Vol. 3 No. 2, Dec. 2023

FUAM Journal of Pure and Applied Science

Available online at
www.fuamjpas.org.ng

An official Publication of
College of Science
Joseph Sarwuan Tarka University,
Makurdi.

FUAM Journal of Pure and Applied Science
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https://fuamjpas.org.ng/
Vol.3. No. 2. December, 2023

Kinetics of Brilliant Green Adsorption onto Activated Carbon, Chitosan and Chitosan-Activated Carbon Blend

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Received: 29/08/2023 **Accepted**: 16/09/2023 **Published online**: 12/10/2023

Abstract
The kinetic adsorption of BG Dye onto chitosan-activated carbon blend was investigated using Batch adsorption process. The adsorbate used is the brilliant green dye (BGD), while Chitosan, Commercial Activated Carbon (CAC) and the blend were used as the adsorbents. A triplicate analysis was made at varying concentration of the Brilliant Green Dye solution (25, 50, 75, and 100mg/L) and at time intervals(10, 20, 30, 40, 50 and 60 minutes) respectively. The effect of temperature at298,308,318,328,338 and 348 K, were investigated.The datawere fitted into Pseudo-second order kinetic model, and their coefficient of correlation (R^2) determined.R^2values for AC and Ch were in conformity with the second order kinetics at all concentrations (0.93-0.99) for AC, (0.98-0.99) for Ch. ChGAC, R^2 of 0.97 fitted at concentration of 100 mg/L. Elovich kinetic models were investigated and R^2values determined.Thehigh R^2 values for Elovich were only confirmed at 25mg/L,5 0mg/L and 75mg/L for AC and with less significant values at Ch and ChGAC, which show non-compatibility to this model, except at 100mg/L. Generally the adsorption of BG dye on AC, and Ch could compete significantly compared to ChGAC.
Keywords: Kinetic, Adsorption, Chitosan-Activated Carbon, Blend, UV-visible spectrophotometer.

Introduction

Textile industries are using large amount of water and organic chemicalsfor colouring. Discharging of dyes into water stream even in small amount can affect the aquatic life. The adsorption technique is an effective and attractive process for the treatment of dye-bearing wastewaters. They are used to colour substrate like textile fibre, paper, leather, hair, fur, plastic material, wax, a cosmetic base and food stuff. Textile dyes constitute a major source of pollution [1]. Many dyes and pigments are hazardous and toxic to human and aquatic life at higher concentrations when discharged to receiving water bodies. Higher concentrations of dyes are known to cause ulceration of skin, mucous membrane, dermatitis, perforation of nasal septum, severe irritation of respiratory tract and on ingestion may cause vomiting, pain, haemorrhage and sharp diarrhea [2].

Dye colours are visible in water concentration as low as 1 mg/L, whereas textile processing wastewater normally contains more than 10 – 200mg/Lof dye concentration resulting in aesthetic problem, affecting photosynthesis in aquatic plants and have toxic and carcinogenic effect in mammals. About 10 – 15% of dyes go unused in textile effluents fungi or their oxidative enzymes can decolorize textile wastewater either by adsorption of dyes on fungal mycelium or by oxidative. A very small amount of dye in water (10 – 50mg/L) is highly visible and reduces light penetration in water systems, thus causing a negative effect on photosynthesis.

Several factors determine the technical and economic feasibility of each single dye removal technique. These include; dye type and its concentration, wastewater composition, operation costs (energy and material), environmental fate and handling costs of generated waste products. Furthermore the dyes and/or their degradation products may be toxic to ora and fauna [2].

Dye contamination in the environment has been a serious environmental concern due to its toxicity, persistency and accumulation. This contamination has increased over decades due to increase in population which has increased the consumption rate and has led to rapid industrialization. It is essential to extend methods for removal of dyes to decrease the pollution load on the environment. Classical techniques of dye removal from solutions include the following processes; solvent extraction, electrochemical reduction, chemical and biosorption, pre-concentration.Of all the treatment methods mentioned above, adsorption had been reported as an efficient method for the removal of dyes from aqueous solution because of their effectiveness even at low concentration [3].The adsorption process has also recorded much attention and has become one of the more popular methods for the removal of dyes from waste water, because of this competitive and effective process. The commercial activated carbon most widely used as an adsorbent has become economically less feasible owing to its relatively high cost. Also the development of surface modified activated carbon has produced a variety of activated carbon with far surpassing adsorption capacity [3].In any agricultural country such as Nigeria, accumulation of waste material is always one of such agricultural by-products of farming activities. The uses of such agricultural by-products have been in their conversion into usable forms. Caustic soda is a widely used industrial alkali, and it serves at least 61 major industrials in the country [11]. It has numerous applications in rayon and other textile

manufacture, pulp and paper production, reducing of rubber, food production, detergents and soap production. In recent years, development of surface modified activated carbon has generated a diversity of activated carbon with far superior adsorption capacity. Among these low cost adsorbents, chitosan has the highest sorption capacity for several sorbates [3]. Chitin (2-acetamido-2-deoxyd-glucose-(N-accetylglucan) is the main structural component of molluscs, insects, crustaceans, fungi, algae and marine invertebrates like crabs and shrimps. Chitosan (2-acetamido-2-deoxy-β-d-glucose-(N-acetylglucosamine) is a partially deacetylated polymer of chitin and is usually prepared by deacetylation of chitin with a strong alkaline solution [3].

Chitosan is slightly soluble at low pH and possess problems for developing commercial applications [4]. Also the active binding sites of chitosan are not readily available for sorption. The sites are reported to be soft and have a tendency to agglomerate or form gel in aqueous solutions. Therefore, it is necessary to provide physical support and increase the accessibility of the metal binding sites for process application. Ion exchange is considered a better alternative technique for such a purpose

Materials and Methods

Materials

All the glassware used in this research work were soaked in 1M HCl overnight and thoroughly washed with detergent, rinsed with distilled water and dried in the oven at 103°C. Similarly all the chemical reagents employed in this work were of analytical grade.

Reagents:

The reagents used include; Commercial activated carbon (CAC), High molecular weight Chitosan, 75% deacetylated(Sigma Aldrich),Brilliant Green dye,NaOH,

Apparatus

Beakers, measuring cylinders (Pyrex), Crucibles, Volumetric flasks, conical flasks, test tubes, pipettes, and funnels were used for this experiment.

Equipment

The equipment used were; Oven,(DHG-9053A, Gallenkomp, England) Desiccators, Glassware, litmus papers, Filter papers, thermometer, filtration apparatus, Analytical Weighing balance (model PX224/E), Stuart Magnetic stirrer/hot plate (Bibby Sterlrin LTD, Stone, Stffordshire, ST15 OSA, UK) Flask shaker, hot plate, sieve, Temperature meter, Hanna instrument, Italy), Electric shaker, water bath (Genlab Thermal Engineers, model WBH6).

Instruments

The instruments used include; Jenway (JENWAY7415) single beam UV-visible spectrophotometer.

Preparation of stock and standard solution

Stock solution of Brilliant green was prepared by dissolving 1g of Brilliant Green in about 100 mL distilled water. The solution was transferred to 1 dm^3 volumetric flask and made to mark with distilled water to obtained 1000 ppm[5,6].Different working standards were prepared from the stock solution ranging from 25, 50, 75 and 100 mg/L using serial dilution.

0.1 M HCl

HCl (8.50 cm^3) (Sp. Gravity 1.18 g/cm^3) was measured, transferred into 1 dm^3 volumetric flask and made to mark with distilled water. The solution was standardized with 0.1 M Na_2CO_3.

0.1 M Na_2CO_3

Exactly 20.50g of anhydrous sodium carbonate was dried in the oven. Then, 10.60 g was dissolved in 100 mL distilled water and transferred to 1 dm^3 volumetric flask and made to mark with distilled water.

0.1M NaOH

Exactly 4.0 g NaOH pellet was dissolved in a beaker, transferred to 1 dm^3 volumetric flask and made to mark with distilled water. The solution was standardized with 0.1 M HCl.

0.5% NaOH

NaOH (5.0 g) was dissolved in 200 mL of distilled water and transferred to 1 dm^3 volumetric flask then made to mark with distilled water.

Oxalic acid 10%

Oxalic acid (100 g) was dissolved in 100 mL distilled water. The solution was transferred to 1 dm^3 volumetric flask and made to mark with distilled water.

Preparation of chitosan gel

About 5g of chitosan was slowly added to 100 mL of oxalic acid in a beaker. The mixture was stirred for homogeneity, and then heated to about 50°C for 10 minutes to facilitate mixing. A chitosan-oxalic acid mixture was allowed to gel for 20 minutes.

Production of grafted chitosan

The chitosan gel (100 mL) was diluted with 500 mL distilled water and heated to about 50°C. Then 50g of activated charcoal was slowly added and agitated using a shaker for 24h. The chitosan engrafted activated charcoal was washed with distilled water severally and dried at 40 °C in a dry oven. It was soaked in 0.5% NaOH solution for 3h to neutralize the acid and thereafter extensively rinsed with deionized water and dried at 100°C for 2h and stored in a dessicator [3].

