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Removal of Acid Orange 7 dye from aqueous solutions by adsorption onto Kenya tea pulps; granulated shape

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Abstract
Background and Aim: Water resources pollution control is one of the main challenges of our time for researchers. Colored wastewater discharges caused by textile industry activities has added to the concern. In this study, removal of Acid Orange 7 dye (AO7) using Kenya Tea residue absorbent (granular) has been studied.
Methods: This cross-sectional study was conducted in 2016. In this work, initially, tea residue was prepared in three forms of raw, treated with concentrated phosphoric acid, and carbonated, at temperatures of 350, 450 and 500 °C in the chemistry laboratory of Gonabad University of Medical Sciences. Then, efficiency of the above absorbents in the removal of Acid Orange 7 dye in initial concentrations of dye as 50-500 mg/l from water samples in terms of pH 2-10 and 1-10 g/l of adsorbent dose within 20 to 300 minutes was investigated. In addition, their subordination from Langmuir and Freundlich absorption isotherms was also determined. Concentration changes in Acid Orange 7 dye at a wavelength of 483 nm was determined by spectrophotometry and results were reported using descriptive statistics.
Results: Results showed that efficiency of Acid Orange 7 dye removal is higher in acidic pH and higher adsorbent dosage. The highest efficiency of Acid Orange 7 dye removal was 98.41% by raw tea residue absorbent at pH 2, reaction time was 120 minutes and initial concentration of dye was 50 mg/l, which was obtained at adsorbent dosage of 10 g/l. It was determined that the mechanism of absorption acceptably follows Freundlich absorption isotherm (R^2=0.97).
Conclusion: Due to the availability and very low price, optimal performance of Kenya tea raw residue (granular) in Acid Orange 7 dye removal, it can be used as an efficient surface absorber in an absorber from colored wastewater.
Keywords: Acid Orange 7, Dye Removal, Aqueous Solutions, Adsorption, Kenya Tea Pulps, Granulated Shape

1. Introduction

Increased production of color products and their high applications in today's world has led to production of colored wastewater and a serious concern for environmental researchers (1, 2). Wastewaters are produced or consumed in various industries, including textile and dyeing industries, pharmaceuticals, food industries, cosmetics production, paper making, tannery and similar industries (3). There is more than one million tons of annual production of dye worldwide used in different industries, especially in the textile industry as the second largest water consuming

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industry in the world (4). Colored wastewater discharged to water resources causes undesirable appearance, loss of ability to use it in urban affairs, agriculture, industry and creates an aesthetically undesirable environment (5, 6). Generally, the dyes, especially azo dyes have a complex aromatic, toxic, mutagenic and carcinogenic structure and have high stability against sunlight (7-9). Discharging wastewater containing azo dyes not only affects the aesthetic aspect of receptor waters, but also causes the blocking of sunlight passing through the body of water and reduces photosynthesis process and dissolved oxygen concentration. About 12% of the dyes used in the textile industry enter wastewater, and about 20% of them are discharged to the environment without treatment (10, 11). Acid Orange 7 is among the acidic azo dyes which causes damage to the eyes and skin, and has chronic toxicity and carcinogenicity (12). Today, under intense pressure from public opinion and regulations of conventions, industries are required to treat their wastewater favorably before discharge into the natural environment (13-15). Thus, finding effective treatment methods is necessary and inevitable. Until now, various processes including electrochemical coagulation processes, oxidation, chemical precipitation, anaerobic treatment, flotation, and ion exchange for removal of dyes from aqueous solutions have been studied (16-19). Surface adsorption process is one of the environmentally-friendly processes with simple and economic mechanisms which, in recent decades, has become very famous in wastewater treatment (20, 21). In this method, using activated carbon or raw and inactivated biological materials such as bentonite, ash, and agricultural waste is very common for effective removal of soluble contaminant particles especially organic contaminants such as paint, from the aquatic environment (22). Some of the advantages of the surface absorption method are its low initial cost, simple design and operation, need for much lower energy source, lack of influence of toxic materials in great removal of contaminants with organic nature compared to other conventional treatment methods and non-selective functioning (21, 23). But restrictions in surface adsorption process such as increased suspended solids in water caused by powder form applications and their difficult separation, especially carbon forms from the water environment, have caused engineers to fix these separation problems and make changes in the form of absorbent application (24). In addition, in order to reduce preparation costs, the use of inexpensive materials such as remains of crops for production biodegradable biological absorbents has been highly regarded as a potential option (25). Meanwhile, using food residues has been considered in several studies due to reduction of biomass management cost reduction, prevention from unsanitary accumulation of biological mass, production of capital from food and agricultural waste. In addition, the carbon obtained from food and agricultural waste has low ash content, acceptable hardness and high surface area and adequate porous structure, thus their use for production of bio-adsorbents has been considered (26). One of the food wastes, which has high production volumes, particularly in Asian countries, is tea residue or waste produced by tea-producing industries, especially in cold tea producing industries. Need for proper discharge of tea residue for prevention of allopathic adverse effects on soil, low cost, its natural quality and its availability has caused the attention of researchers to produce absorbent from it (27). Using tea residue is not permissible in other affairs such as production of compost due to its toxicity and biological resistance (28). Up to now, efficiency of tea residue as active powder carbon on cations and onion dye removal from water environments, has been studied in some works (29, 30). Since cellulosic residues are suitable options for producing surface absorbents, given the basic problem of powder absorbent separation from water environments, efficiency of Kenya tea (CTC tea) residue as granule in surface absorption of Acid Orange 7 dye from water solutions is studied for the first time in this study.

