Int. j. adv. multidisc. res. stud. 2024; 4(6):677-685

Received: 15-10-2024
Accepted: 25-11-2024

International Journal of Advanced Multidisciplinary Research and Studies

ISSN: 2583-049X

Adsorpition and Kinetic Study for Removal Some Heavy Metals by Use in Activated Carbon of Sea Grasses

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DOI: https://doi.org/10.62225/2583049X.2024.4.6.3495
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Abstract
In this study Sea grasses were used to prepared active carbon by burned it in oven at 500 ^{0}C, for used it to removal some of metals solutions including Iron, Copper and Nickel. Different doses (0.1, 0.2, 0.3, 0.4 and 0.5 g) of activated carbon were applied, also the adsorption process was estimated at different times (15, 30, 45, 60, 75 and 90 min), the Langmuir and Frundlish isotherms were calculated. Also, the kinetic order of the adsorption was evaluated. The results of this study recoded different removal percentages of the applied dosages of activated carbon on the studied heavy metals, for the iron solutions, the higher removal percentage of (96 %) was observed after used (0.5 g) of adsorbent followed by dose of 0.1 which showed removal percentage of (70.80 %), meanwhile, the adsorbent dose of (0.1 g) recorded higher removal percentage of (80.71 %).For Nickel solutions the adsorbent dosage of (0.4 & 0.5 g) gave higher removal percentage (78.11 %) comparing with other doses used in this study. The adsorption isotherms of Langmuir and Frindulish were estimated by applied different times of (15, 30, 45, 60, 75 and 90 min), the Langmuir isothers of RL gave favorable adsorption, where the values of RL were 0.90, 0.547 and 0.19 for iron, copper and Nickel solutions, respectively and ranged between (0 – 1).On the other side the Frindulish isotherms showed different results, where the adsorption of metals selected in this study by activated carbon of sea grasses gave favorable results for iron and copper but unfavorable adsorption of Nickel. The kinetic of adsorption was calculated, the results recorded that the order rate of adsorption of metals selected in this study by sea grasses activated carbon follows first order.

Keywords: Adsorption, Heavy Metals, Sea Grasses

Introduction

The industrial revolution and rapid population growth have led to a significantly increased environmental heavy metal contamination since the 20th century, resulting in global health and environmental concerns (Abdelhafez, 2016; Ben Ali *et al*., 2017) [1, 3]. Heavy metal pollution poses a significant threat to ecological systems worldwide. Both developed and developing nations are grappling with this issue, stemming from various sources including industrial emissions, vehicular exhaust, waste incineration, and agricultural practices involving sludge composts and mineral fertilizers (Rezania *et al*., 2016).

Typically, the extent of water pollution is affected by the duration and nature of contaminating activities. To mitigate hazardous impacts and prevent the spread of toxic metals to adjacent agricultural land and groundwater, it is crucial to remediate contaminated sites**.** Numerous industries worldwide, including those involved in electronics, automotive, battery, plastic, paint, and ink manufacturing, rely on heavy metals in their production processes. As a result, heavy metal residues often contaminate water sources, leading to widespread water pollution. Therefore, removing of these toxic heavy metal contaminants from aqueous waste streams has become a significant environmental concern**.** One potential material for this purpose is thioglycolic acid-modified oil palm (Akaniwor *et al*., 2007) [2]. One of the most pressing environmental issues confronting our planet is the presence of elements like iron, nickel, and copper. Several effective methods exist to mitigate this pollution, including the physical technique of adsorption**.** Adsorption refers to the attachment of chemical species to the particles' surface. This process varies from absorption, where substances penetrate a solid or liquid to create a solution (Orth, 2006) [10]. In adsorption, particles of gas or liquid bind to a solid or liquid surface, known as the adsorbent, forming an atomic or molecular adsorbate film (Pardo *et al*., 2003) [11]. Adsorption, as defined internationally, is the phenomenon where the substance concentration increases at the boundary between a condensed phase and a fluid phase because of the influence of surface forces (Ferrari *et al*., 2010) [5]. The aim of this is using low coast activated carbon material of sea grasses for removal some of heavy metals (Iron, Nickel and Copper) from aqueous solutions.

Experimental

Chemical and apparatus:

The Chemicals and solutions used in this study were grade including: Ferric chloride ($FeCl_3.6H_2O$), Potassium thiocyanate (KSCN), concentrated hydrochloric acid (HCl), copper Chloride, ($CuCI_2.2H_2O$), pH buffer (universal), Sea grasses were collecting from beaches of Libya. Atomic absorption (perien Elymener), Nabertherm oven 30-3000°C, pH meter p 600.

