

Biosorption of Reactive Azo Dye from synthetic wastewater using low cost Dead Sludge Biomass

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Abstract:

Reactive azo dye biosorption behavior on dead sludge biomass was studied by sets of the equilibrium and time-based experiments under several conditions. Experimental data were estimated to find out kinetic characteristics. Biosorption process were found to follow the pseudo second-order kinetic model equation. Biosorption isotherm connect well with the Langmuir and Freundlich isotherm model and the maximum sorption capacity of dye estimated is 62.89 mg/g. Thermodynamic parameters were calculated based on Van't Hoff equation. The average change of standard enthalpy ΔH° was -25.89 kJ/mol. Negative ΔH° and ΔG° values indicate the adsorption process for dye onto the studied dead biomass is exothermic and spontaneous. The average change of entropy ΔS° was -64.52 J/mol.K also negative, which suggests a decrease in the freedom of the system. Equilibrium constant K_d was estimated from Langmuir and Freundlich isotherm model constants.

Key words: Reactive Azo Dye, dead biomass, thermodynamics, kinetic; Biosorption.

1. Introduction

Reactive azo dyes are useful and suitable dyes used by industries worldwide. The colorants present in dyeing, textile, leather, food processing, cosmetic, dye, and paper manufacturing industries are main sources of dye pollution [1]. However, it has a high potential to produce pollutants. Treatment of dyed effluents presents numerous problems mostly due to recalcitrance and toxicity of dyestuffs.

Dyes are synthetic aromatic compound water-soluble dispersible organic dyes, having probable application in numerous industries. These chemical are not only toxic to humans but also found poisonous to



aquatic life [2] and the may result in food contamination [3].

It is quite difficult to treat the effluents by the physic-chemical and conventional biological processes, e.g. heat, wash, light, and oxidizing agents, used in regular treatment [4]. That is because of complexity of the colorants aromatic molecular structures [5]. Adsorption is the most effective physical process in the treating these dye wastewaters. Today activated carbon is commonly used for adsorption in various treatment plants [4]. But the producing costs for activated carbon is very high, there is a need of another material that is more cost efficient [4]. A low costs adsorbent is defined as one which is abundant in nature or one that is yields as a byproduct in another industry [5].

Some existing technologies (UV/ozone treatment via oxidative destruction, electrochemical reduction, photocatalytic degradation etc.) may have a certain efficiency in the removal of dyes, but their initial and operational cost are so great, that they constitute an inhibition to dyeing and finishing industries[6]. On the other hand, low cost technologies do not allow a desired degree of dye have removal or certain disadvantages. Therefore, in order to reach the desired degree of treatment,

it is necessary to integrate biological, chemical and physical processes.

In recent years, numerous adsorbents have been recognized as possessing good dye-binding capabilities [7, 8]. In particular, biomaterials of microbial origin have been very effective because of their cell wall constituents.

Biosorption involves a combination of passive and active transport mechanisms starting with the diffusion of the adsorbed component to the surface of the microbial cell. Some dyes, especially azo dyes, are

known to be biorecalcitrant pollutants even with carefully selected microorganism and under favorable conditions. Azo dyes are categorized by the presence of one or more azo bonds (R_1 -N=N- R_2) represent

about 70% by weight [9]. Since little is known on the biosorption of dyes to adsorptive microbial biomass. properties of the microorganisms for studied dyes should be [10]. **Biological** wastewater treatment produced a biological sludge (biosolid) including of inert materials and microorganisms.

The present study has been designed to utilize locally available inactive biomass, biological sludge as biosorbent to remove reactive azo dye from synthetic wastewater.



