

Uptake of dyestuffs by chemically treated human hair waste as a biosorbent

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Human hair is a waste which has no economical application. It even constitutes a solid waste as far as the environment is concerned. Human hair has been investigated as an adsorbent to uptake of dyestuffs from aqueous solutions. Natural human hair was treated with different chemical activators (NaOH, Na₂S and mixture of NaOH and Na₂S) at different conditions to enhance its adsorption capacity. Adsorption of a basic dyestuff (Basic Blue 69) and an acid dyestuff (Acid Blue 25) onto chemically treated human hair has been studied. The equilibrium isotherms have been determined for human hair treated at different conditions where, the maximum adsorption capacity has been determined for each case according to Langmuir model. The pretreatment optimum conditions which give maximum adsorption capacity for each pollutant were determined and were found to be, pretreatment with 0.1N-NaOH/0.1N-Na₂S solution for 20 minutes. High adsorption capacity was observed for basic dyestuff while, lower capacity for acid dyestuff. At the optimum pretreatment conditions, adsorption capacities were found to be 182.9 mg/g and 68.6 mg/g for Basic Blue and Acid Blue respectively. The equilibrium isotherms have been analyzed using Langmuir, Freundlich and Redlich-Peterson models and the experimental data have been compared with the theoretically predicted data. The Freundlich model appears to fit the adsorption better than other models.

المخلفات من شعر الانسان ليس لها جدوى اقتصادية بل وتشكل دائما احدى الملوثات الصلبة المؤثرة علي البيئة. ويهدف هذا البحث دراسة إمكانية استخدام المخلفات من شعر الانسان لازالة الأصباغ من المحاليل المائية. ومن أجل زيادة سعة الامتزاز القسوى للشعر تم معالجة كيميائيا بواسطة منشطات مختلفة وهي هيدروكسيد الصوديوم أو كبريتيد الصوديوم أو خليط منهما معا عند ظروف مختلفة. وقد تم دراسة امتزاز نوعين من الصبغات وهما صبغة قاعدية زرقاء (BB69) وصبغة حامضية زرقاء (AB25) باستخدام الشعر المعالج كيميائيا. حيث تم قياس التوازنات ثابتة درجة الحرارة للشعر المعالج عند الظروف المختلفة ومنها أمكن الحصول على سعة الامتزاز القسوى في كل حالة وذلك باستخدام نموذج Langmuir الرياضي. وقد تم ايجاد الظروف المثلى لعملية المعالجة لكل من الصبغتين والتي تعطى أعلى سعة امتزاز وخلصت الدراسة إلى انها المعالجة بواسطة (0.1NNaOH/0.1NNa₂S) لمدة 20 دقيقة. ووجد ان السعة القسوى لامتزاز الصبغة القاعدية الزرقاء تبلغ (182.9mg/g) اما تلك الخاصية للصبغة الحامضية الزرقاء فكانت (15.4 mg/g) وذلك عند الظروف المثلى للمعالجة. وقد تم تحليل النتائج المعملية مع النماذج الرياضية المعروفة (Langmuir, Freundlich and Redlich-Peterson) ومقارنتها مع النتائج النظرية المستنتجة ووجد انها تتطابق بشكل جيد مع نموذج فرنديتش.

Keywords: Keratinous Waste, Human hair, Chemical Treatment, Dyestuff, Adsorption

1. Introduction

Due to ever-growing demands in textiles, synthetic organic dyes are widely used for dyeing textile fibers such as cotton and polyester. However, these materials pose certain health hazards and environmental pollution. Dye effluents not only are aesthetic pollutants as far as their color are concerned but may also interfere with light penetration in the receiving water bodies, thereby disturbing the biological processes. Further, dye effluents

may contain chemicals that exhibit toxic effects toward microbial populations and can be toxic and/or carcinogenic to mammalian animal. There-for, environmental legislation has imposed stringent limits on the concentrations of pollutants that may be discharged in to aqueous effluents from dyestuff manufacturing and textile industries.

Methods for effluent treatment for dyes and/or their effluents may be divided into three main categories: physical, chemical, and biological [1, 2]. Among them, adsorption

technology is generally considered to be an effective method for quickly lowering the concentration of dissolved dyes in an effluent. The major advantages of an adsorption system for water pollution control are less investment in terms of initial cost and land, simple design and easy operation, no effect by toxic substances, and superior removal of organic waste constituents as compared to conventional biological treatment processes. In this regard, activated carbon has been evaluated extensively for the removal of color resulting from the different classes of dyes, that is, acid, direct, basic (cationic), reactive, and disperse [3]. Despite the prolific use of this adsorbent throughout the water/wastewater treatment and other industrial applications, carbon adsorption remains an expensive process due to the high cost for the use of activated carbon. This has attracted considerable research into low-cost alternative adsorbents for adsorbing or removing coloring matter and undesirable residues from waste water. Other materials studied for adsorbing dyestuffs are activated silica, activated alumina [4], peat [5], wood [6], bagasse pith [7], maize cob [8], Peanut hulls [9], clay [10], and orange peel [11]. To date, no other adsorbent has proved as versatile as activated carbon. It has been reported that it is capable of removing specific contaminants and is, therefore, likely to make significant contributions to water purification in the future.

