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Adsorption of Reactive Dyes from Textile Wastewater Using Corn Stalk Activated Carbon

Robel Legese Meko*

Department of Textile and Apparel Fashion Technology, Federal TVET Institute, Addis Ababa, Ethiopia

Abstract

Extensive use of synthetic dyes in textile industry has created a major pollution problem. Among various treatments, adsorption has been considered as a better process due to its effectiveness of removing color from wastewater. In this present work, the efficiency of activated carbon prepared from corn stalk for removal of reactive dye from textile wastewater was studied. Corn stalk was chemically activated with KOH, followed by carbonizing in a muffle furnace. The carbonized corn stalk was characterized by SEM and FTIR spectroscopy. Adsorption of three reactive dyes were carried out by preparing dye samples in laboratory and taking dye wastewater from BDTSC. Adsorption was carried out under the control of three different factors namely contact time, adsorbent dosage and pH. Optimum time, pH and adsorbent dosage for adsorption process were found to be 60 minutes, 3.8 pH and 4 g/L respectively. Using those optimum operating parameters, the adsorption capacity of prepared activated carbon for Reactive yellow-145, Reactive red-2, Reactive blue-19 and wastewater taken from BDTSC was 96.9%, 95.5%, 97.1% and 88% respectively. Langmuir and Freundlich adsorption isotherm models were used to simulate the equilibrium data for the adsorption process. The result indicates that the adsorption process best fits with Freundlich isotherm. The produce activated carbon was also shown a reduction of BOD, COD, TDS, TSS and turbidity.

Keywords: Activated carbon • Corn stalks • Reactive dye • Adsorption • Textile waste Water • Adsorption isotherm

Introduction

Contaminants such as color, heavy metals, cyanides, toxic organics, nitrogen, phosphorous, phenols and suspended solids from the industries and untreated sewage sludge from the domestic processes have become a great concern to the environment and public health. Color is the first sign of contamination recognized in wastewater, since dye concentrations in watercourse higher than 1mg/L caused by the direct discharges of textile effluent is highly visible and affect the aesthetic merit, water transparency and gas solubility in lakes, rivers and other water bodies [1]. Therefore, textile industry is one of the industries that produce a high volume of waste water and cause water pollution. Dyes in wastewater from textile and dyestuff industries are difficult to remove. This is because dyes are usually synthetic and have complex aromatic structures which make them more stable consequently they are difficult to biodegrade [2].

Over 50% of cotton products are colored with reactive dyes which offer good proportion of the total market ranges from 20% to 30%. The reason behind this popularity of reactive dyes for dyeing of cotton fiber is that its molecules, containing one or several reactive groups, chemically react with the fiber polymers to form a stable chemical linkage (covalent bond) between the dye molecules and fiber polymer [3]. Wastewaters from textiles do not only deface the look of natural waters, but are also highly toxic. Some dyes are reported to harm mammalian cells by causing kidney tumors and reproductive difficulties. These dyes are also potentially carcinogenic in many mammalian species [4].

Accumulation of dyes in wastewater from industries such as textiles, paper, cosmetics, rubber, and plastics has been regarded as a significant source of water pollution. Reactive dyes, an anionic in water, are most commonly used due to their provision of bright colors, excellent color fastness, and easy application [5]. However, many reactive dyes are toxic to organisms and may cause direct harm to aquatic life. As the dyes are structurally complex, are of synthetic origin, and have high water solubility, their removal from effluent by the use of conventional physico-chemical and biological processes is difficult [6]. However, it has been reported that the adsorption technique provides a potential for the removal of dyes from aqueous solutions [7]. It has been estimated that the total dye consumption in textile industry worldwide is more than 10,000 tons per year and about 10-15% of these dyes are released as effluents during the dyeing processes [8]. These effluents can cause potential pollutants to human beings and to aquatic life. Various Physical, Chemical and Biological separation technologies are used in the removal of this effluent [9]. All of these methods have their own advantages and disadvantages. However, adsorption process is considered to be a very effective physical separation technique in wastewater treatment in terms of simplicity of design, ease of operation and insensitivity to toxic substances provided adsorbents are locally available with little or no value [10].

