Equilibrium Study of Low-cost Bio-adsorbents During Adsorption of Synthetic Industrial Dyes

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***Abstract*— The adsorptive power of organic waste products, namely, potato peel, mosambi peel and banana peel was investigated using three separate dye solutions in demineralised water. The dyes used were Methylene Blue, Malachite Green and Methyl Orange. The adsorption capacity was quantified by studying the relative change in concentration of the solutions using UV spectrometry. Mosambi peels were found to have the best adsorption capacity with regards to Malachite Green and Methylene blue and the process followed Redlich-Peterson adsorption isotherm for the former and Temkin adsorption isotherm for the latter dye. For Methyl Orange, the best adsorption capacity was shown by potato peels where the process followed Langmuir adsorption isotherm.**

***Keywords*— Adsorption, dye removal, bio-adsorbents, synthetic dyes, adsorption isotherm.**

I. INTRODUCTION

More than 800-kilotonnes of synthetic dyes are produced every year, catering to various types of industries [1]. These dyes are of various types- acid dyes, basic dyes, azo dyes, reactive dyes, etc. The notable consumers of this product are the textile industry, printing industry, food and pharmaceuticals industry among others. The wide range of available colour shades and longevity are two major reasons why synthetic dyes have dominated the market over natural dyes. However, these do come with their own set of problems. In most cases, it is economical for the industries to dump the excess solution of dyes in water bodies (hydrosphere) after their primarypurpose is served. Initially, after disposal, these solutions cause a change in the colour of the water, increase the chemical oxygen demand, interfere with the transmission of solar rays into the water body and reduce photosynthetic activity. Thereafter, the dye solution runs off into the other spheres of the environment and enters various living organisms at various levels of the food chain through the processes of bioaccumulation and biomagnification. These dyes can cause severe irritation, adversely affect the brain, lungs, kidney and nervous system besides, they have been reported to be carcinogenic and teratogenic.

Commercial dyes like methylene blue have been reported to cause the serotonin syndrome while malachite green can be toxic at various levels of consumption [1-9]. The azo dyes are highly resistant to degradation but under certain conditions, can degrade into various aromatic amines under reductive conditions and about 24 of these amines have been confirmed to be carcinogenic according to EU Directive 2002/61/EC and 2003/3/EC.

Present treatment methods for the removal of these dyes include chemical methods (oxidation, electrolysis etc.), physical methods (adsorption, filtration, flocculation etc.) and biological methods (micro-organism, enzymes etc.). Most of these methods are effective however it is always desirable to look into more economical alternative methods. Adsorption onto agricultural by-products is one such method that is presently being investigated. The key requirements to be a good adsorbent include- presence of strong but easily dissociable adsorption forces, availability of sufficient no. of adsorption sites, the durability of adsorbents under operating conditions and easy availability [10]. The use of by-products essentially serves two purposes, firstly it mitigates the problem of by-product disposal and secondly, it increases the usability of these by-products by utilising them for the purpose of dye removal from wastewater.

Commercial activated carbon (CAC) and indigenously prepared activated carbon (IPAC) from bio-based sources were investigated for the removal of Congo red [11]. It was found that IPAC's can replace CAC's as adsorbents. Activated carbon from coir pith, an agricultural waste, showed adsorption capacity of 6.7 mg/gm with Congo red [12], while bagasse fly ash (BFA) was also a good adsorbent for thesame. The equilibrium data for BFA followed the R-P isotherm [13]. Coir pith also gave a satisfactory result with uranium removal from aqueous solution at acidic pH [14]. The equilibrium data fit the Langmuir and Freundlich isotherms. Rice husk is a common material of investigation for the purpose of dye adsorption [15-21]. The husk may be used without modification or may be activated by steam [16]. Fruit and vegetable peels have also shown good capacity of dye removal [21-22]. Other materials that showed good adsorption capacity included teak tree bark (333.33 mg/gm), wheat straw (71.43 mg/gm), hazelnut shell (76.9 mg/gm) , Bengal gram seed, rubberwood saw dust (35 mg/gm), eucalyptus saw dust, cashew nut shell among others [23-28]. Perlite, a volcanic siliceous rock, could also be used as an adsorbent [29]. Researchers have also used hybrid hydrogels to remove silver from aqueous solutions. With certain modifications, these gels can possibly be employed to remove dyes [30]. In a related development, the disposal of red mud from the bauxite mining industry is a problem and thus, it was important to find an alternate use for the material. Its use as an adsorbent for Congo red was investigated. The adsorption capacity was 4.05mg/gm with ion-exchange being the mechanism of adsorption [31]. Bacterial decomposition of the highly resistant azo dyes is also a subject of interest [32]. In most of the cases, the Langmuir isotherm fit the equilibrium data sets while in some cases the Freundlich or R-P isotherm was a better fit.

