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Comparative Studies on Removal of Fast Green Dye from Aqueous Solutions by Activated Carbon Prepared from *Gloriosa superba* Waste and *Alternaria raphani* Fungal Biomass

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ABSTRACT

A comparative study was done on removal of fast green dye from aqueous solution by activated carbon prepared from *Gloriosa superba* pericarp and live fungal biomass of *Alternaria raphani* was used by adsorption technique under varying conditions of agitation time, dye concentration, adsorbent dose and pH. Adsorption depended on solution pH, dye concentration, adsorbent concentration and contact time. Equilibrium time for fast green dye removal using both adsorbents was attained at 60 to 120 min. Increase in biomass dosage increased the adsorption. Adsorption capacity (Q_0) of fast green by activated carbon (51.02 mg g^{-1}) was higher than fungal biomass (35.59 mg g^{-1}). The second-order kinetic model described well the experimental data. Acidic pH was favorable for the adsorption of fast green studied. Studies on pH effect and desorption show that chemisorptions seems to play a major role in the adsorption process. Among the two adsorbents studied for fast green removal, activated carbon showed better adsorption capacity. Thus utilization of *Gloriosa superba* pericarp waste for fast green dye removal has potential application in dyeing industry wastewater treatment.

Key words: *Gloriosa superba* pericarp, activated carbon, fast green, *Alternaria raphani*

INTRODUCTION

Fast green FCF is widely used for coloring foodstuffs and as a staining agent (quantitative stain) for histones at alkaline pH after acid extraction of DNA. Toxicological data reveal that fast green FCF is highly toxic (Aksu and Cagatay, 2006). It is an allergen to humans and causes eyes and skin irritation and affects the upper respiratory tract. It also acts on a presynaptic locus by inhibiting the release of neurotransmitters in the human and animal nervous system (Van Hooft, 2002). Removal of dyes from environment poses severe problem to humans as well as others. Though there are various methods for removal of dyes, adsorption method has been considered for the removal of textile dye effluents from wastewater and this method is superior compared with other traditional treatment methods due to its low cost, easy availability, simplicity of design, high efficiency, ease of operation and ability to treat a wide variety of dyes.

The most commonly used adsorbent for the removal of dyes is commercial coal based activated carbon due to its high adsorption capacity and high surface area. Due to high cost of commercially

available activated carbons, significant research on using activated carbon prepared from low cost industrial and agricultural by-products is on a rise. Some of the examples include scrap wood of *Acacia auriculiformis* (Dutta *et al.*, 2011), modified lignocellulosic waste such as pericarp of *Gloriosa superba* (Ramalakshmi *et al.*, 2011a), rice husk and sugarcane bagasse (Yeneneh *et al.*, 2011), jack fruit peel waste (Jayarajan *et al.*, 2011a), oil palm empty fruit bunch (Tan and Hameed, 2010), pomelo fruit peel waste (Jayarajan *et al.*, 2011b) and cross-linked chitosan/oil palm ash composite beads (Masitah *et al.*, 2011).

Biosorption has been defined as the property of certain biomolecules (or types of biomass) to bind and concentrate selected ions/other molecules from aqueous solutions. Thus biosorption using microbial biomass especially, fungal biomass (Ramalakshmi *et al.*, 2011b, c) as potential sorbent for removal of dyes from industrial wastewater has gained considerable attention (Rajendran *et al.*, 2011, 2012).

Gloriosa superba which is available in plenty in Africa, southeastern Asia and parts of Malaysia and now widely cultivated in and around areas of Coimbatore. Seeds of plant contain colchicines alkaloid which is used in treatment of gout disease, also in inducing capillary blood circulation. The seeds are exported to foreign countries for drug preparation, after which pericarp is usually dumped, add up to existing pollution problems. The objective of the present study was to compare and evaluate fast green dye removal from aqueous solutions by batch studies using activated carbon from *Gloriosa superba* pericarp waste and *A. raphani* biomass.