The full introduction of adsorption technology in to the practice of deep cleaning of dye wastewater is inhibited by the high cost of activated carbons and by problems with their regeneration. In recent years, attention of chemists has been focused on low-cost adsorbents (bio sorbents) from agricultural waste. The agricultural countries have abundant source of straw, stalks, hulls, leaves etc. Plant biomass is a natural renewable source that can be converted into useful material and energy. Disposal of residue from agriculture is currently a major economic and ecological issue. However, the abundance and availability of corn stalk as agricultural by-product make them good sources of raw materials for a lot of uses, and converting it to adsorbents such as activated carbon represents a possible outlet [11].

In this present work, use of activated carbon from corn stalk for adsorption of reactive dyes from textile, wastewater has been investigated. The main aim this research work is to study the efficiency of carbonized corn stalks for the removal of reactive dyes from textile waste water; to produce activated carbon from corn stalks and to study the effect of the operating parameters, such as absorbent dose, pH and mixing time on the treatment performance of absorption process.

***Address for Correspondence:** *Robel Legese Meko, Lecturer, Faculty of Textile and Apparel Fashion Technology, Federal TVET Institute, Addis Ababa, Ethiopia, E-mail: robellegese1@gmail.com*

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Materials and Methods

Equipment and materials

The equipment and tools used to achieve the objective of this present work were Oven drier, Nabertherm muffle furnace, desiccator, measuring cylinder, beakers, magnetic stirrer, weighing balance, pipette, filter paper, pH meter, DO meter, COD digester, Turbidity meter, TDS meter, Perkin Elmer UV-visible spectroscopy lambda 25, Perkin Elmer FTIR spectroscopy, Scanning Electron Microscope (SEM), BOD incubator and corn stalk.

Chemicals and reagents

Reactive dyes (Reactive yellow 145, Reactive red 2, Reactive blue 19), hydrochloric acid, potassium dichromate, silver sulphate, ferrous ammonium sulphate, sulphuric acid, potassium hydroxide and sodium hydroxide, mono potassium phosphate, sodium hydrogen phosphate, sodium bicarbonate, acetic acid and sodium acetate were used.

Experimental design

Design expert 11 software was used for designing of the experiment conducted for carbonization and adsorption process. Central composite design was selected for designing and optimizing operating parameters.

Experimental procedure

Corn stalk has been collected from local agricultural field found around Bahir Dar city in Zenzelima farm land. The leaves were manually removed from the stalk.The collected corn stalk was chopped into small pieces, washed with tap water to remove dirt and suspended impurities. The washed stalk was sun dried for three days [12]. The stalk was soaked in 3mol/L potassium hydroxide for 8h, followed by neutralizing in 2mol/L HCL. KOH was used as activating agent to reduce the formation of tar, and help to generate pores by partial oxidation [13]. After soaking, the stalk was washed and sun dried for three days. Then, the sun dried stalk was further dried in an oven drier at 1100C for two hours. The dried stalk was carbonized in Nabertherm muffle.

Surface response methodology was used for designing of experiment conducted for the carbonization process. Two factors (temperature and time) were considered for the experimental design. The temperature and time range used for carbonization process were 4000C- 6000C and was 60-120 minutes respectively. Using central composite design, 13 experimental runs were carried out. At the end of each carbonization experiment, the samples were withdrawn from the Nabertherm muffle furnace and put in Dissector for cooling purposes. The produced activated carbon was then crushed to small particles using Jaw crusher (BB50). Then, the samples were sieved to identify its particle size. Sieve of pores size 0.075mm -0.5mm were used, and the smallest particle size was used for adsorption purposes. The sieved activated carbon at particle size of 0.075 mm was stored in plastic bottle for further adsorption process.

Determination of percentage yield

Once, activated carbon was prepared at different time and temperature as per experimental design, its percentage yield was determined. The yield of produced activated carbon was calculated using formula 2.1.