2. MATERIALS AND METHODS

Adsorbent: Heterogeneous cultures, Sewage sludge collected from Al-Rostomia, a third extension drying bed. The collected biomass was thoroughly washed with tap water until all the dirt was removed finely the biomass was washed with deionized water until the entire colour of the material was removed. The result wet biomass subjected to autoclaving for 30 min at 121 °C and used as dead biomass. . The physical, chemical characteristic (dead biomass) were measured and listed in Table 1. Anaerobic and facultative anaerobic microorganisms species, (Aeromonas E-coli, Pseudomonas aerginrsa, Clostridium, Staphylococcus sp and Salmonella sp, Rhizopusarrhizus, Saccharomyces erevisiae) were found in biomass from the drying bed using Absolute Process Instruments, API Instrument (Biomer-ieux, France). Adsorbate: Reactive azo dye (BlueH 3 R, Solid Wave length powder, 585nm, solubility 90g/l, pH 7.2+0.3, Mwt. 763.5, India, the chemical structure shown in Fig.1), was obtained from Al-Hilla textile factory south of Baghdad. Simulated stock solution was prepared by dissolving 10 g of dye in one litter of distilled water then diluted to the desired solution concentration

properties	value		
рН	7.0-7.5		
Surface area, m ² /g (*)	94.53		
Particle porosity	0.584		
CEC, meq/100g ^(**)	51.2		
Particle diameter,	0.45		
mm	0.45		
Heavy metals mg/l	0.02		
Microorganism			
species Total	4 1*10 ⁶		
Colony-forming	4.1 10		
unit(CFU)/ml			

Table 1 Physical chemical and biologicalcharacteristics of dead biomass.

^(*) Surface area analyzer, BET method, Quantachrome.com.(USA),

(**) CEC Cat ion Exchange Capacity



Fig. 1 Chemical stricture of reactive azo dye (Blue H_3R)

Dye solution concentration were determined at the maximum wave length λ =585nm using a spectrophotometer (Model SP-3000 plus, Optima Co., 2003, Japan), using OptiVie 3.2 software



Windows-2000/XP (survey scan at wavelengths 200-1100 nm). The residual dye concentration in the reaction mixture was analyzed by centrifuging (Model: J2-21, BECKMAN) at 5000 rpm, before measuring the absorbance of the supernatant of the sample. Calibration curve were prepared by measuring the absorbance of different known concentration of dye solution at λ_{max} (plotted between absorbance and concentration of the dye solution). These analyses were carried out in duplicate.

2-1:Adsorption Experiments

Batch experiment were carried out using 250 mL Erlenmeyer flasks. 50 mL of dye containing waste water and biomass were added to investigate factors such as initial concentration of dyes, pH, dosage of the biosorbent and agitation time influencing the rate and extent of uptake of dyes by dead biomass (biosorbent.)

These flasks were agitated in a temperature – controlled shaker at a constant speed of 150 rpm to study the effect of the above factors. Samples were withdrawn at suitable time intervals and these samples were centrifuged at 4000 rpm. The supernatant was used for analysis of the remaining dye concentration. The effect of pH on dye removal was studied over a pH range of 2-10. pH was adjusted by adding of dilute aqueous solutions of 0.1N HCl or 0.1N NaOH. For the optimum amount of adsorbent per unit mass of adsorbate ,

a 50 ml dye solution with constant concentration was used with different amounts of biomass till equilibrium reached. The kinetics of was determined adsorption was bv analyzing adsorptive uptake of the dye from the aqueous solution at time different intervals. The adsorption isotherm was found by agitating dyes solution of different concentrations with the known biomass till the amount of equilibrium was attained. The effect of temperature on the sorption characteristics was investigated at temperature range of 25-45°C. Initial dye concentration varied from 0 to 40 mg/L.

Effect of pH: The adsorbate solutions were prepared at various levels of pH. The dye solution were agitated with 1 gram of dead biomass (adsorbents). The amount of the



adsorbed dye was calculated. The optimum pH was determined from the plot of the pH against percent dye removal.

Effect of Adsorbent dosage: The adsorbate solutions were agitated with various dosages of dead biomass of 0.1, 0.2, 0.3, 0.5, 0.75 and 1.0 g / 50 ml at 150 rpm and 30°C for the equilibrium period. After the equilibrium period, the amount of the adsorbed dye and the optimum adsorbent dosage were determined. Then the graph of the adsorbent dosage vs percent adsorbate removal was plotted. The data obtained in the adsorption studies were used for the determination of Langmuir and Freundlich isotherms and for Lagergren rate constant and the second order rate kinetics.