MATERIALS AND METHODS

Preparation and characterization of adsorbent: The pericarp was collected in and around agricultural fields of Coimbatore city, Tamilnadu, India. The samples were cut into small pieces, dried in sunlight until the moisture was partially evaporated and further dried in a hot air oven at 60°C for 24 h. The dried material was ground and sieved. The particle size of 125-250 µm was used for fast green dye removal studies. Physical properties of carbon prepared from pericarp of *Gloriosa superba* (PGS) were analyzed. All the chemicals were of analytical reagent grade procured from Sigma, USA and Merck, India. All the solutions were prepared in double distilled water.

Preparation of inoculum and adsorbate: The fungus was collected from dye industry effluent canals in the textile city, Tirupur, Tamil Nadu. Fungal culture was grown in Czapek-Dox agar medium for 5 days at 27±2°C in petriplates. Czapek-Dox agar medium contained potassium chloride (0.5 g), potassium dihydrogen phosphate (1.0 g), sodium nitrate (2.0 g), ferrous sulphate (0.01 g), magnesium sulphate (0.5 g), yeast extract (1.0 g), dextrose (30.0 g), agar (20.0 g), streptomycin (0.3 g) and distilled water (1000 mL). The plates were then flooded with sterile distilled water, brushed with sterile camel hairbrush smoothly without disturbing the mycelial growth and filtered through sterile filter. The concentration of the filtrate was adjusted to 10⁶ spores/mL with sterile distilled water and used as inoculums for further studies. For the preparation of adsorbent, 1 mL (10⁶ spores) of fungal spore suspension was inoculated in to 100 mL of sterile Czapek-Dox broth (composition is similar to Czapek-Dox agar medium devoid of agar alone) in a 250 mL Erlenmeyer flask and incubated at 27±2°C for 5 days in an orbital shaker at 125 rpm. After the incubation period, the mycelium developed as pellets was separated by filtration through Watman No.1 filter paper and washed with generous amounts of deionized water until free from the media components. The washed, live mycelia pellets were used as biosorbent as such (Sathishkumar *et al.*, 2004).

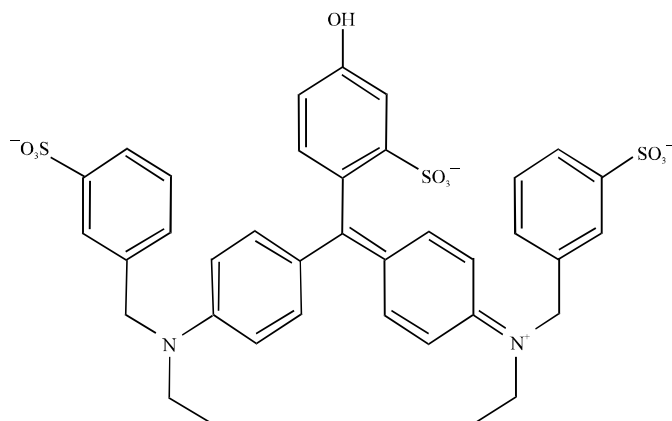


Fig. 1: Structure of fast green dye studied

Adsorbate: Fast green dye was procured from dye industry in Mumbai. The dye structure is given in Fig. 1. Fast green FCF is an anionic sea green triphenylmethane dye. Standard solutions containing 10 to 50 mg L⁻¹ of the dye were prepared by diluting a stock solution of 1000 mg L⁻¹ of fast green dye studied. The analysis of fast green dye was estimated spectrophotometrically by monitoring the absorbance at 620 nm using UV-VIS spectrophotometer (Shimadzu, UV-1601, Japan).