2. Material and Methods

2.1. Materials

Most of the chemicals used in this study, including Acid Orange 7 dye (Orange II) C16H11N2NaO4S, sodium hydroxide and hydrochloric acid were obtained from the reputable Merck Company from Germany. HPLC distilled water was used in all tests. Adjusting the pH was done by Denver Ultra basic-UB10 made in America using 1N HCl and NaOH. At the present time, official or orthodox and non-official or CTC (Crush, Tear, Curl) methods are used in many of the world's tea industries to make tea, especially black tea. In CTC method, the new machines called CTC machines include a pair of opposing rotating rollers. CTC tea resembles an ant's head in appearance thus it also known as ant head tea in Persian language. In this technique, tea seeds are very convoluted. Therefore, in addition to high porosity and specific surface area, it retains its structure and cannot be torn apart in an aqueous environment, even in boiling water. With regard to high production and consumption of tea (there is daily production of 2100 tons of cold tea and tea waste just in iced tea production factories in Fars province in Iran), the use of CTC tea residue bio-absorbent for AO7 dye absorption is investigated in this work. To this end, at first, Kenya tea residue was prepared from the Sophia Iced Tea Factory located in Chabahar city (Sistan and Baluchistan, Iran). Then tea residue seeds were screened with a 30-mesh size of 0.5 mm. To prepare fine-grained tea (ant's head tea (Kenya Tea)) residue absorbent as raw, acid treatment with concentrated phosphoric acid in a ratio of 3 to 1 and carbonation at temperatures of 0, 300, 500, 800 °C (the needed temperature was already obtained) was used. In order to remove possible contamination, initially they were boiled in water so that no color was created, and after acid cleaning and neutralization, they were dried for 24 hours at 120 °C. In the next stage, the dried tea residues were heated for 60 minutes in an electric furnace of the Alfa horizontal type model as necessary and were carbonated and were kept in a desiccator until the needed time (31).

2.2. Procedure

The dye used in this study was Acid Orange 7 (Orange II) of acidic type and now is extensively used in textile and paper industries. Experiments were followed by preparation of different concentrations (50, 100, 150, 200, 500 mg/l) of AO7 dye in the range of pH 2 to 10 and adsorbent dosage of 1 to 10 g/l based on a shaker with revolution of 100 rpm for 120 minutes. At each stage of sampling, they were filtered by 42 micron Whatman filter paper, and dye concentration changes were determined using UV-vis spectrophotometer at a wavelength of 483 nm. Microsoft Excel 2013 was used for data analysis and the results were reported based on descriptive statistics. Also, in order to determine maximum absorption and the optimum conditions for Langmuir and Freundlich isotherm, adsorption on the adsorbent were calculated and presented (13). To determine the structural properties of fine-grained tea residue, Electron Microscope Scanning by Philips XL-30 SEM Company was used and the Bruner -Taller- Emmet (BET) isotherm and fifth edition of Belsorp Japan software was used for determining the specific surface area.

