Adsorptive Removal Of Congo Red From Aqueous Solution By Local Chaff Surface: Thermodynamics And Kinetics Studies

Abdumuhsin A. Al-Haidari
Dept. of Chemistry/College of Education for Pure Science(Ibn Al-Haitham)/ University of Baghdad
Saja S. J. Al-Taweel
Department of Chemistry/ College of Science/ University of Al-Qadisiyah
Laith S. Jassim
Department of Chemistry/ College of Education /University of Al-Qadisiyah

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Abstract

This study is concerned with the adsorption of Congo red from solution on the surface of Chaff. The adsorption isotherm is of L-curve type according to Giles classification and the experimental data were best fitted to Langmuir isotherm model.

The adsorption phenomenon was examined as a function of temperature (25, 40, 55 ^{o}C). The extent of adsorption of Congo red on Chaff was found to increase with the increase of temperature (endothermic process). The basic thermodynamic functions have also been calculated.

The effect of contact time was investigated and found that the adsorption process of dye on Chaff surface reached complete equilibrium within 90 min. The maximum uptake of Congo red by Chaff was found to be 92.9% at 25°C.

The kinetic data were well fitted to the Lagergren, pseudo-second order models. The results indicated that adsorption process followed the second order model. This behavior was discussed depending on the chemical structure of dye and Chaff surface.

The kinetics of dye adsorption was also studied in terms of intraparticle diffusion model. The results indicated that intraparticle diffusion plays a significant role in the adsorption mechanism.

Key word: Adsorption, Congo red, Chaff

Introduction

Approximately one-half of the industrially produced organic chemicals reach the global environment *via* direct and/or indirect routes; for example agricultural practices, municipal and industrial wastes, and landfill effluents. These products include a variety of organic compounds. When these substances reach the natural environment, various degradation and transfer processes are initiated [1].

Major contaminants found in wastewater include biodegradable, volatile, and recalcitrant organic compounds; toxic metals; microbial pathogens; and parasites causing deterioration of the surrounding medium that can present a great danger to the environment and human health [2-4]. Several studies have been undertaken on the toxicity of dyes and their impact on ecosystems [5,6].

These studies show that certain dyes degrade and that their derived products can be toxic and carcinogenic even at law concentrations [6]. Textile industries are the major consumers of water, and they release a fair amount of color in their effluents.

Adsorption is a widely used method for the treatment of industrial wastewaters containing colors, heavy metals and other inorganic and organic impurities [7-10]. The advantages of adsorption are its simplicity of operation, low costs (compared to other separation processes), and absence of sludge formation [11-13]. Liquid-phase adsorption has been used effectively for the removal of dye form wastewater [9].

Sorption data are most often analyzed at equilibrium (or near-equilibrium) conditions. Although concentrations of contaminants in soil, water, and other phases in natural systems frequently deviate from those at equilibrium, the equilibrium data serve as an essential guide to the direction of contaminant movement at a particular point in time and to the likely consequence of an earlier contamination event. A comparison of the field data with equilibrium values also enables one to elucidate whether a compartment (such as soil or sediment) functions as a sink (to receive a given contaminant) or as a source (to release a given contaminant) under specified conditions. Such information is often valued in the characterization of a contamination site [14]. The aim of present work is to explore the feasibility utilizing Chaff as adsorbent for Congo red dye. Equilibrium and kinetic analysis were conducted to investigate the mechanism of dye adsorption and optimization of various parameters in dye recovery.

Materials and Methods

Instruments

1- UV-Visible spectrophotometer, Shimadzu. PC1650, Japan.
2- Shaker water bath, K&K Scientific, Korea.
3- Centrifuge, CL008, Belgium.
4- Electronic Balance, Sartorius Lab., + 0.0001g, Germany.
5- pH-Meter, HANNA, Electronic Ltd, Romania

Materials

Congo red (Scheme (1)) was supplied by Fluka. Chaff was obtained from "Al-diwaniyah Grain silo".

Methodology

Chaff in powder forms was washed with excessive amounts of distilled water; several washings were performed to remove dust and soluble materials. The powder was then dried under sunlight and in an oven at 120°C for a period of 1.5 hours and kept in airtight containers. Chaff surface was ground and sieved to a particle size of 150µm. Wavelength of maximum absorbance (l_{max}) for dye was selected, and found 495nm. This value was utilized

for estimation of quantity of dye adsorbed.