Batch studies: Adsorption experiments were carried out by agitating (100 mg) carbon and (0.5 g) biomass pellets with 50 mL of fast green dye solution of concentration ranging from 10 to 50 mg L⁻¹ and pH 7, 150 rpm and 30±2°C in a thermostated rotary shaker. The flasks were withdrawn at predetermined time intervals. The adsorbate and adsorbent were separated by centrifugation at 3000 rpm for 20 min. The remaining adsorbate concentration in the supernatant was determined from which the amount of dye adsorbed by the adsorbent (q, mg g⁻¹) was calculated. Control experiments were carried out without adsorbent to estimate the adsorbate removal due to adsorption onto the walls of the flasks. It was observed that adsorption onto the container walls were negligible. A study was carried out with different dosages of adsorbent (0.2-1.2 g 50 mL for activated carbon and 0.5-3.0 g 50 mL for fungal biomass) for the equilibrium time to determine the effect of adsorbent dose on fast green removal. The effect of pH on the adsorption process was studied by varying the pH of 50 mL of 50 mg L⁻¹ adsorbate solution in the range of 2 to 9 using dilute HCl and NaOH solutions, while keeping the other experimental parameters at the values described earlier.

Desorption studies: Desorption studies were carried out with adsorbate-laden adsorbent obtained from a batch process, in which the adsorbate solutions (30 mg L⁻¹ of fast green) were treated for the optimum contact time. The dye-laden carbon was washed gently with distilled water to remove unadsorbed dye. Several such samples were prepared. The spent adsorbent was then agitated at 200 rpm for optimum contact time with 50 mL of distilled water and adjusted to different pH values in the range of 2 to 10. The desorbed dye was estimated spectrophotometrically as mentioned earlier. Similarly desorption studies were carried out separately in 50 mL of 0.1-0.5 N NaOH solutions.

All experiments were carried out in duplicate and the mean values are presented. The error obtained was ±2%.

RESULTS AND DISCUSSION

Adsorbent characterization: Characteristics of carbon prepared from pericarp of *Gloriosa superba* pericarp are presented in Table 1. The decolorizing power was 75.9 mg g^{-1} which indicated that the carbon prepared by the activation method had good adsorption capacity and it could be used for adsorption of organic dyes.

Effect of agitation time and initial concentration on dye adsorption: The fast green dye removal increases with time and attains equilibrium at 105 and 60 min for using live biomass of *Alternaria raphani* and activated carbon respectively for all the concentrations studied (Fig. 2a, b). The curves were single, smooth and continuous till the saturation of dye on the carbon surface. Similar results were reported for various dye adsorptions by other adsorbents (Namasivayam and Kavitha, 2002). Equilibrium time is the one of important considerations in the design of water and wastewater treatment systems because it influences the size of reactor, thereby the plant economics (Deepa *et al.*, 2006). The initial rapid phase may be due to availability of more number of adsorption/vacant sites, as a result there exists an increased concentration gradient between adsorbate in solution and adsorbate in the adsorbent (Sathishkumar *et al.*, 2007).

Table 1: Characteristics of carbon

Parameters	Value
pH	7.19
Moisture content	9.4
Ash content	0.282
Decolorising power (mg g^{-1})	75.9
Porosity (%)	84.2
Ion exchange capacity (millieqv/g)	0.2
Apparent density (g mL^{-1})	0.255
Specific gravity	1.618
Water soluble matter (%)	3.75
Acid soluble matter (%)	19.9
Sodium ($\mu\text{g g}^{-1}$)	39.6
Potassium ($\mu\text{g g}^{-1}$)	274

