Batch Scale Removal of an Organic Pollutant Amaranth Dye from Aqueous Solution using *Pisum sativum* Peels and *Arachis hypogaea* Shells as Adsorbents

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Summary: The goal of this study was to utilize low cost and environmentally friendly adsorbents for batch scale removal of Amaranth dye from aqueous medium. Peels of *Pisum sativum* (Pea) and *Arachis hypogaea* (Peanut) were utilized to investigate their dye removing capacity. The optimized adsorption conditions for *Pisum sativum* (P.S.P) and *Arachis hypogaea* (A.H.S) were: adsorbent dose; 0.6 and 0.4 g, contact time; 45 and 10 minutes, pH; 2.0 for both, agitation speed; 150 and 100 rpm and temperature; 60 and 50 °C for P.S.P and A.H.S respectively. The adsorption data well suited to Langmuir isotherm. Maximum adsorption capacities were found to be 144.93 and 10.53mg/g for P.S.P and A.H.S respectively. Feasibility of the process was indicated by negative values of thermodynamic parameters ΔG^0 for both adsorbents. Kinetic studies indicated that adsorption of Amaranth dye from aqueous medium by *Pisum sativum* peels and *Arachis hypogaea* shells followed pseudo-seconder order kinetics. It was concluded that *Pisum sativum* peels are more effective adsorbent for removal of Amaranth from aqueous solution as compared to *Arachis hypogaea* shells.

Key words: Amaranth; Pisum sativum; Arachis hypogaea; adsorption.

Introduction

Increased industrialization is an important need in the modern epoch. So, industrial development has prevailed on the environment with its downsides. Textile, printing and paper industries frequently use toxic chemical dyes. The textile industry is thought to be the major water polluter. 10⁵ types of dves are commercially available and annual production of dyes is seven lakh tons. During dyeing process 10-25% dye is lost. 2-20% is discharged into water bodies as aqueous effluents. Effluents from dyeing and textile industries contain organic and inorganic colorants [1]. Discharge of such industrial effluents in the rivers or oceans can be very toxic for aquatic fauna and flora, which may lead to the serious diseases in human beings. Mostly synthetic dyes contain complex organic compounds which are mutagenic and carcinogenic in nature [2-4]. Reactive dyes form strong covalent bonds with fibre. So, their use is very common in textile industry. People exposed to reactive dyes may suffer with asthma, rhinitis, contact dermatitis and utricaria. Azo dyes are more than 50% of textile dyes. Azo dyes are anionic in nature and soluble in water. Due to their good fastness, variety of colors and low energy consumption azo dyes are frequently used in textile industry. These dves are resistant to photolysis and chemical decomposition and have adverse effects on human health [5-7]. Amaranth is an azo dye. 2-hydroxy-1-(4-sulphonato-1-Trisodium naphthylazo) naphthalene-3, 6-disulphonate is its IUPAC name [8]. It is widely used in coloring textile materials, wood, leather, paper, food etc. [9]. In 1970

Russian research revealed its carcinogenic properties. So, its use was banned in many countries [10]. Some properties of Amaranth dye are given in Table-1 and structure in Fig. 1

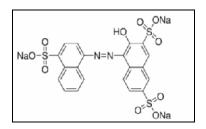


Fig. 1: Structure of Amaranth.

Table-1: Properties of Amaranth dye.

Property	Value
Class	Azo dye
Molecular formula	$C_{20}H_{11}N_2Na_3O_{10}S_3$
Molecular weight	604.473048
Density	1.5
Maximum absorption	525nm
Solubility	Soluble in water, glycerol, glycol,

Other methods to remove dyes like: coagulation, photo-catalytic biological or degradation, ozonation, membrane separation, electrolysis or adsorption on activated charcoal were expensive, difficult to apply, sludge disposal problem and fuel plus electricity consumption make them unattractive for researchers to adopt them in future. Adsorption is an effective technique for treatment of waste water. Features like cost, flexibility, ease of design and operation make this technique better as compared to other techniques. This method also has advantage of harmless end products [11, 12]. Adsorption is a very helpful method for removal of anionic dyes. This method involves the use of many low cost adsorbents like peat steel plant slag, bentonite clay, fly ash, etc. [13]. The passive removal of toxic heavy metals or toxic organic dyes by inexpensive biomaterials is termed as biosorption. Biosorption of these types of hazardous organic materials by selected live and dead micro-organisms and waste of plant materials has now become an emerging technique [14].