Batch adsorption studies

Kinetic studies of Brilliant Green Dye were investigated using Batch adsorption process as reported by [7]. Experiments was conducted in four different 100 mL flasks containing 50 mL varied concentration (25, 50, 75 and 100 mg/L) of Brilliant green dye solution and the adsorbent dosage of 1g was added to the different flasks. The flasks were agitated on a shaker. The mixture was filtered after 10 min, and the filtrate analysed at 624 nm using UV-visible

spectrophotometer (Jenway, model 7415). The adsorption was repeated using the chitosan, activated carbon and chitosan-grafted activated carbon formulation, contact period of 10, 20, 30, 40, 50 and 60 minutes were observed. Triplicate analysis was carried out for each adsorption experiment. Batch adsorption studies for different concentrations of BG dye was carried out. The adsorption capacity (qt) was calculated as follows:

$$qt = \frac{(C_0 - C_t)V}{M} \quad (1)$$

time t, qt (mg/g) is the amount adsorbed at time t, V(L) is the volume of the solution, and M(g) is the mass of adsorbent. The percentage removal of the Brilliant Green dye was calculated using the relation:

$$\% R_e = \frac{C_0 - C_f}{C_0} \times 100 \quad (2)$$

.% R_e represents the percentage adsorption or efficiency, Co the initial dye concentration(mg/L) and C_f is the final dye concentration (mg/L).

Effect of temperature;

The adsorbent (0.5g) was mixed with 50 mL BG dye and treated using the temperature controlled water bath shaker. The water bath was set at 25^{0}c for 1 hr, thereafter the sample was taken off and filtered using Whatman No 2 filter paper, stored in a well labelled glass bottle at normal room temperature for absorbance measurement. This procedure was repeated for samples at different temperatures (35, 45, 55, 65) ^{0}C for 1 hr

Effect of contact time;

Exactly 0.5g adsorbent was placed in 100mL Erlenmeyer flasks containing 25, 50, 75,100 mg/L dye. The flasks were agitated continuously for 10 minutes. The resulting mixture was filtered using Whatman No.42 filter paper and stored in well labelled glass bottles at room temperature for absorbance measurement. The procedure was repeated for 20, 30, 40, 50, and 60 minutes agitated time.

Adsorption kinetics

In order to evaluate the kinetic mechanisms which controls the process, First-order, Pseudo-second order, and Elovich models were tested and the validity of the models were verified by their linear equation plots were based on methods documented by [7].

Results and Discusion.

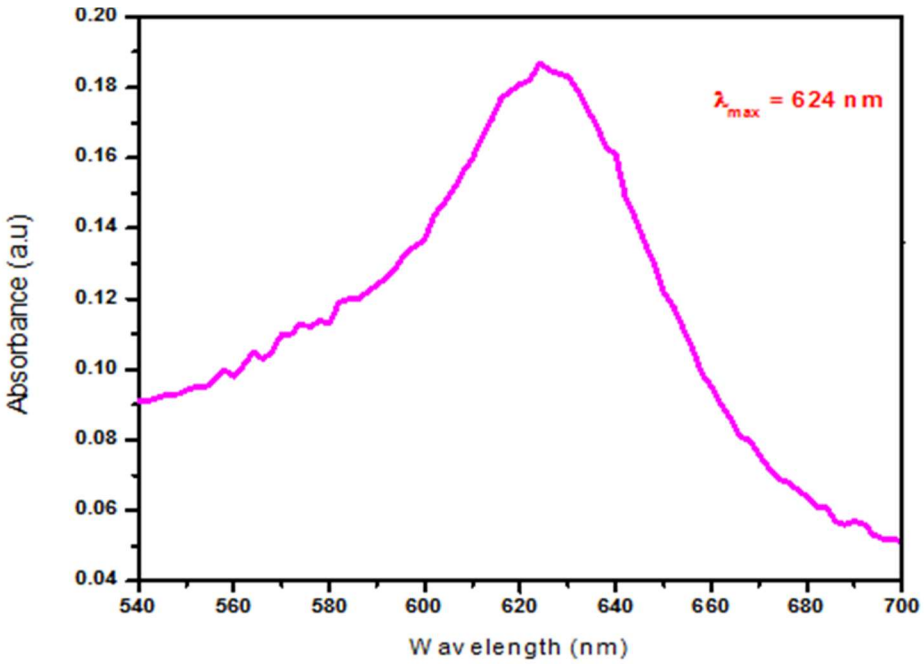


Fig. 1: Absorption Spectrum of Brilliant Green Dye

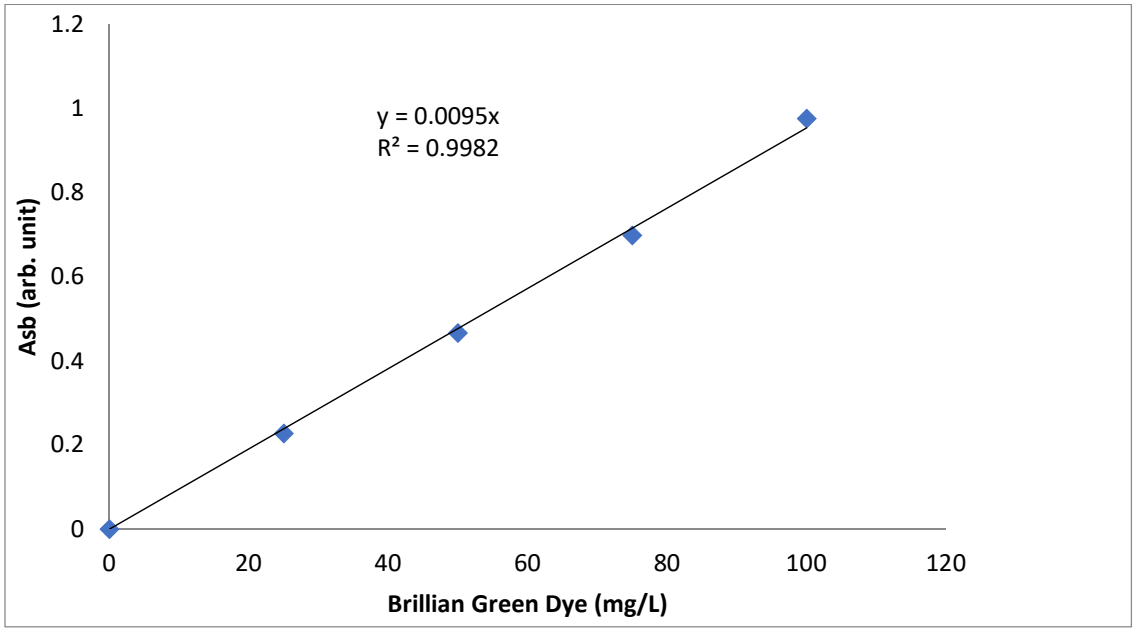


Fig.2 Calibration Curve for Brilliant Green Dye

Table 1: Pseudo Table - Second Order Kinetic Constants for Uptake of BG Dye on AC, Ch and ChGAC at 25 mg/L

Adsorbent	Regr. Equation	R^2	Constant (mg/L)	K_2(g/mg/min)
AC	y = 0.575x +4.229	0.93754	1.7388	0.0889
Ch	0.5438x – 1.67	0.98503	1.8389	8.379
ChGAC	-0.11877x – 18.10133	0.47917	0.11877	102.533

Table 2:Pseudo- second order Kinetic constants for the Uptake of Brilliant Green Dye on AC, Ch and ChGAC at 50 mg/L

Adsorbent	Regression Equation	R^2	Constants Q_m (mg/g)	K_2(g/mg/min)
AC	0.215883x+0.346	0.99954	4.633276	0.1421
Ch	0.22811x +0.00267	0.99936	4.3838	18.5778
ChGAC	-0.7571+10.102	-0.93403	0.7571	0.01609

Table 3: Pseudo-Second order Kinetic Constants for the Uptake of BG Dye on AC, Ch, and ChGAC at 75 mg/L

Adsorbent	Regression Equation	R^2	Constants Q_m (mg/g)	K_2(g/mg/min)
AC	0.13549x+0.148	0.99979	7.3806	0.1269
Ch	0.1436x-0.016	0.99992	6.9638	0.7884864
ChGAC	0.7893x+3.118	-0.7733	0.7893	17.5134942

Table 4:Pseudo-second order Kinetic constants for the Uptake of BG Dye on AC,Ch,and ChGAC at 100 mg/L

Adsorbent	Regression Equation	R^2	Constants Q_m (mg/g)	K_2(g/mg/min)
AC	0.10329x+0.08	0.9999	9.6815	0.1362
Ch	0.1056x+0.012	0.99997	9.46897	1.0990
ChGAC	0.17306x+0.848	0.96665	5.7803	0.0353

Table 5: Elovich Kinetic Experimental Constants ForThe Uptake Of Brilliant Green Dye On AC, Ch, and ChGAC at 25mg/L

Adsorbent	Regression Equation	R^2	α(mglg/min)	Constants β (mg/g)
AC	0.22202x+0.66864	0.5508	4.51145	4.5041
Ch	0.22202x+0.66864	0.5508	4.51145	4.5041
ChGAC	-1.02471x+1.91311	-0.92766	6.93076	1.0347

Table 6: Elovich Kinetic Experimental Constants for the Uptake of Brilliant Green Dye on AC, Ch, ChGAC at 50 mg/L

