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Preparation and Characterization of Acid and Base Modified Fish Scales of *Labeo Rohita* and Their Application as an Adsorbent

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ABSTRACT

The study is carried out to investigate the removal efficiency of the textile dye Congo Red (CR) on fish scales (FS) of *Labeo rohita*. Huge fish scales are dumped as waste from the fish markets and are not converted into valuable products. That is why a fish scale of widespread fish in Bangladesh *Labeo rohita* has been selected as an adsorbent in removing Congo red from an aqueous solution. In the present study, the effect of different parameters such as adsorption dosage, contact time, concentration of dye, and pH on the removal of CR were investigated. The maximum dye removal efficiency (88.53 %) was observed for 3g/100 mL of adsorbent at an initial concentration of 1×10^{-4} M and pH 8.80 after 60 minutes. Modification of the surface of the fish scales was carried out by using HCl and KOH. The surface of the adsorbents was characterized by FTIR spectroscopy. After 60 minutes of contact time, the percentage of adsorption for the acid and base-modified FS was 54.81% and 90.51% respectively. The higher intense FTIR spectra of the base-modified FS denoted the better efficiency of the adsorbent at the basic medium. The adsorption kinetics of CR on FS is studied by following the pseudo-first-order and pseudo-second-order kinetic model. Experimental results showed that fish scales of *Labeo rohita* have satisfying color removal efficiency and might be a better alternative to other adsorbents to detoxify industrial wastewater. A mechanism of adsorption of CR on FS has been proposed.

Keywords: Adsorption, Congo Red, Fish Scale, Kinetics, Surface Modification.

1 Introduction

The change in water quality or composition, either directly or indirectly, because of both natural and human activities is considered water pollution. Nowadays, water pollution is a common problem due to the untreated wastewater released from textile and paint industries during the dyeing and finishing processes [1]. The major contaminants from these industrial effluents are mainly persistent colors of different dyes, organics, toxicants, chrome, starch, acids, surfactants, heavy metals, chlorinated and inhibitory compounds, etc. [2],[3]. Congo red with the molecular formula of $C_{32}H_{22}N_6Na_2O_6S_2$ and molecular weight of 696.68 g mol^{-1} is an azo dye that has widespread applications in many industries like textile, pigment, leather, food, pharmaceutical, pulp, and paper [4]. Toxic effects such as carcinogenic, mutagenic, allergic, and phytotoxicity have been observed with CR dye [5]-[8]. CR dye negatively affects both the human body and animals leading to different diseases involving the eyes, skin, respiratory, and reproductive systems. The wastewater containing azo dye reduces the penetration of light which adversely affects the photosynthesis process of aquatic plant life, and, thus, the ecosystem is negatively affected [9]. That's why untreated wastewater from various industries must be treated before its ultimate release to protect the aquatic environment.

There are several water treatment techniques ozonation, photo-oxidation, electrocoagulation, froth flotation, reverse osmosis, ion exchange, membrane filtration, flocculation, and adsorption to remove colored dye from industrial effluent [10]-[13]. Among all these processes, adsorption is more applicable due to the ease of its procedure and cost-effectiveness. Today, the most commonly used adsorbent is activated carbon but its use is sometimes restricted due to its higher cost [14]. Many researchers prefer to use cost-effective bio-sorbent i.e. walnut shells, sugarcane bagasse, Jackfruit Seed, mango leaves, paper industry waste, rice husk, sawdust, agricultural solid waste, aloe vera leaf, etc. [15]-[18]. Recently, Pal D. et al. and Nadeem et al. reported the use of fish scale (FS) as a biosorbent to remove heavy metals from industrial effluent [19],[20]. Marrakchi et al. discovered the maximum adsorption of methylene blue onto the higher surface area of fishery waste [21]. The FS contains calcium phosphates (hydroxyapatite) and collagen, which makes it possible to exchange the ions and adsorb different compounds to their surface [22]-[23]. Vieira M. G. A. et al. and Senturk H. B. et al. exploited surface modification of adsorbent by different modifying agents such as acid and base which influence the adsorption process [24],[25].

Therefore, this research will explore the adsorption of CR by unmodified fish scales, acid, and base-modified FS of *Labeo rohita* as an adsorbent at different conditions to evaluate the maximum adsorption capacity. Furthermore, the surface of the adsorbents was characterized by FTIR, and the adsorption kinetics of CR on FS was studied by pseudo-first-order and pseudo-second-order kinetic models.

