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ARTICLE

Removal of Metanil Yellow from Aqueous Solution by Batch Sorption on *Caladium bicolour* Corm and Egg Shell: Effect of Nature of Sorbents

Beniah O. Isiuku[1,✉], Ozioma U. Akakuru[2], Festus Emengaha[3] and Benjamin I. Nwabueze[1]

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ABSTRACT

Caladium bicolour is a stubborn weed in farmlands while eggshell biomass is an agricultural waste of no economic importance. Batch sorption of metanil yellow on the biomasses was carried out with a view to determine the effect of nature of sorbents. Effects of initial dye concentration, initial pH and sorbent dosage were investigated. Experimental results were analyzed with three isotherms and three kinetic models. The Boyd model was used to determine the rate-determining step. The *Caladium bicolour* corm contains functional groups while eggshell contains pores. The highest sorption capacities were 3.995 and 1.385 mg/g for *Caladium bicolour* and eggshell, respectively; the optimum pH was 5. Sorption decreased with increase in initial concentration for *Caladium bicolour*, while for eggshell sorption increased with increase in initial concentration. The isotherm and kinetic models applied were good fits. The sorption was controlled by liquid film diffusion. Sorption on *Caladium bicolour* was more than twice that of eggshell. However, the sorptions on both sorbents were low.

KEYWORDS

Batch sorption, Eggshell, *Caladium bicolour*, Metanil yellow.

Author affiliations:

[1]Department of Chemistry, Imo State University, PMB 2000, Owerri, Nigeria
[2]Department of Pure and Applied Chemistry, University of Calabar, Calabar, Nigeria
[3]Department of Clinical Biochemistry, Imo State University, PMB 2000, Owerri, Nigeria

✉To whom correspondence to be addressed:

Tel: + 234 80 35731300
E-mail: obinnabisiuku@yahoo.com

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INTRODUCTION

Dyes are organic compounds made up majorly of chromophores which give colours to the dyes and auxochromes which intensify the colours [1]. Synthetic dyes possess complex aromatic structures that make them stable. The high stability makes these dyes resist degradation [2]. Close to 12 % of artificial dyes are lost during production in industries. From this amount 20 % is received by water bodies as industrial liquid effluent [3].

Metanil yellow is an artificial anionic azo dye used as a biological stain. It is used to impart colour on cosmetics, silk, wool, nylon, paper, fur, ink, detergent and aluminium. Metanil yellow is harmful on ingestion and slightly harmful when inhaled or contacts the eyes [4]. Oral feeding of metanil yellow in rats causes intra-peritoneal and intra-testicular lesions, making seminiferous tubules to suffer damage and reducing the rate of spermatogenesis. On oral intake of metanil yellow, humans suffer methaemoglobinaemia and cyanosis while skin contact causes allergic dermatitis. Metanil yellow is not mutagenic but can change expression of genes. It has tumour-producing potential and can cause intestinal and enzymic disorders in humans [5-7].

Pollution of water bodies with dyes is seriously problematic in water quality. It destroys aesthesy and prevents penetration of light into the water thereby hindering photosynthesis [8,9]. Hen eggshell is made up of the membrane, comprising interwoven protein fibres and spherical masses, and the calcified eggshell made of calcite or calcium carbonate crystals [8]. The chemical components of calcified eggshell is 94 % $CaCO_3$, 1 % Mg CO_3, 11 % Ca_3PO_4 and 4 % organic material. The calcified eggshell is porous containing 7000-17000 pores. Its porous nature qualifies it to serve as a sorbent [9-11]. Eggshell has high nutrition contents such as calcium and magnesium [12]. The mineral content makes eggshell potentially useful as fertilizer, soil conditioner or as an additive for animal feeds [13]. Eggshell weighs about 10-11 % of egg which is about 60 g [9,14].