Adsorbent	Regression Equation	R^2	α(mglg/min)	Constants β (mg/g)
AC	0.0728x+4.20523	0.6879	8.8857	13.7362
Ch	0.07155x+4.1547	0.7172	1.1823	13.9762
ChGAC	0.1322x-2.59936	0.2273	1.0472	7.5643

Table 7: Elovich Kinetic Experimental Constants for the Uptake of Brilliant Green Dye AC, Ch, and ChGAC at 75 mg/L

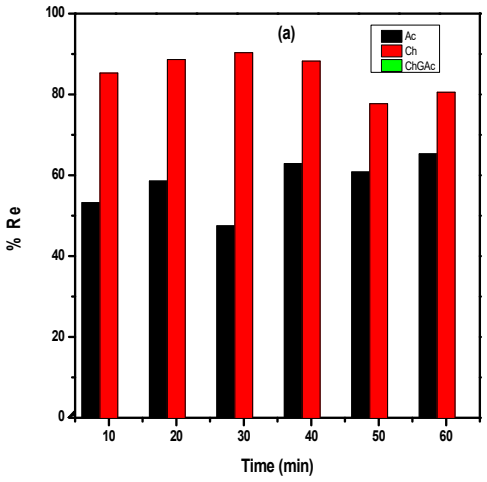
Adsorbent	Regression Equation	R^2	α(mg/g/min)	Constants β (mg/g)
AC	0.15722x+ 6.5457	0.8552	1.9168	6.3605
Ch	-0.02162x +7.0662	-0.3178	53.8859	0.0216
ChGAC	0.1385x – 2.098	0.1036	1.0601	7.2254

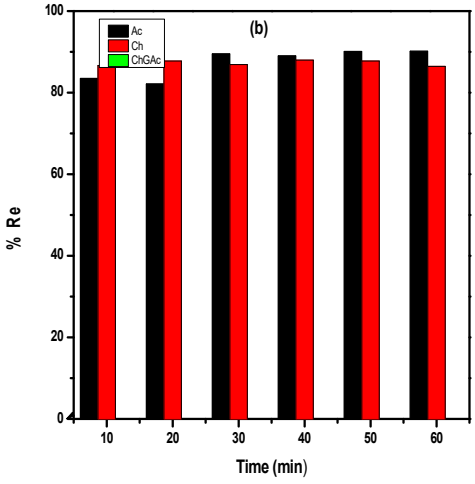
Table 8: Elovich Kinetic Experimental Constants for the Uptake of Brilliant Green Dye AC, Ch and ChGAC at100 mg/L

Adsorbent	Regression Equation	R^2	α(mg/g/min)	Constants β (mg/g)
AC	-0.1874x + 10.1885	-0.5416	35.99	0.1873
Ch	-0.0676x + 9.7666	-0.7920	28.6290	0.06759
ChGAC	0.4238x + 0.3909	0.3909	1167.94	2.3596

Effect of time

The charts in Figure 3.Shows The Effects of removal efficiency of BG Dye on time. Figure 4 shows The Effect of amount of BG Dye adsorbed on time at (a)25mg/L, (b) 50mg/L,(c)75mg/L and 100mg/L Dye concentrations





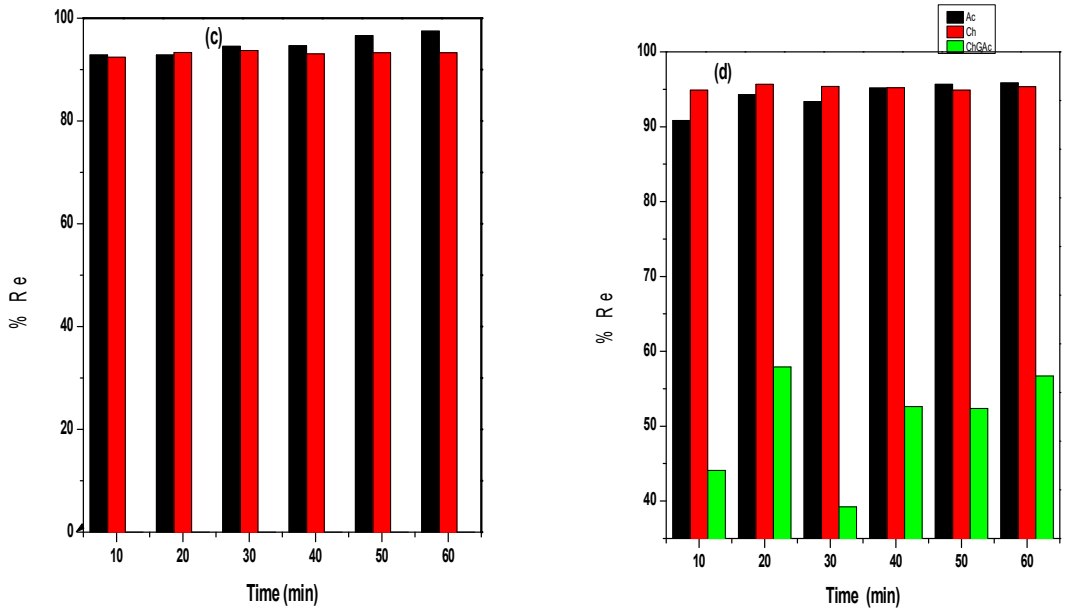
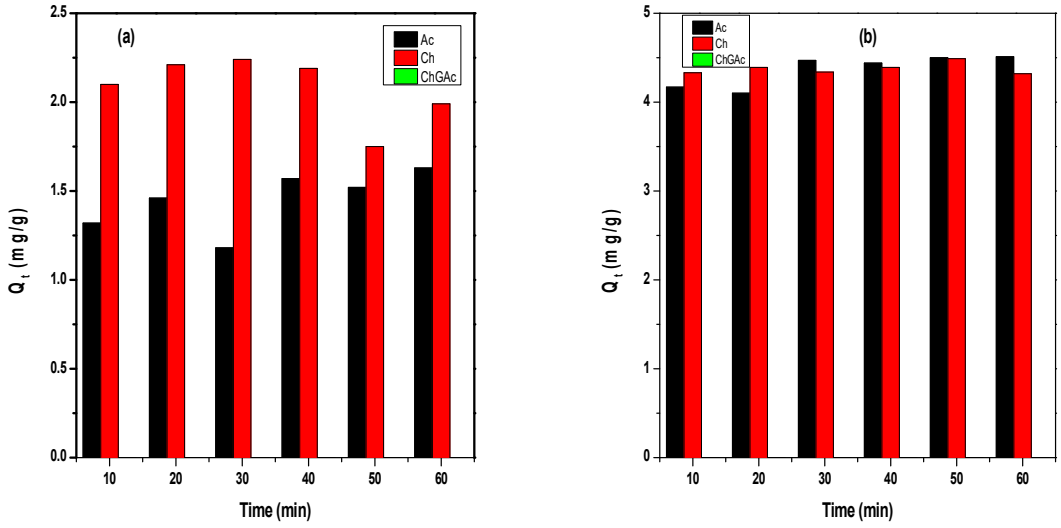


Figure 3: Plots of Adsorption Efficiency (% Re) Against Time onto AC, Ch and ChGAC for (a) 25 mg/L, (b) 50 mg/L, (c) 75 mg/L and (d) 100 mg/L Dye concentrations