This research work includes batch scale removal of Amaranth dye from aqueous solution using Pisum sativum peels and Arachis hypogaea shells. Pisum sativum is commonly known as garden pea, English pea, Green pea and Field pea. It is grown in many parts of the world. It is a cool season fruit. Peas have high levels of nutrition. They contain fiber, protein, minerals, vitamins and lutein. Dry weight is about one-quarter protein and one-quarter carbohydrates (mostly sugars). Peas are helpful in blood sugar regulation. It prevents some diseases like arthritis and Alzheimer. It helps to strengthen the immune system [15]. Arachis hypogaea has many common names such as earth nuts, Ground nuts, and Monkey nuts. It was probably first grown in valleys of Paraguay. It is grown in most of the parts of world. China shows leading production of Peanut plant. Peanut oil is used in cooking. Peanut flour is rich in protein content [16]. This research study was carried out to characterize Pisum sativum peels and Arachis hypogaea shells to evaluate their absorptive characteristics for removal of Amaranth dye.

Experimental

Reagents and Instruments

Amaranth dye (λ_{max} : 525 nm), 0.1M HCl, 0.1M NaOH (Friends laboratory chemicals), UV-Visible double beam Labomed-3500 UVD spectrophotometer, Measuring balance (VELP Scientifica 230 V,50 Hz), pH meter (HANNA pH 211), Orbital flask shaker (model OSM-747), Electric Grinder (Ken-Wood) were used.

Preparation of Biosorbents

Pisum sativum (pea) peels and *Arachis hypogaea* (peanut) shells were obtained from pea and peanut respectively, which were purchased from local markets of Lahore, Pakistan. They were washed and peeled off. Then their waste material was dried in sunlight, followed by oven drying at 80 ^oC. Afterward they were crushed into fine powder with

electric grinder and sieved through 60 mesh (250 microns) in order to get fine powder of homogeneous size.

Characterization of Adsorbents

Characterization of both adsorbents was done by recording their FT-IR spectra shown in Fig. 2 and 3, pH, Ash, volatile organic compounds (VOC) and moisture contents.

Preparation of Adsorbate Solutions

Stock solution of Amaranth dye was prepared by dissolving 0.1 g of Amaranth dye in 4-5ml of ethanol. Then the solution was dilutes up to 100ml with distilled water. 5-25 ppm (5, 10, 15, 20, 25 ppm) solutions were prepared by diluting stock solution.

Adsorption Experiments

Different biosorption experiments were performed to evaluate the effect of different parameters such as adsorbent dose, contact time, pH, temperature and agitation speed. The dye solutions were centrifuge after biosorption experiment and decanted, instead of filtration in order to avoid filter paper adsorption capability interference and then mechanism of adsorption and feasibility of process was determined by isothermal study. After adsorption concentration of dye was determined by measuring absorption of filtrate. Percentage adsorption was calculated by eq.1:

Adsorption (%) = $C_0 - C_e / C_0 \times 10$ (1)

Where " C_0 " (ppm) is initial concentration and " C_e " (ppm) is remaining concentration of dye which is determined spectrophotometrically. Average readings were taken in order to reduce error. Amount of adsorbed dye was calculated by eq.2:

$$q = (C_0 - C_e) V/m$$
(2)

Where "q" is the amount of dye adsorbed by biosorbent in mg/g. "V" is the initial volume of dye solution in litre. "m" is the mass of adsorbent in grams [19].

Results and Discussion

Adsorbents were characterized and optimum conditions for batch scale removal of dye were found. Maximum wavelength of Amaranth dye was found at 525 nm as clear from Fig. 4. For calibration, 5-25 ppm solutions of Amaranth dye were prepared. Their absorbance was measured on UV-Visible double beam Labomed-3500 UVD spectrophotometer. Calibration graph is shown in Fig. 5.