Solutions of different concentrations of dye were prepared by serial dilution. Absorbance values of these solutions were measured at the selected (l_{max}) value for dye and plotted against the concentration values. The calibration curve in the concentrations range that falls in the region of applicability of Beer-Lambert's law was employed.

The adsorption isotherms were determined by shaking 0.2 g of Chaff into 10 ml dye solutions, having concentrations ranging from 1 x 10^{-5} - 15 x 10^{-5}M at pH≈6.8. After 90 min. of shaking, the suspensions were centrifuged at 3000 rpm for 10 min. The dye concentration was determined spectrophotometrically.

The quantity of dye adsorbed was calculated according to the following equation [15]:-

$$Q_e = \frac{x}{m} = \frac{V(C_o - C_e)}{m} \qquad \text{..... (1)}$$

Where:

Q_e: sorption capacity (mg/g).

x: the quantity adsorbed (mg).

m: weight of adsorbent (g).

C_o: initial concentration (mg/L).

C_e: equilibrium concentration (mg/L).

V: volume of solution (L).

Thermodynamic Parameters of Adsorption

Adsorption experiment was repeated in the same manner at temperatures of 25, 40 and 55°C to estimate the basic thermodynamic functions.

Kinetic Studies

Effect of contact time was determined by adding 0.2gm of adsorbent into 10ml dye solution, with initial concentration (34 ppm) under shaking. The temperature of solution was held constant at 25°C with a thermostatic shaker. After different time intervals, the solutions were centrifuged and volumes of 3ml supernatant were taken for spectrophotometrically measurements of dye content.

Results and Discussion

The adsorption isotherm of Chaff for the dye at temperature (25˚C) and pH≈6.8 is shown in Figure (1), where the quantities adsorbed on Chaff are plotted as a function of equilibrium concentration. The results showed an increase in adsorptive capacities of Chaff as the concentration of Congo red increased. The capacity of adsorption depends on several parameters such as the specific surface area, the expansible character, the mobility of dye molecules in the liquid phase and in the interior of the solid, and the forces of attraction between the surface of the solid and the molecules of dye [16,17]. The coulombic forces between dye species and negatively charged Chaff in water are the major interactions which affect the adsorption of dye on the chaff.

The shapes of Congo red adsorption isotherms were found to coincide with the L-type isotherm reported by Giles *et al.* [18]. L-shaped adsorption isotherm indicates the adsorbed solute molecules are most likely being adsorbed in a flat geometry, which is based on the assumption of high adsorption affinity between the dye and the surface [19,20].

The adsorption of Congo red on Chaff followed the linearized Langmuir model as shown in Figure (2). The Langmuir isotherm can be expressed as follows [21]:

$$Q_e = \frac{q_m . K_L . C_e}{(1 + K_L . C_e)} \qquad \text{..... (2)}$$

Where

Q_e: amount adsorbed per unit weight of adsorbent at equilibrium (*mg/g*).
C_e: equilibrium concentration of adsorbate in solution after adsorption (*mg/L*).
q_m *(mg/g)* and K_L *(L/mg)* are the Langmuir isotherm constants.
The Langmuir isotherm constants are evaluated through linearization of equation (2).

$$\frac{C_e}{Q_e} = \frac{1}{q_m K_L} + (\frac{1}{q_m}).C_e \qquad \text{..... (3)}$$

