

ORIGINAL ARTICLE

Adsorption of C. I. ACID RED 2 from their aqueous solutions onto raw, activated and blended bagasse ash based biosorbents

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ABSTRACT

The synthesis and characterizations of activated and blended biosorbents for C. I. Acid Red 2 adsorption have been investigated. The biosorbents were characterized by using modern technique (FTIR, SEM, XRD, BET, BJH). The biosorbents used in this research work are raw bagasse ash (RBA), activated bagasse ash (ABA) and blended bagasse ash (BBA). Effects of operating parameters such as initial dye concentration, amount of adsorbent dose, pH, contact time and temperature on the decolorization of dye (C. I. Acid Red 2) have also been examined. The equilibrium data were analyzed using Langmuir and Freundlich modern isotherms. The BBA shows maximum removal (96%) of dye at pH 4.4. The maximum adsorption capacity was found in the case of BBA is 0.2 mg/g. In all biosorbents the blended bagasse ash was found to be very effective adsorbent for C.I. Acid Red 2 adsorption. Removal of C.I. Acid Red 2 dye in aqueous solutions is important as they cause human harmful effects besides being environmental damage.

Keywords: Adsorption, C.I. Acid Red 2, blended, activated biosorbents.

Received 11.02.2020

Revised 24.04.2020

Accepted 03.06.2020

How to cite this article:

R Sharma, S P. Satsangee and S K Verma. Adsorption of C. I. ACID RED 2 from their aqueous solutions onto raw, activated and blended bagasse ash based biosorbents. Adv. Biores., Vol 11 (3) May 2020: 91-103

INTRODUCTION

Different types of dyes have been used extensively in food industries as food colorant, textile, paper industries, leather and cosmetic industries. The rapid development of industries, population explosion and discharge of pollutants from various industries results in environmental pollution. Among various types of environmental pollution, water pollution is the major distress to the community, because of the existence of dye stuff in the wastewater which is released from the dyeing industries [1]. Almost all the industrial sectors are responsible for the environmental pollution [2]. Among them, textile industry plays a vital role in the release of effluents with a huge amount of additives, surfactants, detergents, suspended solids, carcinogenic amines and dyes that poses environmental threats to aquatic bodies [3]. Without any proper treatment, the bleached wastewaters from textile industries are directly released into water bodies. It makes a severe problem to aquatic life, reduces photosynthetic activity and also has an adverse effect on human health and causes toxicity turbidity problems [4]. Hence most of the dyes are toxic, mutagenic and carcinogenic in nature [5]. Elimination of colour from textile wastewater is one of the major environmental problems and its removal seems to be difficult by treating it with conventional method because the dyes are stable and to heat and oxidizing agents [6].

The physicochemical treatment methods such as adsorption, sedimentation, flocculation, floatation, coagulation, osmosis, neutralization, reduction, oxidation, electrolysis, and ion-exchange are employed for dye removal. But, these methods are expensive and in addition, most of them produce the large quantity of sludge which needs further disposal. Therefore, economically and eco-friendly techniques are needed for the degradation of dye from textile effluent [7-10].

Recently, a number of low costs, easily available and effective adsorbents were used for the removal of different dyes from aqueous solution. Many studies have been focused on the use of non-conventional low-cost adsorbents such as sugarcane bagasse, coconut husk, groundnut shell, banana pith, clay, rice husk, maize cob, coir pith, orange peel and wheat straw dust in wastewater treatment. Instead of

disposing of the sugarcane bagasse as a waste material, it has been used as an adsorbent for the removal of dyes from the wastewater. It mainly consists of cellulose (45%), hemicellulose (28%) and lignin (18%) and also it has carboxylic and hydroxyl groups, which are responsible for the uptake of dyes from wastewater [1, 13]. There are numerous numbers of low cost, commercially available adsorbents which had been used for the dye removal. However, as the adsorption capacities of the above adsorbents are not very large, hence the new adsorbents which are more economical, easily available and highly effective are still needed. Rawbagasse ash, activated bagasse ash and blended bagasse ash based biosorbents are low cost biosorbents that being used in this study to investigate the removal of C.I. Acid Red 2 from wastewater. The aim of the present study is to determine the optimum conditions for the removal of C.I. ACID RED 2 by rawbagasse ash, activated bagasse ash and blended bagasse ash based biosorbents.

MATERIAL AND METHODS

Preparation of adsorbents

Preparation of RBA

The bagasse ash was collected from local sugar mill (The Kisan Sahkari Chini Mills Ltd. Mahmudabad, Uttar Pradesh). The collected bagasse ash was washed with distilled water for several times to remove all the dirt particles. The washed material is dried in oven at 110°C for 24 hours. The dried biomass were grounded to a fine powder by using mixer and sieved through 150 micron sieves. 25 grams of sieved particles were repeatedly washed with hot distilled water. The sample was dried and stored.

Preparation of ABA

The raw material (RBA) was dried overnight in an air oven. To prepare the activated bagasse ash refuse was treated with the solution of 20 % of H_2O_2 . The mixture was stirred with the help of magnetic stirrer at 60 °C for 2 hours and dried in an air oven at 100 °C for 2 hours. The heat treated sample was washed several times with doubled distilled water until the filtrate is neutral ($pH \approx 6.5-7.5$) and its conductivity is minimal. These tests ensure that H_2O_2 and other ions which might be present are completely removed. The sample was dried, ground and sieved to uniform size of 75 micron.