Biosorbents have caught the attention of many researchers for the removal of heavy metals as environmental contaminants from aqueous solutions, with an abundant literatures existing for such processes. For example, spent animal boneds have been used for the removal of copper and zinc [12], lignite for the removal of mercury, cadmium and lead [13], immobilized fungal biomass for the removal of different metal ions [14], and peanut hulls for the removal of the cadmium [15]. Fibrous keratinous materials such as wool and hair are known to adsorb metal ions from solution in their native state and with suitable chemical pretreatment, the adsorption capacity for these ions can be significantly enhanced [16]. Such adsorption characteristics have prompted many research

workers to explore the possibility of using such keratinous waste as wool waste, poultry feather waste, tannery hair waste and human hair waste as cheap substitutes for the more expensive ion exchange resins normally used for the removal of heavy metal pollutants from the waste-water [17]. The uptake of metal ions by these materials was attributed to their constituents which contain such functional groups as carboxyl, hydroxyl, phosphate and amine that act as binders for these ions. In contrast, literature on the use of biosorbents for the removal of dyestuffs is very limited and there appears to be relatively poor information in the literature concerning the adsorption of dyestuffs onto human hair. One of literature used natural human hair, abundant human waste of no commercial use, as biosorbent for BB69 and AB25 [18]. The use of such human hair waste as an adsorbent would help to reduce the cost of wastewater treatment and would make a contribution in cleaning the environment.

Previous work showed that, adsorption capacity of natural human hair (untreated human hair) for dyestuffs is limited, where the maximum adsorption capacity was reported as 16.9 and 5.4 mg/g hair for BB69 and AB25 respectively [18]. In this study, adsorption capacity of human hair for BB69 and AB25 was significantly enhanced using suitable pretreatment reagents, activators, such as NaOH, Na₂S, and mixture of NaOH and Na₂S. The pretreatment process has been carried out at different conditions for different soaking times and reagent concentrations to estimate the pretreatment optimum conditions. The work is directed primarily towards studying the applicability of common adsorption isotherm models (i.e. Langmuir, Freundlich and Redlich-Peterson). A comparison of the experimental data with theoretical isotherms have been studied in order to develop a model which both accurately represents the experimental results and could be used for design purposes.

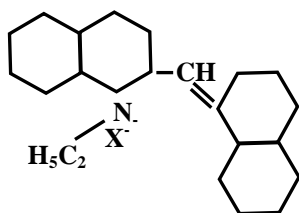
2. Materials and methods

The adsorbates (dyestuffs) and their structures used in the experiments are listed

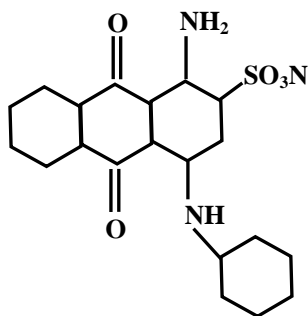
below. The dyestuffs were used as the commercial salts and were supplied by Bayer.

2.1. Basic Blue 69 (Astrazone Blue FRR)

No structure is available for this dye. It belongs to the methane class of which chromophore is a conjugate chain atoms terminated by an ammonium group, and in addition a nitrogen, sulphur or oxygen atom, or an equivalent unsaturated group. A general structure of methane group is,



2.2. Acid Blue 25 (Telon Blue ANL)



The dye was made up in stock solution of concentration 1500 mg/dm^3 and was subsequently diluted to the required concentration using distilled water. The concentration of the dye staffs in aqueous solution as determined using a spectrophotometer (Spectro-Plus MK1A). All measurements were made at the wave length corresponding to maximum absorbance, λ_{max} , which was 585 nm for BB 69 and 600 nm for AB 25. In accordance with the Lambert-Bear law the absorbance was found to vary linearly with concentration and a dilution of concentrated samples were undertaken prior to the analysis in cases where the concentrations were higher than the linear calibration range to give accurate results.

The adsorbent used in this investigation is the chemically treated human hair (activated

human hair). Human hair waste collected from various local barber shops, were mixed together, washed, clean of the adhering dirt with a detergent, rinsed several times with distilled water and finally dried in an electric oven at $100 \text{ }^\circ\text{C}$ before being used. For each pretreatment process, about 4 grams of the dried hair was weighed out and soaked in 250 ml of a pretreatment reagent of a known concentration at room temperature. After a given soaking time, the solution was filtered and the hair washed clean with normal water, deionized water and then dried again in an electric oven. The dried treated hair is stored in a clean plastic bin to avoid the adsorption of moisture from environment, at this its used directly [17].

