

Adsorption Studies Using Biosolids for the Removal of Dyes

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ABSTRACT: Wastewater containing high volume of dye has adverse impacts on water, soil and environment hence it is necessary to develop a scientific solution of low cost materials for removing the dye from the water. The aim of this study is to synthesis a low cost adsorption material using biosolids and to evaluate its efficiency for removing methylene blue, basic violet dyes by adsorption process. Batch experiments were conducted with different dosage of adsorbent ranging from 0.5 to 2g and different contact time ranging from 0 to 180 minutes. Synthetic samples of adsorbate were prepared and the samples were analyzed under different operational parameters like pH, contact time, adsorbent dosage. For Methylene blue dye the optimum pH, contact time, adsorbent dosage was 2, 150 min, and 1 g/L and maximum removal efficiency was 93.5%, similarly for basic violet dye the optimum pH, contact time, adsorbent dosage was 2, 120 min and 2g/L and maximum removal efficiency was 87.5%. Adsorption rate of Methylene blue and Basic violet dyes was found to be considerably faster for chemically activated biosolid than unmodified biosolid. For both the dyes Langmuir adsorption isotherm model fitted well. Based on the results it is concluded that activated biosolid can be used effectively for the removal of methylene blue and basic violet dyes from aqueous solution.

KEYWORDS: Adsorption, Dyes, Biosolids, Removal Efficiency, Wastewater.

I. INTRODUCTION

Wastewater / used water are defined as any water that has been adversely affected by anthropogenic influence. Waste water is obtained from both domestic and industrial activities. Industries like tannery, textile, paper and pulp etc. exhibit effluents which are harmful to the environment. Thus, the waste water has to be treated before their discharge into any source and the principal objective of wastewater treatment is generally to allow municipal and industrial effluents to be disposed of without danger to human health.

The textile industry is one of the largest polluters in the world. The World Bank estimates that almost 20% of global industrial water pollution comes from the treatment and dyeing of textiles. Around 72 toxic chemicals reach our water supply from textile dyeing. Many of these chemicals cannot be filtered or removed. Dyeing, rinsing, and treatment of textiles all use large amount of fresh water. Cotton production accounts for 2.6% of annual global water usage. A single T-shirt made from conventional cotton requires 2700 litres of water, and a large amount of chemicals. Million gallons of wastewater discharged by textile mills each year containing chemicals like formaldehyde (HCHO), chlorine and heavy metals such as lead and mercury. These chemicals cause both environmental damage and human disease. Effluents released from mills are often at high temperatures and pH, which adds the problem. Textile manufacturing dyes releases aromatic amines (benzamine and toluidine), heavy metals, ammonia and alkali salts.

The primary objective of the study is, to study about the potential of utilizing “waste-treats-waste” by using biosolids as an adsorbent, to create a low cost and effective adsorbent, to evaluate the removal efficiency of dye using biosolids.

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II. LITERATURE REVIEW

Xiaoge Chen et al (2002) this paper explains about the preparation of activated carbon from sewage sludge. The objective of this study was to investigate the physical and chemical properties of the activated carbon made from sewage sludge so as to give a basic understanding of its structure. The activated carbon was prepared by activating anaerobically digested sewage sludge with 5 M $ZnCl_2$ and therefore pyrolyzing it at 500 °C for 2h under nitrogen atmosphere. Its adsorption capacities for aqueous phenol and carbontetrachloride were examined. The results indicated that the activated carbon made from sewage sludge had remarkable micropore and mesopore surface areas and notable adsorption capacities for phenol and carbontetrachloride. In comparison with commercial activated carbons, it displayed distinctive physical and chemical properties.

M.Sarioglu (2006) et al this paper explains about the mechanism of methylene blue adsorption on biosolid (waste sludge). The effects of various experimental parameters, such as pH (3-11), biosolid dosage (1-10g/l), contact time (5-1440 min) and initial dye concentration were investigated. The results showed that the dye removal increased with increase in the initial concentration of the dye and also increased in amount of biosolid used and initial pH. Adsorption data was modeled using freundlich adsorption isotherm. The results show that biosolid could be employed effective and low cost material for removal of dyes and colour from aqueous solution.

Yao chen et al (2011) stated that activated carbons derived from municipal sewage sludge with pyrolusite modification were prepared. The impact of pyrolusite amount on the activated carbons' characteristics was evaluated by methylene blue tests. The results showed that activated carbons modified with certain amount of pyrolusite (ACP) had better performance than the ones without pyrolusite addition. These activated carbons prepared were effective in COD and color removal in treating an industrial dyeing wastewater. The significant increase of mesopores in the adsorbents was identified as the reason. The optimum pH value for dye adsorption was pH 5.0 and it took a relatively long contact time to reach equilibrium (8 h). Temperature had no obvious effect on dye removal in this study. The regeneration efficiency for three ACPs after two adsorption/regeneration cycles was above 70%.