Although many different types of agricultural wastes have been previously investigated for their usefulness as adsorbents, it is prudent to look into newer material. In primarily agrarian countries where fruits and vegetables are a substantial part of the domestic produce, fruit and vegetable peels are abundantly available. For eg. in India, among fruits, banana and citrus fruits (eg.Mosambi) are the two major produces while India ranks second in global potato production and hence, it is hugely beneficial if the peels of these products give satisfactory result as adsorbents. This work is a comparative study on the adsorption capacity and dye removal capability of three bio-adsorbents prepared from potato, mosambi and banana peels studied with three different synthetic dyes-Methylene blue, Malachite green and Methyl orange.

II. Materials And Methods

A. Preparation of the bio-adsorbents: The raw materials, i.e. the potato peel, mosambi peel and banana peel were agricultural waste materials obtained from vegetables purchased from the local markets of Kolkata, West Bengal, India. The peels were dried in the sun for 2 weeks followed by drying in a vacuum hot air chamber for 4 hours. These peels were then ground and sieved. The ground materialcollected from the -100 +140 mesh was used as the adsorbent.

B. Dyes used for experiment: RANKEM Chemicals supplied

the dyes used for the study. The three dyes (adsorbate) used were:

1) **Methylene Blue**, also known as methylthionium chloride. It is used as a dye and in medication.

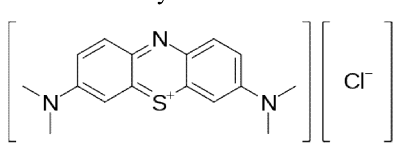

Fig. 1Chemical structure of Methylene Blue

2) **Methyl Orange**, an azo-dye which is also used as a pH indicator.

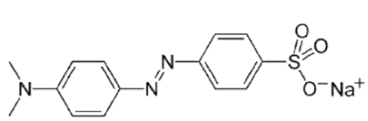

Fig. 2Chemical structure of Methyl Orange

3) **Malachite Green**, which is a cationic dye that is sometimes used as an antimicrobial. The dye is not prepared from malachite, but it is named so because of the similarity of colour.

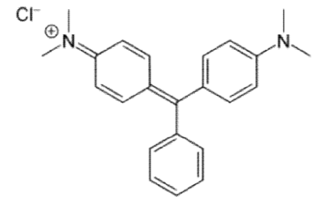

Fig. 3 Chemical structure of Malachite Green

C. Analysis of dyes and adsorbents: The concentration of the dyes in the aqueous solution was determined using a UV-spectrophotometer - Thermo Scientific Evolution 60S model. Scanning Electron Microscopy and Electron Dispersive X-ray Spectroscopy of the bio-adsorbents were done using Zeiss Evo60 SEM with Oxford EDS detector.

D. Sorption Procedure: Adsorption experiments were carried out using 2.5 g/L of adsorbent dose. This adsorbent dose was kept constant for all the concentrations of all the dyes. Five different aliquots of concentrations 50 mg/L, 100 mg/L, 150 mg/L, 200 mg/L and 250 mg/L for all three dyes were prepared and studied. Each of the dye solutions were dosed with the adsorbents and stirred manually for one hour after which they were kept resting for 24 hours.

Thereafter the adsorbent particles were separated from dye solutions after the adsorption operation by filtration using Whatman filter paper following which, the dye solutions were tested under UV spectrophotometer to determine final concentration of the dyes remaining in solution.

E. Adsorption Isotherm models:

a) Langmuir isotherm

Designed by Irving Langmuir in 1918, this is one of the simplest and most popular isotherms used mainly for gases adsorbed on solids. It is semi-empirical with a kinetic basis and has been derived from statistical thermodynamics [33].