Water plants are hindered from producing their food as a result of preventing photosynthesis due to the fact that sunlight does not penetrate the water bodies [15,16]. Several techniques have been developed for removing dyes from wastewaters, which include filtration, sorption, coagulation, advanced oxidation and ozonation [17]. Sorption has been proved to be the best due to its low cost, flexibility, simplicity of design, ease of operation and insensitivity to toxic pollutants [18]. Batch operation is applied easily in laboratory treatment of wastewater in small volumes. It provides some basic information such as initial pH, sorbent dosage, initial sorbate concentration and sorbent particle size for maximum sorption [19]. Several researchers have worked on the removal of dyes from wastewater. Pramanpol and Nitayapat [10] used eggshell and its membrane to remove reactive dyes, Ehrampoush *et al*. [20] successfully removed Reactive Red 123 dye from aqueous solution with eggshell. Salman *et al*. [21] used eggshell and its membrane to remove three different types of dyes. Tsai *et al*. [8] applied powdered calcified eggshell to remove cationic basic blue 9 and anionic acid orange 51 from aqueous solution. Hassan and Salih [9] used eggshell to remove methylene blue from aqueous solution. Horsfall and Spiff [22] used *C. bicolour* to remove heavy metals successfully from aqueous solutions.

The population of Nigeria is growing and many urban cities are coming up. This has generated many industries that produce and use hen eggs. Such industries include beverages, food and hatcheries. Being a developing country, Nigeria generates a huge amount of eggshell as waste. *Caladium bicolour* is a tuberous perennial plant with brightly coloured foliage in warm shady areas. The corm is inedible due to intense irritation it produces in the throat [23]. Profitable uses of *C. bicolor* corm will not only bring about the production in large quantity and practical exploitation of the plant but also encourage local farmers and boost their economy. *C. bicolor* contains a lot of cellulose fiber, protein and some functional groups such as carboxyl and hydroxyl which make sorption possible [22].

Several articles have been reported using *C. bicolour* and calcified eggshell in removing different pollutants, but work on the use of eggshell and *C. bicolour* in removing metanil yellow is not yet reported. The aim of this work was to evaluate the efficiency of calcified eggshell and *C. bicolour* in removing metanil yellow, an anionic azo-dye from aqueous solution, with a view of determining the effect of nature of the biomasses on sorption.

EXPERIMENTAL

Sorbate: The metanil yellow (M/s Merck, Switzerland) was purchased at Onitsha, Anambra State, Nigeria and used without further treatment. The stock solution was prepared by dissolving 1 g dye/L solution with distilled water.

Sorbents: The eggshells with the membranes were collected from restaurants within and around Imo State University, Owerri, Nigeria in polyethylene bags. The *C. bicolour* corms were obtained from Mpam, Ahiazu local Government Area of Imo State, Nigeria.

Preparation of sorbent: The eggshell biomass was washed in a plastic bucket thrice with warm distilled water to remove dirt and soil particles. The biomass was boiled in distilled water for 30 min and allowed to cool. While soaked in water, the membranes were peeled off. The calcified eggshell biomass was rinsed twice with distilled water and spread on a plastic surface for water to drain off. The biomass was dried in a hot-air oven at 80 ºC for 24 h. After cooling, the biomass was ground and sieved to obtain 0.42-0.84 mm particles. The sorbent obtained was packed in a plastic container. The *C. bicolor* corms were washed with distilled water to remove dirt and soil. They were sliced into small pieces and spread under the sun for 5 days to dry. They were further dried in a hot-air oven at 70 ºC for two days. The crispy biomass was ground and sieved to obtain 0.42-0.84 mm particles which were packed in a plastic vessel.

Characterization of *C. bicolor*: Sample of *C. bicolour* and eggshell were proximately analyzed using AOAC 1990 [24] method for moisture, lipid, protein, fibre, carbohydrate, ash and some mineral contents. The infrared spectrum of *C. bicolour* was obtained using (FTIR-8400S, M/S Shimadzu, China). The surface structure of eggshell was examined with a scanning electron microscope (SEM model Phenom Prox, M/s Phenom world, Netherlands).

Batch sorption

Effects of initial dye concentration and contact time: 50 mL portions of metanil yellow solution of initial concentration (C_o) 50, 100 and 200 mg/L at pH 5 and temperature 30 ºC were contacted with 0.2 g portions of *C. bicolor* in 100 mL conical flasks in a water-bath at a speed of 120 rpm. A sample flask was removed at 1 h intervals for a total time of 7 h. Clear solution from sample flask was syringed and the clear filtrate was analyzed with UV/visible spectrophotometer (Shimadzu model 752, M/s Shimadzu, China) at 440 nm. The experiment was repeated replacing *C. bicolor* with calcified eggshell.