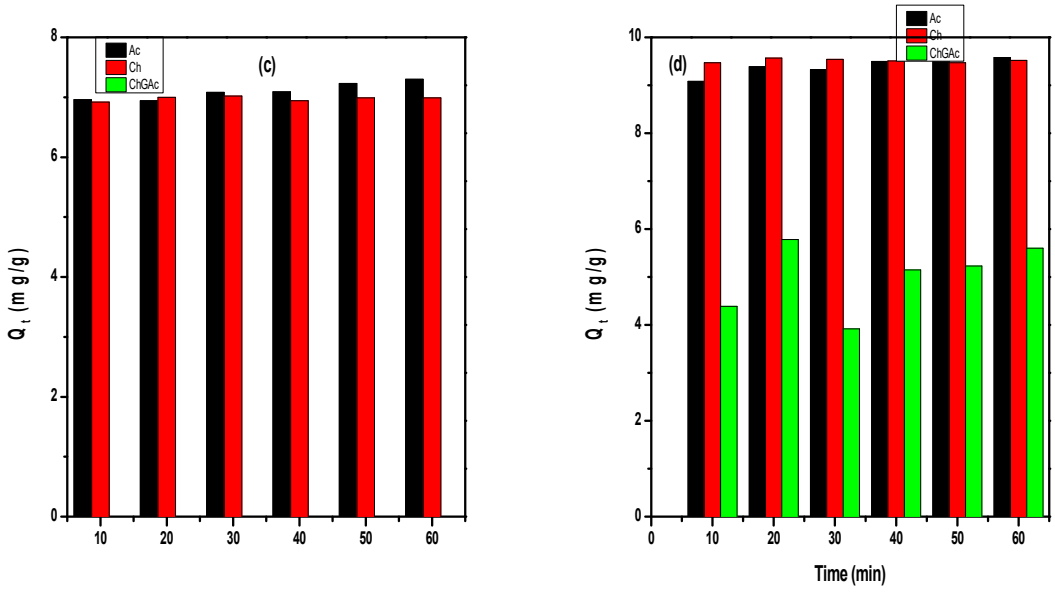
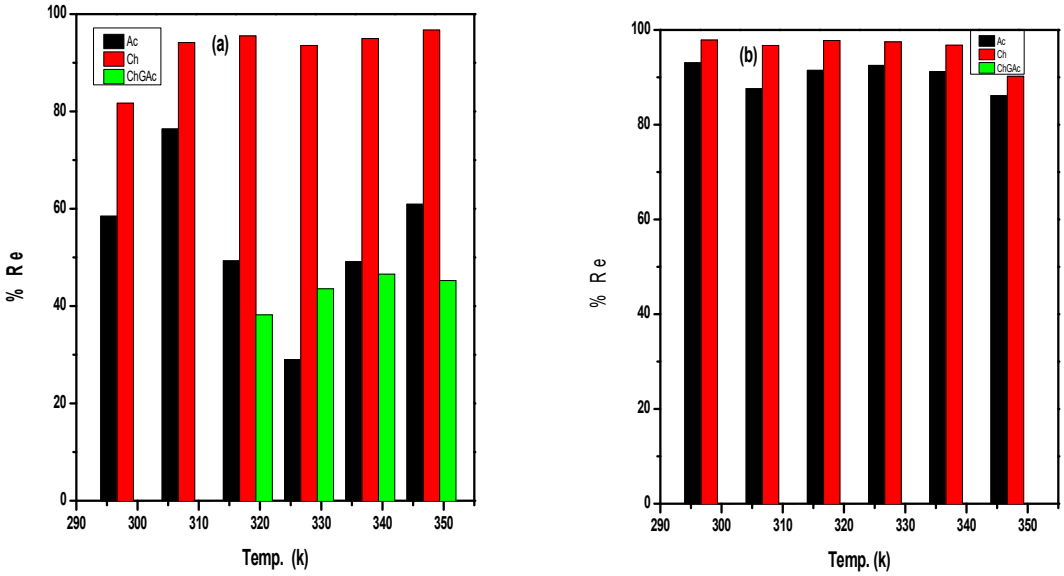
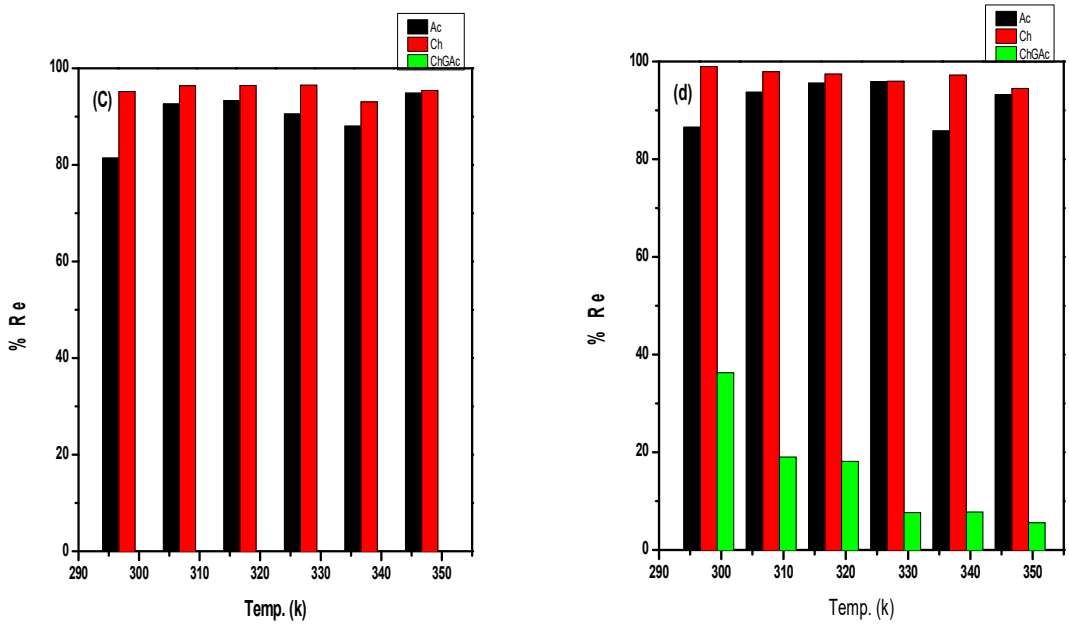


Figure 4: Plots of Adsorption Capacity (q_t) Against Time onto AC, Ch and ChGAC for (a) 25 mg/L, (b) 50 mg/L, (c) 75 mg/L and (d) 100 mg/L Dye concentrations

Effect of temperature.

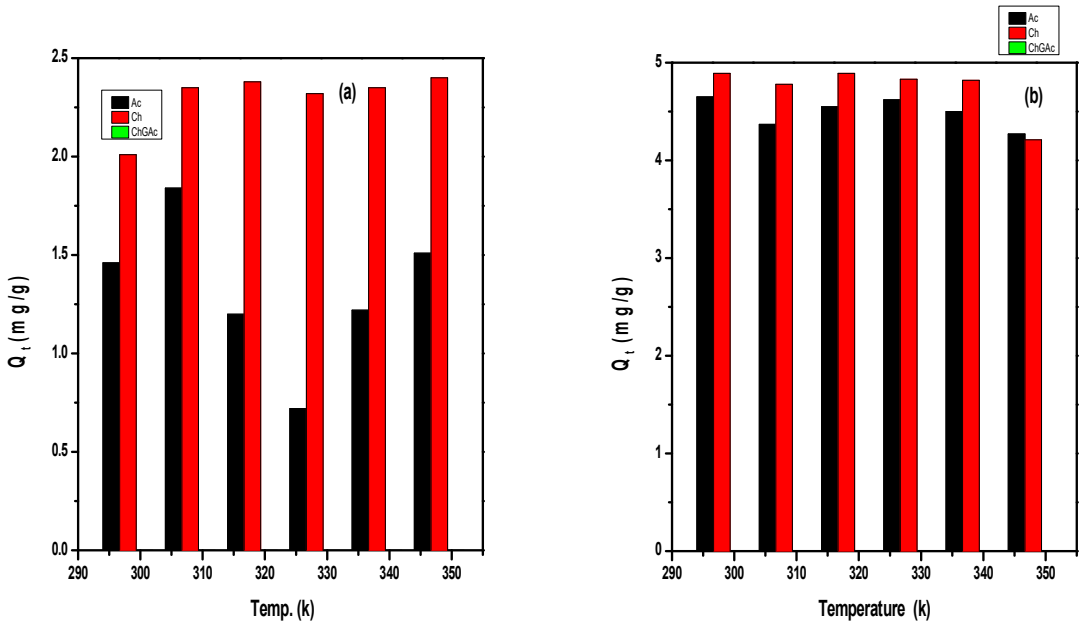
The charts in Figure 5 Shows The Effect of temperature on the removal efficiency of B.G Dye and Figure 6Shows The Effect of amount of B.G Dye adsorbed on temperature onto AC, Ch, ChGAC at (a)25mg/L, (b)50mg/L ,(c) 75mg/L and (d) 100mg/L Dye concentrations





Figure

Figure 5: Plots of Adsorption Efficiency (% R_e) Against Temperature onto AC, Ch and ChGAC for (a) 25 mg/L, (b) 50 mg/L, (c) 75 mg/L and (d) 100 mg/LDye concentrations



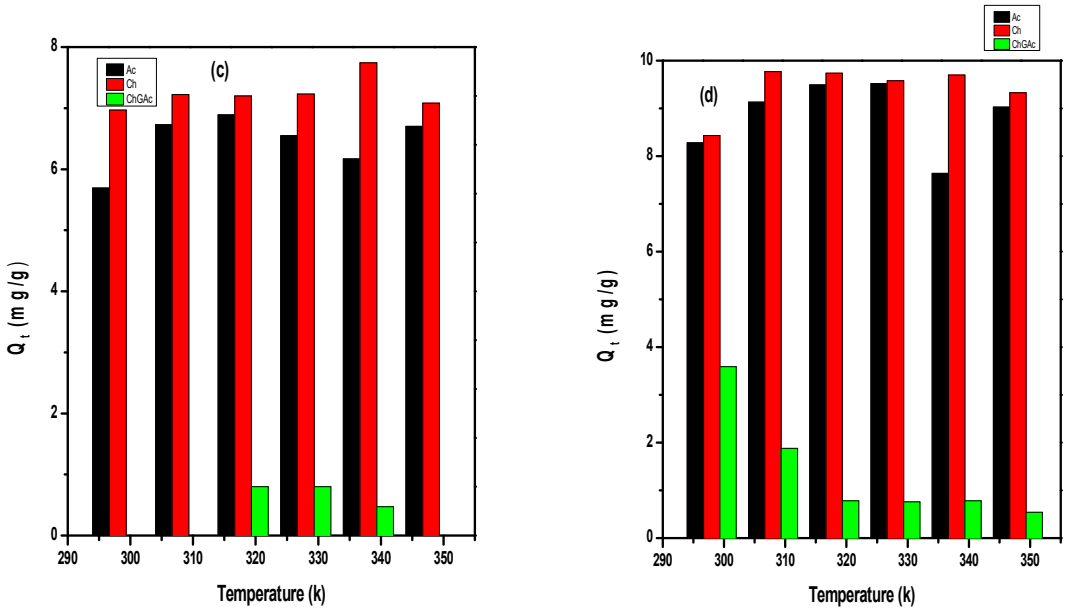


Figure 6: Plots of Adsorption Capacity (q_t) Against Temperature onto AC, Ch and ChGAC for (a) 25 mg/L, (b) 50 mg/L, (c) 75 mg/L and (d) 100 mg/L Dye concentrations

Adsorption kinetics and diffusion studies.
Adsorption kinetics was investigated using Pseudo-second order kinetic model as shown in .Figure (7, 8, 9, and 10) and Elovic models as shown in. Figure (11,12, 13, and 14) respectively.