The isotherm can be represented in a linear form as:

$$\frac{1}{q_e} = \frac{1}{q_o} + \frac{1}{q_o C_e K_l} \ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where,

q_e= The amount of adsorbate adsorbed per gram of the adsorbent (mg/g)

q_0= Maximum monolayer coverage capacity (mg/g)

C_e = Equilibrium concentration of adsorbate (mg/L)

K_l = Langmuir isotherm constant (L/mg)

The values of q_0 and K_l can be obtained from the slope and intercept of the Langmuir plot of $\frac{1}{q_e}$ vs. $\frac{1}{C_e}$. R_L is a dimensionless constant known as separation factor or equilibrium parameter.

$$R_L = \frac{1}{1+(1+K_L c)} \dots\dots\dots\dots\dots\dots\dots (2)$$

Where c = initial concentration.
The separation factor can help determine the nature of the adsorption:

i) $R_L = 0$ indicates the adsorption is irreversible
ii) $0<R_L<1$ indicates the adsorption is favourable
iii) $R_L = 1$ indicates the adsorption is linear
iv) $R_L>1$ indicates the adsorption is unfavourable

b) Freundlich Isotherm

This is an empirical isotherm which assumes that the amount adsorbed at equilibrium has a power-law dependence on

partial pressure p of solute-

$$q_e = K_f . p^{\frac{1}{n}} \dots\dots\dots\dots\dots\dots\dots\dots (3)$$

$$\ln q_e = \ln K_f + \frac{1}{n} \ln c_e \dots\dots\dots\dots\dots (4)$$

K_f, n are adjustable parameters. This is a practically viable representation but thermodynamically inconsistent [33].

c) Temkin Isotherm

This is a three-parameter type empirical isotherm assumes that the heat of adsorption of all molecules in a layer would decrease linearly, rather than logarithmically, i.e. there is a uniform distribution of binding energy [33].

Governing equation:

$$q_e = \frac{RT}{b}(\ln A_T c_e) \dots\dots\dots\dots\dots\dots\dots (5)$$

$$q_e = B\ln(A_T) + B\ln(C_e) \dots\dots\dots\dots\dots (6)$$

where,

$$B = \frac{RT}{b},$$

R = Universal gas constant = 8.314 J/mol/K,

and T = Temperature at 298K.

The adjustable parameters are- A_T, b.

d) Redlich-Peterson Isotherm

Redlich-Peterson (RP) Isotherm is another hybrid of the Langmuir and Freundlich isotherm [33].

$$q = \frac{A}{(1+b_{RP} * c^k)} \dots\dots\dots\dots\dots\dots\dots (7)$$

This can be also be written as:

$$\ln\left(A\frac{c_e}{q_e} - 1\right) = K \ln c_e + \ln b_{RP} \dots\dots (8)$$

$$q_e = \frac{A * c_e}{1 + c_e^K * b_{RP}} \dots\dots\dots\dots\dots\dots\dots (9)$$

A, b_{RP}, and k are RP constants and
$\frac{A}{b_{RP}}$ = adsorption capacity.

III. Results And Discussion

A) Characterisation of adsorbents: Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS) analysis was performed on the adsorbents to study their surface morphology and elemental composition. It can be seen from fig. 3.1 to 3.3 that the potato peel has a rather random and dispersed microstructure with non-uniform microparticle size. The mosambi peel has a very conical and elongated micro-structure which allows for a larger surface area than the other adsorbents. The banana peel shows agglomeration in various places which reduce the effective available surface area available for adsorption.

EDS study was performed to detect the major elements present on the adsorbents. Carbon and oxygen were found to be the major components, as expected in a bio-entity. Additionally, other than those majorly expected, no unusual elements were present thus proving that there was no contamination of the adsorbents during the drying and grinding process and there weren't present, any element that could have negatively interfered with the readings. Presence of oxygen in substantial amounts may aid in the adsorption process since there is the availability of lone electron pairs.