Effect of initial pH: 50 mL portions of metanil yellow solution of initial concentration (C_o) 50 mg/L were measured into five 100 mL conical flasks. The flasks contents pH were adjusted to pH 2, 4, 6, 8 and 10 with 1 M HCl and 1 M NaOH solutions.Eggshell (0.2 g) was quickly measured into each of the flasks which was then stoppered, put in the water-bath shaker and agitated at 30 ºC and speed of 120 rpm for 5 h. A sample flask was removed every hour, syringed and analyzed with UV/visible spectrophotometer.

Effect of sorbent dosage: 50 mL portions of metanil yellow solution of initial concentration (C_o) 50 mg/L were measured into six 100 mL conical flasks. 0.2-2.5 g portions of eggshell were added into the respective flasks. The flasks were stoppered and quickly put in the water-bath shaker which agitated at 30 ºC and speed of 120 rpm for 5 h. A sample flask was removed each hour, syringed and analyzed as above.

RESULTS AND DISCUSSION

Characterization of *C. bicolour* and eggshell: Tables 1 and 2 show the proximate analysis data and some minerals content of *C. bicolour* and hen eggshell. The FTIR spectrum (Fig. 1) shows the peaks at 1010.73, 1627.97, 2924.18 and 3271.38 cm^{-1} representing C-O (in esters and alcohols), C=O (in esters), NH (in amides) and (H- bonded OH in alcohols) functional groups, respectively present in lipid, carbohydrate and protein. The functional groups are responsible for the ability of *C. bicolour* to sorb. Physical properties and surface structure of adsorbents influence their sorption properties. Scanning electron micrographs of eggshell (Figs. 2 and 3) show that eggshell has pores which are not well developed. The pores are responsible for the sorption.

TABLE-1
PROXIMATE ANALYSIS DATA OF *C. bicolour*

Parameter	Value	Parameter	Value
Ash content (%)	7.585	Calcium (mg/kg)	25.23
Moisture (%)	12.03	Magnesium (mg/kg)	5.93
Lipid (%)	1.82	Potassium (mg/kg)	3.34
Fibre (%)	2.11	Sodium (mg/kg)	5.5
Crude protein (%)	1.05	Phosphorus (mg/kg)	12.48
Carbohydrate (%)	7.4		

TABLE-2
PROXIMATE ANALYSIS DATA OF HEN EGGSHELL

Parameter	Value
Ash content (%)	95.85
Total organic matter (%)	4.15
Trimagnesium phosphate (mg/kg)	354.33
Tricalcium phosphate (mg/kg)	418.66
Calcium carbonate (mg/kg)	1268.40
Magnesium carbonate (mg/kg)	552.80
Calcium (mg/kg)	447.60
Magnesium (mg/kg)	134.40
Potassium (mg/kg)	73.20
Sodium (mg/kg)	110.00
Iron (mg/kg)	9.20

Effect of initial dye concentration and contact time: The effect of initial concentration (C_o) and contact time on the sorption of metanil yellow on *C. bicolour* and eggshell at 30 ºC and pH 5 is shown in Figs. 4 and 5. In both cases, q_t increased with increase in contact time; q_t decreased (3.995-1.558 mg/g) with increase in C_o for *C. bicolour* and increased (0.383-1.345 mg/g) with increase in C_o for eggshell. However, q_e for C_o 100 and 200 mg/L were almost equal for *C. bicolour*. For eggshell, C_o 50 and 100 mg/L showed similar sorption patterns. The *C. bicolour* showed a better sorption than eggshell though both sorbents showed low sorptions. The decrease in q_e with increase in C_o at pH 5 might be as a result of competition for low number of positively charged sorption sties. At pH 5, the medium does not generate abundant protons to protonate the surface of the sorbent upon which the sorbate anions compete for the funnel-shaped pores leading to decrease in q_e as C_o increased (Tables 3 and 4).