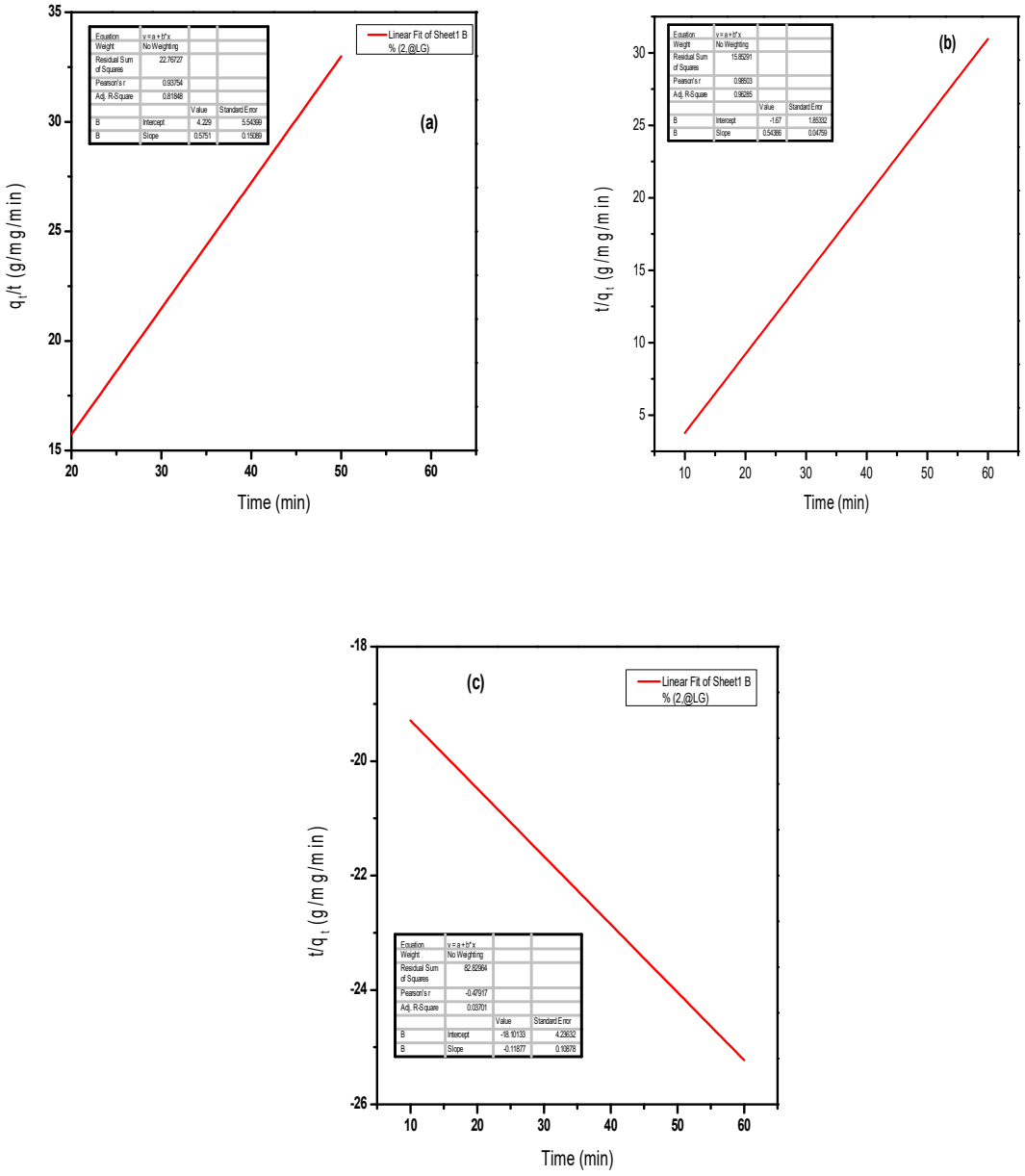


Figure 7: Pseudo-second Order Kinetic plots for the Uptake of BG Dye onto (a) AC, (b) Ch and (c) ChGAC for 25 mg/L

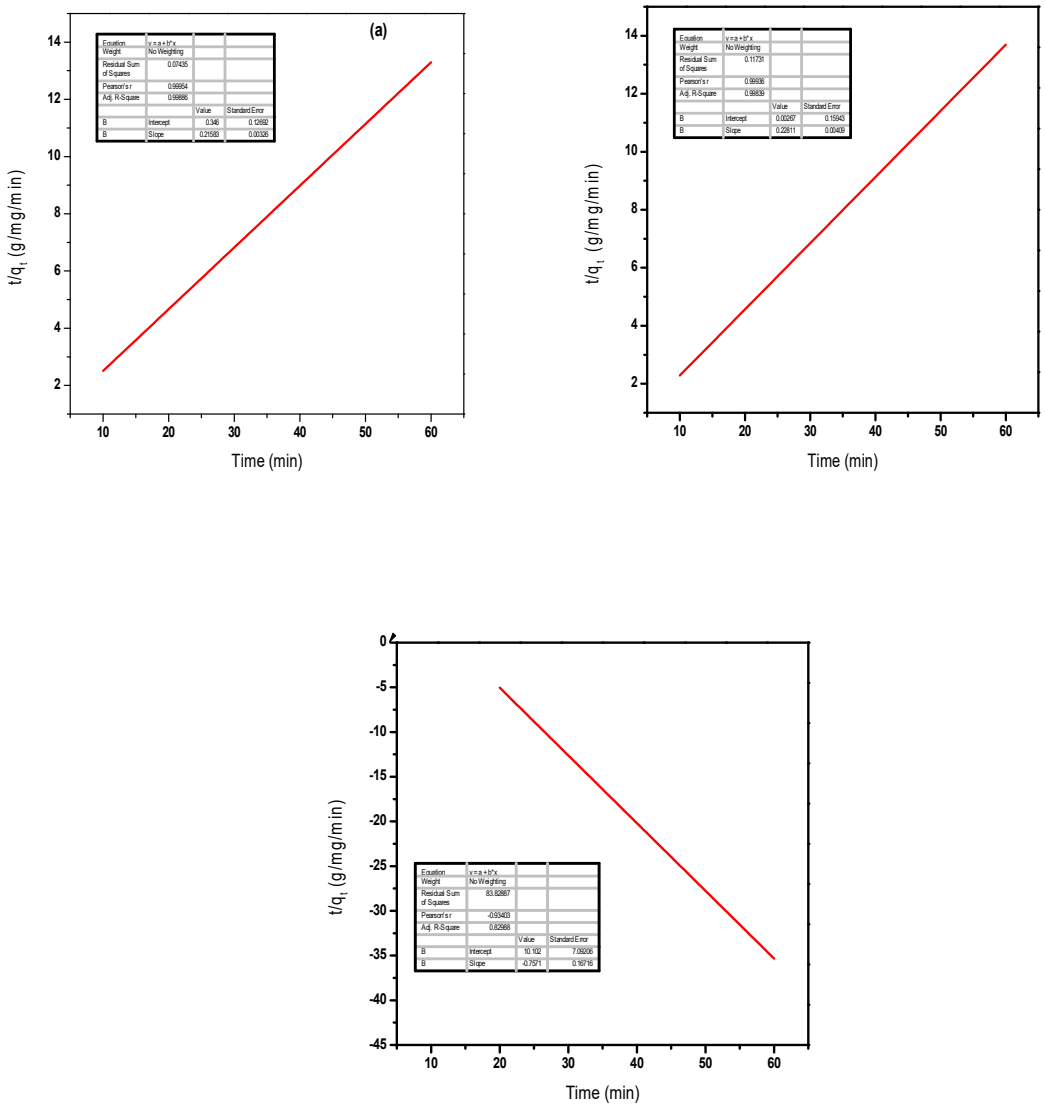


Figure 8: Pseudo-second Order Kinetic plots for the Uptake of BG Dye onto (a) AC, (b) Ch and (c) ChGAC for 50 mg/L