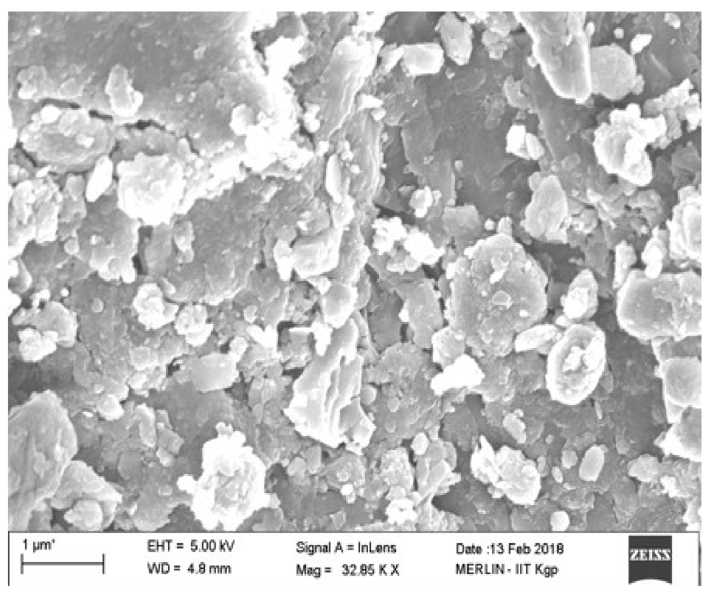

Fig. 4SEM image of potato peel

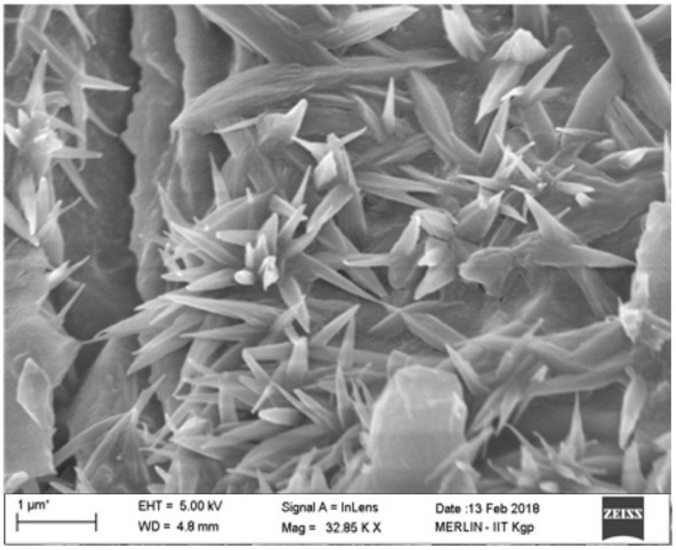

Fig. 5SEM image of Mosambi peel

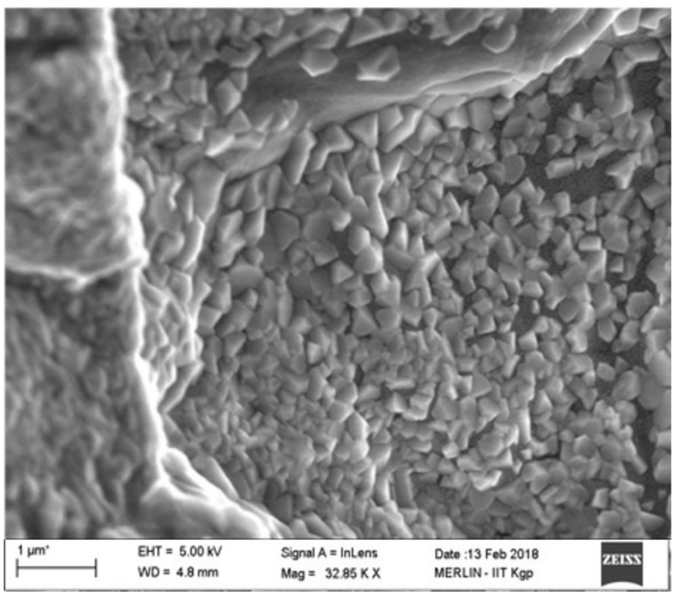

Fig. 6SEM image of Banana peel

TABLE 1

EDS RESULT OF BANANA PEEL

ELEMENT	WEIGHT %	ATOMIC %
Carbon, C	53.43	63.85
Oxygen, O	35.63	31.97
Magnesium, Mg	0.24	0.14
Chlorine, Cl	2.89	1.17

TABLE 2 EDS RESULT OF MOSAMBI PEEL

ELEMENT	WEIGHT %	ATOMIC %
Carbon, C	59.78	66.69
Oxygen, O	39.48	33.06
Potassium, K	0.54	0.18
Chlorine, Cl	0.21	0.07

TABLE 3 EDS RESULT OF MOSAMBI PEEL

ELEMENT	WEIGHT %	ATOMIC %
Carbon, C	55.38	62.66
Oxygen, O	43.51	36.96
Potassium, K	0.64	0.22
Chlorine, Cl	0.45	0.15

B) Preparation of Calibration Curves: Mother solutionsofthe respective dyes were prepared followed by preparing the aliquots by appropriately diluting the mother solution to required concentrations. These were then used to prepare the calibration curve for the three dyes.