The chemical reagents used in this work for pretreatment process are, NaOH solutions of various concentrations (0.05N, 0.1N, 1N and 2N) for 30 minutes soaking time, NaOH/Na₂S solutions of concentrations (0.1N/0.1N) and (0.1N/0.2N) for different soaking times (10, 20, 40 and 60 minutes), and Na₂S solutions of concentration 0.1N for 20 minutes soaking time.

Adsorption isotherms were determined using the bottle- point method. A constant mass (0.5 g) of treated human hair was added to bottles containing fixed volume 50 ml of dye solution, the latter have a range of known concentration. The bottles were sealed and, together with appropriate controls, mechanically shaken for a period of 150 minutes [18]. After equilibrium time, dye concentration in residue solutions was determined, the equilibrium data from each bottle representing one point on an adsorption isotherm. All experiments were carried out at $25 \pm 2 \text{ }^\circ\text{C}$ to eliminate any temperature effects using thermo-state. The study on the effectiveness of the various pretreatment processes, using different reagents such as NaOH, Na₂S, and mixture of NaOH and Na₂S solutions of various concentrations and for different soaking times, was carried out by comparing the adsorption capacity for dyes. The results also helped to establish the optimum pretreatment process for uptake dyestuffs from aqueous solutions. For each of experimental runs, before the adsorbate solution was added to the treated hair, its pH

was adjusted to 7 ± 0.3 using dilute HCl or NaOH to eliminate the effect of pH.

3. Results and discussion

3.1. General

The purpose of studying adsorption isotherm is, firstly, to measure the adsorption capacity of each system and secondly, to ascertain the liquid-solid equilibrium distribution of the solute concerned. Adsorption isotherms were determined for various dye-treated hair systems. Preliminary tests were undertaken to assess the contact time necessary for each system to come to equilibrium and, for experimental purposes, each system was given a contact time in excess of this period. Experiments showed that such equilibrium was established within 100 minutes [18]. However, all equilibrium experiments were allowed to run for 150 minutes.

3.2. Effect of pretreatment method

Previous work showed that, untreated human hair exhibited a limited adsorption capacity for basic dyestuffs (i.e. BB69) and relatively exhibited negligible adsorption capacity for acid dyestuffs (i.e. AB25). However, with chemical treatment, significant softening of the fibers was observed. The extent of softening increased with increase in the reagent concentration and/or soaking time, however, the adsorption capacity was found to increase significantly corresponding to the increase in the softness of the fibers up to limit as shown in table 1. Prolonged soaking and increased pretreatment reagent concentration led to a reduction in the adsorption capacity corresponding to extremely soft fibers which easily disintegrated into fine pieces on slight agitation. These observations showed therefore a close relationship between the adsorption capacity and the extent of softening of the hair fibers. The softening process may result from partial breakage of the various linkages of the keratin structure making available the various active functional groups responsible for dye-keratin interaction. The loosening of the compact

keratin structure also increased the penetrability of the fibers to the diffusion of the dye ions to these active sites, thus increasing the adsorption capacity. However, with extreme loss of fibrous strength on prolonged soaking or increased reagent concentration in the pretreatment process, the weak solid substrate may not be able to support the load of the adsorbed dye ions [17].

3.3. Equilibrium isotherm

Adsorption capacity of chemical by treated human hair for dyestuffs (BB96 and AB25) can be determined by measuring equilibrium isotherms. Figs. 1 to 4 depict the adsorption isotherms measured for BB69 onto human hair treated at different conditions. According to the classification of Giles et al. [19], the adsorption isotherms obtained may be classified as L-type. In this case, adsorption is very efficient at low concentration, but it becomes increasingly difficult, as the concentration increases, for a solute molecule to find a vacant adsorption site. As same isotherms shape (L-type) were observed for AB25 dye. The maximum adsorption capacity for a particular dyestuff was calculated and tabulated in Table 1 corresponding to Langmuir isotherm. The experimental results showed that a favorable pretreatment method would be to soak the hair fibers in a 0.1N-NaOH/0.1N-Na₂S solution for 20 min. The maximum adsorption capacities were found 182.9 mg/g hair with 982.2% removal increasing and 68.6 mg/g hair with 1170.4% removal increasing for BB69 and AB25 respectively.

A comparison between isotherms data for untreated human hair and human hair treated at optimum conditions was carried out as shown in fig. 5 for BB69 and fig. 6 for AB25. Data for untreated human hair was taken from previous work [18]. Effect of the pretreatment on adsorption capacity of both dyestuffs was observed where; it increased from 16.9 to 182.9 mg/g for BB69 and from 5.4 to 68.6 mg/g for AB25. The results showed that BB69 has much greater affinity for treated and untreated human hair than AB25.