Effect of Initial dye concentration:

Fifty ml at various concentrations (10-40 mg/l) were taken. One gram of dead biomass (adsorbent) was added to the aqueous solutions and the flasks were agitated on a rotary shaker (150 rpm) at 30°C. The flasks were withdrawn at 5, 10, 15,, 180 mins. The adsorbent was separated by centrifugation at 4000 rpm for 15 min. The concentration of remaining

dye was determined. Then the amount of the adsorbed dye was calculated. A plot was drawn to determine the optimum contact time to obtain equilibrium in adsorption.

Adsorption isotherms and kinetics: The Langmuir plot was obtained using the equilibrium time curves data (i.e. the adsorbate concentration was fixed, while the adsorbent dose was varied). Freundlich plots were obtained from the equilibrium data of the adsorbent dose effect (i.e. the adsorbate concentration was fixed, while the adsorbent dose was varied). Kinetic studies were also carried out with different initial concentrations of dye, while maintaining the adsorbent dosage at a constant level.

2-2:Biosorption Thermodynamic:

Temperature is one of the important parameters for the biosorption of dyes related with the thermodynamics of the biosorption process. Generally, there are two common types, exothermic and endothermic sorption processes which are determined based on decrease or increase in the temperature through biosorption. Adsorption thermodynamic studies of dye onto dead sludge biomass were



performed at four different temperatures: 298, 308, 318, and 328 K. If the sorption decreases with the increasing temperature it is described as an exothermal process whereas if the sorption increases with the increasing temperature, the sorption is an endothermal process.

3- RESULTS AND DISCUSSION

Effect of pH: pH value of 6.0 was found to be the best removal percent for azo dye biosorption where the maximum percent removal was 85 % at a concentration of 10 mg/L as shown in Fig.2. Dye adsorption on to dead biomass at extreme acidic and alkaline condition, decreased the removal efficiency of dye (10-40 The dependence of the mg/L). biosorption on pH can be associated with both the surface functional groups on the biomass cell walls and also the dye chemistry of the solution [11]. At a higher pH, the surface of biomass becomes negatively charged, which improves the positively charged of dye cations through electrostatic force of attraction and at lower pH, the surface charge may become positively charged (i.e. protonation of the cell wall), thus creating (H+) ions compete

effectively with dye cations toward actives sorption sites causing a decrease in the amount of adsorbed dye [17, 18, 19].



Fig. 2: Effect of pH-value on the removal efficiency of dye using dead biomass (at Temp. 30; contact time 180 min)

Effect of adsorbent dosage: When dead biomass was concerned. an optimum dye removal (94.1 %) was observed at a dosage of 20 g/L, and 10 mg/l (Figure 3). Minimum maximum dye removals of and 50 % and 94.1 % were observed at and 20 of 5 dosages g/50ml, respectively as shown in Fig.3. Consequently the optimum biomass of 20 g per 50 ml was used for biosorption of dye. This can be explaining the large availability of the surface area at higher concentration of adsorbent. Any further addition of the adsorbent beyond this would not cause any significant change in the adsorption due to the overlapping adsorption sites of adsorbent particles



resulting in decrease in total biosorbent surface area available to dye and an increase in diffusion path length [20].



Fig. 3: Effect of adsorbent dosage on the dye removal efficiency using dead biomass (at pH 6; Temp. 30°C; contact time 180 min)

Effect of initial dye concentration and agitation time: The effect of initial dye concentration and agitation time on the biosorption of dye was investigated and shown in Fig.4, the uptake of dye increased with increase agitation time, but remained in constant after the equilibrium time period whereas the percentage dye removal decreased with an increase in the initial dye concentration . At lower dye concentrations solute concentration to biosorbent site ratio is higher, which cause an increase in removal. dye At higher concentrations, lower adsorption yield is due to the saturation of biosorption sites [12,21].

The equilibrium time required for the maximum removal of dye by dead biomass was 140 min at all the dye concentrations studied. The percent dye removal at equilibrium time was found to be 94 % and 90 % for the dye concentrations of 10 and 40 mg/L respectively.



