



Methylene Blue Dye Adsorption in Aqueous System using Microcrystalline Cellulose obtained from Sugarcane Bagasse

ONIGBINDE, MO; FATOYE, EO; *ISOLA, OB

Chemical Sciences Department, Glorious Vision University, Ogwa, Edo State, Nigeria

*Corresponding Author Email: dotmanchope@gmail.com; Tel: + 2347064644527

Co-Authors Email: monigbinde@gmail.com; bukkyfatoye1@gmail.com

ABSTRACT: Microcrystalline cellulose (MCC) was prepared from sugarcane bagasse (SCB) after alkali extraction with sodium hydroxide (NaOH). The prepared MCC was treated with methylene blue solution. Batch experiments were performed to investigate the effect of contact time, initial dye concentration, pH and adsorbent dosage on methylene blue adsorption. The result shows that the adsorption of methylene blue dye onto the adsorbent was influenced by adsorbent dose, dye concentration, contact time and pH values. For higher removal of dye from simulated wastewater, adsorbent dose of 0.4g and dye concentration of 200 mg/l gave optimum adsorption with percentage removal (% R) of 87.19. The adsorption capacity of methylene dye increased with increase in contact time. Also, the duration and pH for optimum adsorption were at 120 mins and pH 9 respectively. The experiment data fitted well into Freundlich isotherm. The results of this work revealed that microcrystalline cellulose from sugarcane bagasse is a potential alternative non-conventional adsorbent for treating dye effluent.

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Microcrystalline cellulose (MCC) is a naturally occurring substance obtained from purified and partially depolymerized cellulose (Trache, 2016). The amorphous regions of α -cellulose are the result of deformity in the structure due to the change in the process of crystallization. The cellulose can lead to Micro cellulose (MCC) and Nano-cellulose (NCC) through its acid hydrolysis, and as a result it improves its performance as reinforcing particle in composites (Wankenne and Aditivos, 2014). The natural disorders of cellulose molecules in the amorphous regions favour the easy penetration of the acid and consequently the hydrolysis of cellulose chains in these regions. The MCC has many properties, such as odorless, tasteless, and whitish crystalline cellulose powder which exhibited strong mechanical properties, low density, less or non – abrasive behavior, high reactivity renewable and biodegradability compared to

other filler such as silica, glass fibers, carbon black etc. It is an aqueous insoluble derivative of cellulose, being rod-shaped particles, which explain its vast application, especially in the food field. Due to the good properties of MCC, they have attracted the attention of various fields like packaging, agriculture, automotive, aerospace, pharmaceuticals, polymers, cosmetics and food composite industries as thickener, binder, reinforcement, emulsifier and stabilizer agent (Trache, 2016). Microcrystalline cellulose is manufactured by controlled acid hydrolysis of α -cellulose with dilute mineral acid solutions which can be obtained from various sources, such as plants, wood, agricultural waste like sugarcane bagasse, corn cob, rice husk cotton linter, coconut shell, silk cotton husk, orange peel, mango mesocarp and banana peel (Dawoud *et al.*, 2022; Kharismi and Suryadi, 2018). Direct use of polysaccharides such as maize straw