C) Dye removal ability of the adsorbents: The graphical representation of the percentage removal of the dyes by the adsorbents are as follows:

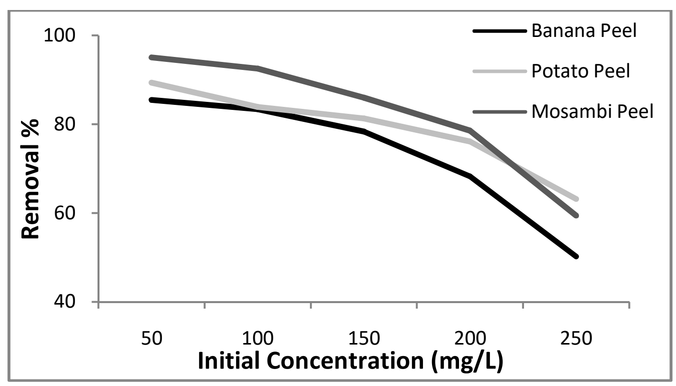

Fig. 7 Amount of Malachite Green removal

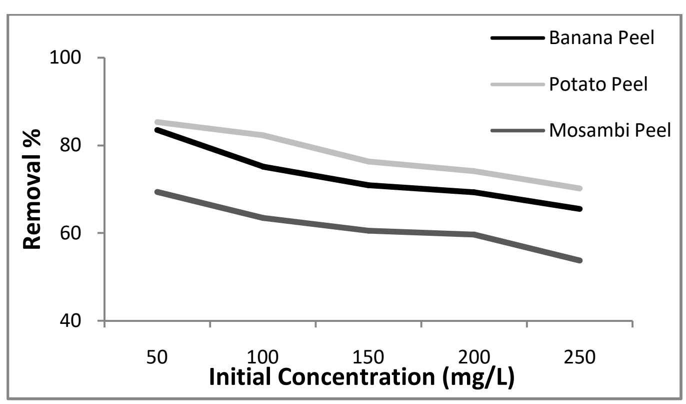

Fig. 8 Amount of Methyl Orange removal

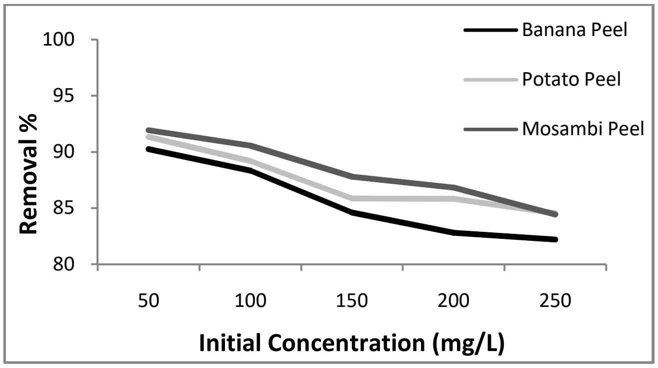

Fig. 9 Amount of Methylene Blue removal

It was observed that for Malachite green, an overall higher percentage removal was shown by mosambi peels while for Methyl orange, an overall higher percentage removal was shown by potato peels and for Methylene blue, an overall higher percentage removal was shown by mosambi peels.

D) Search for appropriate adsorption equilibrium-From the above data (fig. 7,8,9) the best pairs of adsorbate-adsorbent were taken and the adsorption data obtained from these pairs were then fit to the Langmuir, Freundlich, Temkin and Redlich-Peterson isotherms. Based on regression analysis, the best isotherm representing each pair was determined.