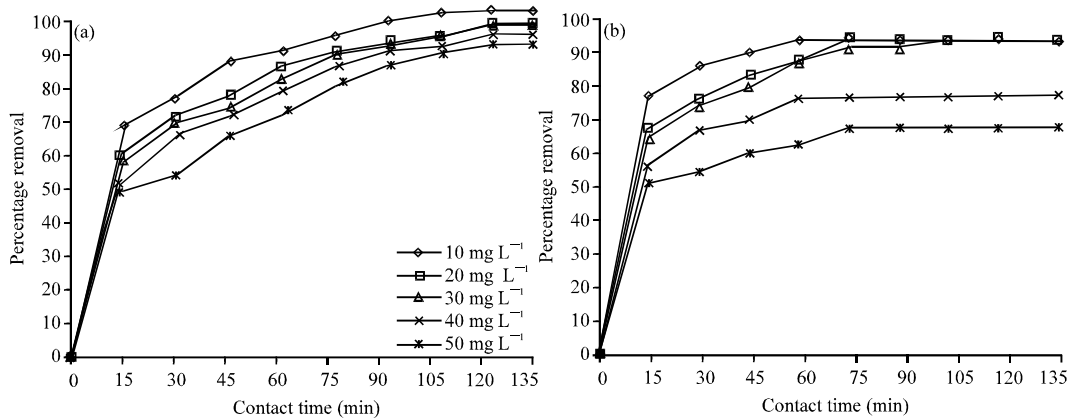


Fig. 2 (a-b): Effect of Agitation time and initial dye concentration of fast green onto (a) Activated carbon and (b) Live biomass

Table 2: Kinetic parameters for the fast green dye adsorption using live biomass at different initial dye concentrations

Initial dye concentration (mg L ⁻¹)	Lagergren model				Pseudo-second-order model		
	(Q _e) _{exp} (mg g ⁻¹)	K ₁ (L min ⁻¹)	Q _e (mg g ⁻¹)	R ²	K ₂ (g mg min ⁻¹)	Q _e (mg g ⁻¹)	R ²
10	10	0.050	10.00	0.9973	0.03	10.33	0.9997
20	20	0.032	20.00	0.9974	6.4	18.79	0.9985
30	30	0.032	30.00	0.9721	2.9	29.00	0.9976
40	37	0.078	32.76	0.8530	5.0	34.72	0.9990
50	46	0.031	43.25	0.9761	3.2	44.91	0.9983

Table 3: Kinetic parameters for the fast green dye adsorption using activated carbon at different initial dye concentrations

Initial dye concentration (mg L ⁻¹)	(Q _e) _{exp} (mg g ⁻¹)	Lagergren model			Pseudo-second-order model		
		K ₁ (L min ⁻¹)	Q _e (mg g ⁻¹)	R ²	K ₂ (g mg ⁻¹ min ⁻¹)	Q _e (mg g ⁻¹)	R ²
10	10	0.042	9.90	0.894	7.7	10.83	0.9986
20	19.87	0.025	19.46	0.9888	2.8	21.74	0.9972
30	30.2	0.025	29.01	0.9874	1.6	32.78	0.9959
40	42.1	0.031	37.60	0.9762	1.2	40.29	0.9969
50	49.5	0.032	45.30	0.9250	6.5	51.94	0.9882

The good adsorption capacity may be attributed to the micropores and mesopores structures present in the prepared carbon (Sathishkumar *et al.*, 2009; Madhavakrishnan *et al.*, 2008).

Adsorption kinetics: To study the adsorption kinetics, two kinetic models were used which included Lagergren and Svenska (1898) and pseudo-second order models. In order to obtain the rate constants and equilibrium dye uptake, the straight-line plots of log were made at different initial dye concentrations.

If the intercept did not equal to the experimental equilibrium dye uptake then the reaction was not likely to be first order even if plot had high correlation coefficient with the experimental data (Ho and McKay, 1998). The rate constants, predicted equilibrium uptakes and the corresponding correlation coefficients for all concentration tested are summarized in Table 2.

For Lagergren plot, correlation coefficients were from 0.853 to 0.9974 but the calculated Q_e was not equal to experimental Q_e, suggesting the insufficiency of the model to fit the kinetic data for the initial concentrations examined. The reasons for these differences in the Q_e values was that there was a time lag, possibly due to a boundary layer/ external resistance controlling at the beginning of the adsorption process (McKay *et al.*, 1999). In most cases, the Lagergren model does not fit the kinetic data well for the whole range of contact time and generally underestimate the Q_e values (Ho and McKay, 1998).