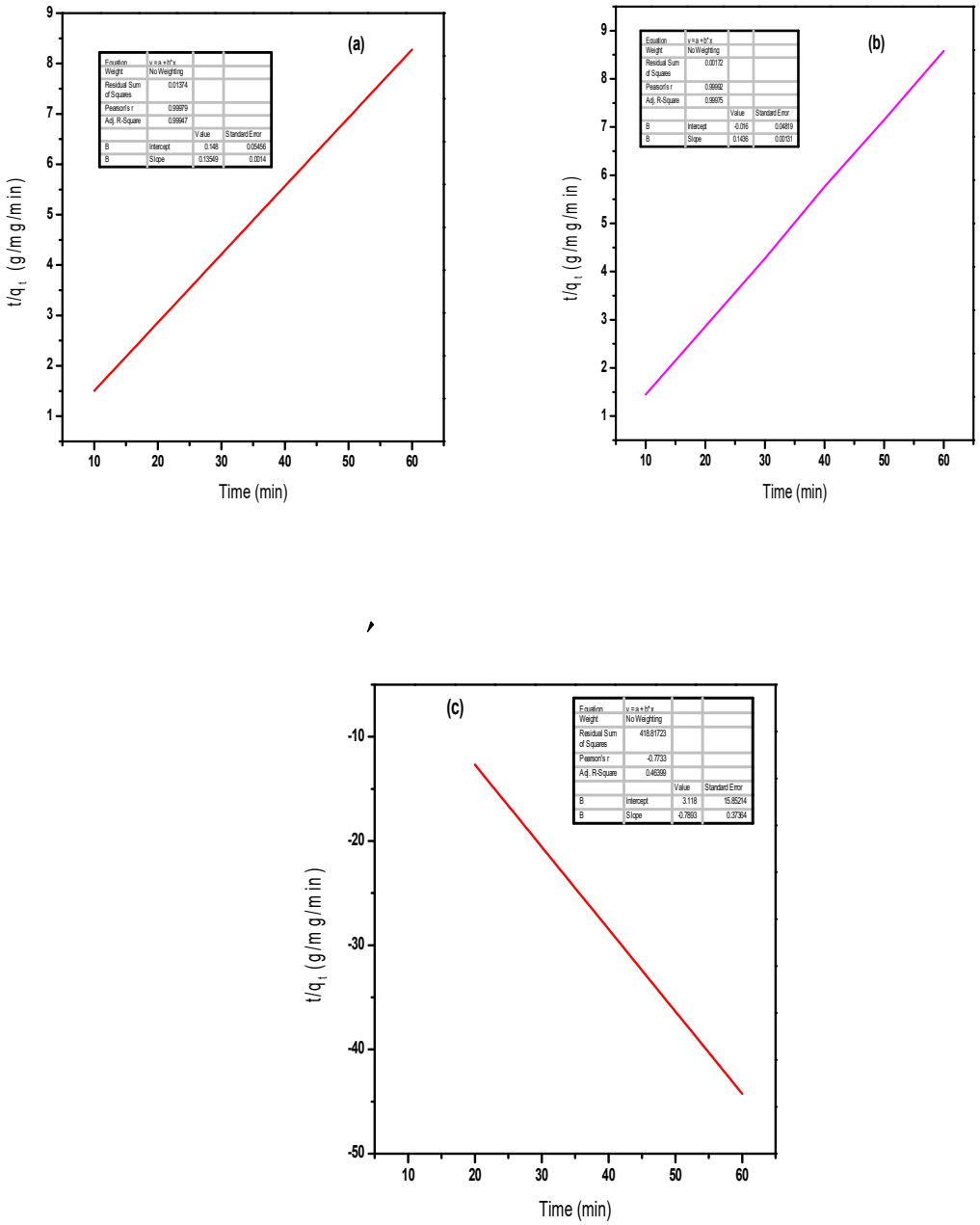


Figure 9: Pseudo-second Order Kinetic plots for the Uptake of BG Dye onto (a) AC, (b) Ch and (c) ChGAC for 75 mg/L

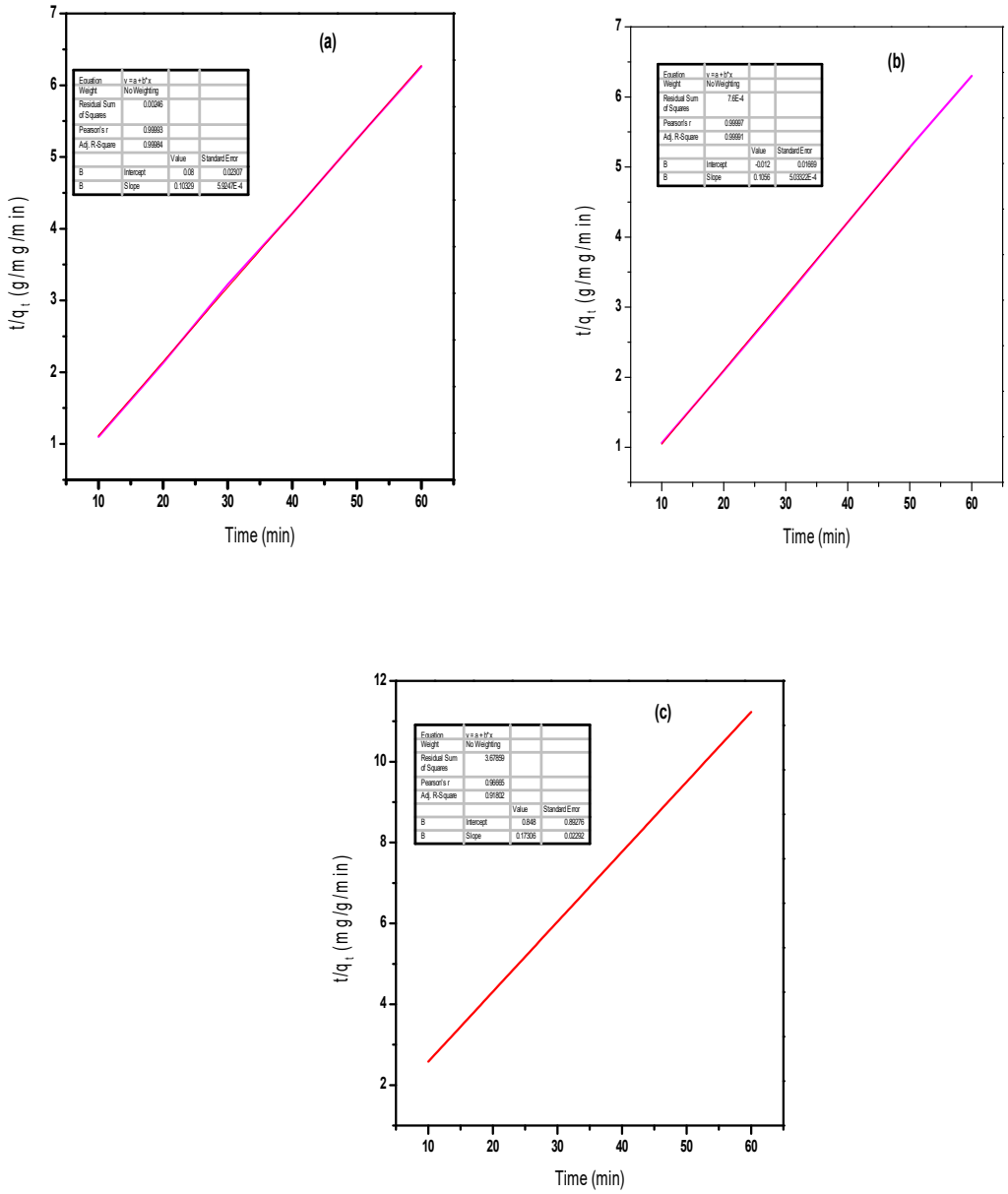


Figure 10: Pseudo-second Order Kinetic plots for the Uptake of BG Dye onto (a) AC, (b) Ch and (c) ChGAC for 100 mg/L