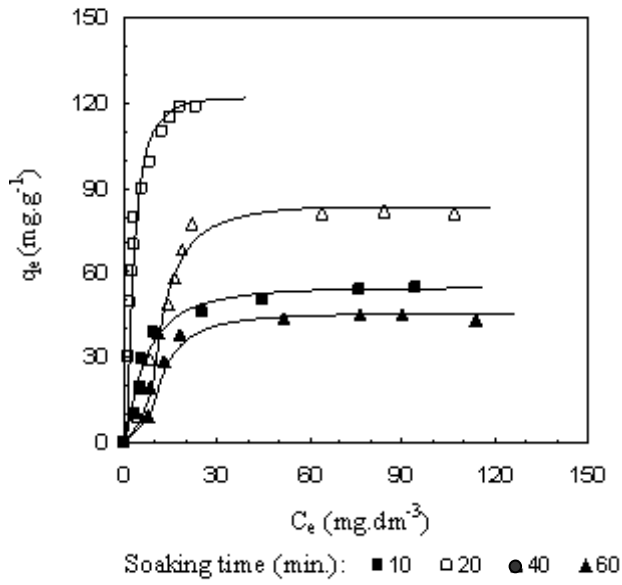


Fig. 1. Adsorption isotherms for BB 69 onto human hair treated with 0.1N-NaOH/0.1N-Na₂S solution for different soaking times.

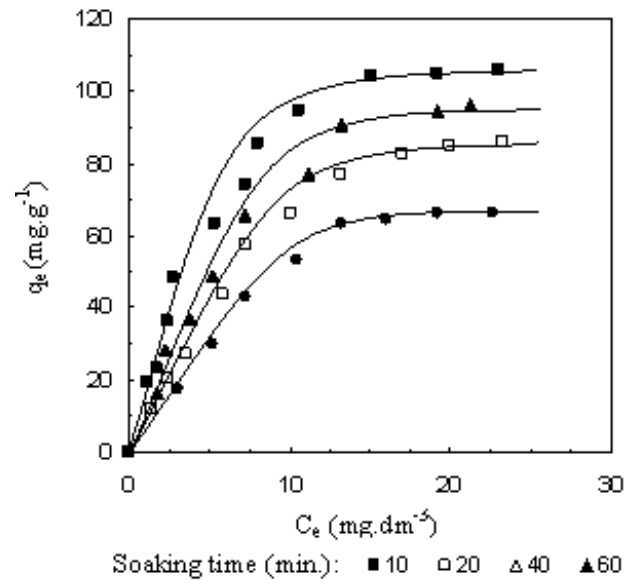


Fig. 3. Adsorption isotherms for BB 69 onto human hair treated with 0.1N-NaOH/0.2N-Na₂S solution for different soaking times.

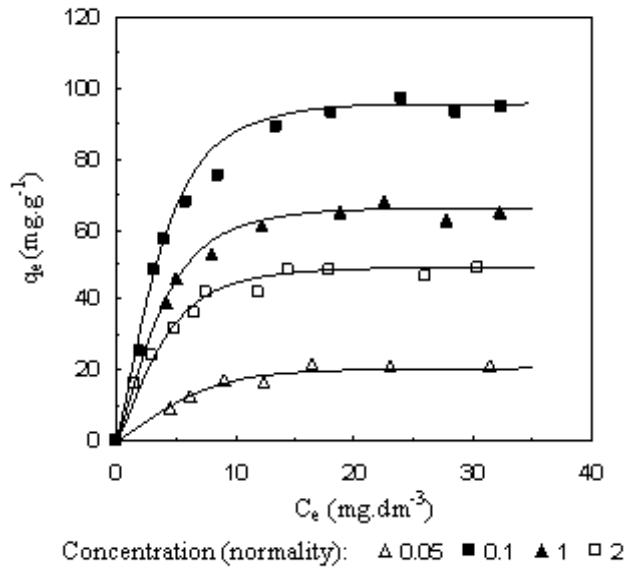


Fig. 2. Adsorption isotherms for BB 69 onto human hair treated for 30 min. with NaOH solution at different concentrations.

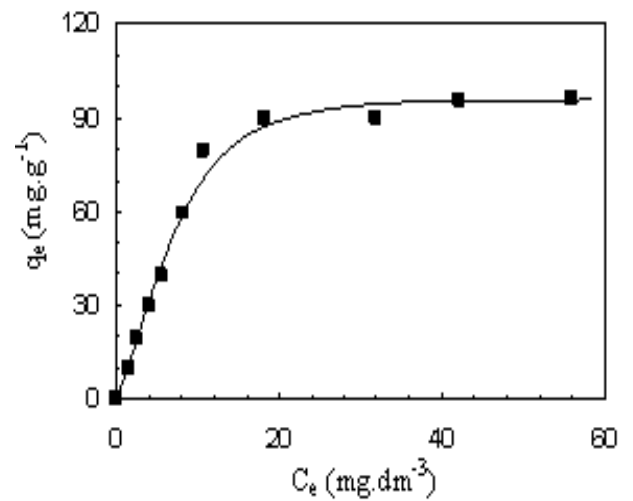


Fig. 4. Adsorption isotherms for BB 69 onto human hair treated with 0.1N-Na₂S solution for 30 min.