The pseudo-second order model is based on the sorption capacity on the solid phase. Contrary to other well-established models, it predicts the behavior over the whole range of studies and it is in agreement with the chemisorptions mechanism being the rate controlling step (McKay *et al.*, 1999). This was consistent with the better results obtained with the pseudo-second order model (Table 2, 3). Correlation coefficients were always higher than 0.99 and the lowest correlation coefficients in this case was better than the first order model correlation coefficients (Fig. 3 a, b). The values of predicted equilibrium sorption capacities showed reasonably good agreement with the experimental equilibrium uptake value.

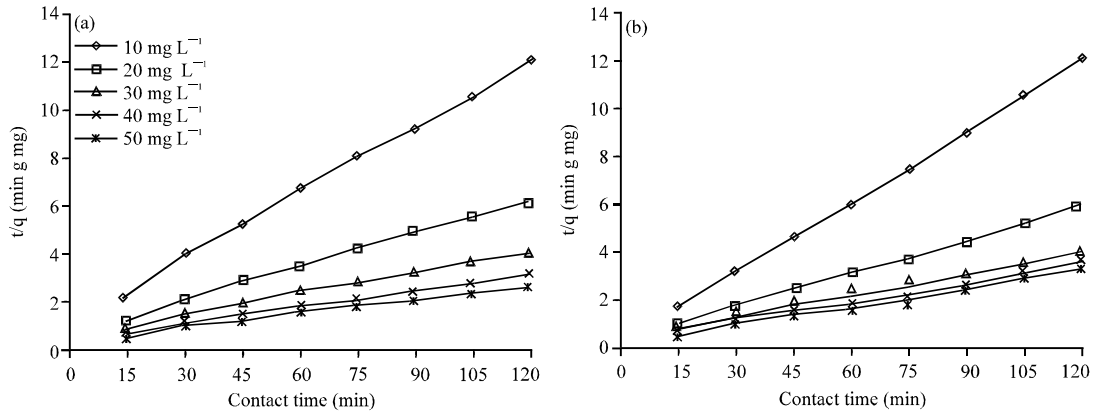


Fig. 3 (a-b): Pseudo second order plots for fast green dye onto (a) Activated carbon and (b) Live biomass

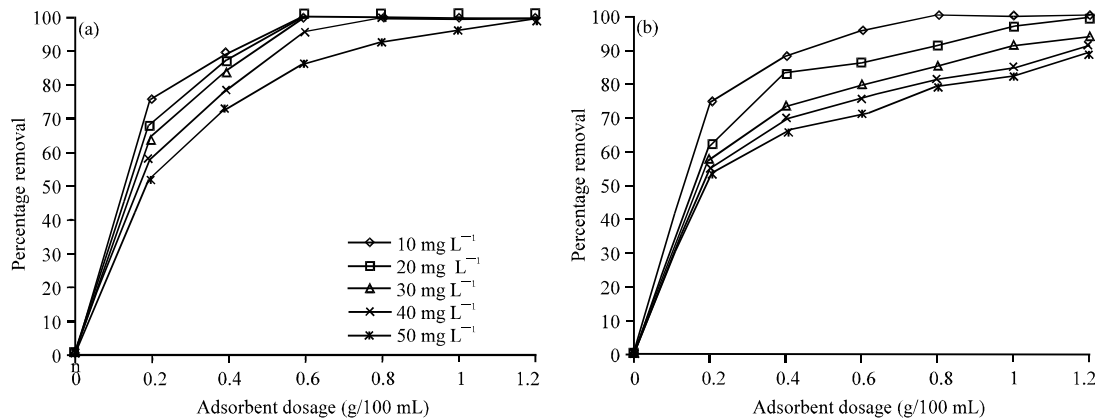


Fig. 4 (a-b): Effect of carbon dosage of fast green dye onto (a) Activated carbon and (b) Live biomass

Effect of carbon dosage on fast green removal: The removal of fast green dye increased with increasing carbon dosage (0.2 to 1.2 g of carbon) and biomass (0.5 to 3.0 mg g⁻¹), respectively (Fig. 4a, b). Increase in adsorbent dosage increased the percentage removal of dye which is due to the increase in adsorbent surface area consequent to the number of carbon particles with more number of active surface sites for the adsorption and the saturation occurs as a result of non-availability of dye molecules for adsorption.