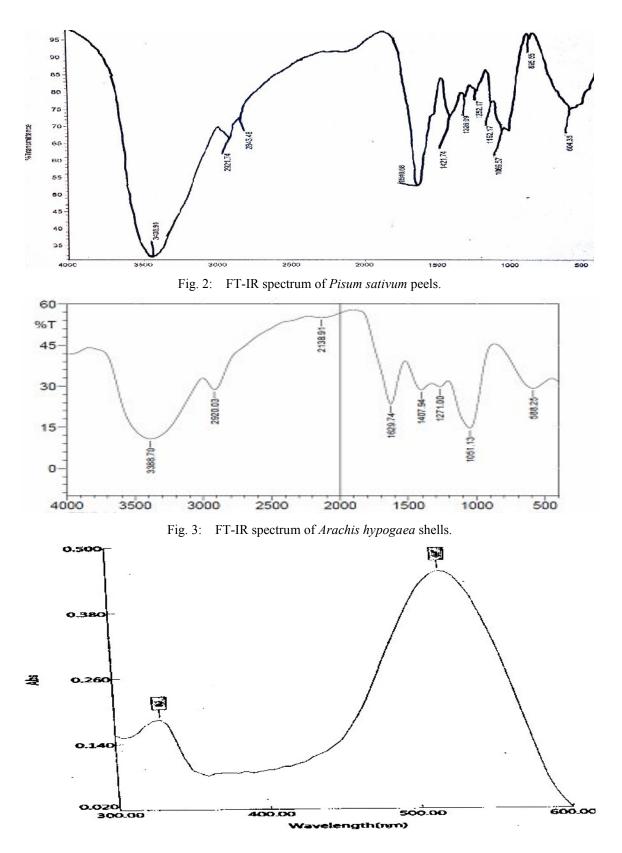


Fig. 4: λ max of Amaranth at 525nm.

1. Characterization of Adsorbents

The results of characterization of adsorbent are given Table-2 and FT-IR spectra in Fig. 2 and 3. FT-IR spectra were compared to literature for evaluating the presence of different functional groups. FT-IR spectrum of Pisum sativum peels as shown in Fig. 2 had an intense band at 3436cm⁻¹ showed the presence of O-H groups. C-H stretching bands were indicated by peak at 2921cm⁻¹. C=O bond was indicated by the peak at 1640cm⁻¹. The band at1069 cm⁻¹ showed the presence of C-O-C. These functional groups act as binding sites for adsorption [17]. Arachis hypogaea shells FT-IR spectrum is shown in Fig. 3. It was found that it mainly constitutes polysaccharides, lipids and proteins. Presence of many polar groups such as hydroxyl, carboxyl, amino and carbonyl groups was indicated. Such groups effectively act as binding sites for removal of dye and other pollutants. At 3388cm⁻¹ broad band appeared which showed the presence of hydroxyl group. At 2920cm⁻¹ a peak was appeared which indicated the presence of C-H groups. The peak at 1407cm⁻¹ assigned to the C-O group. These functional groups acted as binding sites for adsorption of dye molecules [18].

Table 2: Results for characterization of adsorbents.

Characteristics	Pisum sativum	Arachis hypogaea
рН	7	5
Moisture content %	10	6.8
Ash %	3.4	4.40
VOC %	21.6	30

From Table-2, it is obvious that *Pisum* sativum peels had 10% moisture and 3.4% ash content, whereas *Arachis hypogaea* shells had 6.8% moisture and 4.40% ash contents. Ash content is residue left when carbonaceous material is burnt. Lower ash for *Pisum sativum* peels refers the small density of particles indicating it as a good raw material for adsorption. High ash and volatile organic compounds (VOC) acts to reduce the efficiency of adsorbent [20].