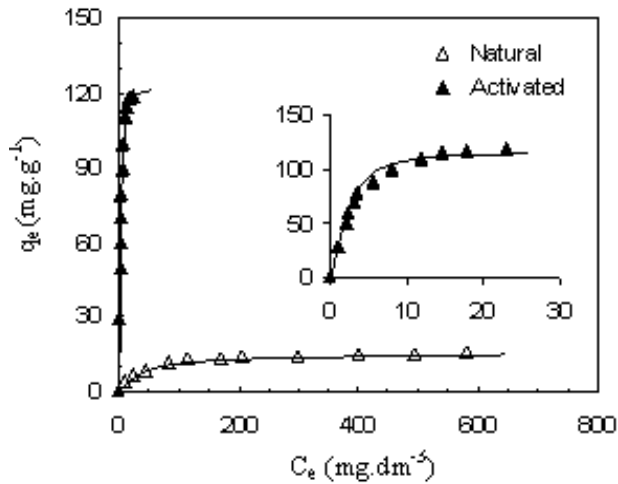


Fig. 5. Comparison between adsorption isotherms for BB 69 onto natural human hair and human hair treated at optimum conditions.

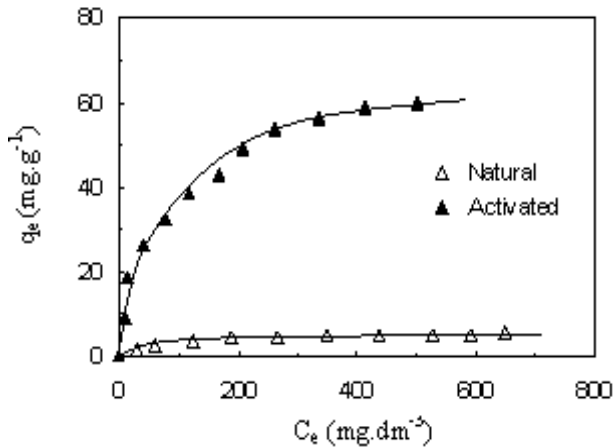


Fig. 6. Comparison between adsorption isotherms for AB 25 onto natural human hair and human hair treated at optimum conditions.

To optimize the design of an adsorption system for the adsorption of each dyestuff, it is important to establish the most appropriate model for the equilibrium data. In this study three isotherm models have been selected to simulate the experimental data, namely the Langmuir, Freundlich and Redlich-Peterson isotherms. The later incorporates features of both the Langmuir and Freundlich equations. Analysis of isotherms data have been done for the two dyestuffs only at the optimum pretreatment conditions.

3.3.1. Langmuir isotherm

The Langmuir isotherm is valid for monolayer adsorption on a surface containing a finite number of identical sites [20]. The model assumes uniform energies of adsorption on the surface and no transmigration of adsorbate in the plane of the surface. The Langmuir isotherm is represented by the following equation:

$$q_e = (K_L.C_e)/(1 + a_L.C_e). \quad (1)$$

This may be converted into a linear form convenient for plotting and determining the constants, K_L and a_L :

$$C_e/q_e = (1/K_L) + (a_L/K_L) C_e. \quad (2)$$

Hence a plot of C_e/q_e versus C_e should yield a straight line with the reciprocal intercept equal to K_L and the slope equal to a_L/K_L . The Langmuir plots for both dyestuffs are represented in figs. 7 and 8. The slopes, a_L/K_L , and intercepts, $1/K_L$, of these straight lines were calculated using the least-squares method and are tabulated in table 2. The values of constant K_L/a_L are important since they represent the maximum or monolayer adsorption capacity (q_{max}) of the treated human hair waste for a particular dyestuff. Referring to table 1, values of maximum adsorption capacity for all particular pretreatments are calculated by fitting of experimental data with Langmuir isotherm.

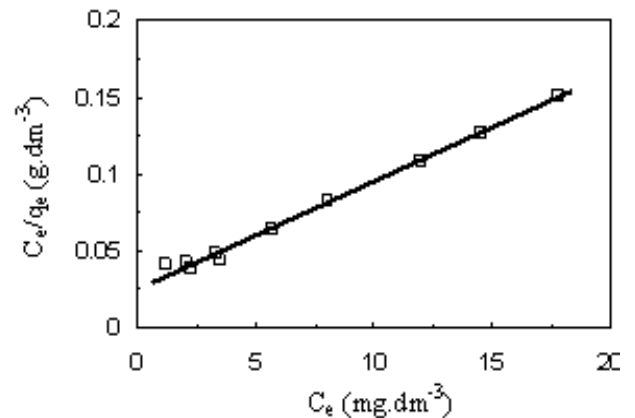


Fig. 7. Langmuir plot corresponding to the adsorption of BB 69 onto treated human hair.