Adsorption isotherms: The data obtained from equilibrium studies were analyzed according to Langmuir and Freundlich adsorption isotherms. The Langmuir and Freundlich equations are commonly used to describe adsorption isotherms at a constant temperature for water and waste water treatment applications. The distribution of dyes between the solid-solution interface equations has been described by the Langmuir equation (Namasivayam and Yamuna, 1995). The well known expression of the Langmuir model is given by Eq. 1:

$$q_e = \frac{Q_o b c_e}{(1 + b c_e)} \quad (1)$$

Table 4: Langmuir and Freundlich model constants for fast green dye

Adsorbent	Langmuir model					Freundlich model		
	Q _o (mg g ⁻¹)	b, (L mg ⁻¹)	R ²	C _o , (mg L ⁻¹)	R _L	K _f , (L mg ⁻¹)	n	R ²
Live biomass	35.59	1.76	0.9974	10	0.008	0.2858	1.536	0.9334
				20	0.004			
				30	0.003			
				40	0.002			
				50	0.001			
Activated carbon	51.02	1.431	0.9893	10	0.065	1.4325	1.9252	1.0000
				20	0.034			
				30	0.023			
				40	0.017			
				50	0.014			

Table 5: Comparison of the Q_o values for fast green dye for various adsorbents

Dyes	Adsorbent	Q _o (mg g ⁻¹)	Reference
Fast green	Pericarp of <i>Gloriosa superba</i>	51.02	This work
	Live biomass of <i>Alternaria raphani</i>	35.59	This work
	Peanut hull	15.60	Gong <i>et al.</i> (2005)
	Tur dal husk	100.39	CES report
	Bengal gram husk	56.06	CES report
	Tamarind husk	101.58	CES report

where, q_e (mg g⁻¹) and C_e (mg g⁻¹) are the amounts of adsorbed dye per unit weight of adsorbent and unadsorbed dye concentration in solution at equilibrium, respectively. Q_o is the maximum amount of the dye bound per unit weight of adsorbent to form a complete monolayer on the surface at high C_e, and b is a constant related to the affinity of the binding sites (L mg⁻¹).

The empirical Freundlich equation based on sorption into a heterogenous surface is given as:

$$q_e = K_F C_e^{1/n} \tag{2}$$

where, K_F and n are the Freundlich constants for the system which are indicators of adsorption capacity and intensity respectively (Aksu and Donmez, 2003).

The values of the Freundlich and Langmuir parameters were obtained respectively from the linear correlation between the values of C_e/q_e and C_e and log q_e and log C_e (Table 4). The linear relationships were evidenced by the R² values (for the Langmuir model, 0.9974 and 0.9893 for live biomass and activated carbon respectively; for the Freundlich model, 0.9334 and 1.0 for live biomass and activated carbon, respectively) (Table 4). This indicates the applicability of the adsorption isotherm and the monolayer coverage on the adsorbent surface.

The Q_o from Langmuir isotherm indicates that the adsorption capacity of fast green dye was greater for activated carbon (51.02 mg g⁻¹) than live biomass (35.59 mg g⁻¹). The observed R_L values indicate favorable adsorption of fast green on activated carbon (0 < R_L < 1). Comparison of the Q_o obtained from this study and other adsorbents was presented in Table 5.