*Corresponding Author Email: dotmanchope@gmail.com

(Gualo *et al.*, 2015), palm oil biomass (Abdullah *et al.*, 2009), chitosan (Chiou and Li, 2002) and many other polysaccharides- based adsorbents; have been studied as potential effective yet renewable adsorbent. Among these agricultural waste, sugarcane bagasse produced from soft cane is regularly cultivated by local famers in Nigeria. The soft cane is mainly chewed raw for its sweet juice while some of it is processed into different crude sugar products. Soft cane production has being reported to accounts for about 60% of total sugarcane production in many years in Nigeria (Wayagari *et al.*, 1999). Synthetic dye pollutants are highly toxic organic substances from textile industrial effluents which constitute serious environmental problem. These dyes are difficult to degrade because they are very stable to light and oxidation (Chen *et al.*, 2010). Methylene blue and crystal violet are typical examples of widely used industrial dyes possessing high toxic effect. Methylene blue is a common cationic dye which is highly soluble in solution and readily aggregates causing harmful effects. It has been reported to cause breathing difficulties, eye burn, vomiting, nausea, and mental bewilderment (Rafatullah *et al.*, 2010; and Crini, 2006). Adsorption is the accumulation of substance on the surface of solid and liquid. This process is one of the most economic, practicable and effective methods for removing dyes or de-colourization of textile mill effluent. Although, techniques such as coagulation (Liang *et al.*, 2014), membrane filtration (Banat *et al.*, 2005), electro-catalytic method (Li *et al.*, 2012) and adsorption (Xiong *et al.*, 2010; Deng *et al.*, 2011; Ai *et al.*, 2011; Theydan and Ahmed, 2012; and Li *et al.*, 2012) are commonly utilized for the removal of dyes, adsorption has an edge over the other methods due to its simple clean operation design, nontoxic, availability and superior removal of pollutant. Activated carbon (powdered or granular) is mostly use as an adsorbent but because its high cost of production, it makes its use in adsorption process to be restrained. However, in this research, the utilization of microcrystalline cellulose obtained from sugarcane bagasse after treatment with sodium hydroxide for the adsorption of methylene blue in aqueous solution was investigated.

MATERIAL AND METHOD

Materials: The raw material used for the MCC preparation in this study is sugarcane bagasse which was obtained from Ibadan, Oyo State, Nigeria. Chemical and reagents used include Sodium hydroxide (NaOH), Sulphuric acid (H₂SO₄), methylene blue and distilled water. They are standard chemicals of analytical grade.

Extraction of α -cellulose: Cellulose powder was isolated from sugarcane bagasse following the

extraction method used by (Fatoye and Onigbinde, 2020). The agricultural waste materials was prepared, 40g was weighed from it and delignified with about 600ml of 2% w/v sodium hydroxide at 100°C for 30minutes using an oven and the resulting slurry was washed with distilled water and filtered. The residue was treated with 100ml of 17.5% w/v sodium hydroxide at 80°C for 1 hour. The residue (cellulose) was excessive washed with distilled water. The extraction process was then concluded by bleaching with aqueous solution of about 150ml of 3.5% w/v sodium hypochlorite at 80°C for 20minutes and subsequent washing with distilled water until filtrate was clear and residue wash neutralized then the residue was dried in oven at 60°C for 6 hours.

Preparation of microcrystalline cellulose: Approximately 10 g of the samples were hydrolyzed in 100 ml of 50% (w/v) sulfuric acid at 45°C and vigorously stirred for 1hour. Cold distilled water (200 ml) was added to stop the reaction. The sulfuric acid was partially removed from the resulting suspension through centrifugation at 3,000 rpm for 30 min. After centrifugation, the supernatant solution gave the NCC while the residue gave the MCC. The resulting MCC was washed to neutral pH and dried at 60°C in an oven for several hours. Then the MCC dried were pulverized and stored at room temperature in the desiccator (Pedro *et al.*, 2016).

Identification and characterization of MCC: Fourier Transform Infrared (FTIR) Spectroscopy: The FTIR spectra were recorded on an attenuated total reflection Fourier transform infrared (ATR-FTIR) to analyze the chemical changes of the sample MCC (SCB) using a spectrum Bx FTIR spectrophotometer. FTIR spectral analysis was performed within the wave number range of 400-4000 cm⁻¹.

Physicochemical properties of MCC: The following physicochemical analysis was carried out on the raw SCB by standard method (AOAC, 2005; Ekebafe *et al.*, 2012): ash content, moisture content, pH, bulk density, water retention value, colour, form and percentage yield.

Preparation of dye standards (Absorbate): Methylene blue (MB) basic dye was used for the purpose of this study without further purification. The stock dye solution was prepared by dissolving 1 g of MB in 1000 ml distilled water each. The experimental solutions were obtained by diluting the stock dye solution with distilled water to give each appropriate concentration of the experimental solutions. The pH of the experimental solution was adjusted by the addition of either dilute 0.1 M NaOH or 0.1 M HCl solutions.