Fig. 4 Effect of initial dye concentration and agitation time on the dye removal efficiency using dead biomass (at adsorbent dosage 20 g/l; pH 6; Temp. 30°C)

Biosorption equilibrium time and isotherm models

The biosorption capacity was investigated with the Langmuir, and Freundlich isotherm models. Table 2 shows the equilibrium parameters for the different isotherm models for biosorption of dye onto dead biomass. For Langmuir equation, it is written as:

$$q_e = \frac{q_m k_L C_e}{1 + k_L C_e} \tag{1}$$



The linear form of Langmuir isotherm model equation can be written as:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m}$$
(2)

Where, Ce and q_e are equilibrium concentration of a dye in solution (mg/L) and equilibrium asdsorption q_m is the maximum capacity, capacity reflecting adsorption a complete monolayer (mg/g); K_L is equilibrium adsorption constant (L/mg) that is related to sorption energy.

A plot $\frac{C_e}{q_e}$ versus C_e should indicate a straight line possessing a slope of $\frac{1}{q_m}$ and an intercept of $\frac{1}{K_L q_m}$ The value q_m and K_L constant and of the correlation coefficients obtained from Langmuir model. Data obtained for the sorption of dye the in concentration range of 10 to 40 mg/L of dye were fitted to the Langmuir isotherm (Figure 5) Table 2. The isotherm was found to be linear over the whole concentration range studies and the correlation coefficient were extremely high as shown in Table 1. The values of q_m decrease with increase in temperature, thereby confirming that the process is exothermic [13].



Fig. 5 Langmuir isotherms for the adsorption of Reactive azo dye (BlueH_3 R). (at initial concentration 10 mg/l; pH 6; contact time 180 min)

Another dimensionless equilibrium parameter (RL) can be estimated using the following relation.

$$R_L = \frac{1}{1 + k_L C_0} \tag{3}$$

Where k_l is the Langmuir constant and C0 is the initial dye concentration in the solution (mg/l). According to Hall et al.[14] mathematical calculations show that this parameter gives an indication of isotherm. The values of RL (Table 2) were between 0 and 1 for selected dye which indicate the applicability (favorable) of the Langmuir isotherm.



Dimensionless equilibrium Table 2 different initial parameters at dye concentration

Initial conc.(mg/l)	10	20	30	40
RL	0.25	0.14	0.1	0.077

The equilibrium parameter, RL, is used to predict if an adsorption system is "favorable or unfavorable". RL=1 (Linear), 0<RL<1.0 (Favorable) and RL=0 (Irreversible).

Freundlich isotherm: The Frundlich isotherm model is another form of Langmuir approach for adsorption on amorphous surface. The model is applied to evaluate the adsorption on heterogeneous surfaces considering interactions between adsorbed molecules. The application of the Freundlich equation also suggests that sorption energy exponentially decreases on completion of the sorption centres of an adsorbent. This isotherm is an empirical equation, which can be employed to describe heterogeneous and systems is expressed as follow:

$$=K_F C_e^{1/n}$$

 $q_e =$ Where Ce the equilibrium is concentration of a dye in solution (mg/l), qe is the amount of dye sorbed on to biomass (mg/g), K_F is the Freundlich constant (L/g) related to the bonding energy. K_F can be defined as adsorption distribution the or

coefficient and represents the quantity of dye adsorbed onto adsorbent for unit equilibrium concentration. 1/n is the heterogeneity factor and n is a measure of deviation from linearity of adsorption. Equation 2 can be linearized in the logarithmic form as equation (5) and Freundlich constants can be determined:

$$Logq_e = LogK_F + \frac{1}{n}LogC_e$$
 (5)

The plot of $Logq_e$ versus $LogC_e$ was employed to generate the intercept value of K_F and the slope of 1/n. The computed correlation coefficients and Freundlich constants for the dyes studies are presented in the Table (3). The constant 1/n showed the sorption intensity and its fractional value (0<1/n<1) showed the heterogenous nature of sorbent surface.







Table 3 Langmuir and Frendlich parameters for Reactive azo dye (Blue H_3R).

Т	Langr	Langmuir isotherm		Freundlich isotherm		
(K)	K_L	q_m	R ²	K_F	$1/_{n}$	R ²
298	0.06	62.89	0.9413	5.22	06115	0.9794
308	0.096	55.87	0.9941	4.33	0687	0.9898
318	0.117	51.81	0.9964	3.94	0.6969	0.9862
328	0.137	48.78	0.9920	3.20	0.7671	0.9883

Effect of Temperature (Adsorption thermodynamic study)

The thermodynamic parameters are calculated using the following equations

 $\Delta G^{\circ} = -RT ln K_d$ (6) Where ΔG° is the free Gibbs energy change (kJ/mol). R is the universal gas constant (8.314 J/mol.K), K_d the thermodynamic equilibrium constant and T is the absolute temperature (K). K_d was derived from the constant of Langmuir and Freundlich model, respectively using the method from literature [16]. The thermodynamic calculation was based on the linear plot of ln K_d versus 1/T (Fig.7).