Yield of activated carbon (%)=(Mi-Mf)/Mi*100 Where Mi- Original weight of corn stalks; Mf- Final weight after carbonization (Figure 1).

Preparation of wastewater sample

The color removal capacity of the produced activated carbon was determined by measuring absorbance values of samples using UV visible spectroscopy lambda 25. The absorbance of treated and untreated waste water was measured, and the adsorption capacity was evaluated using formula (2.2).

Dye removal(%)= ((Ao-A))/Ao*100………(2.2)

Where A0: is the absorbance value before treatment, and A: value after treatment process.

Effluent's parameters such Biological oxygen demands (BOD), Chemical Oxygen Demand (COD), pH, Turbidity, Total dissolved solid (TDS) and Total suspended solid were evaluated before and after adsorption process (Figure 2).

Conducting adsorption

Batch adsorption experiments were conducted to study the influence of parameters such as; contact time (min), adsorbent dose (g/L) and pH on the removal of color from textile wastewaters. Three factors; contact time, pH and adsorbent dosage were considered for conducting the experiment. The contact time, pH and adsorbent dosage used for adsorption process were 30-60 min, 3-11 and 2-4 g/L respectively. Surface response methodology was used for analyzing and optimizing the operating parameters; each experiment was conducted with three replicas. Totally, 45 experimental runs were conducted for adsorption process.

Results and Discussion

Physico-chemical characteristics of produced activated carbon

The physic-chemical properties of activated carbon prepared from corn stalk are shown in the moisture content of raw corn stalks was found to be higher than the activated product. The value of moisture content of raw corn stalks was 9.01% (Table 1).

Yield of activated carbon

Using formula (2.1), 28-40% yields were obtained by varying carbonization time and temperature as per experimental design. The optimized percentage yield was found to be 34.4% at 90 min and 500 oC respectively. The optimum temperature and time were selected depending on percentage yield and dye adsorption capacity (Table 2).

Effect of carbonization temperature and time on per - centage yield

From the experiment, it was found that as carbonization temperature increases, the percentage yield of the activated carbon decreases. This is because a large amount of a volatile material release occurred at a higher temperature.

Carbonization time has also the same effect as the temperature on percentage yield of the produced activated carbon. The percentage yield of the activated carbon slightly decreases as carbonization time increases. The effect of both time and temperature on the percentage yield of produced activated carbon can also be indicated by a 3D graph in (Figure 3.1).

Surface characterization

The spectrum had shown the peaks typical for hydroxyl groups at the range 3390 cm-1 to 3415 cm-1 confirming the presence of the free hydroxyl groups of carboxylic acids, alcohols and phenols on the surface of raw corn stalk. For activated carbon prepared from corn stalks, the intensity of peaks observed at 3390 cm-1 to 3415 cm-1 were reduced and low stretching is observed, showing that the stretching intensity at that point is slightly reduced because of carbonizing temperature. Strong signal was obtained at 1000 cm-1 to 1058 cm-1 indicates the C-O linkage in the raw and carbonized samples. Moreover, the presences of functional groups of the samples were presented in (Figure 3.2).

Scanning electron microscope analysis

Scanning Electron Microscope is widely used to study the morphology features and surface characteristics of the materials. The image of activated carbon prepared from corn stalks under scanning electron microscope is illustrated Figure. Shows a sample with a different degree of magnification. For instance, (a) shows image with x250 magnification, (b) with x500 magnification, (c) with x1000 magnification and (d) with x2000 magnification. The sample has many folds and irregular structure. This irregular structure and folds are produced by chemical activation using KOH and physical activation using a temperature at 500 0C in Nabertherm muffle furnace.

Characteristics of wastewater

The pH of the effluent was found between pH 8.4 -11.6 for all dye prepared in laboratory and effluent was taken from Bahir Dar textile share company

Table 1. Definition, categories of main independent variables .