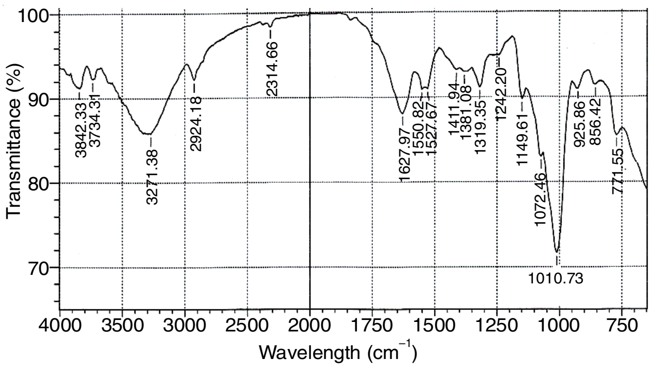

Fig. 1. FTIR spectrum of *C. bicolour*

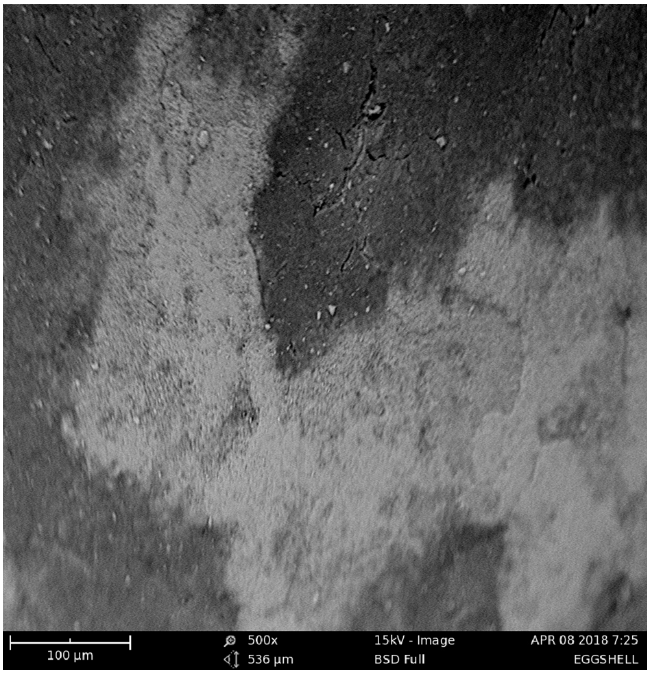

Fig. 2. Scanning electron micrograph of eggshell at 500x

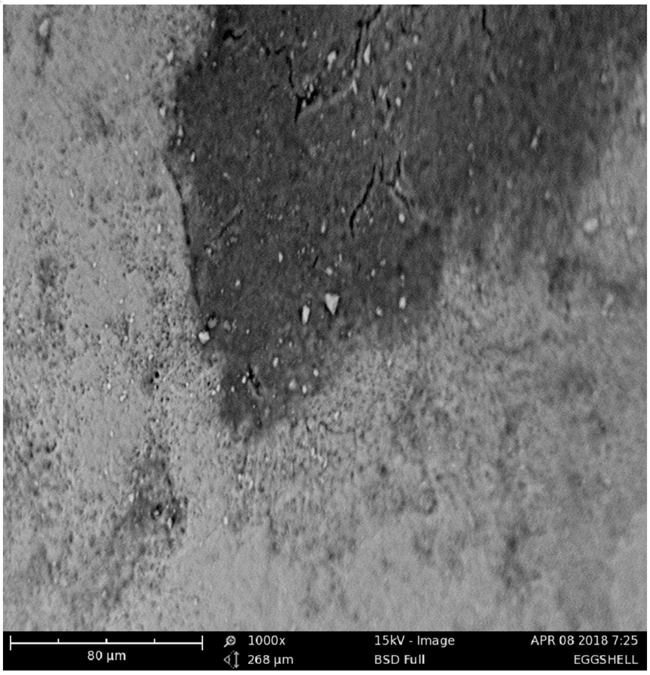

Fig. 3. Scanning electron micrograph of eggshell at 1000x

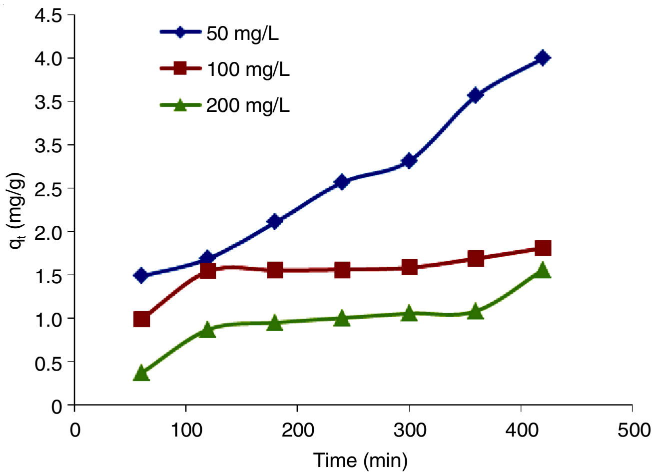

Fig. 4. Batch sorption of metanil yellow on *C. bicolor* at various initial dye concentrations