3. Results

3.1. Properties of Activated Carbon

Investigation of Electron Microscope Scanning images prepared from tea residue indicates that this absorbent has amorphous structure and appropriate porous levels for appropriate and adequate absorption. The level of available surface based on the calculations by Belsorp software was determined as 832 m2/g. Since the specific surface area of the adsorbent is low, functional groups may have a more prominent role in the dye ion absorption from the liquid surface.

3.2. Effects of Carbon Modifications

The highest absorption rate in the raw adsorbent, acid treatment and carbon 350, 450 and 500 °C at different temperatures on the dye absorption by absorbent was related to the raw, acidic, carbonated type with temperature of 450 °C, carbonated type with a temperature of 350 °C and carbonated with temperature of 500 °C equivalent with 80.27, 68.50, 45.60, 34.47 and 29.47% respectively. Thus, results showed that acidic and thermal treatments had impact on absorption level of dye by tea residue, and highest absorption efficiency was obtained in raw absorbent as 98.41%.

3.3. Effect of pH

Figure 1 indicates impact of pH changes on Orange dye absorption rate in pH range between 2-10. Figure 1 indicates that dye absorption is highly reduced by increasing pH from 2 to 3, and then the reduction trend is mild. After reaching to neutral pH, absorption level has low reductive trend with increasing pH. Thus, the highest efficiency of removal was obtained at pH 2 as 98.41%.

3.4. Effect of Absorbent Dosage

Figure 2 indicates impact of absorbent dosage on absorption rate of Acid Orange 7 dye. Figure 2 indicates that dye removal efficiency is increased by increasing absorbent dosage from 1-10 g/l, and concentration of residual dye was reduced to 0.795 mg/l. Highest efficiency of removal was 98.41% in optimal dosage of 10 g/l.

3.5. Effect of Initial Dye Concentration

Figure 3 indicates impact of dye initial concentration change on absorption rate. Figure 3 indicates that dye removal efficiency is reduced to 79.65% from about 99% with increasing initial dye concentration from 50-500 mg/L.

3.6. Effect of Contact Time

Figure 4 indicates impact of contact time change on dye adsorption rate. Figure 4 indicates that dye removal efficiency is increased by increasing time. Acid Orange 7 dye absorption efficiency was obtained as 98.31% at contact time of 120 minutes, while changes in dye absorption efficiency was low with increasing contact time as 120–300 minutes, and it is almost fixed. Dye removal efficiency was obtained as 99.14 after 300 minutes. Thus, optimal contact time was specified as 120 minutes.

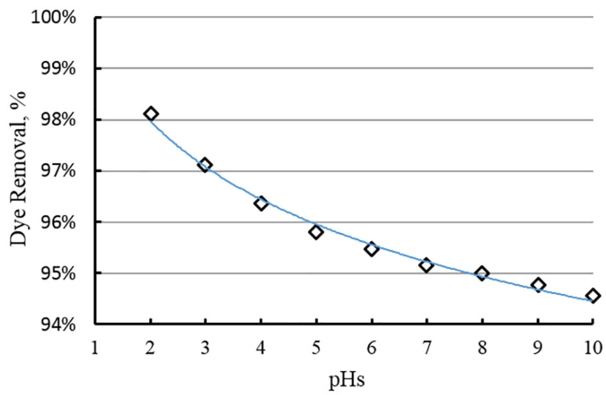

Figure 1. Effect of initial pH on Orange dye adsorption with tea pulp (Adsorbent dosage 10 g/l, dye con 50 mg/l, Time 120 min)