Adsorption study: Adsorption measurement was determined by batch experiments of known amount of the adsorbent in 25 ml of aqueous dye solution (methylene blue) of known concentration. The mixture was shaken for 30 minutes at room temperature. At the end of the adsorption, the conical flasks were withdrawn from the shaker and filtered. The residual dye concentration in the reaction mixture was analyzed at 665nm using an ultra-visible spectrophotometer model 6305 jenway spectrophotometer. Sorption experiments were performed by varying initial solution's pH (5, 6, 7, 8 and 9), contact time (30, 60, 90, 120 and 150 minutes), adsorbent dose ranging from 0.1 to 0.5g and initial MB concentration. The pH value of suspension was adjusted with either dilute HCl or NaOH solution. The percentage color removal and sorption capacity q_e mg/g was expressed as:

$$\text{Colour Removal, \%} = \frac{C_0 - C_e}{C_0} \times 100 \dots 1$$

$$q_e = \frac{(C_0 - C_e)V}{W} \quad 2$$

Where C_e is the concentration after adsorption, C_0 is initial concentration (mg/l), W is the amount of adsorbent and V is the volume of the solution.

Adsorption isotherms: The equilibrium data were evaluated using adsorption isotherms. Equilibrium isotherm equations are used to describe experimental sorption data. Among several models that have been published in literature, Langmuir and Freundlich are the most frequently used models. The equation parameters and the underlying thermodynamic assumptions of these equilibrium models often provide some insight into both the sorption mechanism and the surface properties and affinity of the sorbent.

Freundlich isotherm: In 1906, Freundlich presented the earliest known sorption isotherm equation. This empirical model can be applied to non-ideal sorption on heterogeneous surfaces as well as multilayer sorption (El-Nafaty *et al.*, 2014; Dawodu and Akpomie, 2014) and is expressed by Equations 3 and 4.

$$q_e = K_f C_e^{1/n} \quad 3$$

Where, K_f is adsorption capacity, C_e equilibrium liquid phase concentration of the solvent, (mg/l), $1/n$ adsorption intensity values for $n > 1$ and q_e is the amount of sorbate per unit mass of bio-sorbent (mg/g). The linearized Freundlich equation is shown in Equation 3.

$$\log q_e = \log a_f + b_f \log C_e \quad 4$$

A plot of $\log q_e$ versus $\log C_e$ is a straight line with slope, b_f and intercept, $\log a_f$.

Langmuir isotherm: Langmuir developed a theoretical equilibrium isotherm relating the amount of gas sorbed on a surface to the pressure of the gas. The Langmuir model is probably the best known and most widely applied sorption isotherm. It has produced good agreement with a wide variety of experimental data and may be represented as in Equation 4. The Langmuir adsorption isotherm assumes that adsorption takes place at specific homogeneous sites within the adsorbent, and it has been used successfully for many monolayer adsorption processes. The linearized Langmuir can be used as expressed in Equation 5.

$$q_e = \frac{q_m b C_e}{1 + b C_e} \quad 5$$

$$C_e = \frac{1}{q_e} + \frac{1}{b q_m} \cdot \frac{C_e}{q_e} \quad 6$$

Where, C_e is the concentration of the sorbate at equilibrium (mg/l), q_e is the amount of sorbate per unit mass of bio-sorbent (mg/g), q_m is a constant representing the strength with which the solute is bound to the substrate (l/mg) and b is the adsorption capacity of the substrate (gram solute/gram adsorbent). Plotting against C_e , a straight line graph with slope and intercept are obtained.

RESULT AND DISCUSSION

Physicochemical properties of microcrystalline cellulose: The intrinsic physical and chemical properties of the microcrystalline cellulose of sugarcane bagasse were evaluated and the results presented in Table 1. The results show that, the pH of the MCC is acidic (3.25). pH determination is crucial because adsorption process is pH dependent. An extreme pH is capable of inducing undesirable physical and chemical effect which may affect the adsorption efficiency of the adsorbate. The ash content (0.03%) showed a minimal presence of inorganic materials and it is less than what was reported by Arif and Ferdausee, (2021) 1.5-5%. The percentage yield of 77.76% is higher than the value reported by liu *et al.*, (2006) 47.8-55.4% and it's comparable with value obtained for microcrystalline cellulose from rice husk (97.55%) and cotton wool (87%) as reported by Arowona *et al.*, (2018).