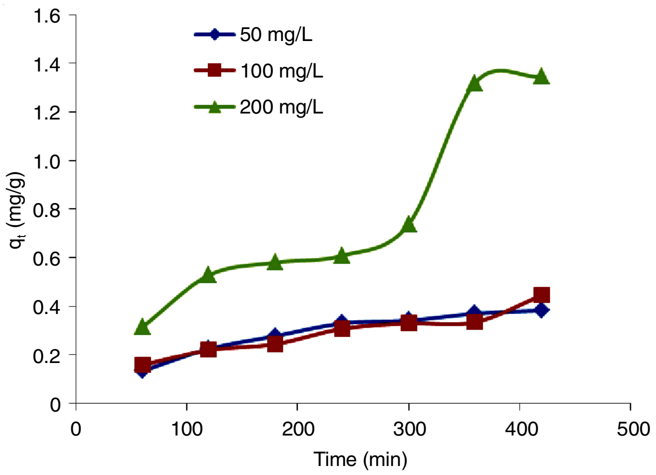

Fig. 5. Batch sorption of metanil yellow on eggshell at various initial dye concentrations

TABLE-3
KINETIC MODELS PARAMETERS FOR THE SORPTION OF METANIL YELLOW ON *C. bicolor* AT 30 °C AND pH 5

Model	Parameter	C_o (mg/L)		
		25	50	100
PFO	q_e (mg/g)	3.995	1.808	1.558
	q_o (mg/g)	4.275	1.358	1.114
	k_1 (min^{-1})	0.0053	0.0048	0.0027
	R^2	0.8408	0.7577	0.8077
	SSE	0.106	0.17	0.167
PSO	$q_{e\ exp.}$	3.995	1.808	1.558
	q_o (mg/g)	6.105	1.954	2.084
	k_2 (g/mg/min)	0.00057	0.01	0.002
	R^2	0.8017	0.988	0.7784
	SSE	0.797	0.055	0.199
Elovich	α	26.835	0.353	5.085
	β	0.795	2.915	2.137
	R^2	08509	0.8345	0.838
Boyd	B	0.0026	0.0042	0.0023
	Di × 10^{-9} (m^2/min)	3.286	5.309	2.907
	R^2	0.9574	0.6194	0.8849

Effect of sorbent dosage: The effect of sorbent dosage on the sorption of metanil yellow on eggshell is shown in Fig. 6. Equilibrium sorption capacity decreased with increase in sorbent dosage. As sorption capacity increased from 0.4 to 5 %,

TABLE-4
KINETIC MODELS PARAMETERS FOR THE SORPTION OF METANIL YELLOW ON EGGSHELL AT 30 °C AND pH 5

Model	Parameter	C_o (mg/L)		
		25	50	100
PFO	$q_{expt.}$ (mg/g)	0.383	0.445	1.345
	q_o (mg/g)	2.031	2.903	1.105
	k_1 (min-1)	0.0093	0.0034	0.0019
	R^2	0.9635	0.9616	0.9229
	SSE	0.622	0.929	0.089
PSO	$q_{e\ exp.}$	0.383	0.445	1.345
	q_o (mg/g)	0.552	0.481	1.001
	k_2 (g/mg/min)	0.102	0.018	0.038
	R^2	0.9951	0.8321	0.9575
	SSE	0.064	0.013	0.13
Elovich	α	0.128	0.128	4.216
	β	7.813	7.813	2.4
	R^2	0.8815	0.8815	0.6524
Boyd	B	0.0071	0.0032	0.0012
	D_i × 10^{-9} (m^2/min)	8.974	4.045	1.517
	R^2	0.9815	0.967	0.9281