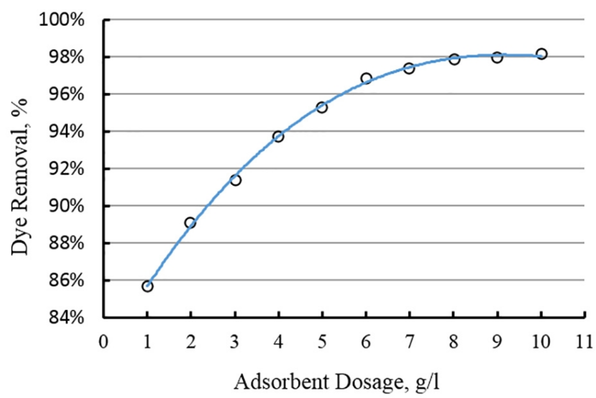

Figure 2. Effect of adsorbent dosage on AO7 dye with tea pulp (Adsorbent dosage 10 g/l, dye con 50 mg/l, Time 120 min, pH 2)

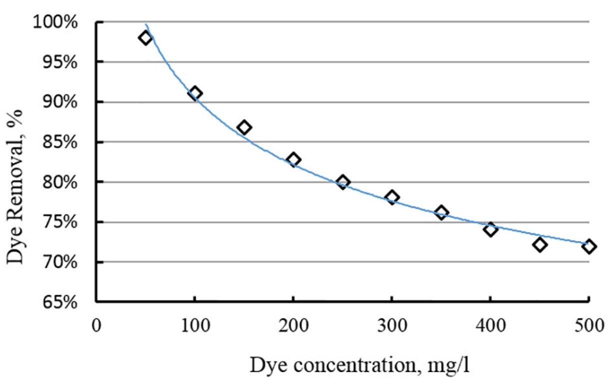

Figure 3. Effect of dye concentration on AO7 dye removal with tea pulp (Adsorbent dosage 10 g/l, Time 120 min, pH 2)

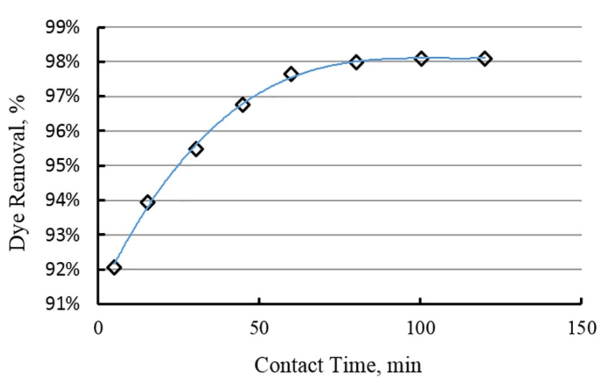

Figure 4. Effect of dye contact time on AO7 dye removal with tea pulp (Adsorbent dosage 10 g/l, dye con 50 mg/l, pH 2)

3.7. Effect of Temperature

Figure 5 indicates impact of variations in liquid ambient temperature on the absorption rate of dye. Figure 5 shows the dye removal efficiency increased with increasing temperature. So that the temperature increase from 30 to 50 C and dye absorption efficiency also increases from 98.41 to 99.69%.

3.8. Adsorption Isotherm

Figures 6 and 7 indicate amount of absorbed material on the surface in field temperature. Based on correlation coefficients (R^2) of isotherms of Langmuir and Freundlich adsorption models, as 0.85 and 0.97, respectively, it can be concluded that the adsorption rate of Acid Orange 7 dye on fine-grained tea residue follows Freundlich absorption isotherm considering higher correlation coefficient and n and k constant coefficients according to the equation curve is 2.46 and 4.31, respectively.

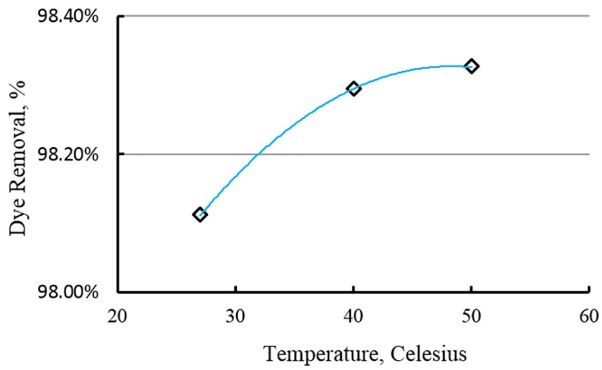

Figure 5. Effect of temperature on AO7 dye removal with tea pulp (Adsorbent dosage 10 g/l, dye con 50 mg/l, contact time 120 min, pH 2)