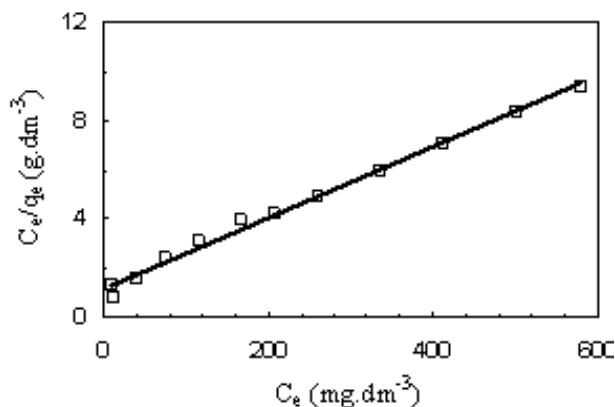


Fig. 8. Langmuir plot corresponding to the adsorption of AB 25 onto treated human hair.

$$q_e = K_F \cdot C_e^{1/n} \tag{3}$$

A logarithmic plot linearizes the equation enabling the exponent, n , and the constant, K_F , to be determined from eq. 4.

$$\log q_e = \log K_F + (1/n) \log C_e \tag{4}$$

Inspection of the results derived from the Freundlich analysis represented by eq. 4 shows that a plot of $\log q_e$ versus $\log C_e$ exhibits some curvature. Indeed, the results can be better represented by more than one straight line. A general equation for the entire concentration range may be expressed as:

$$q_e = K_{F,i} \cdot C_e^{1/n_i} \tag{5}$$

3.3.2. Freundlich isotherm

The Freundlich isotherm is valid for systems in which the surface energies are heterogeneous and vary as a function of q_e due to variations in the heat of adsorption. The Freundlich equation represented as:

Table 1. Pretreatment and adsorption capacity of human hair waste for BB69 and AB25

Reagent	Pretreatment concentration (normality)	Soaking time (min)	Adsorption capacity (mg/g)		Removal increasing (%)	
			BB69	AB25	BB69	AB25
Untreated	-	-	16.9	5.4	0.0	0.0
NaOH	0.05	30	26.6	-	57.4	-
	0.1	30	109.9	7.5	550.3	38.9
	1.0	30	72.1	27.9	326.6	416.7
	2.0	30	54.3	35.1	221.0	550.0
NaOH/Na ₂ S	0.1/0.1	10	62.4	11.9	269.2	120.4
	0.1/0.1	20	182.9	68.6	982.2	1170.4
	0.1/0.1	40	99.1	54.5	486.4	909.3
	0.1/0.1	60	52.3	25.7	209.5	375.9
NaOH/Na ₂ S	0.1/0.2	10	154.0	12.8	811.2	137.0
	0.1/0.2	20	147.6	22.8	773.4	322.2
	0.1/0.2	40	145.5	-	760.9	-
	0.1/0.2	60	116.2	15.3	587.6	183.3
Na ₂ S	0.1	20	168.7	10.1	898.2	87.1

notes: $d_p=40-80\mu\text{m}$, temperature= 25 ± 2 and $\text{pH}=7\pm 0.3$

Table 2. Parameters of the Langmuir adsorption model

Dye	K_L (dm ³ /g)	a_L (dm ³ /mg)	q_{max} (mg/g)	\bar{R} (-)	Correlation Coefficient
BB69	38.40	0.21	182.9	0.13	0.98
AB25	0.961	0.014	68.6	0.02	0.97

Figs. 9 and 10 show the Freundlich isotherms for adsorption of BB69 and AB25 onto treated human hair respectively on the basis of eq. 5. The Freundlich parameters, K_F and n have been calculated using the least-squares method and are tabulated in table 3 together with correlation coefficients obtained. The magnitude of exponent, n , gives an indication of the favorability and capacity of the adsorbent-adsorbate system. Values of $n > 1$, represent favorable adsorption according to Treybal [21]. In both systems studied, the values of n are greater than one ($n > 1$) which indicates that both dyestuffs were favorably adsorbed by treated human hair. The disadvantage of the later model is that it can't be reduced to Henry's law at concentration approaching zero. This condition is fulfilled by the Redlich-Peterson model, which has a linear dependence on concentration in the numerator and an exponential function in the denominator as it be discussed later.

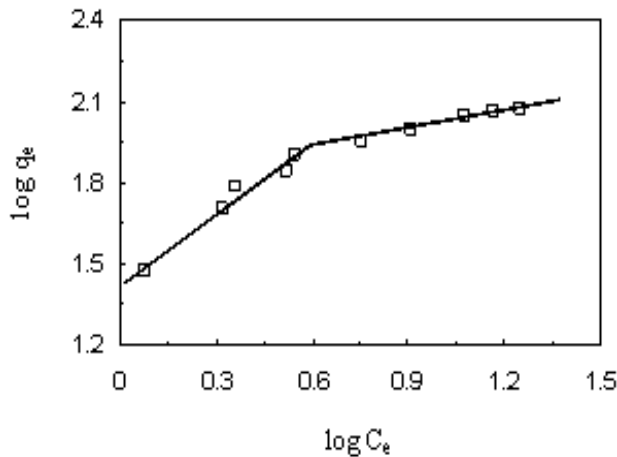


Fig. 9. Freundlich plot corresponding to the adsorption of BB 69 onto treated human hair.