Preparation of BBA

This is one of the attractive ways of physical modification is to provide new desirable characters to chitosan and to synthesize chitosan and bagasse ash based biocomposite by mixing or blending of chitosan and bagasse ash in the weight ratio of 1:1. In blending, at least two materials are mixed to obtain a new material with different physical properties. At thermodynamic equilibrium, the two materials of amorphous nature appear to be as a single phase or homogeneous on blending with a new set of improved properties from the individual components. The miscibility and compatibility between the blended materials are decided by their mechanical and thermal properties. The method of blending is effective in practical application due to its simplicity in operation and availability of various organic compounds and natural polymers. Blending permits the wide range of properties by union of both the components viz. chitosan and bagasse ash to achieve physically and chemically stable biopolymers required for the specific applications.

Adsorbate (C.I. Acid Red 2)

C.I. Acid Red 2 or Methyl red is an azo dye (IUPAC Name-(2-((E)-[4-(dimethylamino)phenyl]diazenyl)benzoic acid) used as pH based indicator, it is yellow in colour in pH more than 6.2, red in pH values below 4.4 and orange in between (pH 4.4-6.2). It was selected as a model system due to its intense colour in aqueous systems and low biodegradability because of the benzene rings. The molecular structure of C.I. Acid Red 2 is depicted in Figure (1), [14]. The chemical formula of C.I. Acid Red 2 is $C_{15}H_{15}N_3O_2$ with molecular weight 269.31 g/mole [15].

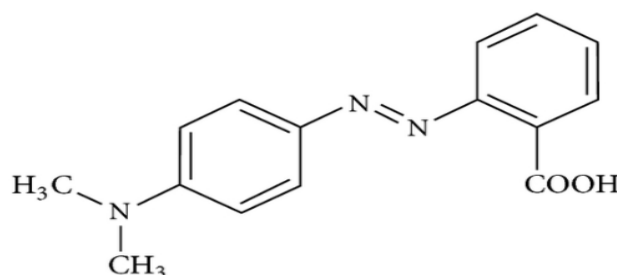


Fig 1: C.I. Acid Red 2 dye Molecular structure.

Table 1: Physico-chemical properties of C.I. Acid Red 2 [16]

S. No.	Properties	Values
1.	Molecular Weight	269.3 g/mol
2.	Hydrogen Bond Donor Count	1
3.	Hydrogen Bond Acceptor Count	5
4.	Rotatable Bond Count	4
5.	Exact Mass	269.116427 g/mol
6.	Monoisotopic Mass	269.116427 g/mol
7.	Formal Charge	0
8.	Isotope Atom Count	0
9.	Covalently-Bonded Unit Count	1

Table 2: Removal of C.I. Acid Red 2 onto various surfaces

S. No	Adsorbents	pH	Temperature (°C)	Maximum % Removal	Maximum Adsorption Capacity	References
1.	Modified Zeolites	6.5	25°C	99	0.250 (g g ⁻¹)	[17]
2.	Activated Carbon and Multiwalled Carbon Nanotubes	1.0	30°C	96	14.08 (mg g ⁻¹)	[18]
3.	Modified Durian Seed	6	30°C	92.52	401.65 (mg g ⁻¹)	[19]
4.	Tree Bark Powder	5	30°C	93.3	125.22 (mg g ⁻¹)	[20]
5.	Iron Oxide Nanoparticle	4	298-328 K	93	625 (mg g ⁻¹)	[21]
6.	Sugarcane Bagasse	-	-	91	54.60 (mg g ⁻¹)	[22]
8.	Modified Banana Trunk Fibers	5	25 - 27 °C	85	555.56 (mg g ⁻¹)	[23]
9.	Eichornia Crassipes Biomass	8.0	30°C	99.7	8.85×10 ⁻² mol g ⁻¹	[24]
10.	Banana Pseudostem Fibers	2.08	27±2°C	98.98	93.515 (mg g ⁻¹)	[25]
11.	CNTs and Activated Carbon	-	-	95	200.0 mg g ⁻¹	[26]
12.	MWCNTs Oxidized	-	-	97.5	108.7 (mg g ⁻¹)	[27]
13.	Annona Squamosa Seed	4.0	27±2°C	82.81	40.486 (mg g ⁻¹)	[28]
14.	Pomelo Peels	6.5	25 °C	93.5-94.8	207.02 (mg g ⁻¹)	[29]
15.	Rice Hulls	3	293 K	89	596.05 (mg g ⁻¹)	[30]
16.	Guargum Powder	4.2	34 °C	76	66.66 (mg g ⁻¹)	[31]
17.	Sugarcane Bagasse Pith	5	30 °C	86	75.55 (mg g ⁻¹)	[32]

Table 3: Functionalized and chemically active bagasse ash based adsorbents, targeted pollutants and their corresponding maximum adsorption capacities