2. Effect of Adsorbent Dose

The effect of adsorbent dose on adsorptive removal of Amaranth dye using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S) as adsorbents was studied within range of 0.2-2.0 g with variation of 0.2 g. Results are given in Fig. 6. The graph shows comparative study of adsorbent dose effect of both adsorbents. Amaranth removal increased up to a certain extent then started decreasing to almost constant values. Maximum percentage adsorption 45.6% was obtained at lower adsorbent dose 0.6g for Pisum sativum peels (P.S.P) and 39.8% at 0.4g for Arachis hypogaea shells (A.H.S). Percentage adsorption increases with increase in adsorbent dose due to availability of more adsorption sites. After the equilibrium is maintained biosorption is less affected with addition of more adsorbent dose. Because adsorbent-adsorbent interaction increases than adsorbent-adsorbate interaction and at this stage more adsorbent dose interferes with binding sites hence decrease in percentage removal starts beyond an optimum value [21, 22].

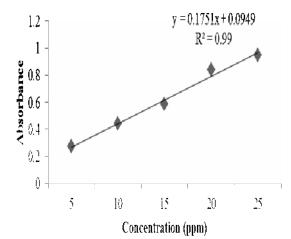


Fig. 5: Calibration Curve for standard solutions of Amaranth dye.

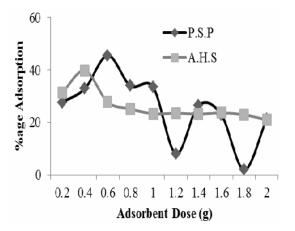


Fig. 6: Graphical comparison of the effect of adsorbent dose on adsorption of Amaranth dye using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

3. Effect of Contact Time

Effect of contact time was studied from 5-60 minutes with variation of 5 minutes. Fig. 7 explains

the result of comparative effect of contact time on the adsorption of Amaranth by two different adsorbents. It is clear from the results that *Pisum sativum* peels showed more percentage removal than *Arachis hypogaea* shells. Optimized contact time for *Pisum sativum* peels was 45 minute with %age removal of 77 % and for *Arachis hypogaea* shells 10 minute with percentage removal of 69.48 %. Adsorption of Amaranth increased with increase in contact time till an optimum contact time was reached. The reason behind the fact is that in increased contact time there is more time for dye molecules to make complex with the biosorbent [23, 24].

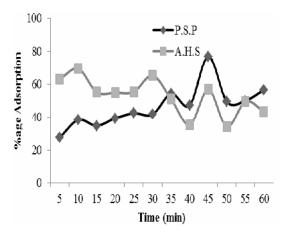


Fig. 7: Graphical comparison of effect of contact time for adsorption of amaranth dye using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

4. Effect of pH

Its effect was studied within pH range of 1-10. Results are shown in Fig. 8. From the results, it is obvious that optimized pH value for both adsorbents Pisum sativum peels and Arachis hypogaea shells is 2.0. Pisum sativum peels showed 88.84 % and Arachis hypogaea shells showed 88.80 % adsorptive removal of Amaranth. There is gradual decrease in percentage adsorption towards higher pH. The reason behind the phenomenon is that at acidic pH Amaranth dye is stable. It becomes precipitated at basic pH. The pH conditions cause the structural changes in dye due to which it undergoes instability when pH is changed. Additionally at higher pH weakening of the attractive forces between adsorbent and adsorbate take place which results in the decrease of adsorption of the dye on the adsorbent [25, 26].

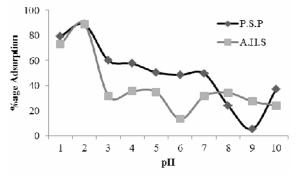


Fig. 8: Graphical comparison of effect of pH on adsorption of Amaranth dye using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

5. Effect of Temperature

Temperature is an important factor. It influences the adsorption process on large scale. Its effect was studied in the range of 20-70 °C. Fig. 9 shows that maximum removal of Amaranth dye occurred at 60°C for *Pisum sativum* peels (P.S.P) and percentage removal was noted 88.2%. Arachis hypogaea shells (A.H.S) gave maximum adsorption 72.48% at 50° C. It was observed that adsorption decreased with further increase in temperature. As the temperature increased from 20° C- 60° C in case of Pisum sativum peels (P.S.P) and 20-50^oC in case of Arachis hypogaea shells (A.H.S) we can notice the increasing trend of adsorption. It is because of the increase in the rate of diffusion of adsorbate molecules around the outer boundary and into the pores of adsorbent particles. Moreover, temperature change causes change in equilibrium capacity of the adsorbent for a particular adsorbate [27, 28].