Effect of pH on fast green dye removal: Adsorption of fast green dye decreased with increasing pH. Lower adsorption of fast green dye at alkaline pH was probably due to the presence of excess

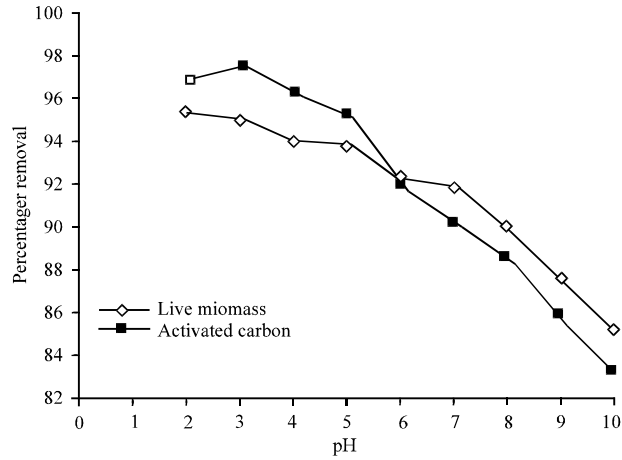


Fig. 5: Effect of pH of fast green dye onto activated carbon and live biomass

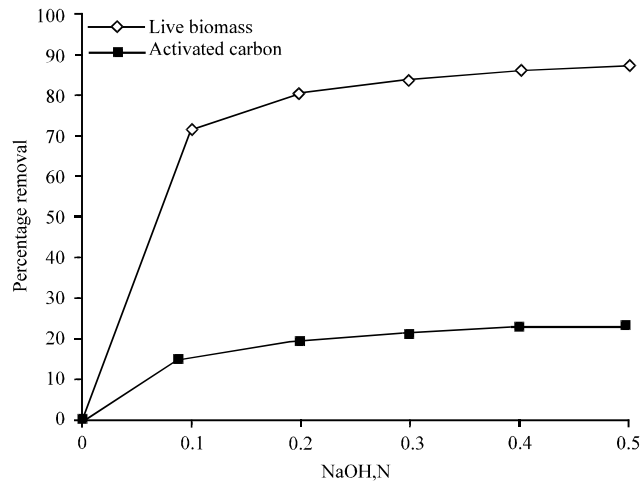


Fig. 6: Desorption studies of fast green dye onto activated carbon and live biomass

of hydroxyl ions competing with the dye anions for the adsorption sites (Fig. 5). The anionic dyes namely amaranth, fast green and sunset yellow was maximally adsorbed by powdered peanut hull at pH 2.0 (Gong *et al.*, 2005).

Desorption studies: Regeneration of the adsorbent may take the treatment more economical. Attempts were made to degenerate color from the dye laden carbon using various strengths of NaOH (0.1-0.6 N). The percent desorption increased with increasing NaOH concentration in the aqueous medium (Fig. 6) and attained a maximum desorption at 0.6 N NaOH solution. The effect of percentage desorption was inversely correlated to pH effect, indicating that ion exchange was probably the major mode of adsorption process. Similar results were observed for the adsorption of Congo red by coir pith carbon (Namasivayam and Kavitha, 2002).

CONCLUSION

The comparative capability of activated carbon from Pericarp of *Gloriosa superba* and biomass of *Alternaria raphani* was examined for removal of fast green dye removal through equilibrium

and kinetic studies. Based on the experimental data, activated carbon from Pericarp of *Gloriosa superba* had higher dye adsorption efficiency on fast green dye removal. The Langmuir and Freundlich isotherm models were used to investigate the adsorption capacity of fast green dye removal, where adsorption capacity was found to be 51.02 and 35.59 mg g⁻¹, respectively for activated carbon and live biomass. The adsorption kinetics of fast green dye obeyed the second order adsorption model. Pericarp of *Gloriosa superba* is an agricultural waste after the seeds have been used for medicinal properties. Thus as a low-cost adsorbent, has potential application of pericarp carbon for fast green dye removal due to high availability, disposed problem and also higher adsorption capacity at lower concentration.

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