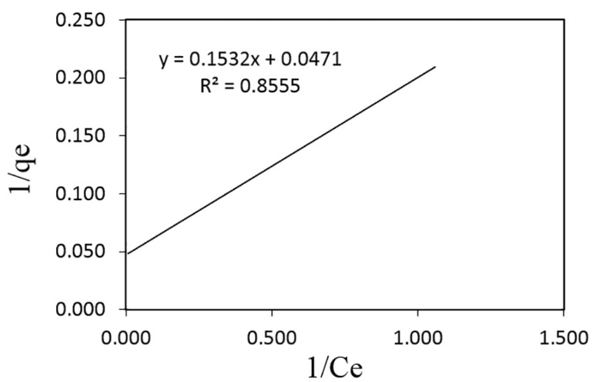

Figure 6. Isotherm of Langmuir of AO7 dye removal with tea pulp (Adsorbent dosage 10 g/l, contact time 120 min, pH 2)

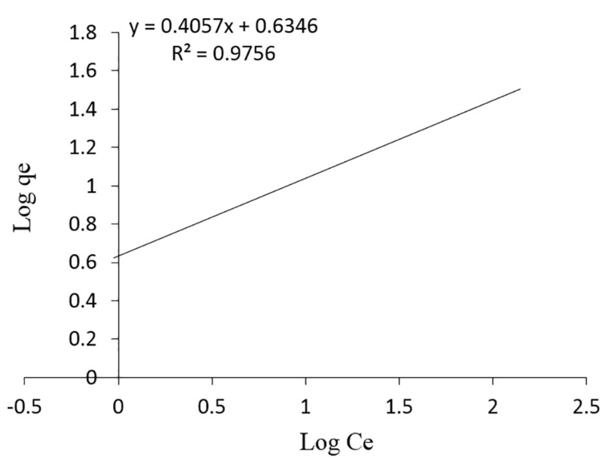

Figure 7. Isotherm of Freundlich of AO7 dye removal with tea pulp (Adsorbent dosage 10 g/l, contact time 120 min, pH 2)

4. Discussion

4.1. Effect of Modifications

Impact of change in raw absorbent, acidic treatment and carbonation in different temperatures on dye absorption level by fine-grained tea absorbent is shown in Figure 3. Carbonating bio-absorbents not only increases absorbent surface, but also can change acidic properties of the absorbent (32). Review of different studies has shown that the carbon prepared from tea residue in presence of activation agent causes increased dye absorption. In a study on adsorption of cationic and anionic dyes using activated carbon of tea residue reported that phosphoric acid as the activation agent in preparing activated carbon from tea residue have had good efficiency in removing contaminants (29). In another study, potassium acetate was used to produce activated carbon from tea for removal of methylene blue (27). But in this study, temperature without the activating factor (carbonation) was used. The results showed that this action does not affect the increasing dye removal efficiency. In the study on removal of cationic dye removal with the absorbent produced from rice bran carbon, it was reported that carbonating rice bran in temperatures of 600 and 800 causes reduction of absorption capacity compared to the raw samples, and absorption capacity is increased at temperature of 1000. They consider the reason for this as the physical properties of the absorbent (specific surface area and average diameter of the pores), and the absorbent surface area has a key role in dye removal (33). Also, acidic modification of absorbents causes improvement in their physical chemical and morphological properties leading to increased dye absorption (34). In the current study, acidic treatment had no significant impact on removal amount in the initial conditions of the experiment. Considering the fact that the highest removal efficiency was obtained with raw absorbent, and un-modified tea residue is more economical than carbonated and acidic samples, raw tea residue was used in this work for the next steps.