S. No	Adsorbates	Adsorbents	Maximum Adsorption Capacity (mg/g)	References
1.	Malachite Green	Natural and modified sugar cane bagasse	89	[33]
2.	Copper	Modified and unmodified sugarcane bagasse	38.84	[34]
3.	Cadmium	Chemically modified sugarcane bagasse	SHS-80.3 and CAS -90.9	[35]
4.	Copper	Sulphuric acid treated sugarcane bagasse	94.4	[36]
5.	Congo Red	Sugar cane bagasse	38	[37]
6.	Fe(III) and heavy-metal ions	Sugar cane bagasse as a natural solidphase extractor	>95.0%	[38]

Preparation of adsorbate standard solution

C.I. Acid Red 2 (azo dye) supplied by local manufacturer was used for the study. 1×10^{-5} M stock solution was prepared by dissolving desired amount of it in 250 ml of distilled water and the required standard solutions were prepared by dilution method ($V_1M_1 = V_2M_2$). Prepared standard solution was used for bio-adsorption and for the study of adsorption isotherm.

Adsorption experiments

Adsorption experiments were performed by the batch technique. The adsorption of a C.I. Acid Red 2 on rawbagasse ash, activated bagasse ash and blended bagasseash based biosorbents were obtained after stirring the beakers containing 50 ml of sample with different amount of adsorbents, different values of pH and different contact time at room temperature. The stirring proceeded for different periods of time after which the mixture was left to settle and filtered. The absorbance of the filtrate was determined by using UV-Vis spectrophotometer at adjusted λ_{max} (410nm).

Equilibrium studies

A fixed amount of adsorbent (0.5 g) was added into a set of each 250 ml beakers containing 50 ml of different concentrations of C.I. Acid Red 2 solution with adjusted pH of 4.5. The beakers were stirred on a magnetic stirrer for 60 minutes until the equilibrium was reached. The absorbance was measured by a double beam UV-Vis spectrophotometer (Model DR 4000 U, HACH). Each experiment was triplicated under identical conditions. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by:

$q_e = (C_0 - C_e) V / W$, where, C_0 and C_e (mg/L) are the liquid-phase concentrations of dye at initial and at equilibrium time (t) respectively, V is the volume of the solution in liter (L) and W (g) is the mass of adsorbent used. The percentage of dye-removal is calculated by following formula:

Removal percentage = $C_0 - C_t / C_0 \times 100$, Where, C_t (mg/L) is the liquid-phase concentrations of dye at time (t).

Effect of contact time:

The contact time ranged from 60 minutes, pH of the aqueous dye solution was adjusted to pH 4.5. The amount of adsorbent was 0.5g/50 ml. the dye concentration was 1×10^{-5} M.

Effect of pH:

The pH ranges from 2.0 to 5.0; the amount of adsorbent was 0.5g/50 ml, where the contact time was 60 minutes. The pH was adjusted by adding a few drops of 0.1 M NaOH or 0.1 M HCl.

Effect of amount of adsorbent:

The range of amount of adsorbent was 0.5 g for 50 mL solution. The pH was fixed at 4.5 and the contact time for the experiment was 60 minutes.

RESULTS AND DISCUSSION

Characterization of new adsorbents

FTIR analysis of RBA, ABA and BBA

To analyze the functional groups and confirm modification of biosorbent, FTIR spectra of RBA, ABA and BBA were recorded (Figure 2). The functional groups assignment has been summarized in Table 4.

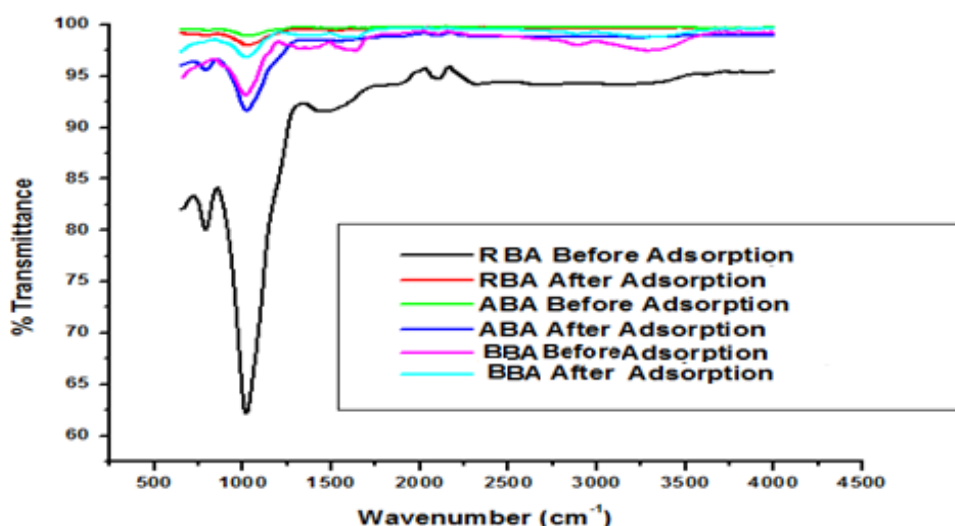


Fig 2: FTIR of RBA, ABA and BBA biosorbents

Table 4: FTIR spectral characteristics of RBA, ABA and BBA before and after adsorption.