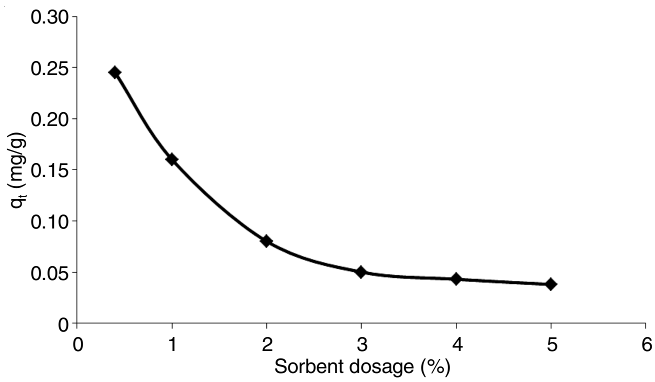

Fig. 6. Batch sorption of metanil yellow on eggshell at various sorbent dosages

q_e decreased from 0.245 to 0.038 mg/g. This observation is supported by the work of Kumar *et al.* [25]. As the ratio of sorbent concentration to that of sorbate increased, the superficial sorption of sorbate onto the sorbent surface increased creating a lower sorbate concentration ratio. The reason for this phenomenon is that for a fixed mass of sorbent, a fixed quantity of sorbate could be sorbed. Hence, the higher sorbent dosage, the larger effluent volume that could be treated by a given mass of sorbent. The decrease in q_e with increase in sorbent dosage was due to split in concentration gradient between sorbate concentration in aqueous phase and in sorbent surface [25,26].

Effect of initial solution pH: Initial solution pH is an important factor in sorption processes. The efficiency of sorption process depends on the sorbate solution pH in that pH change causes variation in the degree of ionization of sorbate particles and sorbent surface properties [27,28]. In Fig. 7, the effect of initial solution pH on the sorption of metanil yellow on eggshell at fixed C_o and sorbent dosage was observed. The best pH was pH 5. From pH 2 to 5, q_e increased (0.138-0.245 mg/g). Further increase in pH caused drop in q_e. In an acid medium, calcite reacts and ionizes to generate H_2CO_3, HCO_3^- and CO_3^{2-}, the proportions of which are determined by the pH of resulting solution. The lower the pH of the solution, the higher concentration of HCO_3^- and CO_3^{2-} which are negatively charged, and the

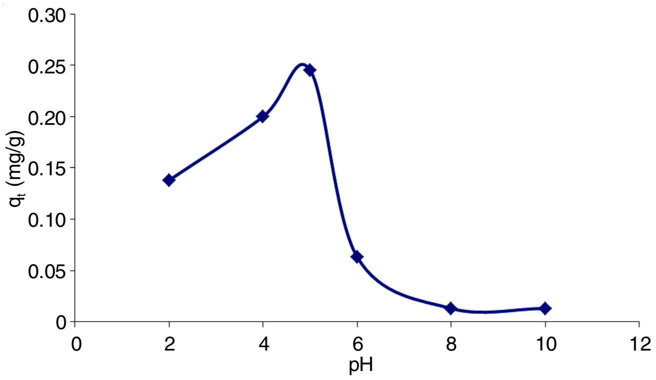

Fig. 7. Batch sorption of metanil yellow on eggshell at various pH values

more the repulsion of metanil yellow ions leading to decrease in q_e [10]. Above dye solution pH 5, the ^-OH ions on sorbent surface increased causing repulsion between anionic sorbate ions and sorbent surface leading to decrease in q_e.

Conclusion

Batch sorption of metanil yellow from aqueous solution on unmodified *Caladium bicolour* corm and eggshell was carried out to evaluate the effect of the nature of sorbents. The eggshell is porous and hard while *Caladium bicolour* corm is soft, and has functional groups. Sorption on unmodified *Caladium bicolour* was higher than for eggshell. Sorption on *Caladium bicolour* increased with increase in time, but decreased with increase in initial dye concentration while for eggshell, sorption increased with increase in time and initial dye concentration. Sorption increased with decrease in sorbent dosage. Langmuir, Freunlich and Temkin isotherms, as well as pseudo-first order, pseudo-second order and Elovich kinetic models analyzed the experimental results well (Table-5). The sorption was controlled by liquid film diffusion. Though sorption on unmodified *Caladium bicolour* corm was higher than on eggshell, the sorptions were very low.

TABLE-5
ISOTHERM PARAMETERS FOR SORPTION OF METANIL YELLOW ON *C. bicolour* AND EGGSHELL AT 30 °C

Model	Parameters	Values	
		C. bicolor	Eggshell
Langmuir	K_L (L/mg)	0.034	0.006
	q_m (mg/g)	1.316	1.618
	R_L	0.128	0.455
	R^2	0.9966	0.7509
Freundlich	1/n	0.634	0.9002
	n	1.578	1.111
	K_F [mg/g(L/mg)$^{1/n}$]	40.185	102.453
	R^2	0.8886	0.8354
Temkin	A_T (L/g)	57.037	3.657
	b_T (J/mol)	1532.6	3654.638
	R^2	0.8522	0.7951

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