Fig. 7 Van t Hoff plot for the Reactive azo dye (Blue H_3R) biosorption on dead biomass.

The values of other parameters such as enthalpy change (ΔH°) and entropy change (ΔS°) , may be determined from Van t Hoff equation:

$$lnK_d = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{RT}$$
(6)

The thermodynamic parameters are listed in Table 4.

Table 4 Thermodynamic parameters of the reactive azo dye biosorption on the studied dead biomass at different

temperatures

K _d	Т	K _d	ΔG°	ΔS°	ΔH°
	(K)		(kJ/mo	(J/m	(kJ/
			I)	ol.K)	mol)
Langm.	298	16.67	6.97		
$K_d =$					-
$1/K_l$				-50.00	21.68
	308	10.42	-6.00		
	318	8.55	-5.67		
	328	7.3	-5.42		
Frendli.	298	14.91	-3.314		
$\begin{array}{l} K_d = \\ K_F^n \end{array}$				-79.04	- 30.11
	308	8.44	-0.986		
	318	7.15	-1.089		
	328	4.56	-0.309		

The standard free energy ΔG° at all is negative. This temperatures suggests the biosorption process confirm the spontaneous nature and the spontaneity decrease with the increase in temperature. The standard sorption enthalpy and entropy are negative value too, this indicates the whole process is exothermic. The negative value of standard enthalpy indicates the whole process is exothermic [22].



Adsorption kinetics:

The kinetics of dye biosorption on the adsorbent, biomass were analyzed by intra-particle kinetic models: diffusion, fractional power, pseudofirst order, and pseudo second order. The kinetic results shown in Table 5 and figures 8. The confirmation between experimental data and the predicted values using different models were expressed by the correlation coefficients (R^2 value close or equal to 1).

The intra-particle diffusion model is expressed as

 $q_t = k_{id} t^{0.5} + C$ (7)

Where C is the value of intercept (mg/g) which gives an idea about the boundary layer thickness, the large intercept; the greater is the boundary layer effect. q_t (mg/g) is the amount adsorbed at time t (min), k_{id} (mg/g min^{0.5}) is the rate constant of intraparticle diffusion.



Fig. 8 Intraparticle diffusion model for biosorption of reactive azo dye onto dead

biomass (at initial concentration 10 mg/l; pH 6; Temp. 30°C)

The plot of q_t versus \sqrt{T} was employed to generate the intercept value of C and the slope of k_{id} (Table 5).

The fractional power kinetic model may be expressed as $q_t = Kt^x$, the linearization form:

 $\ln q_t = \ln K + x \ln t \tag{8}$

Where qt (mg/g) is the amount adsorbed at time t (min.), K is the constant of power function model, and x is the rate constant of power function [22].

The plot of lnq_t versus lnt was employed figure 9 to create the intercept value of lnK and the slope of x(Table 5).



Fig. 9 Fractional power model for biosorption of reactive azo day onto dead biomass

Pseudo-First-Order Kinetic Model is one of the most usually used sorption rate equations for the sorption of solute from a liquid solution. It may be represented to get a linear form as:



 $\ln(q_{eq} - q_t) = \ln q_{eq} - K_L t$ (9) Where q_t is the amount of pollutant adsorbed at time t (mg/g); q_{eq} the amount of pollutant is adsorbed at equilibrium (mg/g); and K_L is the equilibrium rate constant of pseudofirst sorption (min-1).



Fig. 10 Pseudo first order model for biosorption of reactive azo day onto dead biomass

The plot of $ln(q_{eq} - q_t)$ versus t was employed to generate the intercept value of $ln q_{eq}$ and the slope of K_L (Table 5).