Peaks			Peaks difference	Peak assignment
RBA	ABA	BBA		
1456	1520	-	+64	Symmetric bending of CH ₃
2915	2945	2960	+30	Aliphatic CH group
1623	1642	1765	+19	C=O stretching
1087	1100	1095	+13	C-O stretching
1526	1528	-	+2	Secondary amine group
791	1100	1150	+50	Si-O bending
-	-	3235	-	Si-OH[39]

X-Ray Diffraction analysis of RBA, ABA and BBA

The surface area, particle size and pore size of the biosorbents took play an imperative role in the adsorption process. In this conception, the particle size of the used biosorbents was ascertained by XRD analysis. The XRD spectra of RBA, ABA, and BBA are presented in Figure 3 and the plane and corresponding crystallized fractions assignment has been summarized in Table 5.

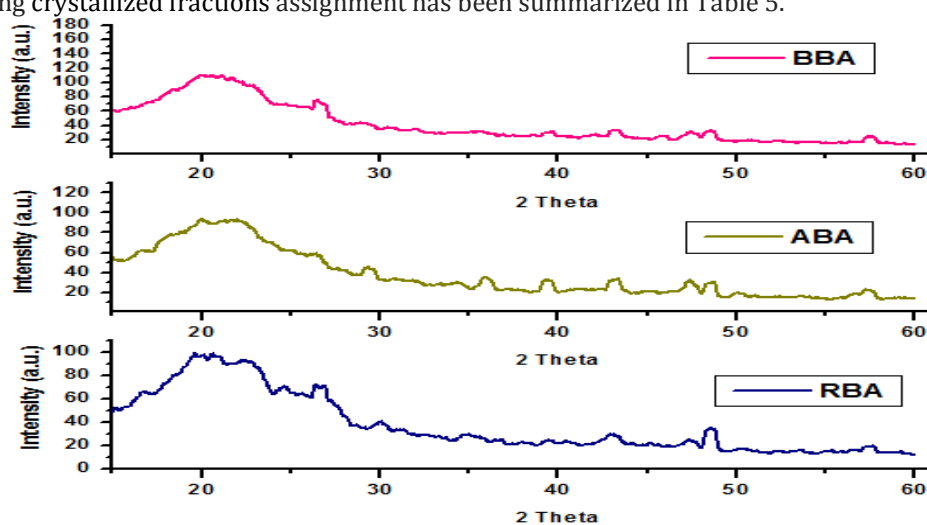


Fig 3: X-Ray Diffraction analysis of RBA, ABA and BBA biosorbents.

Table 5: XRD spectral characteristics of RBA, ABA and BBA.

Value of $2\theta^\circ$	Plane	Crystallized Fractions
40-50	101	Carbon
15-30	002	Carbon (C-H)
20	110	Semi crystalline chitosan
25	002	Graphite crystallite
49.5	002	Graphite crystallite

Scanning Electron Microscopy analysis of RBA, ABA and BBA

SEM micrographs of RBA, ABA and BBA are presented in Figure 4-6. There are irregular pores distributed which enhance the process of adsorption. RBA, ABA and BBA surfaces resemble sponge like structure wherein ions may be trapped in the holes.

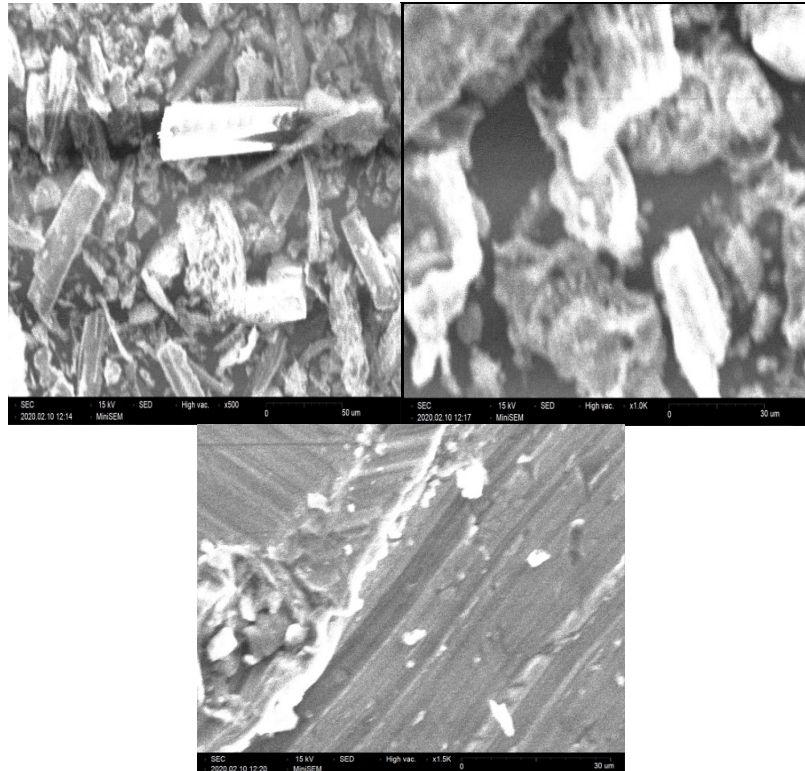


Fig 4: Scanning Electron Microscopy analysis of RBA.



Fig 5: Scanning Electron Microscopy analysis of ABA.