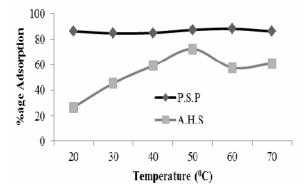


Fig. 9: Graphical comparison of effect of temperature on adsorption of Amaranth dye using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

6. Effect of Agitation speed

Effect of agitation speed was studied from 0-200 rpm. From the results in Fig. 10 it is noted that optimum agitation speed for removal of Amaranth using Pisum sativum peels (P.S.P) was 150 rpm and using Arachis hypogaea shells (A.H.S) was 100 rpm. Adsorption increased with increase in agitation speed. At lower shaking speed the adsorbent become accumulated in the bottom hiding several binding sites below the mass of upper layers of adsorbent. So, the agitation speed must be sufficient enough to expose maximum binding sites. However agitation speed beyond the optimum value will make adsorption process difficult because of unavailability of sufficient time for adsorbent to uptake adsorbate. Agitation speed controls the adsorption either by film diffusion or pore diffusion. In case of lower agitation speed thick fluid film is formed around the particle and film diffusion becomes the rate limiting step. With the increase in agitation speed the thickness of outer film is decreased, turbulence is increased and rate of diffusion is increased. Equilibrium was also attained rapidly at high agitation speeds than at lower agitation speeds.

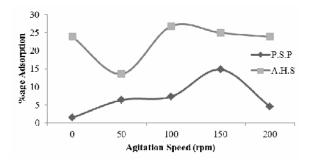


Fig. 10: Graphical comparison of effect of agitation speed on adsorption of Amaranth dye using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

7. Isothermal study

Adsorption isotherms were studied by applying optimized conditions of each factor. Mechanism of adsorption was determined.

(A) Langmuir Isotherm

It was plotted and with the help of graph parameters of standard straight line equation were evaluated. Langmuir equation is written as:

$$1/q_e = (1/bq_m) Ce + 1/q_m$$
 (3)

Where " q_e " is the amount of dye adsorbed in mg/g. " C_e " is the concentration of dye at equilibrium in mg/L. " q_m " (mg/g) and "b" (L/mg) are Langmuir parameters. From the graph values of these parameters were evaluated. R_L is used to describe important characteristic of Langmuir isotherm. It has no unit. It is given by the formula:

$$R_{L} = 1/(1 + bC_{0}) \tag{4}$$

The type of isotherm is indicated by R_{I} value. If $R_L > 1$ the isotherm is unfavorable. If $R_L = 1$ the isotherm is linear. If $0 \le R_L \le 1$ the isotherm is favorable and it is irreversible if the $R_L = 0 R_L$ value in this study was in the range of 0.2 to 0.6. Comparative graph of Langmuir isotherm for Pisum sativum and Arachis hypogaea is shown in Fig. 11 and Langmuir parameters are given in Table-3. R² values of the linearized Langmuir isotherms for both adsorbents (Pisum sativum peels and Arachis hypogaea shells) showed that experimental data could be better fitted in Langmuir isotherm. It is also obvious that R^2 and q_{max} values for *Pisum sativum* peels were higher indicating it a better adsorbent than Arachis hypogaea shells for removal of Amaranth [30].

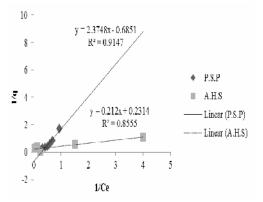


Fig. 11: Comparative Langmuir isotherm plot for the adsorption of Amaranth from aqueous solution using *Pisum sativum* peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

Table-3: Langmuir parameter for adsorption of Amaranth by Pisum sativum peels (P.S.P) and *Arachis hypogaea* shells (A.H.S).