4.2. Effect of pH

The pH of colored waters is effective in surface absorption and capacity absorption of dye. It results from change in the amount of ionization of factor groups available in absorption positions and thus, changing absorbent surface load and amount of ionization of the materials in the solution (35). Figure 4 indicates the impact of pH of coloring solutions in activated carbon absorption capacity prepared from fine-grained tea residue. Figure 4 shows that Acid Orange 7 dye is separated from aqueous environments in higher efficiency acidic pHs, so that absorption level was decreased to 93.6% from 98.41% by increasing pH from 2 to 10. In the study on Acid Orange 7 dye removal from aqueous environments using bio-absorption technology, it has been reported that the highest dye removal was obtained in acidic pH. The reason has been assigned to the presence of $H3O+$ ion, and in some cases, due to bonding $H+$ ion with giving proton with the dye cations. Such bonding causes the removal of dye cations in greater amount and more efficiently (36). In the study on thermodynamic and equilibrium equations for removal of black 5 using acid modified banana peel, it is reported that removal efficiency decreased with increasing pH so the pH 2 and 7 respectively give 69.18 and 30.43% efficiency. It happens because of the close relationship between the binding sites of the adsorbent and hydrogen ions in the acid conditions which act as the ligand bond between the adsorbent surface and color molecules (37). In addition, sulfonate functional group in Acid Orange dye is ionized in water which causes that water molecules to become ionized. On the other hand, the pH rise increases negative charge in the absorbent and repulsive forces prevent acid orange absorption by the absorbent. The highest efficiency is obtained in acidic pH in this study, and it is consistent with the results of the above studies. Thus, in this study, the optimal pH 2 is introduced as the optimal pH.

4.3. Effect of Absorbent Dosage

Absorbent dosage is one of the main parameters in absorption process, which is due to determining role of absorbent capacity for absorbing contaminants at its surface (38). Figure 5 indicates the impact of absorbent concentration in the removal of dye in a fixed concentration of 50 mg/l. It shows that efficiency of dye removal is increased by increasing absorbent dosage, so that removal efficiency was increased to 98.41 and 96.97% respectively by increasing concentration from 1 to 10 g/l. In the study on Direct Black 22 dye removal from aqueous solution by activated carbon made from orange peel, it is reported that by increasing the adsorbent dosage from 0.1 to 1 g/l, respectively, removal efficiency increased as 87.1 and 95.2% (39). In the study on removal of acid orange 2 using egg shells from aqueous solutions, it is reported that with increasing adsorbent dosage, dye removal efficiency increased so that by increasing the adsorbent dose from 10 to 50 g/l, dye removal efficiency has been achieved as 35-74%. It happens because of increase in the available or active surface area (40). The highest efficiency was obtained in high concentrations in the current work which corresponded with the results of the above studies. Thus, the initial concentration as 10 g/l is introduced as the optimum concentration.

4.4. Effect of Dye Concentration

In the adsorption process, the initial concentration of ions of the adsorbed material in the solution has a key role as the driving force to overcome the resistance of mass transfer between the fluid phase and solid phase (38). Figure 6 shows the initial concentration of dye for absorption by tea residue in the range of 50-500 mg/L. Figure 6 shows that by lowering dye concentration, absorption efficiency is increased. In a study on removal of Reactive Orange 3 from aqueous solutions using bio-sorption technology, it has been reported that dye removal efficiency was reduced by increasing dye concentration in the fixed condition. The reason is justified as follows: the lower the concentration of contaminant in contact with a specific amount of absorbent is, there is higher probability for absorption on the absorbent (36). In the study on pomegranate seed powder used in removal of Reactive Red 198 dye from aqueous solutions it is reported that with increasing initial concentration of dye from 25 to 50 mg/L, dye removal efficiency decreased. It is due to reduction of the absorbent resistance against the absorption of the pollutants considered (41). In addition, adsorbed molecules in the solution compete with each other at higher concentrations to achieve the absorption positions, and absorption level reduces at higher doses. The highest efficiency was achieved at low concentrations in the current work that corresponded with the above studies. Thus, the initial concentration of 50 mg/l with 41.98% efficiency is introduced as the optimal concentration.