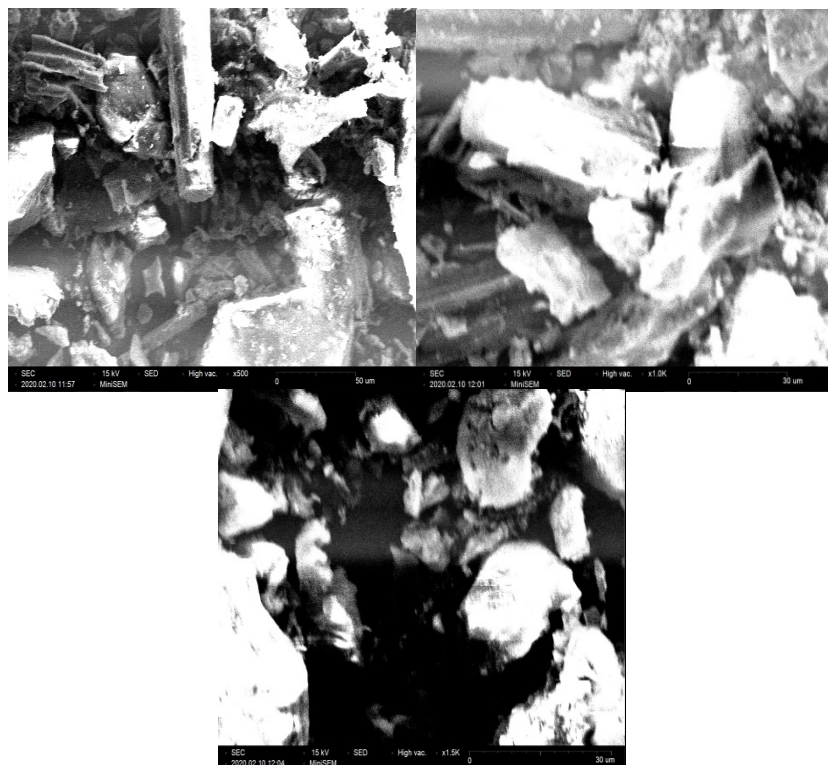


Fig 6: Scanning Electron Microscopy analysis of MBA

ADSORPTION RESULTS

Effect of contact time

50 mL of C.I. Acid Red 2 dye solution with known initial concentration was put in a series of 250 mL Erlenmeyer flasks. The amount of adsorbent that was added to each flask was fixed at 0.5 g. The flasks were placed in an isothermal water bath shaker at constant temperature of 40°C, until equilibrium point was reached. Samples are withdrawn at intervals to determine the residual concentration of the dye at 410 nm wavelength using an UV-Vis spectrophotometer (Model DR 4000 U, HACH). Figure (7) shows the plot of contact time versus percentage of the dye removed C.I. Acid Red 2.

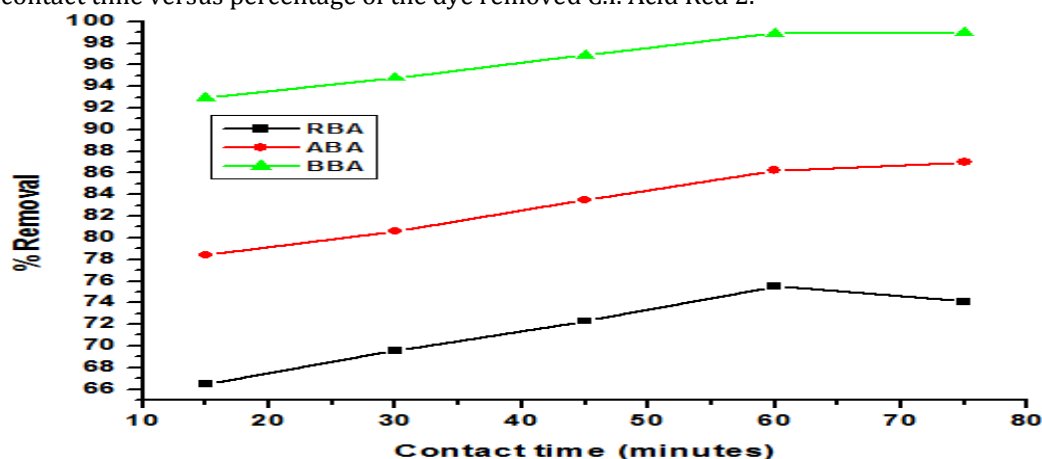


Fig 7: Effect of contact time for the removal of C.I. Acid Red 2 onto RBA, ABA and BBA.

Effect of Adsorbent Dose

An increase in the adsorbent dose (RBA, ABA and BBA) from 0.1 to 0.6 g/50 mL increases the percentage of the dyes removed. This may be attributed to increased sorbents surface area and availability of more sorption sites resulting from increased dose of the sorbent. The increase in adsorbent dose at constant dye concentration and volume results to increased availability of sorption sites and result to unsaturation of the sites. Greater adsorption rate was observed when the adsorbent/dye concentration ratio is greater

than when the ratio is lower. Figure (8) shows the plot of adsorbent dosage versus percentage of the dye removed C.I. Acid Red 2.