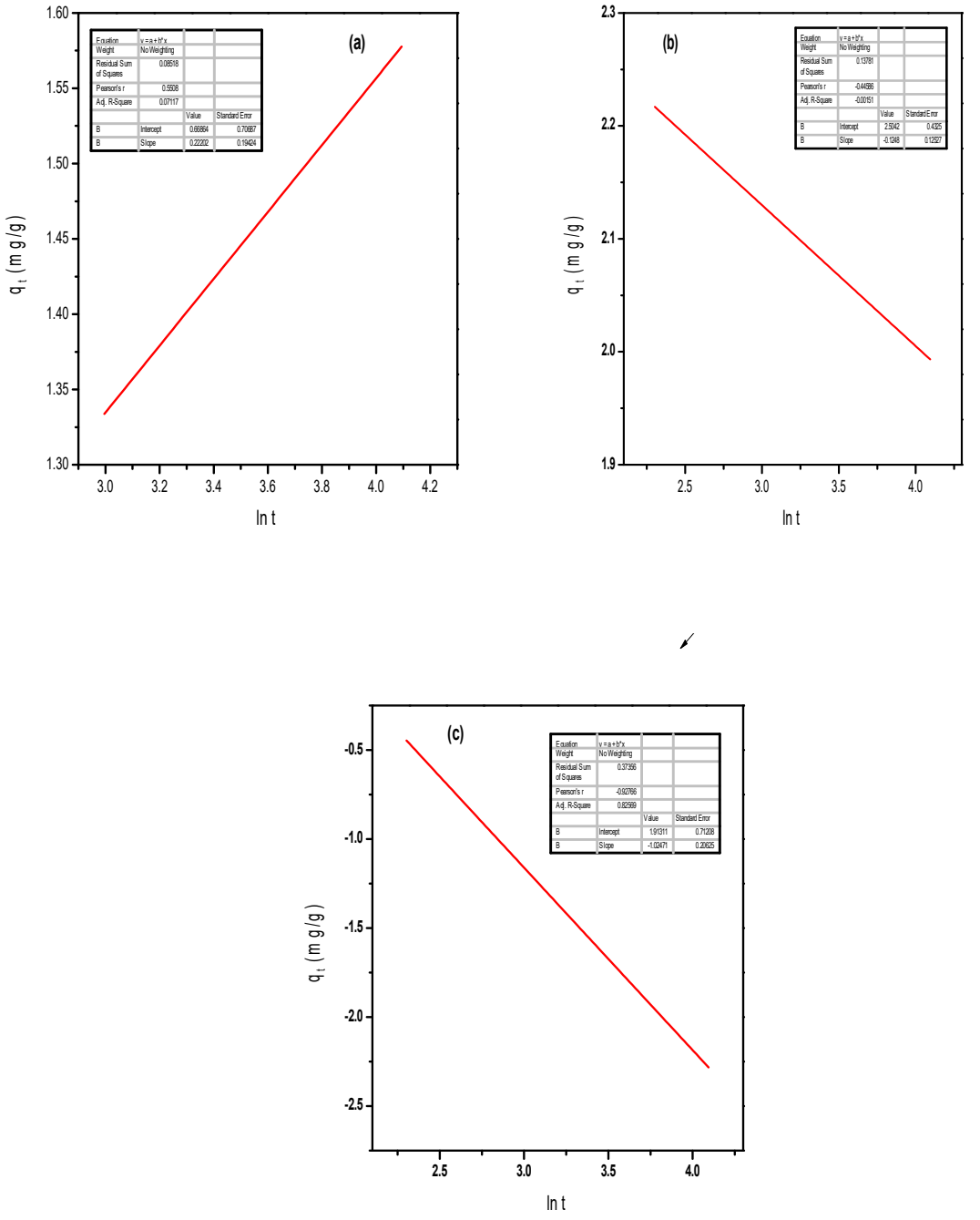


Figure 11: Elovic Model plots for the Uptake of BG Dye onto (a) AC, (b) Ch, and (c) ChGAC for 25 mg/L

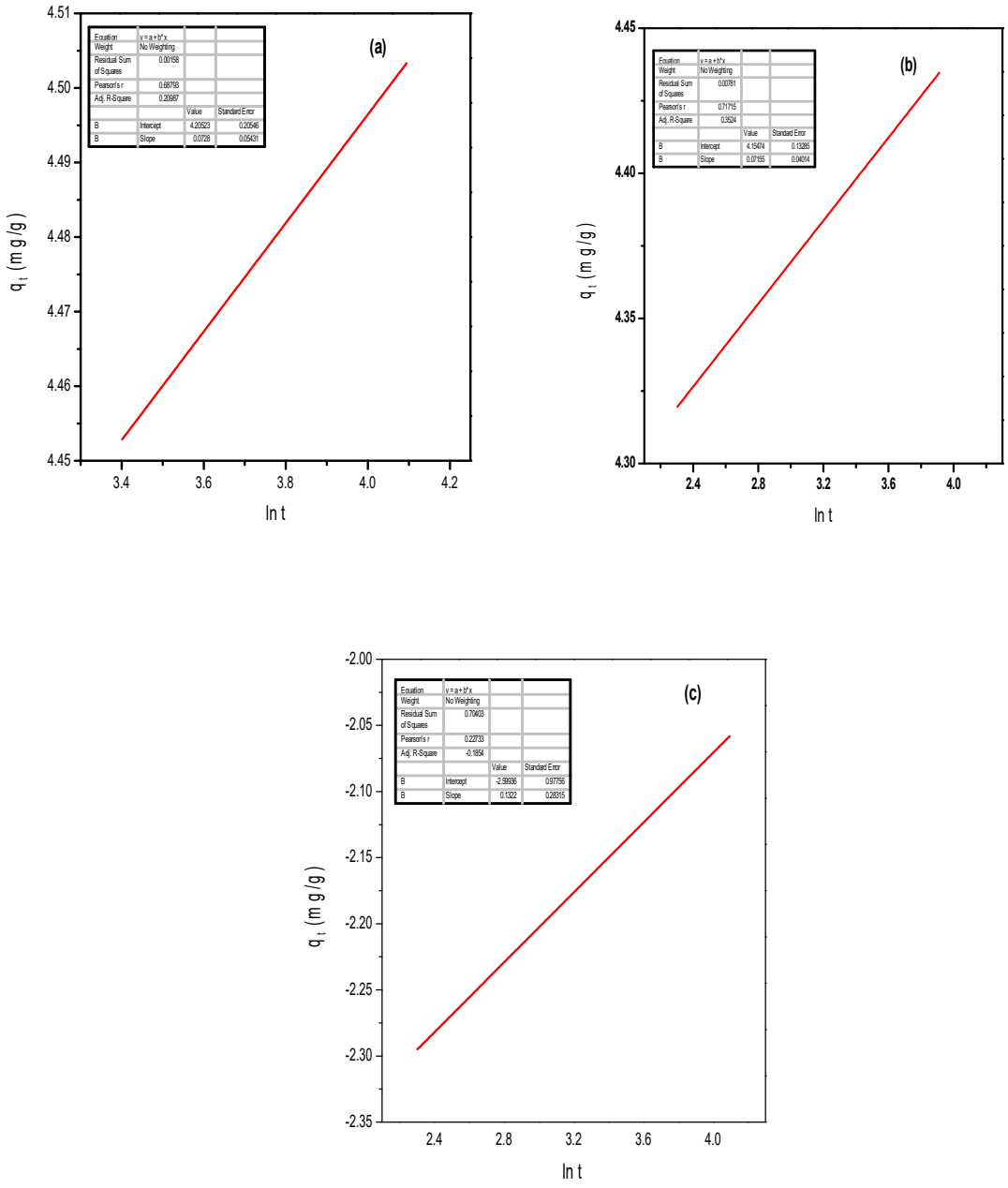


Figure 12: Elovic Model plots for the Uptake of BG Dye onto (a) AC, (b) Ch, and (c) ChGAC for 50 mg/L

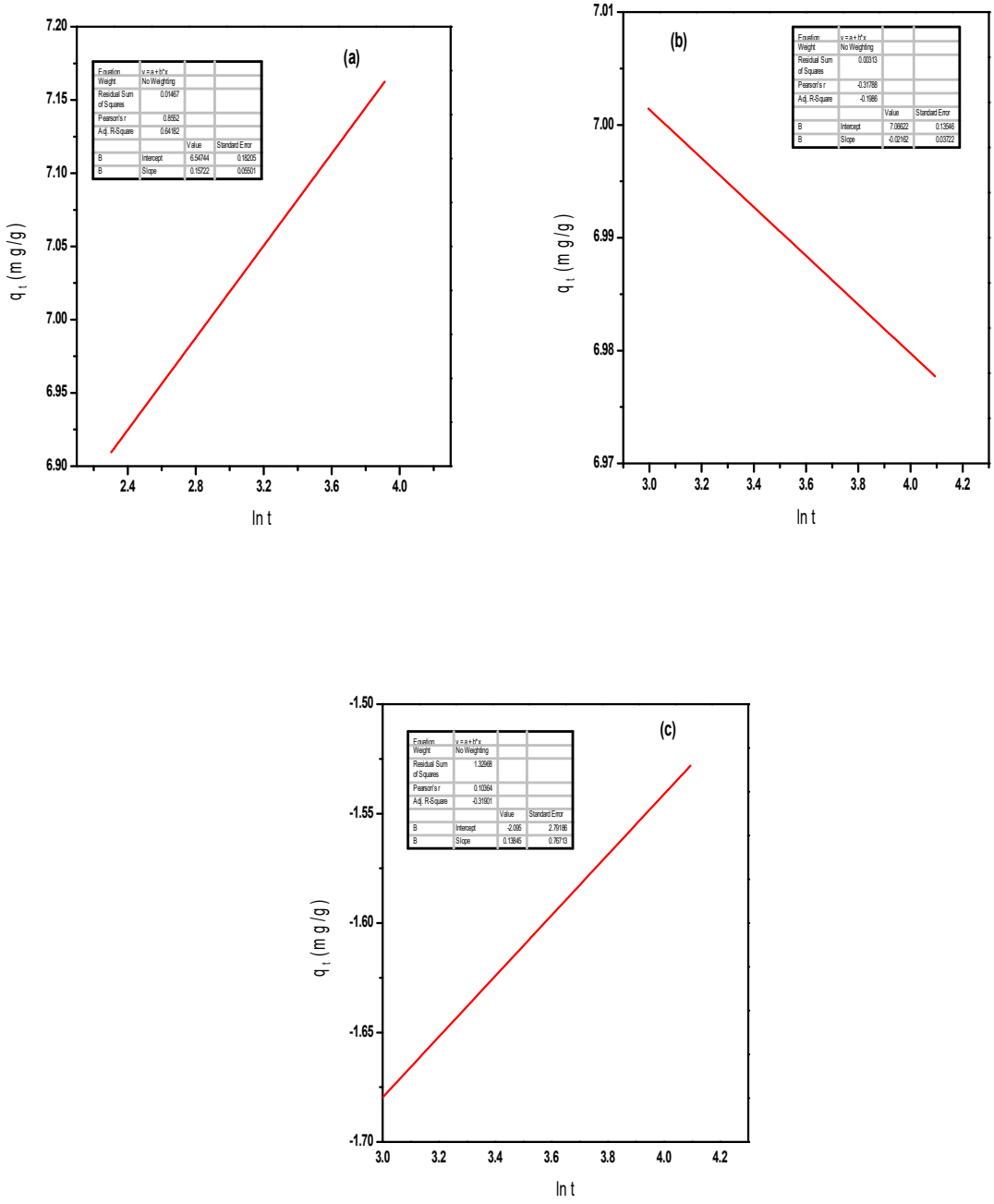


Figure 13: Elovic Model plots for the Uptake of BG Dye onto (a) AC, (b) Ch, and (c) ChGAC for 75 mg/L