The values obtained for moisture content (43.6%), bulk density (0.26 mg/l) and water retention capacity

of (662.47 g/g) showed that sugarcane bagasse is a suitable adsorbent for methylene blue removal in aqueous system.

Table 1: Physicochemical properties of microcrystalline cellulose (MCC)

Physicochemical parameter	MCC
Bulk density (mg/l)	0.26
pH	3.25
Water retention (g/g)	662.47
Ash content (%)	0.03
Moisture content (%)	43.6
Colour	White
Form	Powder
Percentage yield (%)	77.76

Characterization of Microcrystalline Cellulose of Sugarcane Bagasse using Fourier Transform Infrared (FTIR): The characterization of the prepared

microcrystalline cellulose using FTIR is shown in the Fig. 1. The FTIR spectra of the sample indicate the absorbance region of the prepared MCC from sugarcane bagasse before the adsorption process. The wavelength of 3429.00 cm^{-1} and 1642.66 cm^{-1} represented the stretching and bending of hydroxyl group respectively in the MCC structure (Shan *et al.*, 2007) which is similar to the FTIR report of (Tan *et al.*, 2016) that uses pure industrial cellulose for the adsorption of methylene blue. A peak was observed in 2917.33 cm^{-1} , 2354.47 cm^{-1} due to the asymmetric stretching vibration of C-H in pyranoid ring, while the stretching of $1436.17 - 1372.71\text{ cm}^{-1}$ represent the C-O-C bond of the cyclic alcohol of cellulose (Kačuráková *et al.*, 2000).

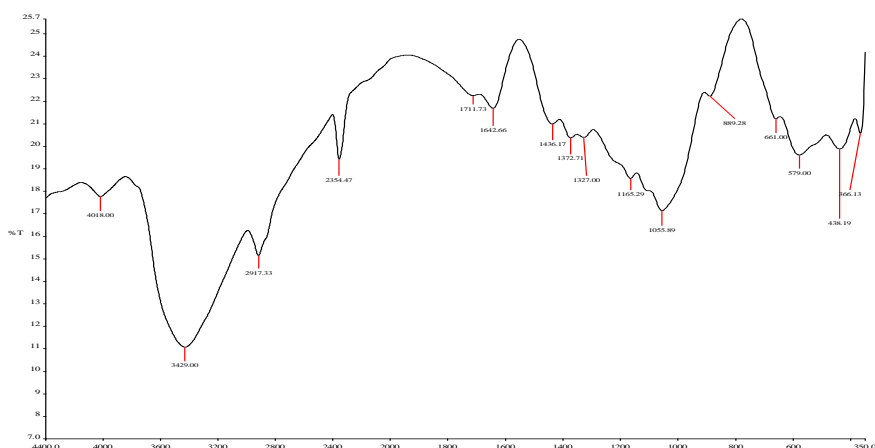


Fig 1: Microcrystalline Cellulose from Sugarcane Bagasse

Effect of amount of adsorbent (dose): The adsorption capacity, adsorption rate and adsorbent cost are very important factors in the selection of the adsorbent to be used in any adsorption experiment. The absorption cost however relates with adsorbent dosage. The ability to adsorb and remove large quantities of adsorbate or any harmful substance with a small amount of adsorbent has many economic advantages (Hee-Jeong Choi and Sung-Whan Yu, 2019). In this study various trails of experiments were conducted using different adsorbent doses from 0.1g to 0.5g. The effect of this variation on adsorption of methylene blue is shown in Fig. 2. The results show that the quantity of active site of the adsorbent increases with increase in dosage. As the dosage increase from 0.1g, the active site also increased leading to an increase in percentage dye removal. But, above 0.4g of SMCC dose, the percentage removal decreased; therefore, 0.4g of SMCC indicates the optimal amount of adsorbent. It implies that the adsorption sites have attained

saturation at 0.4g. Also a similar trend was observed by (Tan *et al.*, 2016) using commercial MCC for adsorption.