Variables	Defination
Bio-demographic	
Mother's age	Age in years (15-24, 25–34, 35–49)
Mother's age when first pregnant	Age when she first conceived a child, in years (15-24, 25–34, 35–49)
Mother's marital status:	Coded as married, and non-married (including divorced, single, widowed, and separated)
Sex of the child	Girl, boy
Birth order	The birth order number of the child in the family (1^{st} - 2^{nd}, $\geq 3^{rd}$)
Socio-economic variables	
Mother's education	Formal level of education/schooling (no education, primary, high school, tertiary (includes university, diploma, certificate), monastic/non-formal education)
Mother's working status	Not working, agricultural jobs and self-employed, professional, technical and sales jobs, and others including manual labor and unskilled jobs)
Wealth index	Economic/wealth status of the household categorized as poor, middle, rich.
Place of residence	urban and rural areas
Environmental variables	
Improved source of drinking water	Piped into dwelling, piped into yard/plot, public tap/standpipe, tube/piped wells or boreholes, protected wells, protected spring, and rainwater. Other sources were classified as non-improved.
Improved toilet facility	Includes flush toilet connected to sewage or septic tank and ventilated/improved Latrine or toilet. Other types of toilet, or no toilet, are considered non-improved.
Health-related variables	
Maternal TT immunization	Tetanus toxoid injection status of mothers who were or became pregnant in the past year preceding the survey (not received, received on dose, received two or more dose)
Contraceptive use	Use of contraceptives (ever used, never used)
Place of delivery	For the birth two years preceding the survey included institutions (i.e. health centers), and non-institutional (i.e. home, others)
Skilled delivery	Delivery was attended by skilled persons for the child born two years preceding the survey (Yes, No)
Antenatal care (ANC)	Any antenatal care by the mother for the last child born two years preceding the survey ((At least 4 visits vs. Less than 4 visits)

Figure 1. Corn Stalk.

Figure 2. Corn Stalk Activated Carbon.

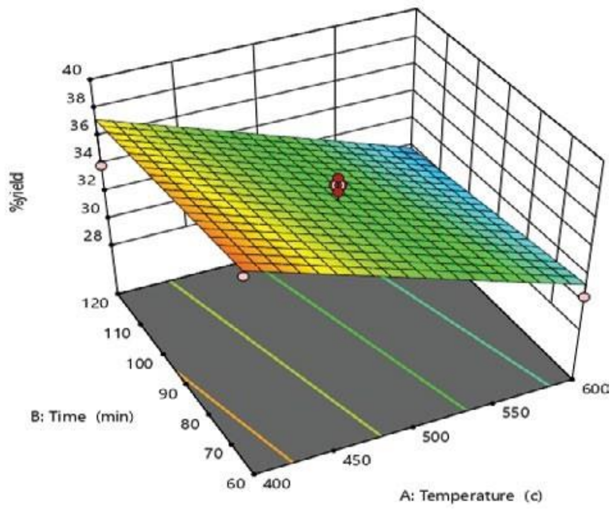


Figure 3.1. Effects of Temperature and Time on Percentage Yield of Activated Carbon.

(BDTSC). Shows the characteristics of raw wastewater prepared in laboratory and sample taken from BDTSC. F. Effect of Operating Parameters on Adsorption Process (Table 2).

Effect of ph and contact time on dye adsorption

The result indicates that variation of pH affects the adsorption capacity of the prepared activated carbon. It was indicated that the prepared activated carbon has higher adsorption capacity in an acidic media. At low pH there is an increase in the H+ ions in solution. The dye molecule is negatively charged; this result in electrostatic interaction between the dye molecule and the adsorbent resulting in higher percentage dye removal Similar result was obtained by [14].

The efficiency of adsorption is dependent on the solution pH, since variation in pH leads to the variation in the degree of ionization of the adsorptive molecule and the surface properties of adsorbent. Solution pH determines the surface charge of the adsorbent and the degree of ionization of the adsorbate, which affects the adsorption of dyes on activated carbon (Table 3).