1) *Malachite Green-Mosambi Peel system-*

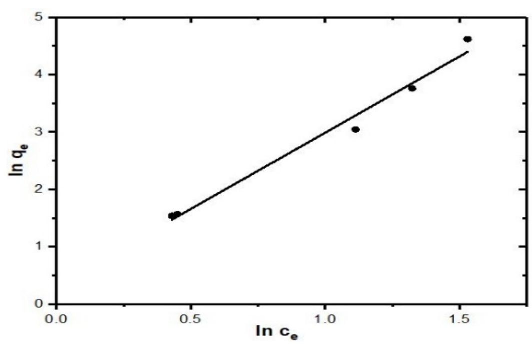

Fig. 10 Plot of Langmuir Isotherm

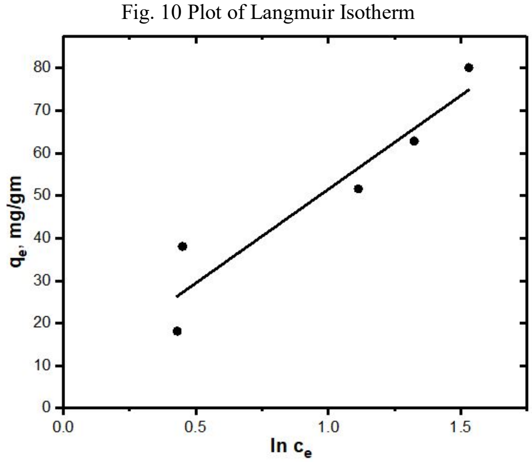

Fig. 11Plot of Freundlich Isotherm

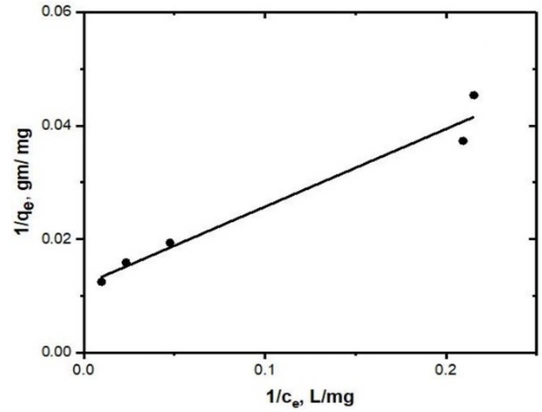

Fig. 12Plot of Temkin Isotherm

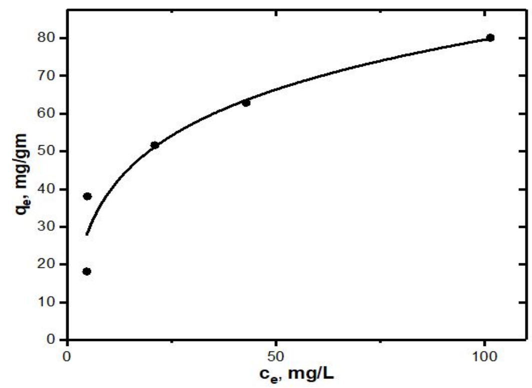

Fig. 13 Plot of Redlich-Peterson Isotherm

The Redlich-Peterson isotherm was seen to have the best fit with the experimental data of this adsorbate-adsorbent pair. This three-parameter isotherm takes into effect the lateral interaction among the adsorbed solute molecules.

From the chemical structure of Malachite Green, the presence of steric effect can be inferred. This effect and the conical micro-sructure of mosambi peel may be the reason for the lateral interaction of the solute molecules in the adsorbed layer.