2 Materials and Methods

The raw fish '*Labeo rohita*' was collected from the local fish market, and scales were separated and washed with distilled water to remove the unwanted particles from the surface of the scales as well as dust and dirt. The scales were dried in an oven at 60°C for 24 hours and then powdered by using an electric

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grinder to reduce their size [26]. After that, 10g of processed FS was soaked in 100 mL of 0.01 M HCl solution overnight and then the solution was filtered. The wet FS was dried at 60°C in an electric oven. The acid-modified scales were taken in a mortar to prepare fine particles of FS. By following the same procedure 0.01 M KOH was used to prepare base modified FS.

A stock solution of CR having 1×10^{-2} M concentration was prepared in a 500 mL volumetric flask. The stock solution was diluted by adding appropriate amount of deionized water to prepare different concentrations of CR for further investigations. The adsorption experiment was carried out with a fixed amount of FS of *Labeo rohita* in an orbital shaker (Model No-JSOS-300). The adsorption efficiency was determined by using a UV-visible spectrophotometer (Lamda-365) at 200-800 nm wavelength. The percentage of dye removal efficiency was determined by using Eq. (1). The experiments were carried out by changing different parameters like dosage of FS, initial concentration of CR, pH, contact time, and temperature.

$$\%\ of\ adsorption = \left(\frac{A_{\circ} - A_{t}}{A_{\circ}}\right) \times 100\% \tag{1}$$

where, A_o and A_t are the absorbance of CR solution at 0 minutes and any time, *t* respectively.

The FTIR spectra of untreated fish scale, acid, and base-treated FS were investigated by IR Tracer-100 (Shimadzu) to characterize the functional groups present on the surface of the fish scale.

3 Results and Discussion

The absorption maximum of Congo red was determined spectroscopically from its absorption spectrum. The molar extinction coefficient (ε) at λ_{max} = 498 nm was obtained to be 2.352×10^{4} L mol^{-1} cm^{-1}. This value was used to study the kinetics of CR on FS.

3.1 Effect of dosage of FS

The adsorption capacity is found to be augmented and reached maxima at 60 minutes with the increase of adsorbent dose till 3g of FS. Then the percentage of adsorption was decreased with the increase of adsorbent dose (Fig. 1).

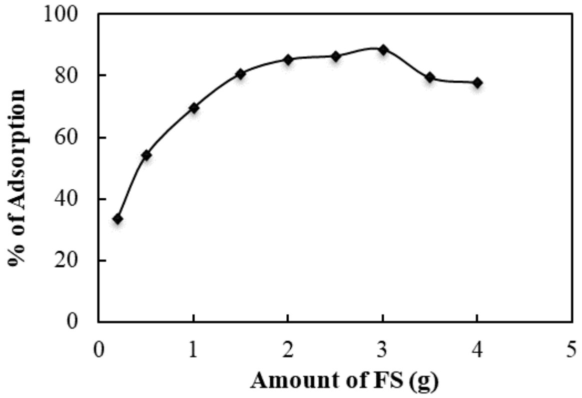

Fig. 1 Percentage of adsorption at various amounts of FS. [$[CR]_o = 1\times10^{-4}$ M, t = 60 minutes, pH = 8.80, T = 25ºC]

At lower adsorbent concentrations, several active sites is abundant. With the increase in adsorbent dosage, aggregation of particles takes place which diminishes the entire surface area of FS and increases diffusion path length [27]. As a result, adsorption efficiency and dye uptake decrease.

3.2 Effect of concentration of CR

The dye removal efficiency decreases from 88.53% to 1.13% with the increase in dye concentration (Fig. 2). The initial concentration of CR imparts an important driving force to encounter all mass transfer resistance of the dye between the dye molecules and solid-phase adsorbent [28]. However, the rise in dye concentration leads to a reduction in the adsorption process, indicating the adsorption of CR is highly dependent on dye concentration. The adsorption of dye molecules involves the unoccupied active sites on the bio-sorbent surface. After a certain concentration, the surface of the bio-sorbent should be saturated [29]. Thus, a further increase of dye molecules decreases the percentage of adsorption because there is competition between the dye molecules to adsorb on the surface.