The result indicates that adsorption capacity of the prepared activated

Table 2. Household and children's characteristics of the final study samples, CDHS 2014

Variables	(N=5,958a) Freq.	%
Place of residence		
Urban	874	14.67
Rural	5084	85.33
Wealth index		
Poor	2565	43.04
Middle	1132	19
Rich	2262	37.96
Source of drinking water in wet season		
Improved	3026	50.79
Unimproved	2932	49.21
Sanitation facilities		
Improved, not shared	3001	50.37
Non-improved or shared	2957	49.63
Child birth weight (grams)		
Mean birth weight (SD)	3,658 (1928)	
> 2500	5217	87.55
< 2500	742	12.45
Sex of the child		
Boy	2993	50.22
Girl	2966	49.78
Birth order		
1-2	3945	66.21
> 3	2013	33.79
Birth interval		
< 2 children	2585	43.37
3 children	850	14.26
≥ 4 children	2524	42.36
Child is twin		
Single birth	5895	98.93
Multiple birth	64	1.07

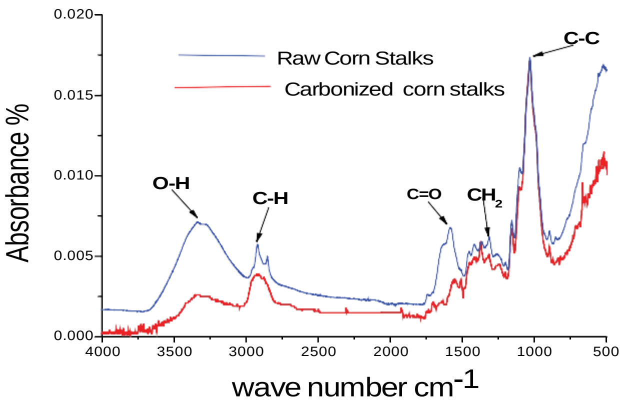


Figure 3.2. FTIR Graphs of Raw and Carbonized Corn Stalks.

carbon was increased with increasing contact time. As time increases from 30 to 60 min the adsorption capacity increases since it reaches saturation point at this point.

Effect of adsorbent dosage on adsorption capacity

Adsorbent dosage has positive effect on adsorption capacity of the adsorbent. The obtained result indicates that percentage of dye removal increases with increasing adsorbent dosage. This is because; the quantity

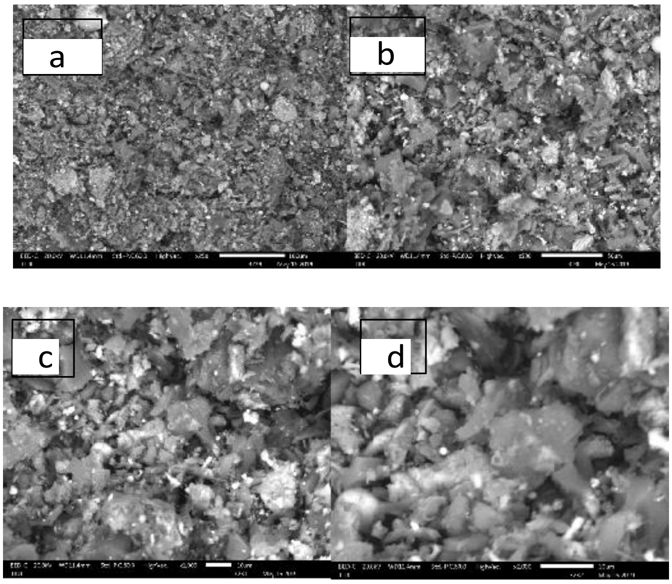


Figure 3.3. Scanning Electron Microscope Image of Corn Stalks Activated Carbon at Different Magnification.