4.5. Impact of Contact Time on Absorption Rate

Contact time is one of the main factors affecting absorption processes (42). Figure 7 indicates impact of optimal contact time for Acid Orange absorption in pH 2 and concentration of 50 mg/L. Figure 7 shows that dye removal efficiency was increased by increasing contact time. Ghaneian et al. (2012), in their study on application of pomegranate seed powder for removal of Reactive Red 198 dye from aqueous solution it was reported that with increasing contact time, removal efficiency increased. However, the maximum adsorption was reached in 30 minutes and adsorption equilibrium was reached in about 2 hours. It is due to quick absorption of dye molecules on the external surface of the absorbent at the beginning of the absorption process. Speed of the pollutant emissions inside the pores and thus the rate of absorption is reduced gradually due to relative electrostatic repulsion forces of surface negative charges absorbed on the absorbent surface and negative charges in the fluid mass (41). In this study on removal of acid orange 2 dye using egg shells from aqueous solutions, it has been reported that with increasing the duration time from 15 to 90 minutes, dye removal efficiency increased from 61 to 74%. It happens because of the emptiness of many absorbent sites at the time of initial contact and change in the pollutant concentration in the liquid phase (40). In addition, contact between pollutants and absorbent is more likely by increasing the contact time and absorption of the pollutant by absorbent increases. In this study, efficiency was obtained as 41.98% at 120 minutes, which corresponds with the results of the above studies. Therefore, in this study, the retention time of 2 hours will be introduced as the optimal exposure time.

4.6. Impact of Temperature on Absorption Rate

Figure 8 indicates impact of optimal temperature on absorption efficiency. Figure 8 shows that absorption is increased by increasing temperature. In a study on basic red 46 dye removal from contaminated water using hardened Portland cement pieces as absorbent, it was reported that absorption was increased by increasing temperature. It is due to endothermic and the chemical absorption nature of the process (43). In a study on the application of egg shell as a natural absorbent for removal of Reactive Red 123 dye from synthetic textile wastewater, it was reported that in the temperature range of -45 °C, removal efficiency increased with a temperature increase and then decreased. It is assigned to the change in the contaminant structure or surface characteristics of the absorbent used at higher temperatures resulting in decreased absorption efficiency (41). In addition, number of contacts between particles and absorbent surface is increased ad absorption is increased by increasing temperature. Highest removal efficiency was obtained at 50 °C in this study which is consistent with the above studies.

4.7. Absorption Isotherms

Adsorption isotherms indicate absorbent molecules in equilibrium between liquid and solid phases. Basis of isotherms is in the description of the behavior of the absorbed participle and the absorbent as well as providing an important plan of absorption type (44). Freundlich and Langmuir adsorption isotherm models are investigated in Figures 9 and 10. Figures 8 and 9 show that dye adsorption is more following the Freundlich isotherm. In the study of absorption of Acid Red 18 dye using bio-adsorbent Sargassum Glaucescens from aqueous solution, it was reported that the absorption process follows Freundlich isotherm (R^2=0.992) which represents the multi-layer nature of dye removal process (45). In the study on methylene blue dye removal from synthetic wastewater using bone ash it was reported that the dye absorption on bone ash follows the Freundlich isotherm due to higher correlation coefficient (R^2=0.99) (46). In this study, the adsorption of acid orange 7 dye on tea residue followed the Freundlich isotherm with a regression coefficient of 0.993, and corresponded with the results of the above studies.

5. Conclusions

This study aimed to evaluate the efficiency of Acid Orange 7 dye by surface adsorption process using tea residue. Study results showed that the optimum conditions for a reduction of 50 mg/l of the dye with an efficiency of 98.41% is obtained under pH 2, 10 g/l as adsorbent dose and contact time of 120 minutes, at a temperature of 30 ˚C. Thus, the results show that adsorption of Acid Orange 7 dye by the respective absorbent is influenced by various parameters such as pH, absorbent dose, initial dye concentration, contact time and temperature such that dye removal efficiency has direct relationship with increasing the adsorbent dose, contact time and temperature, and it is inversely related with increasing pH and initial dye concentration. Equilibrium data in this study was obtained to follow Freundlich isotherm. According to the granular structure of CTC tea residue (fine-grained tea), raw absorbent is an appropriate and cheap adsorbent in surface adsorption of Acid Orange 7 dye from an aquatic environment.

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Conflict of Interest:
There is no conflict of interest to be declared.

Authors' contributions:
All authors contributed to this project and article equally. All authors read and approved the final manuscript.

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