LiliXu (2014) et al The study was undertaken to propose a potential approach for resource utilization and becomes one of the most widely used adsorbent for wastewater treatment. However, effects of environmental factors and its relative mechanism for pollutant removal have not been thoroughly studied. In this study, carbonized sludge derived from sewage sludge was investigated. The absorption process of Methylene Blue was a spontaneous and endothermic reaction. The values of ΔH° , ΔS° , and ΔG° for adsorption of Acid Scarlet GR were 32.78 kJ/mol, 104.00 J/mol and 2.26 kJ/mol, respectively. The adsorption process of Acid Scarlet GR was less effective.

Glaydsonsimoaes et al (2015) proposed that the Sewage sludge was pyrolyzed in an inert atmosphere at 550°C after chemical treatment with $ZnCl_2$ or KOH using an organic precursor to inorganic weight ratio of 1:1. The materials were used as adsorbents for removal of Cu (II) from aqueous solution. The adsorbents were characterized using adsorption/desorption nitrogen isotherms, thermal analysis, scanning electron microscopy, and elemental analysis. The results showed that copper removal was linked to surface pH and compounds present on the surface of the adsorbent. The highest desorption was obtained using 1.50mol/L of HNO_3 as the eluent. The recoveries of Cu (II) loaded adsorbents were 98.9% for $ZnCl_2$ -treated and 95.5% for KOH-treated adsorbents.

III. METHODOLOGY AND MATERIALS USED

The waste activated sludge (sewage sludge or biosolids) from the secondary treatment unit (Sequential Batch Reactor) of Avaniyapuram Wastewater Treatment plant (125MLD) was collected with prior permission from The City Engineer of the Madurai Corporation. The mostly used dyes were identified such as Methylene blue (basic blue), Basic violet (fuchsine) and are purchased from the local store. Synthetic stock solutions of the dyes were prepared by dissolving the dye in the distilled water. All other chemicals used for the experimental analysis are of Analytical

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grades. Equipment used for this experiment is UV-vis spectrophotometer which was used to determine the concentration of the dyes.

ICP-OES, Orbital shaker for mixing adsorbent and adsorbate, Muffle furnace and oven for activation of biosolids, pH meter for pH adjustment, COD digester for COD analysis were also used. The sewage sludge was obtained from nearby wastewater treatment plant and it was generated from the process of sequential batch reactor. The waste activated sludge was collected from the centrifuge. The raw sewage sludge was firstly sun dried for three days and then dried in oven at 105°C for approximately 4 hours and sieved. The dried sewage sludge was acid digested to analyse the heavy metal concentration using ICP-OES. For thermal treatment approximately 50g of raw sludge was placed in a steel reactor to a muffle furnace. The sample was heated in a closed system at 500°C under low vacuum in the muffle furnace for 60 minutes. 50g of sludge was soaked stillly to 100 ml of 3 mole zinc chloride (25%) solution for 24 hours at room temperature. Then, it was dried again at 105°C for 8 hours to constant weight. The obtained dried thermally and chemically activated sludge was stored in a plastic container for further experiments.

Synthetic solutions of all the dyes were prepared by diluting a stock solution with distilled water with concentration of 200 mg/L. The pH of each solution was adjusted to the desired value by adding diluted H₂SO₄ or NaOH with the aid of pH meter. For the optimization of the experimental parameters on the adsorption of dyes, batch experiments were performed at the ambient temperature. In this experiment, the effects of adsorbent dosage (0.5, 1, 2g), pH of the solution (2,7,10) agitation speed 110 rpm and contact time (3 hours) of the adsorption of the dyes were studied.

IV. RESULTS AND DISCUSSIONS FOR METHYLENE DYE REMOVAL

Table 1 Removal of Methylene Blue for various dosage of raw biosolids @pH=2

Reaction Time (min)	Dosage of raw biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	146	27	160	20	147	26.5
60	136	32	149	25.5	117	41.5
90	100	50	118	41	083	58.5
120	084	58	114	43	082	59
150	070	65	097	51.5	082	59
180	070	65	072	64	069	65.5

At a 2g dosage of raw biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Methylene blue (65.5%) was obtained at pH 2. As time increases the removal efficiency of methylene blue dye increased. The concentration decreased from the initial concentration of 200 mg/L to 69 mg/L after the batch experiment.

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Table 2 Removal of Methylene Blue for various dosages of chemically activated biosolids @ pH=2

Reaction Time (min)	Dosage of Chemically activated biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	149	25.5	119	40.5	136	32
60	096	52	092	54	114	43
90	095	52.5	062	69	070	65
120	039	80.5	044	78	057	71.5
150	042	79	031	84.5	040	80
180	042	79	013	93.5	013	93

At a 1g dosage of chemically activated biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Methylene blue (93.5%) was obtained at pH 2. As time increases the removal efficiency of methylene blue dye increased. The concentration decreased from the initial concentration of 200 mg/L to 13 mg/L after the batch experiment.