Table 3. Mother's and health related characteristics of the final study samples, CDHS 2014

Variables	(N = 5,958a) Died (n= 108) n (%)	P-value	OR	95% CI
Place of residence				
Urban	4 (0.50)	< 0.001	1	-
Rural	104 (2.04)		4.17	2.15-8.08
Wealth index				
Poor	69 (2.69)	0.005	1	-
Middle	15 (1.31)		0.48	0.21-1.07
Rich	24 (1.08)		0.39	0.22 - 0.71
Source of drinking water				
Unimproved	55 (1.86)	0.82	1	-
Improved	54 (1.77)		0.95	0.60 - 1.50
Sanitation facilities				
Unimproved	73 (2.46)	0.007	1	-
Improved	35 (1.18)		0.47	0.27 - 0.82
Child birth weight (grams)				
> 2500	67 (1.29)	< 0.001	1	-
< 2500	41 (5.49)		4.43	2.58 - 7.63
Sex of the child				
Boy	66 (2.22)	0.084	1	-
Girl	42 (1.41)		0.63	0.37 - 1.07
Birth order				
01-Feb	49 (1.25)	< 0.001	1	-
> 3	59 (2.92)		2.37	1.44 - 3.90
Birth interval				
< 2 children	40 (1.54)	0.507	1	-
3 children	18 (2.10)		1.38	0.68 - 2.76
> 3 children	50 (2.01)		1.31	0.75 - 2.27

of sorption sites at the surface of adsorbent will increase by increasing the amount of the adsorbent.Moreover, the result is shown in (Table 4). effect of adsorbent dosage gives an idea for the ability of a dye adsorption to be adsorbed with the smallest amount of adsorbent [6]. At constant initial dye concentration, increasing the adsorbent dosage provide greater surface area and large number of sorption site which results in enhancement of dye adsorption.

Effect of initial dye concentration on adsorption

The results indicate that the absorbance values of the samples decreases, i.e. adsorption capacity increases as initial dye concentration decrease. The adsorption capacity of the adsorbent at different initial dye concentration is illustrated in (Figure 3.4 - 3.7).

The adsorption capacity of the adsorbent decreases with increasing concentration of the dyes, because of increased ratio of initial adsorption number of moles of dye to available surface area of the adsorbent.

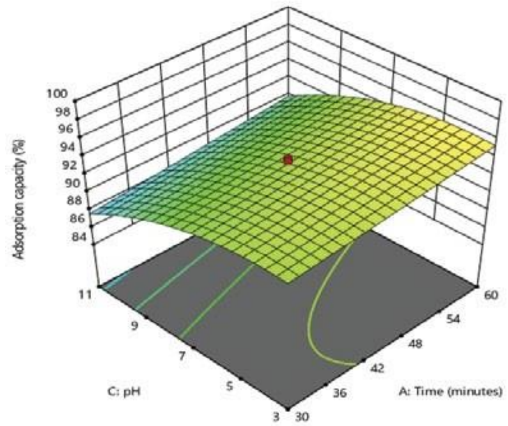


Figure 3.4. Effect of pH and Contact Time on Adsorption Capacity.

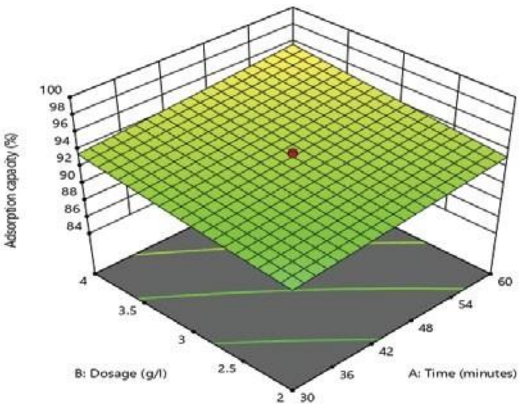
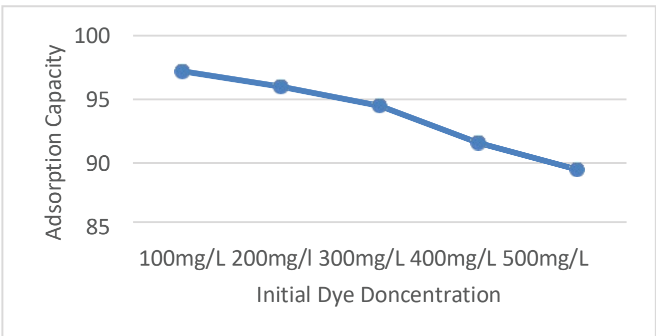


Figure 3.5 Effect of Adsorbent Dosage on Adsorption Capacity.