Figure (2) showed the linear relationship of C_e / q_e versus C_e. The values of Langmuir constants as well as the correlation coefficient are presented in Table (1). The isotherm data fit the Langmuir model well as indicated from the value of correlation coefficient.
The general shapes of Congo red adsorption isotherms at three different temperatures are given in Figure (3). The results showed a slight increase in the amount of dye adsorbed on Chaff with the increase of temperature; hence the adsorption process appeared endothermic.
The extent of adsorption of some dyes was found to increase with the increase of temperature [22]. This means the interaction between Chaff and the dye molecules requires an appreciable energy in order to take place. Endothermic dye uptake can also be attributed to the possibility of occurring absorption or sorption process by the surface [23].
The basic thermodynamic quantities of adsorption of Congo red on Chaff were estimated through calculating X_m values at different temperatures. The heat of adsorption (ΔH) may be obtained from Van't Hoff equation: $\ln X_m = constant - \frac{\Delta H}{RT}$, the change in free energy (ΔG) could be determined from equation (DG = -RTlnK) and the change in entropy (ΔS) was calculated from Gibbs equation: ($\Delta G = \Delta H - T.\Delta S$).
Table (2) and Figure (4) demonstrate these calculations.
Table (3) shows the basic thermodynamic values of adsorption of Congo red on chaff. An adsorption of van der waals type is suggested to take place as indicated by these values. The adsorption of Congo red on Chaff is endothermic as indicated by the positive value of enthalpy (ΔH^o). The negative value of free energy (ΔG^o) indicates the spontaneous nature of the adsorption process of dye and the positive value of entropy (ΔS^o) suggests the increase randomness at the solid-solution interface during the adsorption of dye onto adsorbent surface [24].
To evaluate the effectiveness of an adsorbent, the adsorption of Congo red dye on Chaff surface was studied as a function of contact time, and the result is shown in Figure (5). The adsorption rate of dye onto Chaff are observed to be very fast within the first few minutes and gradually decrease and become almost constant after a period of 60 min.
The data for q_t versus t during the initial hour of contact show a very fast increase in q_t with time, followed by a gradual plateau at quasi-equilibrium situations. The initial uptake is attributed to surface adsorption. When the dye adsorption at the exterior surface reached the saturation level, the dye begins to enter the pores of the Chaff surface and adsorbed by the interior surface of the adsorbent particles. As the surface saturates with dye molecules, the adsorption rate decreases due to an increase in the diffusion resistance. This means that the pore diffusion is the rate-controlling step during dye adsorption [25].
Figure (5) showed rapid adsorption of dye in the first 10 min., and thereafter, the adsorption rate decreased gradually. Nearly 91.8% of Congo red is removed from an aqueous solution within 90 min. Numerous kinetic models have been proposed to elucidate the mechanism by which pollutants are adsorbed. The mechanism of adsorption depends on the physical and / or chemical characteristics of the adsorbent, as well as on the mass – transport process. The rate constant of the dye removal from the solution by Chaff was determined using first order and pseudo – second order equations.
The Lagergren first order rate equation was used to fit the experimental results. The integral form of the model is [26]:

$$\ln (q_e - q_t) = \ln q_e - k_1 t \qquad \text{..... (4)}$$

Where q_e (mg/g) is the equilibrium sorption capacity and q_t (mg/g) is the amount of dye adsorbed at time t (min), and k_1 is the equilibrium first-order constant. Values of k_1 for dye-Chaff system were obtained from the slope of the plot of $ln\ (q_e-q_t)$ *vs.* t Figure (7). The adsorption kinetic parameters from Figure (6) are indicated in Table (4).
The adsorption data were also analyzed in terms of a pseudo - second order mechanism [27-29]. The linearized-integral form of this model is:

$$\frac{t}{q_t} = \frac{1}{k_2\, q_e^2} + \left(\frac{1}{q_e}\right)t \qquad \text{..... (5)}$$

Where k_2 ($g.mg^{-1}.min^{-1}$) is the rate constant of the pseudo – second order adsorption.
If the initial adsorption rate is

$$h = k_2\, q_e^2$$

Then equation (5) becomes:

$$\frac{t}{q_t} = \frac{1}{h} + \left(\frac{1}{q_e}\right)t \qquad \text{..... (6)}$$

By plotting t/q_t versus t Figure (7), a straight line could be obtained and q_e, k_2 and h can be calculated. The adsorption kinetic parameters from Figure (7) are listed in Table (4). The fit goodness of the models was at first expressed by the liner regression coefficients of determination (R^2): a relatively high R^2 value indicated that the model successfully described the kinetics sorption of dye. The R^2 values Table (4) suggested that the process of dye adsorption followed second-order kinetics. The structure of Chaff is cellulose based, and the surface of cellulose in contact with water is negatively charged [30]. Congo red is an acidic dye and contains negatively charged sulfonated group. So, it may be expected that the dye have a low sorption affinity for the adsorbent surface.
In general, the mechanism of adsorption of dye by porous adsorbent may be determined by the following four stages: (i) diffusion of molecules from the bulk phase towards the interface space so-called external diffusion; (ii) diffusion of molecules inside the pores internal diffusion; (iii) diffusion of molecules in the surface phase-surface diffusion; and (vi) adsorption desorption elementary processes [30].
One or more of the previous steps may control the rate at which dye is adsorbed and the quantity of dye adsorbed onto the solid particle. In many cases, there is a possibility that intraparticle diffusion will be the rate-limiting step, which is normally determined using the equation proposed by Weber and Morris [31]:

$$q_e = k_p\, t^{1/2} + C \qquad \text{..... (7)}$$

Where q_e (mg/g) is the amount adsorbed at time t, k_p is the intraparticle rate constant ($mg.min^{-1/2}.g^{-1}$) and C is the intercept.
q_e was found to be linearly correlated with $t^{1/2}$. The k_p values were calculated using correlation analysis Table (5). The R^2 value reveals the occurrence of an intraparticle diffusion process [31]. The intraparticle diffusion plots are presented in Figure (8). It can also observed that the plots did not pass through the origin, this was indicative of some degree of boundary layer control (the larger the intercept, the greater the boundary-layer effect) and this further showed that the intraparticle diffusion was not the only rate-limiting step, but other processes might control the rate of adsorption. This confirms that sorption mechanism was a multi-step process, involving adsorption on the external surface, diffusion into the interior, ion exchange and inclusion complex formation, as previously reported [32].

Conclusions

The Chaff could be employed as adsorbent in wastewater treatment for the removal of congo red dye.

1. Langmuir isotherm model adequately described the adsorption of Congo red on chaff.
2. Congo red-Chaff reaction is endothermic and spontaneous as indicated by the positive value of enthalpy (ΔH) and negative value of free energy change (ΔG)
3. The process of adsorption is relatively fast and nearly 92.9 % of Congo red removal is achieved within 90 min. of contact between Chaff and the dye solution.
4. The kinetic of dye adsorption followed pseudo-second - order rate expression and demonstrated that intraparticle diffusion plays a significant role in the adsorption mechanism.

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Table (1): Langmuir constants for the adsorption of Congo red on Chaff

R^2	q_o	K_L
0.8990	10.25641	0.115412

Table (2): Effect of temperature on the maximum adsorbed quantity for adsorption of Congo red onto Chaff

T (k)	$10^3/T$ (k^{-1})	X_m(mg/g)	$\ln(X_m)$
		C_e =15.40	
298	3.36	3.24	1.18
313	3.19	3.42	1.23
328	3.05	3.59	1.28

Table (3):Values of thermodynamic functions of adsorption process of Congo red on Chaff at 25 °C

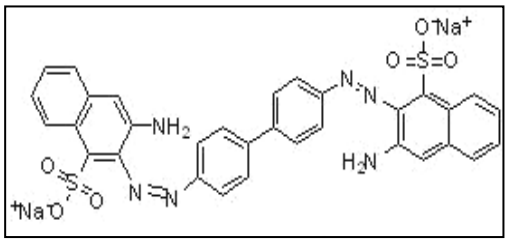
ΔH $kJ.mol^{-1}$	ΔG $kJ.mol^{-1}$	ΔS $J.mol^{-1}.K^{-1}$
+2.779	-3.560	+21.272

Table (4): Adsorption kinetic parameters of dye on Chaff

Pseudo-first order			*Pseudo-second order*			
k_1 (min^{-1})	q_e (mg/g)	R^2	k_2 ($g.mg^{-1}.min^{-1}$)	q_e (mg/g)	R^2	h ($mg.g^{-1}.min^{-1}$)
0.0203	0.3229	0.6427	0.9169	1.6268	1	2.4266

Table (5):The intraparticle rate constant for the adsorption of dye onto Chaff

k_p ($mg.g^{-1}.min^{-1/2}$)	*Intercept*	R^2
0.045	1.3096	0.8557



Scheme (1): The chemical structure of Congo Red

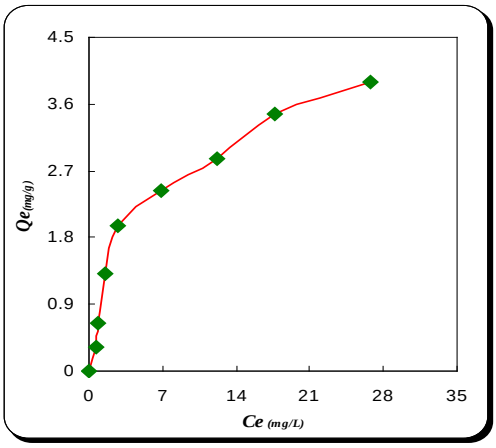


Fig. (1) :Adsorption isotherm of Congo red on Chaff at pH ≈6.8 and constant temperature (25°C)