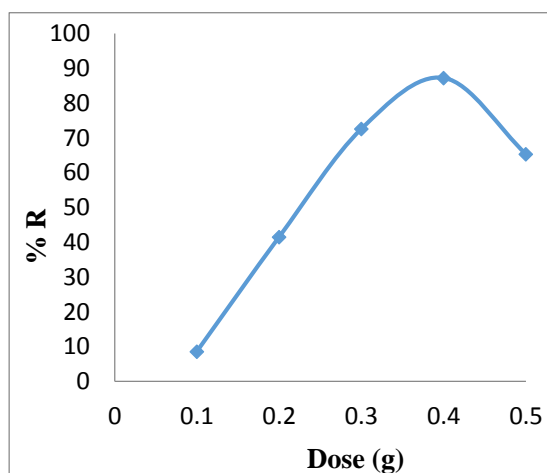


Fig. 2: Effect of Sugarcane bagasse from MCC dose on methylene

Effect of dye-adsorbent contact time: The adsorption experiments were carried out at different time intervals (30, 60, 90, 120 and 180 mins). The result of the effect of contact time on adsorption capacity (Q_e) was graphically captured in fig. 3. Adsorption capacity was seen to increase with increasing contact time. For a fixed amount of dye, the dye removal increased with contact time as there were more adsorption sites available for the methylene blue dye molecules. Therefore, the adsorption sites were able to achieve saturation at higher contact time with higher dye percentage removal. From the results the optimum adsorption for SMCC was achieved at 120 mins contact time.

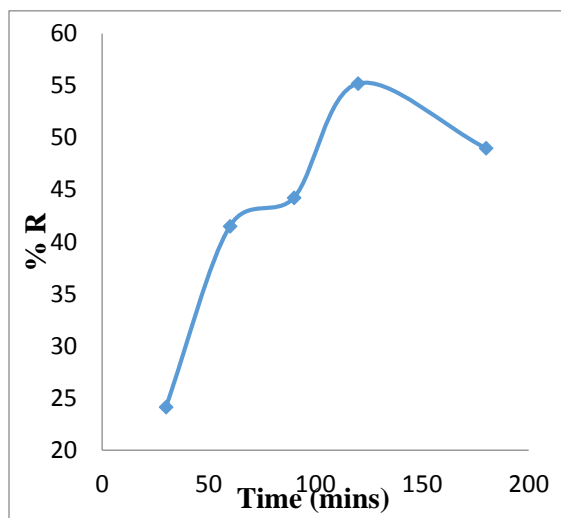


Fig. 3: Effect of time on adsorption on methylene with SMCC

Effect of dye concentration: The methylene blue dye concentration gives an important driving force in overcoming mass transfer resistance between the adsorbent surface and the dye solution. In this study the percentage dye removal of the microcrystalline adsorbent at different dye concentration was investigated. The effect of concentration on adsorption is shown in fig. 4. SMCC at 100mg/l has the highest effective dye removal. As the initial concentration increases, the percentage dye removal decreases and this trend conform to the work (Tan *et al.*, 2016).