Figuer 3.6. Adsorption Capacity of Reactive Yellow-145 at Different Concentration.

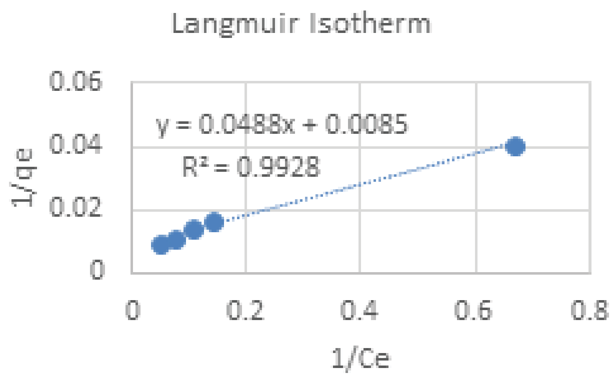


Figure 3.7. Langmuir Adsorption Isotherms.

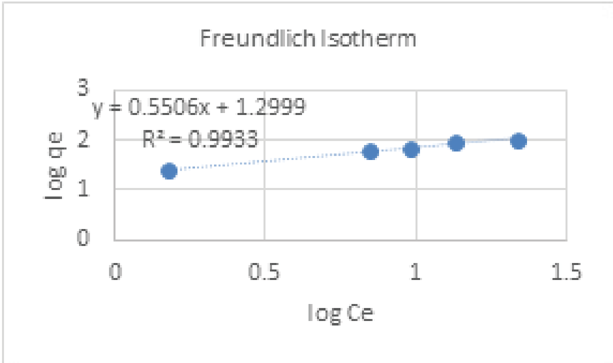


Figure 3.8. Freundlich Adsorption Isotherms.

Table 4. Univariate logistic analysis: Association between household and children's characteristics and U5M.

Variables	(N = 5,958a) Died (n= 108) n (%)	P-value	OR	95% CI
Place of residence				
Urban	4 (0.50)	< 0.001	1	-
Rural	104 (2.04)		4.17	2.15-8.08
Wealth index				
Poor	69 (2.69)	0.005	1	-
Middle	15 (1.31)		0.48	0.21-1.07
Rich	24 (1.08)		0.39	0.22 - 0.71
Source of drinking water				
Unimproved	55 (1.86)	0.82	1	-
Improved	54 (1.77)		0.95	0.60 - 1.50
Sanitation facilities				
Unimproved	73 (2.46)	0.007	1	-
Improved	35 (1.18)		0.47	0.27 - 0.82
Child birth weight (grams)				
> 2500	67 (1.29)	< 0.001	1	-
< 2500	41 (5.49)		4.43	2.58 - 7.63
Sex of the child				
Boy	66 (2.22)	0.084	1	-
Girl	42 (1.41)		0.63	0.37 - 1.07
Birth order				
01-Feb	49 (1.25)	< 0.001	1	-
> 3	59 (2.92)		2.37	1.44 - 3.90
Birth interval				
< 2 children	40 (1.54)	0.507	1	-
3 children	18 (2.10)		1.38	0.68 - 2.76
> 3 children	50 (2.01)		1.31	0.75 - 2.27

Adsorption isotherm model

The two well-known models of Freundlich and Langmuir isotherm were evaluated to know the isotherm models for the adsorption process using the produced activated carbon. The graph of 1/qe versus 1/Ce is the linear form of Langmuir adsorption isotherm (Table 5, 6).

The essential features of the Langmuir isotherm can be expressed in terms of a dimensionless constant called separation factor R_L, also called equilibrium parameter, which is defined by equation (3.1). The value of R_L indicates the favorability of the adsorption process (Table 3.4).