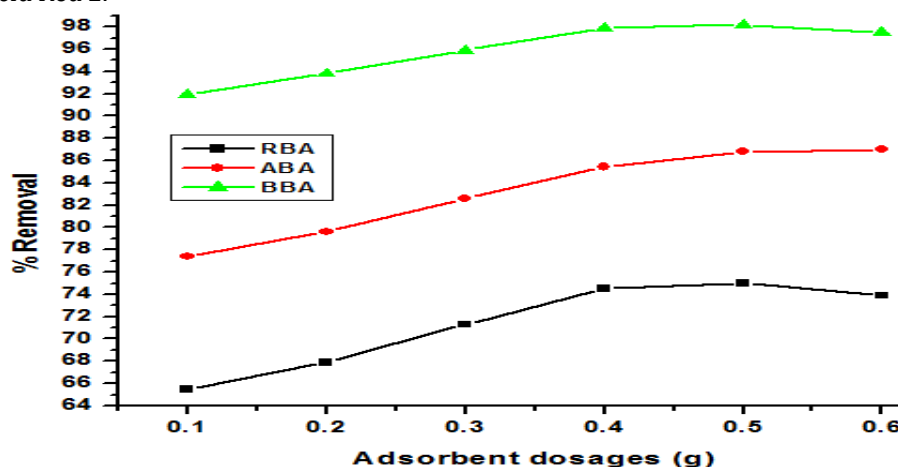


Fig 8: Effect of Adsorbent dosages for the removal of C.I. Acid Red 2 onto RBA, ABA and BBA

From the plot, the slope is positive indicating that adsorption increases with an increase in the adsorbent dose. It can be deduced that a fixed mass of the adsorbent is needed to adsorb certain amount of the dye, which means the higher the adsorbent dose, the larger the volume of effluent can purify [40]. The increase in sorbent dose at constant dye concentration and volume will lead to unsaturation of sorption sites through the sorption process.

Effect of initial dye concentration

A mass of 0.5 g of each adsorbent (RBA, ABA and BBA) was contacted with 50 mL of the dye concentration 1-6 mg/ml at pH 2 and 5 for the adsorbents respectively, using water-bath maintained at 40°C. The agitation speed was kept constant. At predetermined intervals of time, samples were analyzed for the final concentration of dyes by a UV-Vis spectrophotometer Model DR 4000 U, HACH).

From the results obtained, an increase in initial dye concentration or dosage from 1-6 mg/ml leads to a decrease in the percentage of the dyes removed C.I. Acid Red 2. Figure (9) shows the plot of initial concentrations versus percentage of the dye removed C.I. Acid Red 2.

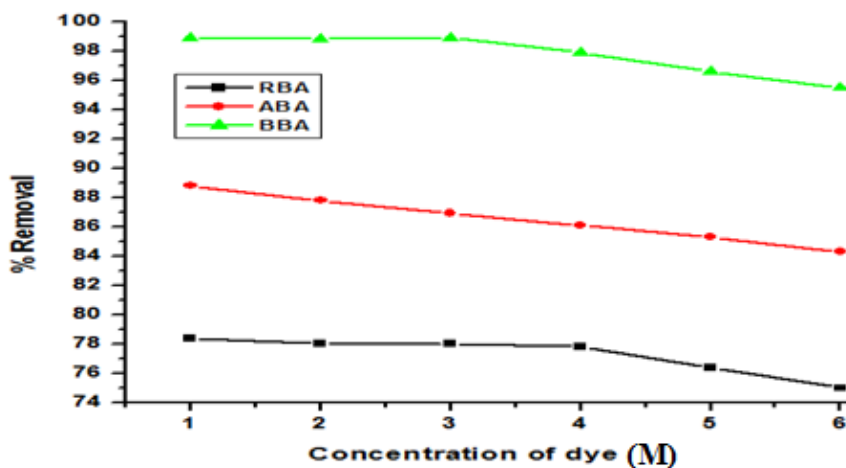


Fig 9: Effect of concentrations for the removal of C.I. Acid Red 2 onto RBA, ABA and BBA.

Effect of temperature

Adsorption of dye C.I. Acid Red 2 (methyl red) increased with an increase in temperature for the adsorbents (RBA, ABA and BBA) respectively. This indicates that adsorption is endothermic. The solubility of the dyes increases with an increasing temperature, thus at high temperature the dyes solution reaches an equilibrium where they can be adsorbed out by the adsorbent particle. In addition, at higher temperature the adsorbents active sites are activated thereby increasing the concentration of the dye removed. The enhancement of the adsorption might also be due to enhancement of the adsorptive

interaction between the active sites and the adsorbate ion. Figure (10) shows the plot of temperature variation versus percentage of dyes (C.I. Acid Red 2) removed respectively.