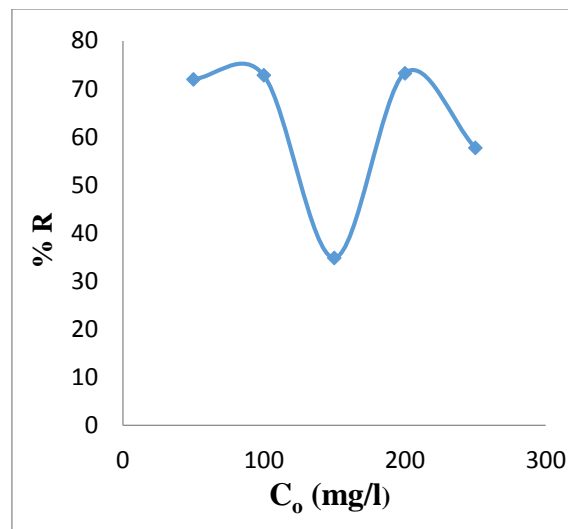


Fig. 4: Effect of concentration on adsorption of methylene blue with SMCC

Effect of pH: Alkalinity, pH, stirring condition and the presence of static materials are major influencing factors in the adsorption and removal of inorganic materials and oil in aqueous solution. More importantly, pH and temperature of aqueous solution can greatly influence the removal of dye (Hee-Jeong Choi and Sung-Wan Yu, 2019). The effect of pH on dye adsorption was investigated at different controlled pH from 5 to 9. The results showed an increase in percentage dye removal as the pH increases. In fig. 5, the highest percentage dye removal for SMCC is shown to be achieved at pH 9. The optimal percentage dye removal was achieved at pH 9. At lower pH, functional oxidized groups of the adsorbents were promoted and thus active site of adsorbents for binding of dye becomes less available. It was also proposed that adsorption mechanism of methylene on MCC involved electrostatic attraction which takes into consideration the net zeta potential values of MCC (Tan *et al.*, 2016). The result shown in MCC fairly follows the trend on previous study by (Tan *et al.*, 2016). The result also compares well with the work of (Zakariyya and Saifullahi, 2018), which made use of microcrystalline cellulose from groundnut shell with optimum pH of 8.

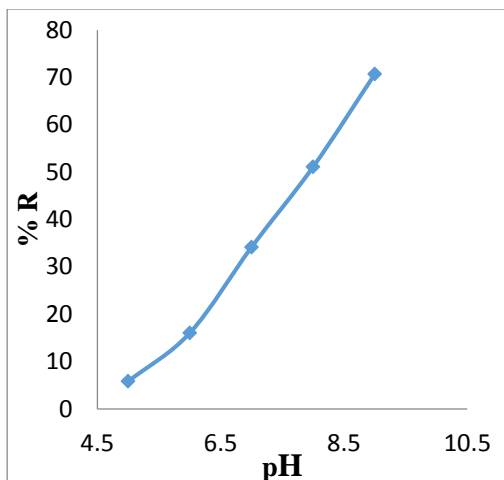


Fig. 5: Effect of pH on adsorption of methylene blue with SMCC

Adsorption isotherm: Adsorption isotherm model was used to investigate the interaction between adsorbate and adsorbent, maximum adsorption capacity of the adsorbent and dynamic equilibrium of adsorption

system. From literatures, it has been established that the best fitted adsorption isotherm model can provide information about the nature of the adsorption system (Hee-Jeong Choi and Sung-Whan Yu, 2019). In this study, two models were used; The Langmuir and Freundlich isotherm models. The Langmuir isotherm represents the equilibrium distribution of methylene dye between the solid and the liquid phase. Langmuir describes the adsorption in homogenous phases while Freundlich described adsorption in heterogenous surface. From the linearized form of their equations, their isotherm constants can be graphically deduced. For this study, a comparison of the coefficient of regression (R^2) for both isotherms is shown in Table 2. The value of the Langmuir constants q_m , K and the Freundlich constant K_f and n calculated are also shown. According to the value of R^2 , Methylene blue dye adsorption best fit into Freundlich isotherm for SMCC than Lagmuir. This indicates that the adsorption process is more multilayer in nature and methylene molecules have the tendency of interacting with each other at neighboring adsorption sites.

Table 2: Langmuir and Freundlich isotherm constants obtained

Adsorbents	Langmuir		Freundlich			
	q_m	K	R^2	n	K_f	R^2
SMCC	27.78	13.67	0.44	-9.90	12.13	0.98

Conclusion: This research work has established the possibility of preparing microcrystalline cellulose (MCC) from sugarcane bagasse for utilization as bio-adsorbent in dye adsorption. From this work, it is clear that the adsorption of methylene blue dye onto the adsorbent (SMCC) is influenced by adsorbent dose, dye concentration, contact time and pH values. The experiment data of this work, fitted well into Freundlich isotherm.

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