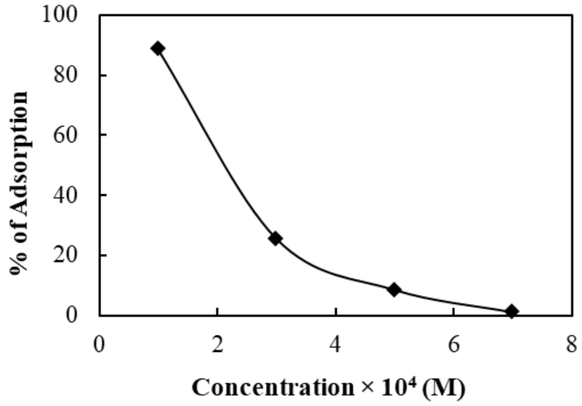

Fig. 2 Percentage of adsorption at different concentrations of CR. [Amount of FS = 3g, t = 60 minutes, pH = 8.80, T = 25ºC]

3.3 Effect of contact time

The effect of contact time on dye removal using fish scales was investigated at different contact times (0 to 60 minutes) for 100 mL of dye solution (Fig. 3).

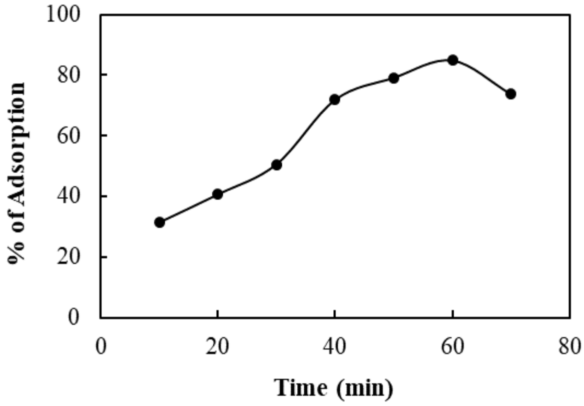

Fig. 3 Percentage of adsorption vs. contact time (min). [Amount of FS = 3g, $[CR]_o = 1\times10^{-4}$ M, pH = 8.80, and T = 25ºC]

The percentage of dye removal was found to increase with contact time. After 60 minutes, about 88.53 % of dye molecules were adsorbed on the surface of FS for 3g of adsorbent. This might be due to the interaction between the dye molecule and many free surface sites of bio-sorbent [30]. After 60 minutes of contact time, the percentage of adsorption reaches at maximum and then decreases. So, the equilibrium is established at 60 minutes, after that desorption of CR molecules takes place from the surface of FS.

3.4 Effect of pH on adsorption of dye

Maximum removal efficiency of Congo red with FS is obtained at pH 8.80. As the pH was increased from 8.80 to 11.44, efficiency decreased as shown in Fig. 4. As pH rises the surface becomes more negatively charged. This causes increased repulsion between the dye and the adsorbent surface. Hence removal efficiency decreases with the increase of pH. Similarly, removal efficiency decreases with a decrease in pH from 8.80 to 2.46. The increase in percentage removal of the adsorbate up to pH=8.80 may be explained by the fact that at higher pH the adsorbent surface is deprotonated and negatively charged. Therefore, there is an attraction between the positively charged ions of the adsorbate and the negatively charged ions of the adsorbent [31].

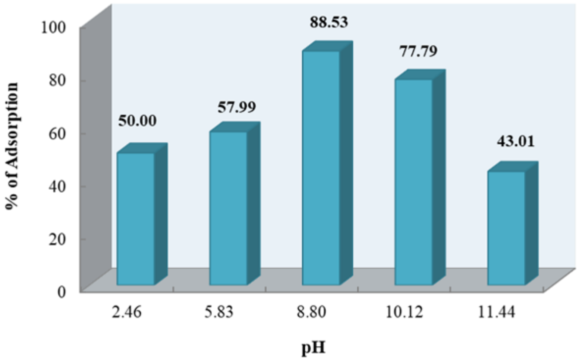

Fig. 4 Percentage of adsorption at different pH. [Amount of FS = 3g, $[CR]_o = 1\times10^{-4}$ M, t = 60 minutes, and T = 25°C]

3.5 Effect of temperature

The adsorption of CR on the surface of FS as a function of temperature is explored in Fig. 5. The dye adsorption decreased with the rise in temperature revealing the exothermic nature of CR adsorption onto FS. The maximum adsorption capacity was observed at low temperatures (25°C). The percentage of dye adsorption is reduced with the increase in temperature.