Table 3 Removal of Methylene Blue for various dosages of raw biosolids @ pH=7

Reaction Time (min)	Dosage of raw biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	157	21.5	156	22	149	25.5
60	118	41	138	31	135	32.5
90	113	43.5	123	38.5	095	52.5
120	088	56	081	59.5	076	62
150	075	62.5	070	65	076	62
180	075	62.5	071	64.5	076	62

At a 1g dosage of raw biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Methylene blue (64.5%) was obtained at pH 7. As time increases the removal efficiency of methylene blue dye increased. The concentration decreased from the initial concentration of 200 mg/L to 71 mg/L after the batch experiment.

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Table 4 Removal of Methylene Blue for various dosages of chemically activated biosolids @ pH=7

Reaction Time (min)	Dosage of Chemically activated biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	139	30.5	136	32	130	35
60	124	38	127	36.5	096	52
90	115	42.5	115	42.5	082	59
120	087	56.5	084	58	082	59
150	078	61	084	58	082	59
180	078	61	084	58	082	59

At a 0.5 g dosage of chemically activated biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Methylene blue (61%) was obtained at pH 7. As time increases the removal efficiency of methylene blue dye increased. The concentration decreased from the initial concentration of 200 mg/L to 78 mg/L after the batch experiment.

Table 5 Removal of Methylene Blue for various dosages of raw biosolids @ pH=10

Reaction Time (min)	Dosage of raw biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	158	21	150	25	148	26
60	139	30.5	136	32	136	32
90	137	31.5	129	35.5	097	51.5
120	127	36.5	100	50	088	56
150	095	52.5	092	54	075	62.5
180	095	52.5	092	54	075	62.5

At a 2g dosage of raw biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Methylene blue (62.5%) was obtained at pH 10. As time increases the removal efficiency of methylene blue dye increased. The concentration decreased from the initial concentration of 200 mg/L to 75 mg/L after the batch experiment.

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Table 6 Removal of Methylene Blue for various dosages of Chemically activated biosolids @ pH=10

Reaction Time (min)	Dosage of Chemically activated biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	163	18.5	158	21	148	26
60	149	25.5	136	32	137	31.5
90	115	42.5	125	37.5	100	50
120	089	55.5	115	42.5	086	57
150	082	59	104	48	087	56.5
180	082	59	104	48	087	56.5

At a 0.5g dosage of chemically activated biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Methylene blue (59%) was obtained at pH 10. As time increases the removal efficiency of methylene blue dye increased. The concentration decreased from the initial concentration of 200 mg/L to 82 mg/L after the batch experiment.

V. RESULTS AND DISCUSSIONS FOR BASIC VIOLET DYE REMOVAL

Table 7 Removal of Basic Violet for various dosage of raw biosolids @ pH=2

Reaction Time (min)	Dosage of raw biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	162	19	158	21	156	22
60	146	27	138	31	134	33
90	128	36	124	38	120	40
120	116	42	110	45	106	47
150	116	42	102	49	100	50
180	116	42	102	49	100	50

At a 2g dosage of raw biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Basic violet (50%) was obtained at pH 2. As time increases the removal efficiency of Basic violet dye increased. The concentration decreased from the initial concentration of 200 mg/L to 100 mg/L after the batch experiment.

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Table 8 Removal of Basic Violet for various dosage of chemically activated biosolids @pH=2

Reaction Time (min)	Dosage of Chemically activated biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	156	22	130	35	110	45
60	108	46	88	56	62	69
90	86	57	50	75	34	83
120	68	66	28	86	25	87.5
150	68	66	28	86	25	87.5
180	68	66	28	86	25	87.5

At a 2g dosage of chemically activated biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Basic violet (87.5%) was obtained at pH 2. As time increases the removal efficiency of basic violet dye increased. The concentration decreased from the initial concentration of 200 mg/L to 25 mg/L after the batch experiment.

Table 9 Removal of Basic Violet for various dosage of raw biosolids @pH=7

Reaction Time (min)	Dosage of raw biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	186	7	178	11	178	11
60	172	14	160	20	162	19
90	166	17	156	22	158	21
120	158	21	146	27	148	26
150	142	29	126	37	136	32
180	136	32	120	40	126	37

At a 1g dosage of raw biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Basic violet (40%) was obtained at pH 7. As time increases the removal efficiency of basic violet dye increased. The concentration decreased from the initial concentration of 200 mg/L to 120 mg/L after the batch experiment.