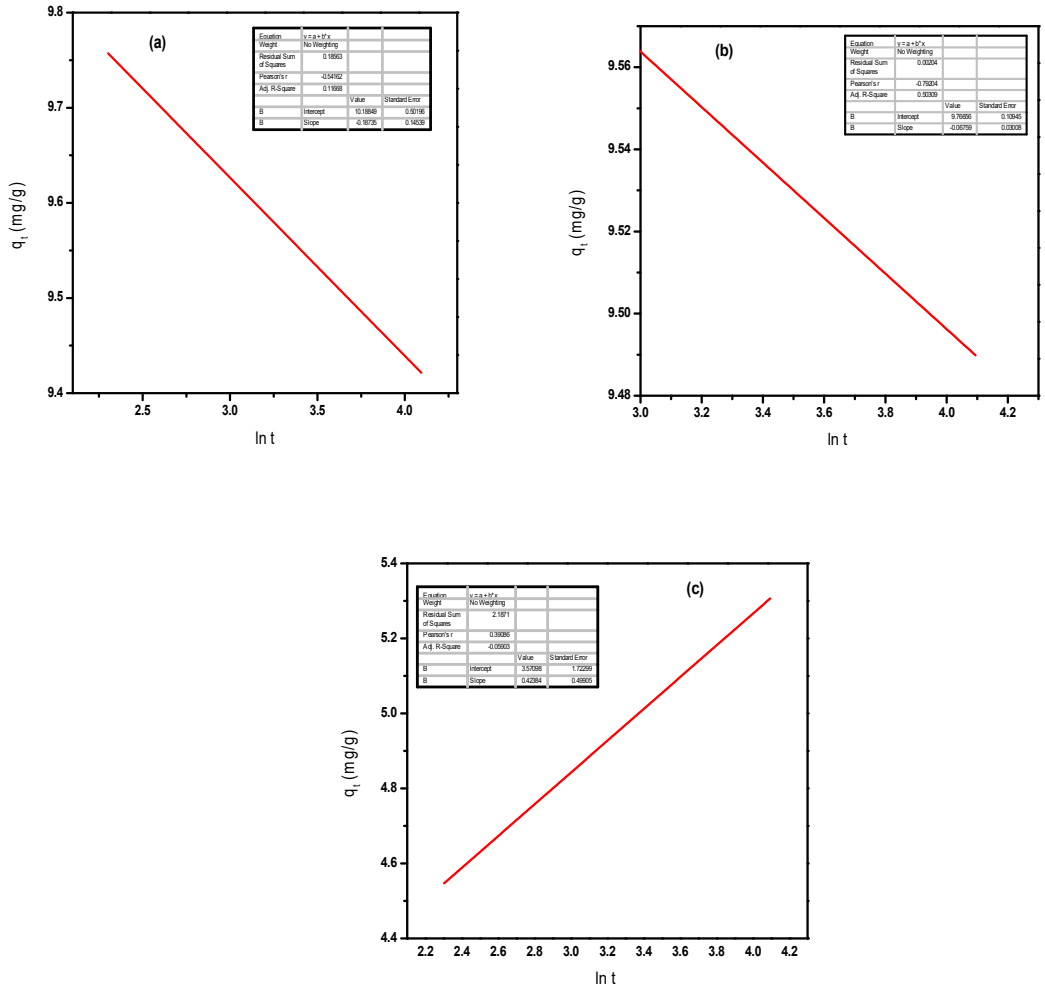


Figure 14: Elovic Model plots for the Uptake of BG Dye onto (a) AC, (b) Ch, and (c) ChGAC for 100 mg/L

Batch adsorption studies

Effect of contact time on uptake of BG dye on AC, Ch and ChGAC

With the uptake of BG dye onto Activated Carbon, Chitosan and Chitosan Grafted Activated Carbon at a contact time of 10, 20, 30, 40, 50 and 60 min and varying concentration of 25, 50, 75, 100 mg/L, (Fig 3, 4). The %Re for AC gave highest values 65.33, 90.18, 97.49, and 95.83% at 60 min for 25, 50, 75, and 100 mg/L respectively. The %Re values for Chitosan gave highest 90.31, 87.98, 93.74 and95.83% at 30min for 25, 50, 75, and 100 mg/L respectively. The %Re for ChGAC gave highest values of -96.83, -49.72 and -31.82%, at 30min and 57.91% at 20min for 25,50,75, and 100 mg/L respectively. The uptake of the BG dye with the stated adsorbents was favored with increasing time and better with Activated carbon and Chitosan compared to ChGAC. The increase in the trend of the BG dye uptake as a result of increasing time (10-60minutes), was achieved due to the adsorbent general monolayer on the adsorbent surface. Thus the removal of BGD from aqueous solution was controlled by the transport rate of the adsorbate species from the exterior to the interior site of the adsorbent.

Effect of temperature on uptake of BG dye on AC, Ch and ChGAC

Figures 5 and 6 Shows a plot of %Re against temperature and amount adsorbed (qt) against temperature. The dye adsorbed increased with increasing temperature. This indicates that the adsorption is endothermic and high temperature is favorable for the adsorption of the dye with Activated Carbon and Chitosan. The enhance adsorption of BGD with temperature is due to increased mobility of BG dye molecules and enlargement of pore size due to activated diffusion which causes pores to widen and deepen thereby creating more surface for adsorption. This result is similar to that carried by [9] on 'The studies of adsorptive removal of cationic dyes using a novel non-conventional Activated Carbon. The temperature increases the level of adsorbate

uptake by altering the interaction and solubility of the adsorbate. The efficient uptake of the adsorbate due to the increasing temperature causes more interaction between the adsorbate and the adsorbent.The high removal efficiency as a result of the increasing temperature is as a result of the increased mobility of the ions of the effluents and yielded a swelling impact with the interior structure of the adsorbent, thus allowing the large molecules to move deeper. The rise in temperature causes thermal energy as though higher temperature induces higher mobility of adsorbate.

Pseudo second-order kinetic model

The coefficient of regression suggested the applicability for Pseudo Second-order kinetic model for the uptake of B.G dye onto AC,Ch and ChGAC. From fig (7, 8, 9 and 10), These trends shows that the adsorption of BG Dye on AC and Ch at all concentration and ChGAC at a concentration of 100mg/L was controlled by chemisorptions and involves valence forces through exchange of electrons between the adsorbate and the adsorbents[3]B.G Dye uptake on ChGAC followedthetrend non-conformity with the Pseudo-Second order kinetic model.ThePseudo-second order kinetics is applicable to AC, and Ch at all concentrations, and with ChGAC at a concentration of 100mg/L. Since a plot of t/qt(g/mg/min) Vs time(sec) with a linear relationship [7].A fairly close agreement between the qt(exp) and qt(cal) were recorded.

Elovic kinetic model:

Figures11, 12, 13 and 14Shows thatthe high correlation values observed with AC at 25mg/L, 50mg/L, and 75mg/L shows conformity with a significant effect of the diffusion mechanism of B.G dye uptake with AC and determining diffusion as a parameter for the uptake of BG dye on AC. Another significant value is felt with Chitosan at 50mg/L corresponding to 0.7172. The very low regression value felt with ChGAC at all concentrations and Ch at (25, 75 and 100)mg/L has less significant effect indicating that Elovich model does not determine the rate of adsorption and does not show good applicability to this model given by their R^2 values.

Conclusion

Physical observation and physico-chemical parameters are signs that discharging dye effluent into the environment (water bodies and agricultural soils) could be detrimental to the environment, agricultural produce and human health respectively.The comparable level of percentage removal using the adsorbent is an indication of economic viability of the adsorbent.The kinetic adsorption studies showed that the dye uptake from AC, Ch. ChGAC as adsorbents best fit the Pseudo-second order kinetics with high correlation coefficient (R^2) values. The Elovich model correlation coefficient (R^2) values were high at (25, 50, and 75) mg/L for AC but less significant with Ch and ChGAC which shows non-compatibility to this model, except at concentration of 100mg/L.The adsorbent are effective for the uptake of BG dye from aqueous solutions. The Batchadsorption method was employed.The effect of temperature on the adsorbents shows that the adsorption process was endothermic and most favoured with an increase in temperature.In general, the uptake of BG dye on Ch, AC, ChGAC and its adsorption studies were significantly carried out. Results obtained compare favorably with studies on adsorption kinetics, and other commercial adsorbents for BG dye effluent remediation.

Declaration of Conflicting Interests

The authors declare no potential conflict of interest

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Cite this article

Akanya E.A., Itodo A.U. Sha,Ato R. Tsaviv N.J. (2023). Kinetics of Brilliant Green Adsorption onto Activated Carbon, Chitosan and Chitosan-Activated Carbon Blend. FUAM Journal of Pure and Applied Science, **3**(**2**):68-87