of adsorption with no transmigration of adsorbate in the plane surface (Hameed et al. 2007). The applicability of the isotherm equation is compared by judging the correlation coefficient R^2 value. Langmuir equation is written as:

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \left(\frac{1}{Q_o}\right) Ce \qquad (6)$$

Where q_e is the Where q_e is the uptake of MB adsorbent, mg g^{-1} at equilibrium, C_e = final concentration of MB at equilibrium, Q_o is the Langmuir adsorption capacity constant, mg g^{-1} and b is the constant related to rate of adsorption, L m g^{-1}.
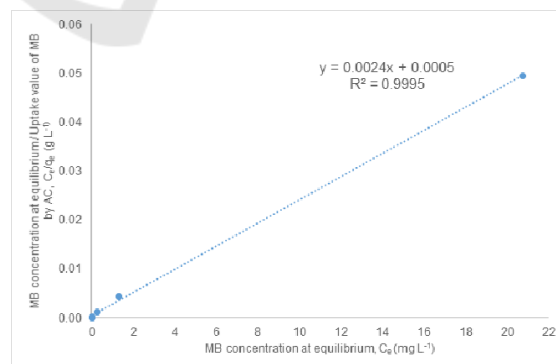

Figure 4: Langmuir isotherm constant for methylene blue-activated carbon.

Figure 4 was plotted using values from adsorption of methylene blue by activated carbon prepared by activation temperature of 750 ℃ and 30

% concentration of KOH. The figure indicated that the adsorption of methylene blue followed the Langmuir isotherm. The constant Q_o and b were calculated using equation and their values are as in Table 3 Confirmation of the experimental data into Langmuir isotherm model indicates the homogenous nature of the rice husk carbon surface. This also demonstrate the formation of monolayer coverage of dye molecule at the outer surface of the activated carbon [Hameed et. al]. The experimental data fit well with the isotherms with R^2 values of 0.9995.

Table 3: Langmuir adsorption isotherm parameters for methylene blue-activated carbon.

Q_o (mg g^{-1})	416.67
b (L mg^{-1})	4.8
R^2	0.9995

The Q_o value obtained was 416.67 mg g^{-1}. Similar result was reported by adsorption of methylene blue onto activated carbons prepared from bamboo-based [Hameed et. al] and by the adsorption of direct dyes on activated carbon prepared from stawdust (Malik et al. 2004). Table 4 shows that the activated carbon prepared has a very large adsorption capacity compared with some data from references.

Table 4: Comparison of the maximum mono layer adsorption of some dyes on various adsorbents.

Dyes	Adsorbent (Activated Carbon)	Maximum monolayer adsorption capacity (mg g^{-1})	Reference
Methylene blue	Bamboo	454.20	Hameed et al. 2007
Methylene blue	Coconut shell	277.90	Adamson 1990
Methylene blue	Groundnu-tshell	164.90	Adamson 1990
Methylene blue	Rice husk	343.50	Adamson 1990
Methylene blue	Jute fiber	225.64	Tsai et al. 2001

4 CONCLUSIONS

From this study, it is concluded that the produce of activated carbon from silica extracted rice husk is possible and doable. The produce activated carbon from silica extracted rice husk shows promising adsorption capacity for the methylene blue removal. The results obtained were even better than the commercially bought activated carbon. The optimum experimental conditions from the study for the produced activated carbon were from the impregnation of the rice husk with 30 % concentration of KOH solution and activation temperature of 750 ºC. The maximum adsorption capacity value, Q_o is recorded to be 416.67 mg g^{-1}. The results obtained from this study suggest that the silica extracted rice husk is a suitable precursor for preparing an activated carbon.

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