The pseudo second-order model gives:

$$\frac{dq_t}{dt} = k_2(q_e - q_t) \tag{10}$$

Where k_2 is the equilibrium rate constant for pseudo second order adsorption (g/mg.min). Integration of Equation 10 by applying boundary conditions t=0 to t and q_t =0 to q, gives:

 $\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$ (11)

 $h_i = k_2 q_e^2$

Where h_i is the initial dye biosorption (mg/g.min). the slop and intercept of plot t/q_t vs t where used to calculate the pseudo second-order rate constant k_2 and q_e . The result shown in Figure 11 and Table 5.



Fig. 11 biosorption of reactive azo day onto dead biomass (Pseudo second order model)

Table 5 Kinetic model parameters forreactive azo dyebiosorption onto deadbiomass.

Model	Parameters		
	C (mg/g)	6.9816	
Intra-particle diffusion	K _{id} (mg/g min ^{0.5})	0.8133	
	Correlation	0.8687	
	Coefficient		
Fractional power	K (mg/g)	2.94	
	x (min⁻¹)	0.159	
	Correlation	0.8163	
	Coefficient		
Psedo-first- order	q _{eq} (mg/g)	1.97	
	K _L (min⁻¹)	0.0563	
	Correlation	0.8515	



	Coefficient	
Psedo-second- order	q _e (mg/g)	4.29
	k_2 (g/mg.min).	22.05
	Correlation	0.9903
	Coefficient	

The kinetic results shown in Table 5 and figures 8, 9, 10, and 11. The data obtained show a good compliance with pseudo second-order equation.

These result determined that the experimental data for the biosorption kinetics of reactive azo dye on dead sludge biomass were fitted by the pseudo second-order kinetic model. Similar phenomenon was observed for the adsorption of basic dyes onto pomegranate peel, apricot stone and cotton [15]. The intraparticle diffusion model is not zero, suggesting that biosorption proceed from boundary layers mass transfer across the interfaces to the intraparticle diffusion within the pores of dead sludge biomass (biosorbent).

Conclusions

The result obtained from this investigation demonstrate that dead sludge biomass prepared from sewage sludge is very efficient in biosorption of reactive azo dye from simulated wastewater. On the comparison of Reactive azo dye uptake capacity of dead sludge with other adsorbent [23-26] used, the dead sludge is a good

biosornent and it removal very high quantity of dye was found in this investigation. The biosorption capacity of dead sludge biomass is strongly dependent on the pH-value. The uptake of dye increased with agitation time increase in and decreased with an increase in the initial dye concentration. The maximum biosorption capacity under the condition of this experiment is with and decrease 62.89 mg/g, increase in temperature, and the sorption enthalpy and entropy are negative, thereby confirming that the process is exothermic. The process confirm the spontaneous nature and the spontaneity decrease with the (∆**G**°is increase in temperature negative). The system is favorable for dye, selected the biosorption isotherms follow both Langmuir and Freundlich model.

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الامتزاز الحيوي للصبغة من فضلات المياه الصناعية باستخدام الكتلة الحيوية للحمأة الميتة قليلة الكلفة

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الخلاصة:

تم دراسة الامتزاز الحيوي لصبغة Reactive azo على الكتلة الحيوية اللاهوائية الميتة. من خلال اجراء سلسلة من التجارب تحت ظروف مختلفة لدراسة تاثير المتغيرات مثل التركيز الابتدائي للصبغة، جرعة المادة المازة، الدالة الحامضية، ودرجة الحرارة. ومن التجارب تم الحصول على الخصائص الحركية. ووجد ان عملية الامتزاز الحيوي تتبع نموذج pseudo second order وهو الاكثر نموذج رياضي لتمثيل النتائج العملية. وجد ان أقصى امتزاز للصبغة بسعة امتزاز 62.89 (ملغم/غم). تم اعتماد ثوابت لانكمير وفريندلش لحساب ثابت التوازن وايجاد معدل تغير الانثالبي 25.89- (كيلوجول/مول) ومعدل تغير الانتروبي 5⁴⁰ - 5⁴⁰ - 5⁴⁰ در ريلوجول/مول) ومن القيم السالبة للمعاملات الحرارية تبين بان العملية تلقائية مستمرة باعثة للحرارة.

الكلمات الرئيسية: الامتزاز الحيوي، صبغة، كتلة حيوية مازة، ديناميكا حرارية حركية