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Table 10 Removal of Basic Violet for various dosage of chemically activated biosolids @pH=7

Reaction Time (min)	Dosage of chemically activated biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	182	9	172	14	178	11
60	164	30	148	26	144	28
90	150	25	138	31	130	35
120	122	39	114	43	116	42
150	106	47	98	51	96	52
180	106	47	98	51	96	52

At a 2g dosage of chemically activated biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Basic violet (52%) was obtained at pH 7. As time increases the removal efficiency of basic violet dye increased. The concentration decreased from the initial concentration of 200 mg/L to 96 mg/L after the batch experiment.

Table 11 Removal of Basic Violet for various dosage of raw biosolids @pH=10

Reaction Time (min)	Dosage of raw biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	186	7	180	10	182	9
60	178	11	166	17	178	11
90	174	13	158	21	166	17
120	170	15	150	25	148	26
150	148	26	148	26	140	30
180	146	27	138	31	134	33

At a 2 g dosage of raw biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Basic violet (33%) was obtained at pH 10. As time increases the removal efficiency of basic violet dye increased. The concentration decreased from the initial concentration of 200 mg/L to 134 mg/L after the batch experiment.

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Table 12 Removal of Basic Violet for various dosage of chemically activated biosolids @pH=10

Reaction Time (min)	Dosage of chemically activated biosolids					
	0.5 g		1.0 g		2.0 g	
	mg/L	% Effi.	mg/L	% Effi.	mg/L	% Effi.
0	200	0	200	0	200	0
30	186	7	184	8	184	8
60	178	11	178	11	174	13
90	172	14	174	13	168	16
120	166	17	162	19	158	21
150	158	21	152	24	150	25
180	148	36	144	38	138	31

At a 1g dosage of chemically activated biosolids as a adsorbent and at a time of 180 minutes, the maximum removal efficiency of Basic violet (38%) was obtained at pH 10. As time increases the removal efficiency of basic violet dye increased. The concentration decreased from the initial concentration of 200 mg/L to 96 mg/L after the batch experiment.

VI. CONCLUSION

Experimental results of the adsorption experiments, which took into account the effects of equilibrium concentration on the adsorption capacity, were evaluated with two popular adsorption models, Langmuir and Freundlich models. Through the analysis of isotherm data, an equation was developed to accurately represent the experimental results and be used for design purposes. The linearized forms of the equations representing the models were used. Langmuir isotherm model assumed that adsorption occurs at specific homogeneous adsorption sites within the adsorbent and intermolecular forces decrease rapidly with the distance from the adsorption surface. Freundlich equation was derived to model the multiplayer adsorption and the adsorption on heterogeneous surfaces. From the comparisons of the correlation co-efficients (R^2) values, the adsorption isotherm for Methylene blue dye were fitted satisfactorily well with Langmuir equation with a correlation coefficient 0.92. Hence, for the adsorption of Methylene blue, Langmuir equation gave a better fit to the experimental results than Freundlich equation.

From the comparisons of the correlation co-efficients (R^2) values, the adsorption isotherm for Basic violet dye were fitted satisfactorily well with Langmuir equation with a correlation coefficient 0.89. Hence, for the adsorption of Basic violet, Langmuir equation gave a better fit to the experimental results than Freundlich equation.

The selectivity of raw and activated biosolid for adsorption of Methylene blue and Basic violet was examined and the influences of contact time, pH and adsorbent dosage on adsorption capacities were examined. The maximum removal capacity of methylene blue for chemically activated biosolids was achieved at optimum pH = 2, contact time 180 minutes and adsorbent dosage 1g and with a maximum removal efficiency of 93.5%. Similarly for basic violet the maximum removal efficiency of 87.5% was obtained at pH = 2, contact time 120 minutes and adsorbent dosage 2g. On increasing the solution pH from 2 to 10 the adsorption capacity decreased for both Methylene blue and Basic violet. The pH trend followed by the chemically activated biosolids for the removal of both the dyes was $2 > 7 > 10$. The trend for contact time followed by the chemically activated biosolids for the removal of Methylene blue dye was $180 > 150 > 120 > 90 > 60 > 30$ minutes. The trend for contact time followed by the chemically activated biosolids for the removal of Basic violet was $120 > 90 > 60 > 30$ minutes. The trend for adsorbent dosage followed by Methylene blue dye was $1 > 2 > 0.5$ g. The trend for adsorbent dosage followed by Basic violet was $2 > 1 > 0.5$ g. When compared with rice husk which has the maximum removal efficiency of 98%, sewage sludge has a maximum removal efficiency of 93.5 %, therefore sewage sludge has a minimum removal efficiency compared to rice husk. The Langmuir adsorption isotherm

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model fitted well for both the dyes. The results obtained indicated that the adsorption capacity of biosolids can be effectively used for the adsorption of methylene blue and basic violet providing a low-cost alternative to the use of commercial-grade activated carbon.

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