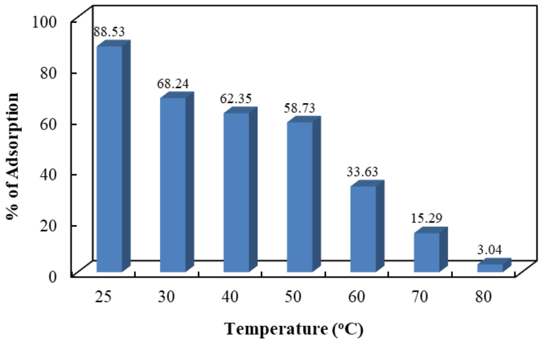

Fig. 5 Percentage of adsorption at different temperatures. [Amount of FS = 3g, $[CR]_o = 1\times10^{-4}$ M, t = 60 minutes, and pH = 8.80]

At higher temperatures, the supportive forces between the active sites of the FS and CR, and between the adjacent CR molecules become weak which reduces the adsorption rate [32]. A reduction in the surface activity of FS with the increase in temperature indicates both the exothermic nature and physical adsorption of CR molecules onto the active sites of FS [33]. Therefore, the desorption of CR molecules from the surface of FS at elevated temperatures was due to the weakening of adsorptive forces between the CR molecules and the functional groups present on the surface of FS.

3.6 Kinetic study

Two different kinetic models including the pseudo-first-order equation and pseudo-second-order equation were tested to find out the mechanism of adsorption. The kinetic equations for the pseudo-first and pseudo-second-order models are as follows [34]-[35].

$$log\,(q_e - q_t) = log\,q_e - k_1 t \tag{2}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{3}$$

Where, q_e and q_t are the amount of adsorbate adsorbed on adsorbent (mg/g) at equilibrium and any time t respectively. k_1 and k_2 are the rate constants for the pseudo-first and second-order respectively.

The value of q_t and q_e are evaluated by using Eq. (4) and Eq. (5) respectively [36]. C_o and C_e represent the initial and equilibrium concentration of CR in mg/L respectively. V is the volume of the solution in liter used for adsorption and W is the mass of adsorbent in g.

$$q_t = \frac{(C_o - C_t)\,V}{W} \tag{4}$$

$$q_e = \frac{(C_o - C_e)\,V}{W} \tag{5}$$

The values of pseudo-first and pseudo-second-order kinetic parameters are determined from the plot of $log(q_e - q_t)$ vs. $log\,q_e$ and t/q_t vs. t (Fig. 6). It is found that the value of R^2 is closer to 1 for the pseudo-first-order model than that of the pseudo-second-order model which suggests that the kinetics of adsorption of CR on FS surface follows pseudo-first-order kinetic model.

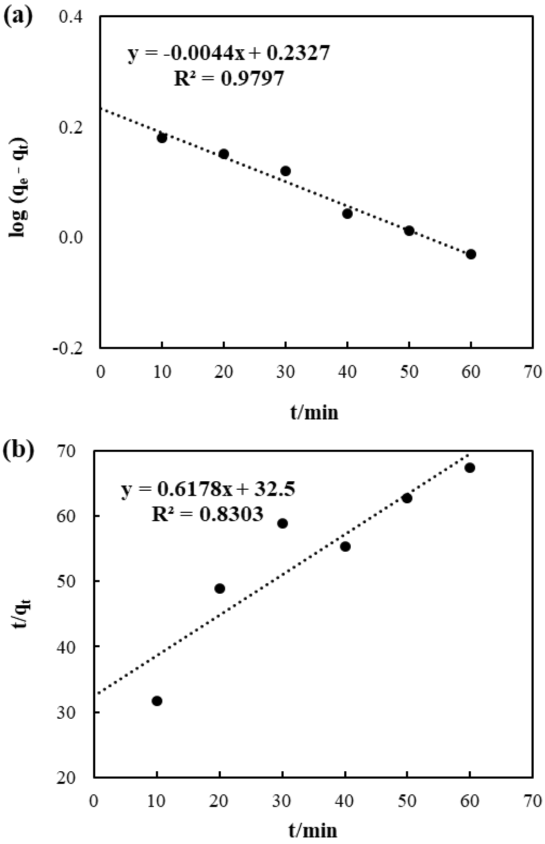

Fig. 6 (a) Pseudo-first order and (b) Pseudo-second order kinetic model for the adsorption of CR on FS