Table 3
Parameters of the Freundlich adsorption model.

Dye	First section of plot			Second section of plot		
	K_F (dm ³ /g)	n (-)	Corr. Coeff.	K_F (dm ³ /g)	n (-)	Corr. Coeff.
BB69	26.30	1.15	0.98	58.13	3.97	0.99
AB25	3.92	2.08	0.97	20.80	5.81	0.98

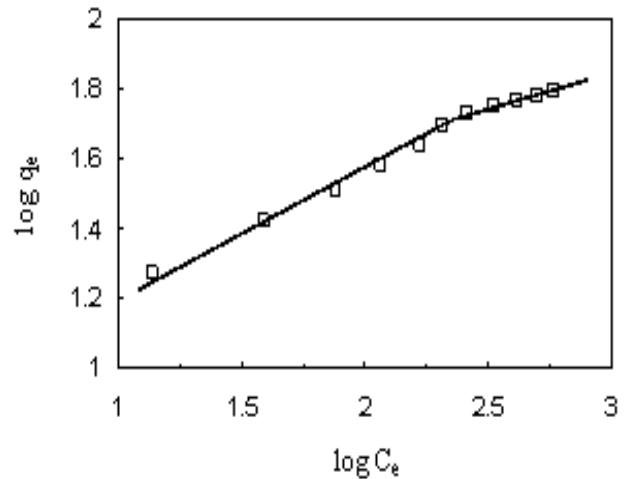


Fig. 10. Freundlich plot corresponding to the adsorption of AB 25 onto treated human hair.

3.3.3. Redlich-peterson isotherm

A third isotherm which incorporates intermediate terms between the Langmuir and Freundlich equations often describes the isotherm better than the classical isotherm models because of its mathematical flexibility. The Redlich-Peterson isotherm has been used in this work and is represented by eq. 6,

$$q_e = (K_{RP} \cdot C_e) / (1 + a_{RP} \cdot C_e^\beta) \tag{6}$$

For $\beta=1$, eq. 6 converts to the Langmuir isotherm; for $1 \gg a_{RP} \cdot C_e^\beta$ it simplifies to Henry's law; and for $1 \ll a_{RP} \cdot C_e^\beta$ it is identical with the Freundlich isotherm. The linear form is shown in eq. 7 from which the constants, K_{RP} , a_{RP} and β , which characterize the isotherm, can be determined:

$$\log \left[\frac{K_{RP} \cdot C_e}{q_e} - 1 \right] = \log a_{RP} + \beta \log C_e \quad (7)$$

The plots of $\log \{ [K_{RP} \cdot C_e / q_e] - 1 \}$ against $\log C_e$ are shown in figs. 11 and 12 and are seen to be linear over the whole concentration range. The Redlich-Peterson parameters for each particular system were analyzed using the least-squares method and are tabulated in table 4.

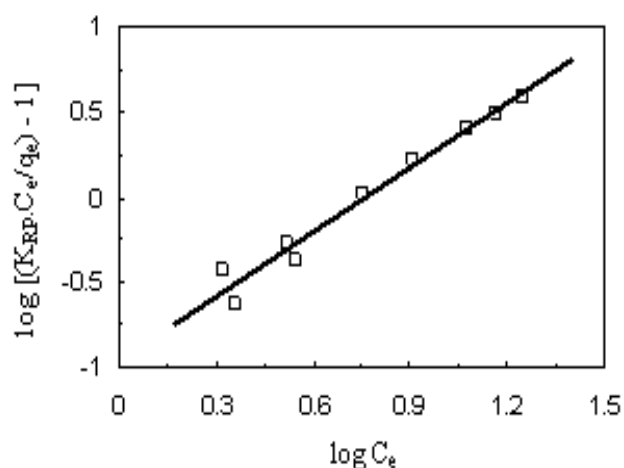


Fig. 11. Redlich-Peterson plot corresponding to the adsorption of BB 69 onto treated human hair.

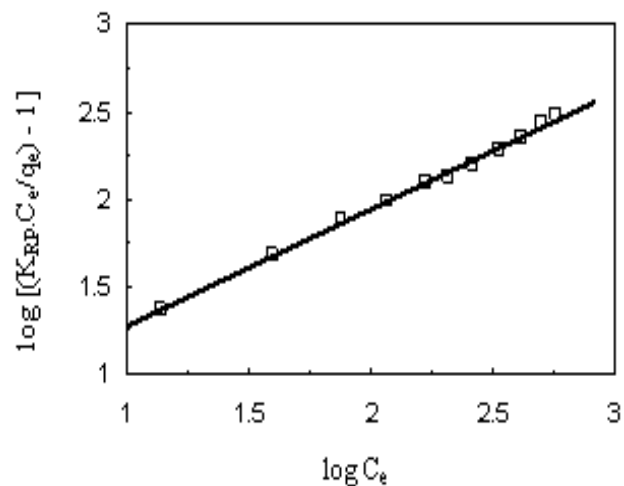


Fig. 12. Redlich-Peterson plot corresponding to the adsorption of AB 25 onto treated human hair.