Adsorbents	Slope	Intercept	\mathbf{R}^2	q _m (mg/g)	b (L/mg)
Pisum sativum peels	2.3748	-0.6851	0.9147	144.93	0.042
Arachis hypogaea shells	0.212	0.2314	0.8555	10.53	0.54

(B) Freundlich Isotherm

It is expressed by the following equation: $lnq_e = ln K_F + (1/n) lnC_e$ (5)

where " K_F " represents adsorption capacity and adsorption intensity is described by "1/n". Both are Freundlich parameters and their values are given in Table-4. Comparative graph for both adsorbents is given in Fig. 12. Their values are derived from the intercept *ln* K_F and slope 1/n. Adsorption intensity or the heterogeneity of the surface is evaluated by slope (1/n). If the value of slope is closer to zero it shows greater heterogeneity. Comparison of K_F values of both adsorbents showed that in case of biosorptive removal of Amaranth dye using *Arachis hypogaea* shells value of K_F was higher. It indicated that *Arachis hypogaea* shell powder showed more physical adsorption than chemical adsorption [31].

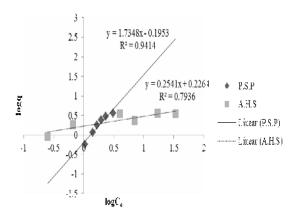


Fig. 12: Comparative graph showing adsorption of Amaranth from aqueous solutions using Pisum sativum peels (P.S.P) and Arachis hypogaea shells (A.H.S).

Table-4: Freundlich parameters for adsorption of Amaranth dye from aqueous solution.

Adsorbent	Slope	Intercept	R ²	K _F	n
				$(mg/g)(L/mg)^{1/n}$	
P. sativum peels	1.7348	-0.1953	0.9414	0.64	0.74
A. hypogaea shells	0.2541	0.2264	0.7936	1.684	3.94

8. Thermodynamic Study

For thermodynamic investigations, 0.5 g of *Pisum sativum* peels and *Arachis hypogaea* shell powder was mixed with 25 mL of 100 mg L⁻¹ of Amaranth dye solution separately and agitated in water-bath shaker with temperature adjusted to 298 to 348 K. The solution was then filtered and the filtrate was analyzed using UV-VIS Spectrophotometer. Thermodynamic parameters such as Gibbs free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°)

were calculated to determine the adsorption process using equations 6-8 and results are presented in Table-5.

Table-5: Thermodynamic parameters for adsorption of Amaranth dye from aqueous solution.

Adsorbent	∆H° (kJmol ⁻¹)	ΔS° (JK ⁻¹ mol ⁻¹)		∆G° (kJ	mol ⁻¹)	
			298K	318K	328K	348K
Pisum sativum peels	36.67	162.4	-11.01	-11.82	-3.14	- 13.69
Arachis hypogaea shells	51.53	213.54	10.94	12.57	13.77	14.26

$$K_{D} = \frac{(C_o - C_e)}{C_o} \times \frac{V}{m}$$
(6)

$$ln K_{\rm D} = -\Delta H^{\rm o} R T + \Delta S^{\rm o} R \tag{7}$$

$$\Delta G^{o} = \Delta H^{o} - T \Delta S^{o} \tag{8}$$

Here, Where ' K_D ' is biosorption equilibrium constant, 'V' is the volume of metal ion solution in Litre, 'm' is weight of biosorbent in grams. ' ΔG^{0} ' is the change in Gibbs free energy (kJmol⁻¹), ' ΔH^{0} ' is the change in enthalpy (kJmol⁻¹), ' ΔS^{0} ' is the change in entropy (J mol⁻¹ K⁻¹), 'T' is the absolute temperature (K) and R is the gas constant (8. 314 x 10^{-3} kJmol⁻¹ K⁻¹). Negative value of ΔG° shows that the reaction is spontaneous.