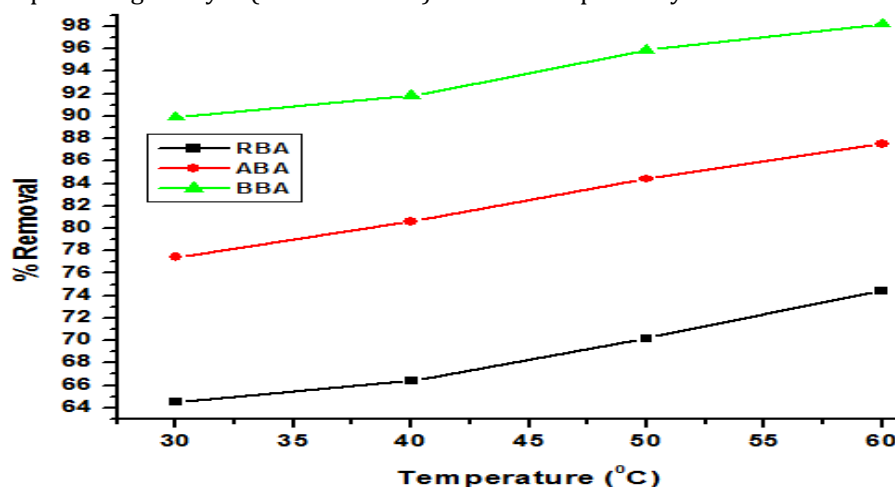


Fig 10: Effect of Temperature on the removal of C.I. Acid Red 2 onto RBA, ABA and BBA

Effect of pH

The result of the experiments done at different pH values, which were conducted to determine the optimum pH range for the dyes adsorption on RBA, ABA and BBA. The percentage removal of C.I. Acid Red 2 by RBA, ABA and BBA were optimum at 2.5, and 4 respectively, it could be as a result of the degree of ionization of the species at the different pH. More C.I. Acid Red 2 was absorbed by RBA, ABA and BBA in an acid medium. Figure (11) shows the plot of pH variation versus percentage of dyes (C.I. Acid Red 2) removed respectively.

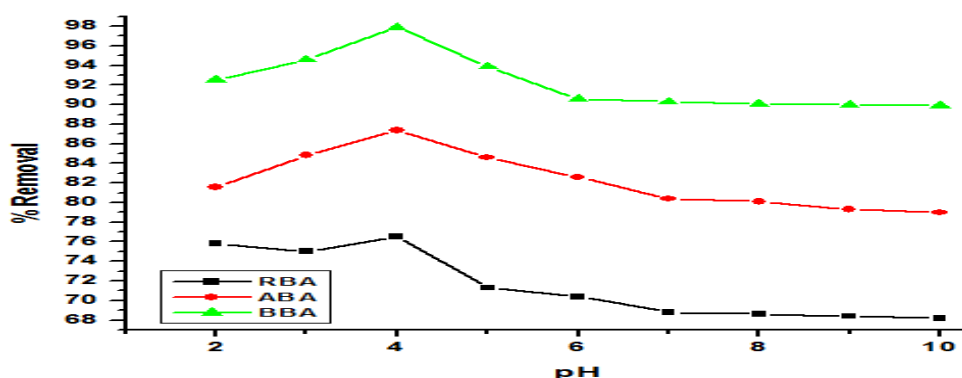


Fig 11: Effect of pH on the removal of C.I. Acid Red 2 onto RBA, ABA and BBA

The dyes sorption behaviour as exhibited by the three biosorbents relative to solution pH could be attributed to several reasons. The surface of all the powdered materials may contain a large number of active sites and the solute (dye ions) uptake can be related to the solute in the solution [41].

ADSORPTION ISOTHERM

Langmuir Isotherm

Adsorption isotherm is important in describing how solutes interrelate with the adsorbent and so is critical in optimizing the use of adsorbents. Correlation of isotherm data by empirical or theoretical equations is thus essential for the operation of adsorption systems and practical design [40]. The Langmuir equation assumes that maximum adsorption occurs when surface is covered by the adsorbate. The distribution of dyes has been described by the linear form of Langmuir equation given as:

$$(C_e/q_e) = (1/bQ_0) + (C_e/Q_0)_2$$

Where C_e is concentration of dye solution (mol L^{-1}) at equilibrium, q_e is amount of dye adsorbed per unit weight of adsorbent (mol g^{-1}), b is related to the energy of adsorption (l mol^{-1}). Figure (12) shows the Langmuir isotherm for the adsorption of C.I. Acid Red 2 by RBA, ABA and BBA respectively.

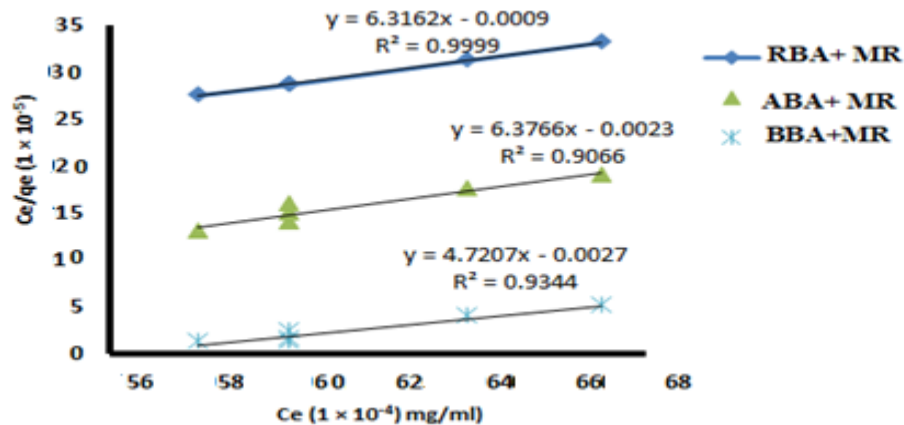


Fig 12: Langmuir isotherm for the adsorption of C.I. Acid Red 2 onto RBA, ABA and BBA