2) *Methyl Orange-Potato Peel system-*

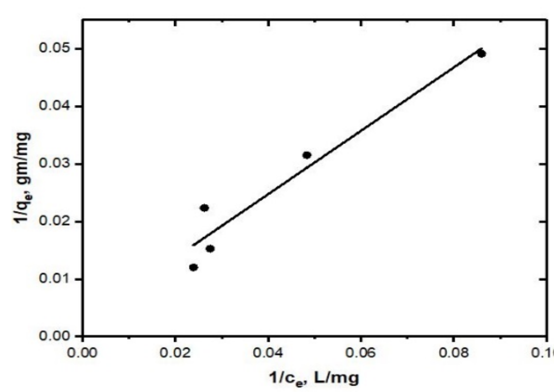

Figure 14 Plot of Langmuir Isotherm

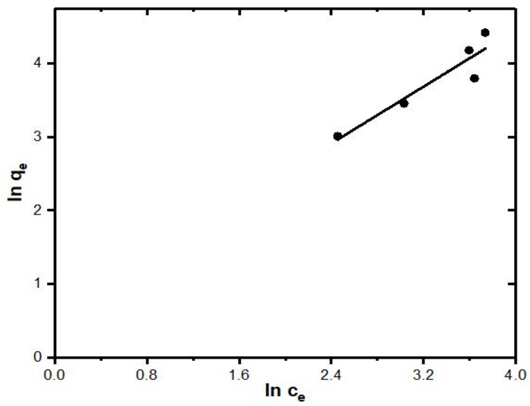

Figure 15 Plot of Freundlich Isotherm

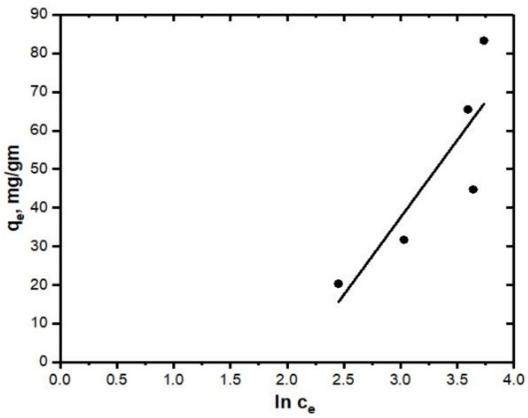

Figure 16 Plot of Temkin Isotherm

The Langmuir isotherm best describes monolayer adsorption of adsorbate onto adsorbent and was the best fit forthe data obtained from adsorption of methyl orange onto potato peels.The potato peel has a dispersed micro-structure which might enable an even, mono-layered distributionof the dye particles.The R_L (eq.2) values varied between 0.45 to 0.33 for the different initial concentrations studied, confirming the favourability of the adsorption process.

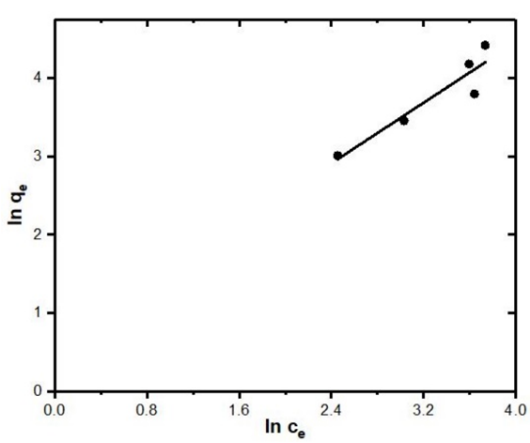

Figure 17 Plot of Redlich-Peterson Isotherm

3) Methylene Blue-Mosambi Peel system-

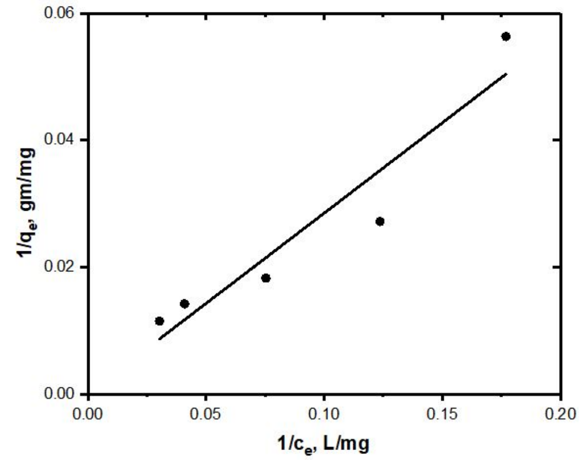

Figure 18 Plot of Langmuir Isotherm

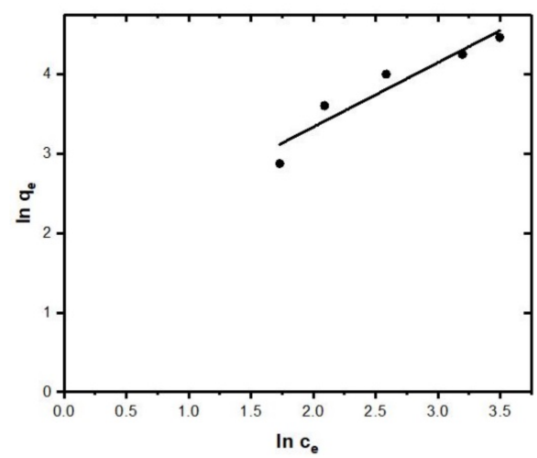

Figure 19 Plot of Freundlich Isotherm

The Temkin isotherm best fit the equilibrium data of the Methylene Blue – Mosambi Peel system.