9. *Kinetic Study*

In order to analyze the controlling mechanism of the adsorption of Amaranth dye using *Pisum sativum* peels and *Arachis hypogaea* shells, pseudo-first-order equation and pseudo-second-order equation were used to fit the adsorption data. In general, the kinetics follows a pseudo-first-order model when adsorption is preceded by diffusion through a boundary. The second-order model is used to predict the whole adsorption process, and chemisorption is the rate-limiting step. The pseudo-first-order model can be written as,

$$ln(\mathbf{q}_{\mathrm{e}} - \mathbf{q}_{\mathrm{t}}) = ln\mathbf{q}_{\mathrm{e}} - \mathbf{k}_{1}\mathbf{t}$$
(9)

Where k_1 (1/min) is the rate constant of the pseudofirst-order adsorption, and q_t and q_e (mg/g) are the adsorbed amounts at time t and at equilibrium. k_1 and q_e can be obtained from the slope and the intercepts of the plots of $ln(q_e-q_t)$ versus t. The related parameters are listed in Table-6. It was observed that the R^2 values were low. This indicated that the adsorption of Amaranth dye using *Pisum sativum* peels and *Arachis hypogaea* shells did not fit a pseudo-first-order kinetic model.

The pseudo-second-order model can be expressed by the following equation 10:

$$t/qt = 1/k_2 q_e^2 + t/q_e$$
 (10)

where k_2 (g/mg min) is the rate constant of the pseudo-second-order model. The slope and the intercept of the plots of t/qe versus t were obtained to calculate k_2 and q_e as shown in Table-6, there was an excellent agreement between the experimental and the calculated q_e values. All R² values obtained from the pseudo-second-order model were closed to one. This indicated that the adsorption of Amaranth dye using *Pisum sativum* peels and *Arachis hypogaea* shells followed the pseudo-second-order model very well.

Table-6: Kinetic parameters for adsorption ofAmaranth dye from aqueous solution.

Kinetic models	Pisum sativum peels			Arachis hypogaea shells			
Temperature	30 °C	40 °C	30 °C	40 °C	50 °C	50 °C	
qe experimental (mg/g)	17.489	16.175	17.309	15.675	16.662	15.562	
Pseudo-first order							
k1 (1/min)	0.007	0.006	0.007	0.008	0.005	0.009	
q _e calculated (mg/g)	2.104	2.237	2.104	2.237	1.231	1.231	
\mathbf{R}^2	0.814	0.723	0.734	0.823	0.682	0.682	
Pseudo-second order							
k ₂ (mg/g min)	0.0120	0.0132	0.0120	0.0132	0.0184	0.0153	
q _e (mg/g)	17.531	15.710	17.575	16.750	18.657	15.699	
t _{1/2} (min)	4.71	4.63	4.82	4.12	4.25	4.35	
R ²	0.987	0.968	0.987	0.981	0.928	0.937	

Conclusion

In this study, two adsorbents Pisum sativum peels and Arachis hypogaea shells were compared for adsorptive removal of Amaranth dye from aqueous solution. Characterization of adsorbents showed Arachis hypogaea shells had less moisture and more ash content. Experimental studies showed higher percentage removal of Amaranth with Pisum sativum peels. Higher K_F value in case of Arachis hypogaea shells indicates greater physio-sorption instead of chemi-sorption. So, Arachis hypogaea shells showed maximum removal of Amaranth with a contact time of 10 minutes. Maximum adsorption capacity of Pisum sativum peels was 144.93 and 10.53 mg/g in case of Arachis hypogaea shells. Adsorption data was best described by Langmuir isotherm. Negative values of ΔG^0 showed that it was exothermic and spontaneous process. Kinetic studies showed that adsorption of Amaranth dye using Pisum sativum peels and *Arachis hypogaea* shells followed the pseudo-second-order model. Hence, from the results of this study *Pisum sativum* peels were found to be better adsorbent than *Arachis hypogaea* shells for removal of Amaranth dye from aqueous solutions. Moreover, *Pisum sativum* pods are very easily available because Pea crop is abundantly grown in Pakistan. So, *Pisum sativum* peels are cost effective and efficient adsorbent for removal of Amaranth dye from aqueous medium.

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