The adsorption was found linear over the entire concentration range studied with good regression coefficient ($R^2 = 0.9481$ and 0.8524) for RBA, ABA and BBA respectively on C.I. Acid Red 2. The result shows that the correlations fit in well into the Langmuir isotherm for C.I. Acid Red 2; this can then imply that the RBA, ABA and BBA are better used to adsorb C.I. Acid Red 2 of the same concentration. It was observed to fit in correctly into Langmuir isotherms. The fact that Langmuir isotherms fits the monolayer coverage of dye very well confirms the monolayer coverage of dye on adsorbent particles and also the homogenous distribution of active sites on the material, since Langmuir equation assumes that the surface is homogenous [42].

Freundlich Isotherm

The Freundlich model is an empirical equation based on adsorption on heterogeneous surface or surface supporting sites of varied affinities. It is assumed that the stronger binding sites are occupied first and that the binding strength decreases with the increasing degree of site occupation. The Freundlich isotherms expressed as

$$\log q_e = \log K_F + 1/n \log C_e$$

where q_e is amount of adsorbate adsorbed per unit mass of adsorbent (mg/g); K_F is Freundlich isotherm constant (mg/g) $(L/mg)^{1/n}$, which indicate the relative adsorption capacity of the adsorbent related to the bonding energy; C_e is equilibrium concentration of the adsorbate (mg/L) and n_F is the heterogeneity factor representing the deviation from linearity of adsorption and is also known as Freundlich coefficient. If the plot of $(\log q_e)$ against $(\log C_e)$ gave straight line, it indicates that the Freundlich isotherm fit the adsorption data. Other constants can be calculated from the slope ($1/n$) and intercept ($\log K_F$) of the linear plot of experimental data. The slope of $1/n$ ranging between 0 and 1 is a measure of adsorption intensity, becoming more heterogeneous as its value gets closer to zero. Figure (13) shows the Freundlich isotherm for the adsorption of C.I. Acid Red 2 by RBA, ABA and BBA respectively. Table 6 shows the Langmuir and Freundlich adsorption parameter for C. I. Acid Red Dye 2.

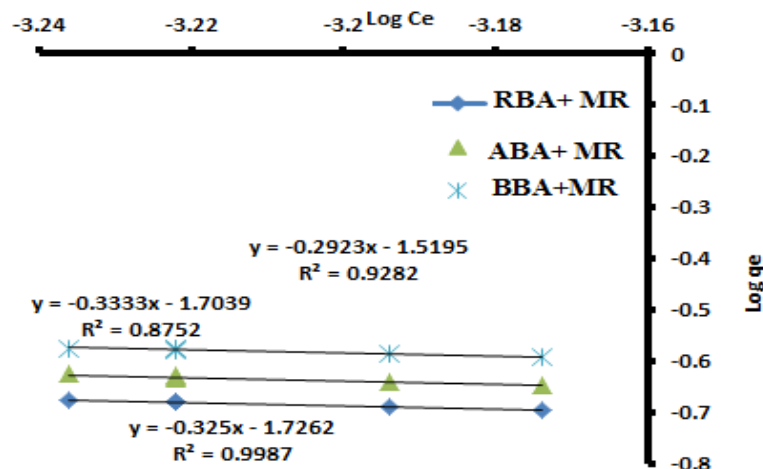


Fig 13: Freundlich isotherm for the adsorption of C.I. Acid Red 2 onto RBA, ABA and BBA

Table 6: Langmuir and Freundlich adsorption parameter for C. I. Acid Red Dye 2

Experimental Condition	Langmuir				Freundlich		
	q _m (mg /g)	b (L/mol)	R _L	R ²	K _F	n	R ²
WBA+MR	0.158	42.25	8.62	0.99	5.64	-3.07	0.99
ABA+MR	0.156	57.14	6.140	0.90	5.47	-3.00	0.87
MBA+MR	0.212	54.38	6.756	0.93	4.57	-3.42	0.92

CONCLUSION

The result revealed the potential of RBA, ABA and BBA, an agricultural waste material to be an effective bio-adsorbent for removal C.I. Acid Red 2 from aqueous solution. Equilibrium data are in well agreement with Langmuir adsorption isotherm model at 25°C. The adsorption capacity was found to be maximum at pH 4.4 and at adsorbent mass of 0.5 g/50 ml dye concentration within 60 minutes of physical adsorption. The major advantage of using bio-adsorbents for removing dyes is due to their highly selective nature of adsorption. Preliminary results indicate that RBA, ABA and BBA are a good bio-adsorbent for removing dyes from textile wastewater.

ACKNOWLEDGEMENTS

The authors would like to place thanks to Head, Department of Chemistry and Director, Dayalbagh Educational Institute, Dayalbagh, Agra, (Deemed University) India for providing required facilities to carry out this research work. The Financial support from the University (DEI) under research division sustainability grant [AR. No. 81/Plan/Capital assets/2018/2019] is gratefully acknowledged.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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