Table 4
Parameters of the Redlich-Peterson adsorption model.

Dye	K_{RP} (dm ³ /g)	a_{RP} (dm ³ /g) ^{1/β}	β (-)	Correlation Coefficient
BB69	32.31	0.11	1.26	0.97
AB25	0.926	0.015	0.91	0.96

3.4. Comparison of theoretical isotherms with experimental results

Using the appropriate constants of the Langmuir, Freundlich and Redlich-Peterson models, the theoretical isotherm curves were predicted using known values of C_e . Figs. 13 and 14 show a comparison of the experimental points with the Langmuir, Freundlich and Redlich-Peterson models, in order to establish which model yields the "best fit". Obviously, it can be seen that, for BB69, Freundlich and Redlich-Peterson models yield somewhat better fit than the Langmuir model, on the other side for AB25, Langmuir and Freundlich models relatively fit better than Redlich-Peterson. As reflected by correlation coefficients (R^2) in tables 2, 3 and 4, it appear that Freundlich model gave a good agreement with the experimental data for the two used dyestuffs.

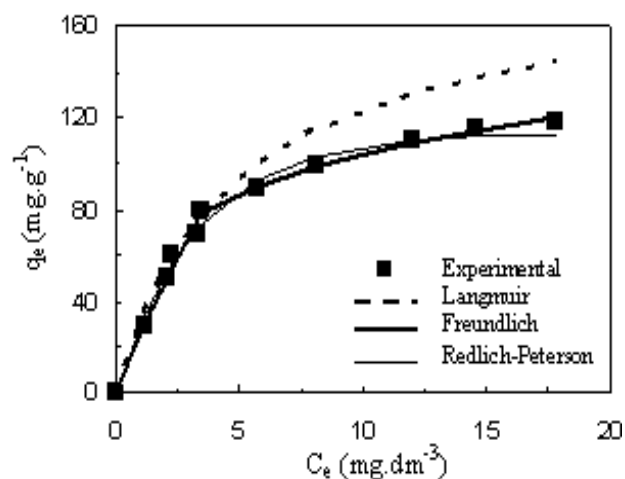


Fig. 13. Comparison of theoretical isotherm plots with experimental data for the adsorption of BB 69 onto treated human hair.

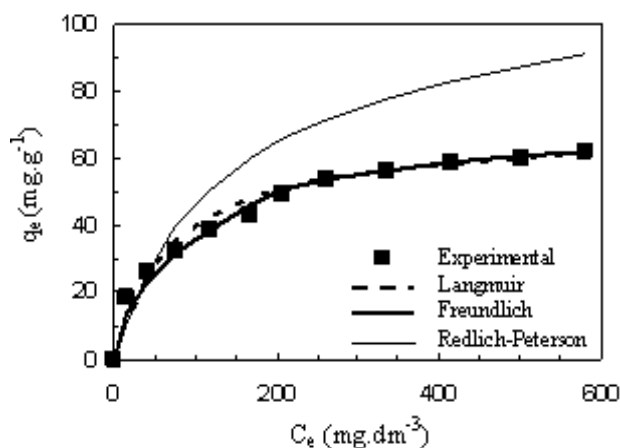


Fig. 14. Comparison of theoretical isotherm plots with experimental data for the adsorption of AB 25 onto treated human hair.

5. Conclusions

Among the various pretreatment processes studied, the data shows that pretreatment of human hair waste with suitable chemical reagents enhances its adsorptive capacities for basic and acid dyestuffs, and pretreatment with 0.1N-NaOH/0.1N-Na₂S solution for 20 minutes gave the best adsorption capacity for both dyestuffs. High adsorption capacity was observed for the adsorption of basic dyestuff (BB69), namely, 182.9 mg/g where, lower capacity observed for acid dyestuff (AB25), namely, 68.6 mg/g. Equilibrium isotherms have been measured for each particular system where, data of optimum pretreatment conditions were analyzed using three common models, Langmuir, Freundlich and Redlich-Peterson models. The theoretical isotherms were compared with the experimental data and the general results of this study reveal that the Freundlich isotherm fitted the experimental adsorption data better than others, hence it is recommended to use it for design purposes.

Nomenclature

a_L parameter of Langmuir isotherm (dm^3/mg),

a_{RP} parameter of Redlich-Peterson isotherm [$(\text{dm}^3/\text{mg})^{1-\beta}$],

C_e equilibrium liquid-phase concentration (mg/dm^3),

K_F parameter of Freundlich isotherm (dm^3/g),

K_L parameter of Langmuir isotherm (dm^3/g),

K_{RP} parameter of Redlich-Peterson isotherm (dm^3/g),

n Freundlich exponent (dimensionless),

q_e equilibrium solid-phase concentration (mg/g), and

β Redlich-Peterson exponent (dimensionless).

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