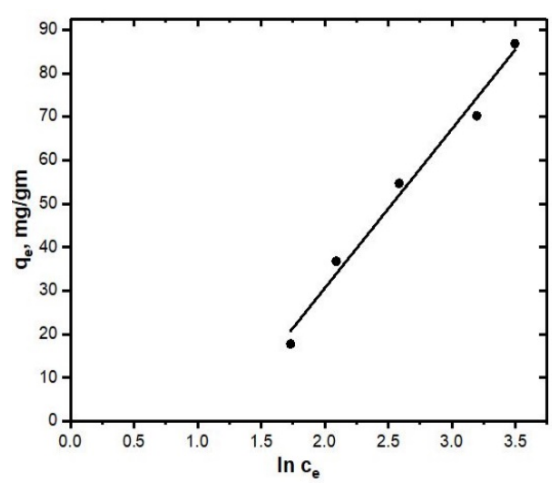

Figure 20 Plot of Temkin Isotherm

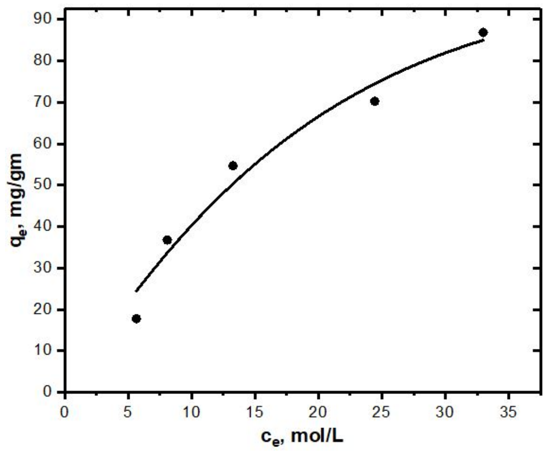

Figure 21 Plot of Redlich-Peterson Isotherm

The Temkin isotherm is a derivative of the Langmuir isotherm. It assumes that the heat of adsorption decreases linearly with increasing surface coverage of the adsorbent, as might be happening in this case. The positive value of the coefficient b confirms the exothermic nature of the process.

TABLE 4

COEFFICIENT OF DETERMINATION (R^2) VALUES OBTAINED FOR DIFFERENT ISOTHERMS AND ADSORBATE-ADSORBENT SYSTEMS

	LANGMUIR ISOTHERM	FREUNDLICH ISOTHERM	TEMKIN ISOTHERM	REDLICH-PETERSON ISOTHERM
Malachite Green with Mosambi peel	0.631	0.787	0.912	**0.914**
Methyl Orange with Potato peel	**0.939**	0.870	0.731	0.817
Methylene Blue with Mosambi peel	0.905	0.908	**0.985**	0.954

TABLE 5

LIST OF PARAMETERS OBTAINED FROM THE PLOTS OF THE ISOTHERMS

	Parameters	MG-MP	MO-PP	MB-MP
Langmuir isotherm	q_0 (mg/g)	72	370	16666
	K_L (L/mg)	0.108	0.004	0.0002
	R^2	0.631	0.939	0.905
Freundlich isotherm	K_f (mg/g)	15.02	1.81	5.56
	n	2.66	1.03	1.23
	R^2	0.787	0.871	0.908
Temkin isotherm	A_T (L/g)	1.11	0.13	0.32
	b	148	62	68
	R^2	0.912	0.731	0.985
Redlich-Peterson isotherm	A	16.55	31806	12.85
	b_{RP}	0.523	50625	0.838
	K	0.789	-0.269	0.433
	R^2	0.914	0.817	0.954

(MG=Malachite Green; MP=Mosambi Peel; MO=Methyl Orange; PP=Potato Peel; MB=Methylene Blue)

IV. CONCLUSION

Using the equilibrium concentration values (c_e)and the amount of adsorbate adsorbed per gram of the adsorbent (q_e), and standard least-squares curve fitting techniques, the best possible isotherm for the different systems are determined. The Langmuir, Freundlich, Temkin and Redlich-Peterson plots are investigated.

It was observed that of all the adsorbents used, mosambi peel showed the best overall performance as an adsorbent. The conical microstructure and consequent availability of a larger surface area make mosambi peel a good adsorbent and it performs better with two of the three dyes.

The potato peel gives satisfactory performance with methyl orange, but the agglomeration seen in the banana peel reduces its effective surface area and the number of available adsorption sites and thus it does not perform well.

From the EDS analysis performed potassium, calcium, magnesium and chloride ions were found to be present in the adsorbents. These may play a role in the adsorption ability of the adsorbent although this is beyond the scope of this paper.

This work provides a basic investigation into newer sources of bio-adsorbents. The performance of these adsorbents was studied without their modification since the primary aim was to check for the viability of the materials as bio-adsorbents. With proper modification of the adsorbents (i.e. doping etc.) and variation of the adsorbate solution (i.e. pH, temperature etc.) the performance may be enhanced. However, with a preliminary investigation with unmodified adsorbents, it can be concluded that mosambi peel and potato peel can be used as a bio-adsorbent while the banana peels may function as adsorbents after some pre-treatment that breaks down the agglomeration of the microstructure.

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