Table 1 Kinetic parameters for the pseudo-first and pseudo-second-order models

	Pseudo-first-order model		Pseudo-second-order model	
$C_o \times 10^4$ (M)	k_1	R^2	k_2	R^2
1	0.0044	0.9797	0.0387	0.8303

3.7 Effect of acid and base-modified FS on adsorption

In surface chemistry, a chemical technique involves the addition of acid, base, or salt to modify the functional groups of the surface [37]. The surface modification of the fish scale was performed by using HCl and KOH solution. The base-modified FS showed a higher adsorption capacity of 90.51% than the acid-modified FS of 54.81% (Fig. 7). The adsorption of Congo red dye by tunics of the corm of the saffron after 60 min was 68%, which is about 20% and 22% less than the maximum adsorption by unmodified and base modified fish scales of *Labeo rohita* respectively [38].

A base-modified adsorbent adsorbs a positive charge on the surface that enhances the adsorption of negatively charged species [39]. For the base-modified surface of FS, there might also be a significant decrease in the surface oxygen-containing functional groups, hence, the specific surface area and the pore volume concentration are raised which results in a higher adsorption capacity. For the acid-modified FS, the total pore volume may have very low values [40]. Thus, the surface of the adsorbent may be considered as a non-porous material, hence, adsorption capacity decreases.

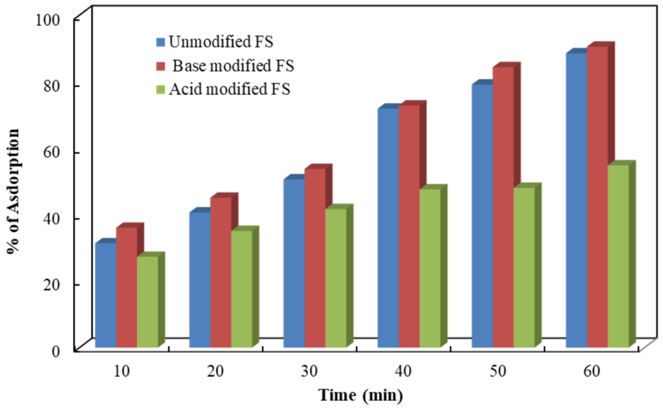

Fig. 7 The adsorption efficiency of unmodified FS, acid, and base-modified FS at different time intervals.

3.8 FTIR analysis

The FT-IR spectra of untreated FS, acid, and base-treated FS were recorded in the wavelength range 4000-400 cm^{-1} (Fig. 8). The data were collected at 2.0 cm^{-1} resolution. Intensive absorption bands were observed at 1021 to 601 cm^{-1} which correspond to the vibrational mode of the phosphate (PO_4^{3-}) groups in the hydroxyapatite network. In addition, the absorption band was also observed at 1657 cm^{-1} which corresponds to the C=O bond stretching of the carbonate groups of the apatite structure [41]. Three bands were observed at 1637, 1567, and 1241 cm^{-1} corresponding to the amides I, II, and III of the collagen's structure. A broadband detected around the 3600-3200 cm^{-1} region indicates the presence of O-H stretching frequency. Again, a band for N-H stretching vibrational frequency of primary amide was observed around the same region i.e., 3540-3050 cm^{-1}. That's why these two intense broad bands overlap in the same region. Therefore, both the O-H and N-H groups are present on the surface of the FS of *Labeo rohita*. Since collagen contains both the O-H and N-H groups, it is evident that collagen is present on the surface of FS. A band is found at near 2877 cm^{-1} due to C-H stretching. The bending mode of N-H gives a band at 1501 cm^{-1}. The C-O stretching is determined at 1013 cm^{-1} [42]. The acid and base used for surface modification were able to interact with different functional groups of the adsorbent and so the intensities of the IR band were changed. In Fig. 8 (b), the comparison of the FTIR spectra of FS before and after adsorption is shown. The intensity of the adsorbed surface of FS is increased due to the interaction between the functional groups of the FS and CR. The overlapping spectra of O-H and N-H groups on the surface of the FS of *Labeo rohita* in the range of 3000-3600 cm^{-1} is not observed after adsorption.