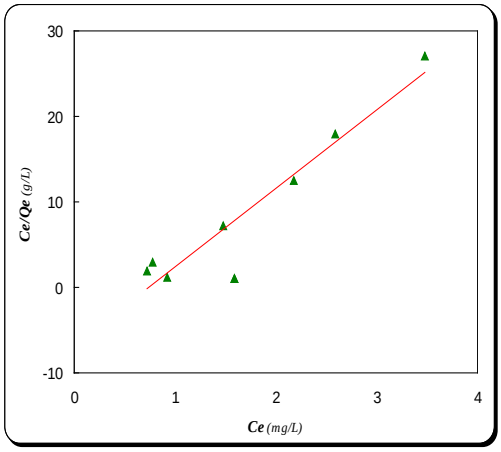


Fig.(2):Linear form of Langmuir isotherm of Congo red on chaff

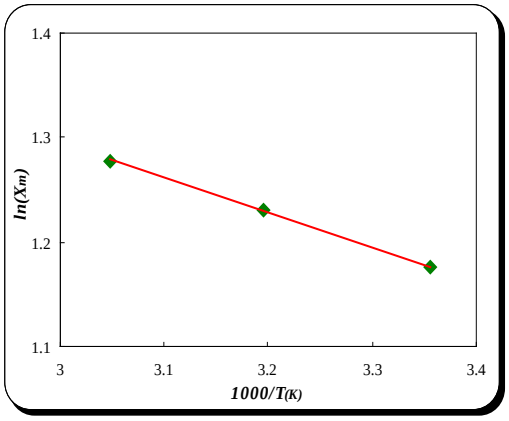


Fig. (3): Adsorption isotherms of Congo red on Chaff at pH» 6.8 and different temperatures

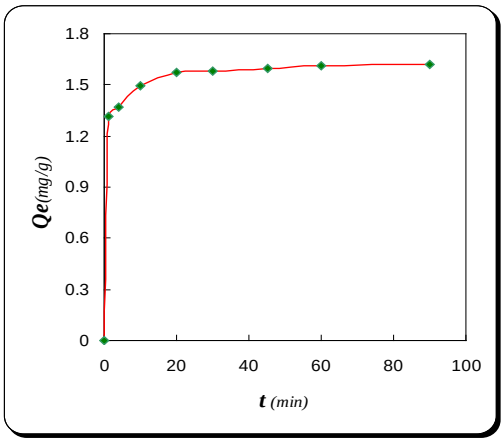


Fig. (4): Plot of ln X_m against reciprocal absolute temperature for adsorption of Congo red on Chaff

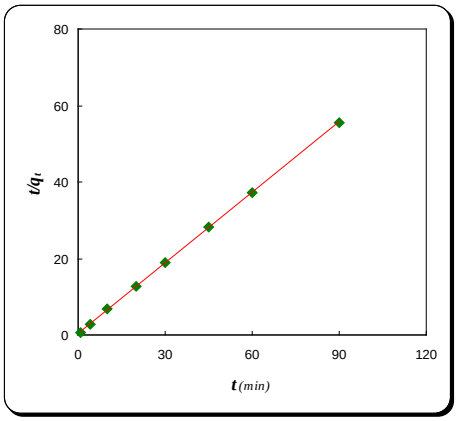


Fig.(5): Adsorption kinetics of Congo red on Chaff

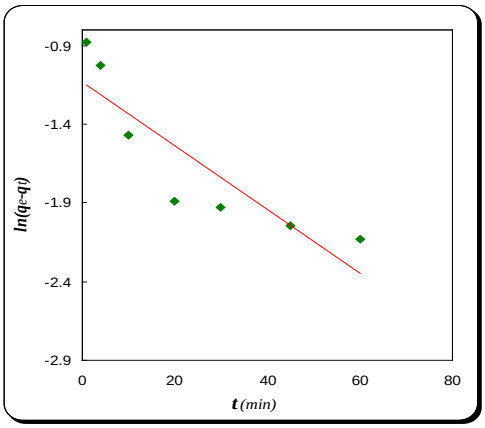


Fig.(6):The applicability of the pseudo first order kinetic model to Congo red on chaff