Table 5. Univariate logistic analysis: Association between mother's characteristics and health related factors with U5M

Variables	(N = 5,958a) Death (n=108) n (%)	P-value	OR	95% CI
Mother's age				
15-24	27 (1.65)		1	-
25-34	45 (1.34)	< 0.001	0.81	0.46 - 1.43
35-49	37 (3.76)		2.33	1.23 - 4.44
Age at the time of giving birth				
15-24	32 (1.30)		1	-
25-34	44 (1.50)	< 0.001	1.15	0.67 - 1.99
35-49	33 (5.42)		4.34	2.31 - 8.14
Mother's education				
No education	19 (2.35)		1	-
Primary	54 (1.75)	0.66	0.74	0.36 - 1.53
Secondary and higher	35 (1.71)		0.72	0.33 - 1.58
Mother's working status				
Professional, technical and sales jobs	20 (1.44)		1	-
No job/not working	16 (1.15)		0.8	0.37 - 1.70
Agricultural jobs and self-employed	49 (2.37)	0.128	1.67	0.81 - 3.45
Others (manual labor and unskilled jobs)	23 (2.12)		1.49	0.65 - 3.38
Marital status of mother				
Married	100 (1.77)	0.27	1	-
Not married@	8 (2.71)		1.55	0.71 - 3.39
Maternal TT immunization				
Not received	31 (3.12)		1	-
Received one dose	16 (1.35)	0.019	0.43	0.23 - 0.81
Received two or more does	62 (1.63)		0.51	0.30 - 0.90
Contraceptive use				
No	77 (3.28)		1	-
Yes	31 (0.87)	< 0.001	0.26	0.16 - 0.43
Place of delivery				
Institutional*	86 (1.71)	0.224	1	-
Non-institutional (home, other)	22 (2.41)		1.42	0.80 - 2.50
Skilled delivery				
Unskilled	17 (3.07)		1	-
Skilled	91 (1.68)	0.036	0.54	0.30 - 0.97
Number of ANC visits				
< 4	48 (3.35)		1	-
> 4	60 (1.32)	< 0.001	0.39	0.23 - 0.65
Health insurance coverage				
No	74 (1.52)		1	-
Yes	34 (3.21)	0.009	2.15	1.20 - 3.86
Perceived problems in accessing health service				
No barrier	24 (1.62)		1	-
≥ 1 barrier (distance/money/waiting time)	84 (1.88)	0.579	1.16	0.68 - 1.98
Smokes cigarettes				
No	101 (1.74)		1	-
Yes	7 (4.71)	0.044	2.79	1.00 - 7.90

Table 6. Associated factors with U5M in the multivariate logistic regressions

Variables	(N = 5,958a) Died (n= 108) n (%)	P-value	OR	95% CI
Place of residence				
Urban	4 (0.50)	< 0.001	1	-
Rural	104 (2.04)		4.17	2.15 - 8.08
Wealth index				
Poor	69 (2.69)	0.005	1	-
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> 3 children	50 (2.01)		1.31	0.75 - 2.27

$R_L = 1/(1+K_L C_o$

Where, K_L is the Langmuir constant related to the energy of adsorption (L/mg) and Co is the highest initial dye concentration (mg/L).

The values of qm and KL have been computed from the intercept and slope of the Langmuir plot of 1/qe verses 1/ce respectively. The favorable adsorption of Freundlich model can expressed in terms adsorption intensity n. the nature of adsorption for the model.

As it could be seen from the adsorption intensity of the Freundlich model was 1.32 which is greater than 1/n. It indicates that the nature of the adsorption intensity was cooperative adsorption (Table 3.7, 3.8).

Conclusion

Corn stalks activated carbon can be used as a very efficient and cheap adsorbent for the removal of reactive dyes and other several pollutants from textile wastewater. Carbonization temperature and time are important parameters in corn stalks activated carbon preparation. Parameters such as contact time, pH and adsorbent dosage should be controlled for effective adsorption processes.

This present research work establishes that corn stalk activated carbon was excellent low cost adsorbent for removal of reactive dyes from textile wastewater.

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