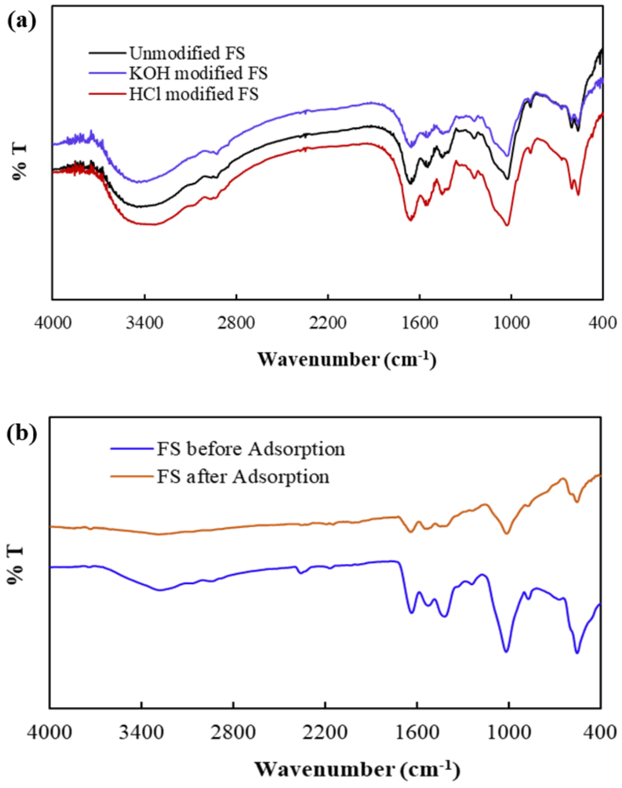

Fig. 8 FTIR spectra of (a) unmodified, acid & base modified fish scale (b) comparison of FS before and after adsorption.

3.9 Mechanism of Adsorption of CR on FS of *Labeo rohita*

The surface characteristics of adsorbents play a significant role in adsorption processes [43]. The FS contains a variety of functional groups on its surface having acidic and/or alkaline characteristics. These groups influence the dye′s adsorption processes through the formation of (a) strong hydrogen bonds, (b) electrostatic interactions, (c) n-π interaction, (d) π-π interaction, etc. Electrostatic interaction occurs between the cationic center of the adsorbent and the negatively charged groups of the dye molecules or vice-versa. The π-π interaction occurs between the π-electron clouds of the adsorbent and aromatic rings of the dye molecule [44]. The FS surface is rich with acidic oxygen functional groups which are responsible for the formation of strong hydrogen bonding between the adsorbent and the dye molecule. The n-π interaction between the adsorbent and the dye molecule can also facilitate the adsorption processes.

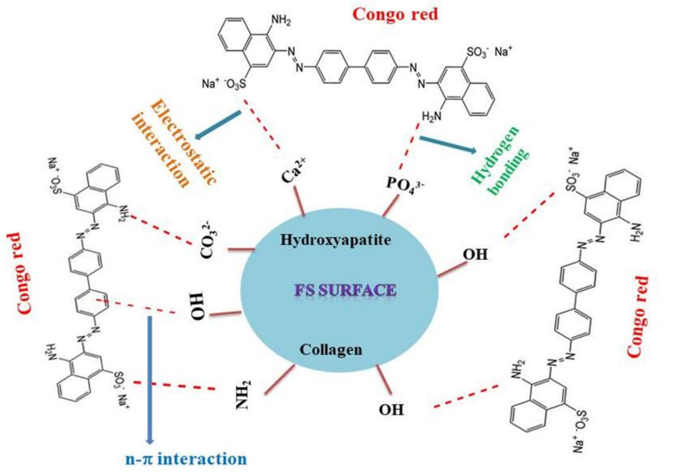

Fig. 9 Proposed mechanism for the adsorption of CR on FS surface.

4 Conclusions

The development of appropriate material to resist the fast-rising aqueous toxicity is a growing demand in the current age of industrialization. In the present study, the waste fish scales were used as an adsorbent to remove color from textile wastewater. It is a low-cost adsorbent and environmentally friendly compared to other adsorbents. For 1×10^{-4} M initial concentration of CR, the maximum percentage of adsorption, 88.53% was achieved within 60 minutes at optimum adsorbent dosage of 3g of fish scales. The adsorption capacity is inversely proportional to the initial concentration of the dye molecule. The optimum pH for favorable adsorption of CR was 8.80. Characterization of FS by IR spectra provided important information about the presence of collagen, hydroxyapatite, and many other functional groups that are responsible for the adsorption on its surface. The correlation coefficient (R^2) value suggests that the adsorption of CR on FS follows the pseudo-first-order kinetics. All of the results support that the fish scales of *Labeo rohita* can be used as an adsorbent for wastewater treatment.

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