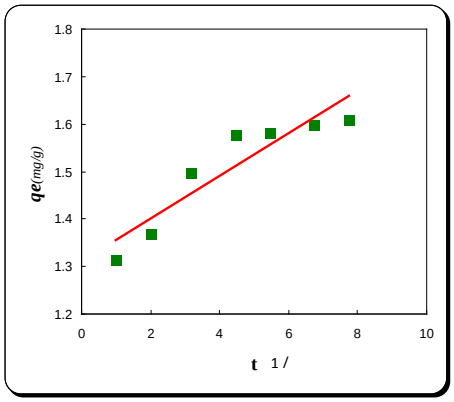


Fig. (7):The applicability of the pseudo second order kinetic model to Congo red on chaff

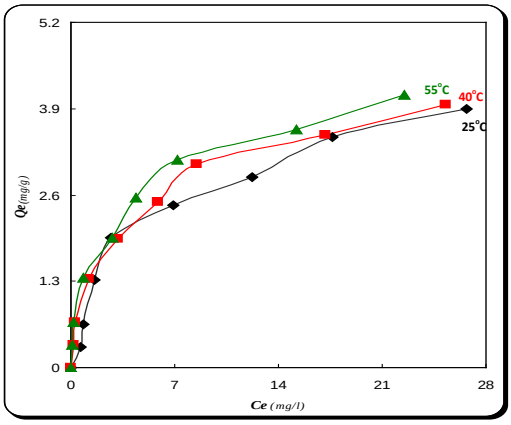


Fig. (8): The intraparticle diffusion model for adsorption of Congo red by Chaff

ازالة صبغة الكونغو الحمراء من محلولها المائي باستخدام قشور الحنطة المحلية : دراسة حركية وثرموديناميكية

عبدالمحسن عبدالحميد الحيدري
قسم علوم الكيمياء / كلية التربية للعلوم الصرفة (أبن الهيثم) / جامعة بغداد
سجى صالح جبار الطويل
قسم الكيمياء/ كلية العلوم/ جامعة القادسية
ليث سمير جاسم
قسم الكيمياء / كلية التربية / جامعة القادسية

استلم البحث في : 20 تشرين الثاني 2011 ، قبل البحث في : 18 آذار 2012

الخلاصة

يعنى هذا البحث بدراسة أمتزاز صبغة الكونغو الحمراء من محلولها المائي على سطح قشور الحنطة، وكان الغرض من الدراسة هو البحث عن أفضل الظروف الواجب توفرها في عملية تنقية المياه. بينت الدراسة ان ايزوثيرم امتزاز الصبغة نوع (L) طبقا لتصنيف Giles وان عملية الامتزاز تتبع ايزو ثيرم لانكماير.
تم دراسة عملية امتزاز الصبغة على سطح قشور الحنطة عندة ثلاث درجات حرارية ($55, 40, 25^oC$)، كما حسبت الدوال الثرموديناميكية ($\Delta H^o, \Delta S^o, \Delta G^o$) لعملية الامتزاز. وقد وجد ان كمية امتزاز الصبغة تزداد مع زيادة درجة الحرارة (امتزاز ماص للحرارة).
أن الزمن اللازم لحدوث الاتزان في عملية امتزاز الصبغة المذكورة أنفاً هو 90 دقيقة. وقد وجد ان كمية الامتزاز القصوى لصبغة الكونغو الحمراء على سطح قشور الحنطة هي 92.9% عند درجة حرارة 25 م^o.
أظهرت نتائج حركية الامتزاز وبتطبيق قوانين السرعة من المرتبة الاولى والثانية بان عملية امتزاز الصبغة على سطح قشور الحنطة تتبع قانون السرعة من المرتبة الثانية. نوقشت النتائج على ضوء التركيب الكيميائي للصبغة وطبيعة سطح قشور الحنطة. طبق قانون الانتشار الضمني للدقائق لدراسة حركية امتزاز الصبغة، وقد أظهرت النتائج ان الانتشار الضمني للدقائق يلعب دوراً رئيسياً في عملية الامتزاز.

الكلمات المفتاحية : الامتزاز، صبغة الكونغو الحمراء، قشور الحنطة