Preparation of sea grasses activated carbon:

The Sea grasses were collected from cost line of Derna city, the samples were washed by tap water several times, then by distilled water, the samples were left to dry, cut in to small pieces, and burned in an oven for two hours at a temperature of 500 °C.

Adsorption procedure:

Effect of Dosage:

Different doses of Adsorbent (0.1, 0.2, 0.3, 0.4 and 0.5 g) were used in this study, with 20 ml metal ion solution of 100 ppm, 5 ml of ligand and supplement with distilled water to 50 ml. The bottles was shacked for 15 min at room temperature then they were filtered. The Absorbance value of complex compound solution were recorded by UV-VIS spectrometer.

Effect of time:

To establish the effect of time on the adsorption. Effect of times of (15, 30, 45, 60, 75 and 90 min) on the adsorption process was carried.

Adsorption studies:

A curve showing the relationship between concentration and absorption of different doses of iron complex.

Measurement of removal % of adsorption:

The amount of complex adsorbed per gm (q_e) was calculated on based C_o and C_e the initial and equilibrium concentration of adsorbate (metal); V is the solution volume (in liter); W adsorbent weight. the removal percentage of complex was calculated based on the following equation (Masoud *et al*., 2016) [8].

$$Removal\,\% = \frac{C-C}{C}\times 100$$

Adsorption Isotherms:

Adsorption isotherms, grounded in theoretical principles, can be constructed. This research explores two specific isotherm equations - Langmuir and Freundlich - to characterize the equilibrium behavior of adsorption. (Butraba *et al*., 2023, Hanan *et al*., 2018).

Langmuir adsorption isotherm: The most commonly employed isotherm equation for modeling equilibrium adsorption is the Langmuir equation. This equation, in its linear form, is typically expressed as follows:

$$\frac{Ce}{qe} = \frac{1}{ki} + (\frac{at}{kl})Ce$$

A plot of C_e versus C_e/q_e was linear showing the applicability of Langmuir adsorption isotherm for complexes adsorption. of adsorption, respectively which are calculated from slop and intercept of the plot C_e versus C_e/q_e. The important characteristics of Langmuir adsorption isothermcan be expressed in terms of a dimensionless constant, separation factor or equilibrium parameter (RL) which is defined by;

$$R = \frac{1}{1+al.Ci}$$

Where, C_i =initial concentration of the complex and a_l =Langmuir constant. $R_L>1$ Unfavorable, R_L =1 Linear, $0<R_L<1$ favorable, $R_L=0$ Irreversible. (Hasan *et al*.,2019)

Freundlich adsorption isotherm:

The Freundlich isotherm model, one of the earliest equations to describe the adsorption process, is an empirical model applicable to non-ideal sorption involving heterogeneous adsorption. It posits that the adsorbent surface is heterogeneous, comprising adsorption sites with varying adsorption potentials. The given equation, similar to the Langmuir equation, posits that each adsorption site class independently adsorbs molecules. This nonlinear equation is presented below:

$$q = KF$$

KF, a system constant linked to bonding energy, can be defined as the adsorption or distribution coefficient. It signifies the amount of complex adsorbed per unit equilibrium concentration on the adsorbent. The 1/n exponent reflects the adsorption intensity and surface heterogeneity of the complex on the adsorbent. A value of 1/n less than 1 suggests cooperative adsorption. Linearization through logarithmic transformation enables the determination of the Freundlich constant:

$$\log q_e = \log K_F + 1/n \log C_e$$

A plot of log C_e versus log q_e was linear, where K_F is measure of adsorption capacity (mg /g) and n is adsorption intensity. 1/n values indicate irreversible isotherm to be (1/n = 0), favorable (0 < 1/n < 1), unfavorable (1/n > 1). The values of K_F and 1/n can be calculated from the intercept and slope, respectively (Bell *et al*., 1998).

Results & Discussion

Effect of dosage adsorption of iron:

According to the results of removal (%) for the effect of doses on the adsorption of the iron. The results indicate that the 0.5 g dose of sea grass (active carbon) yielded the highest percentage (96%) of removal followed by dose of 0.1 g (70.80%). On the other hand, the lowest percentage value (17.85% and 26.96%) were recorded for doses of (0.3g and 0.4g), respectively. Table (1) and Figures (3.1 & 3.2).

Table 1: Effect of adsorbent doses on iron adsorption

Dose (g)	Absorbance	Final (C_e) concentration	Removal %	q_e (mg/g)
0.1	1.548	45.335	70.80	1.797
0.2	1.633	48.952	6.79	0.452
0.3	1.439	43.143	17.85	0.781
0.4	1.280	38.359	26.96	0.885
0.5	0.066	2.0119	96.15	2.585

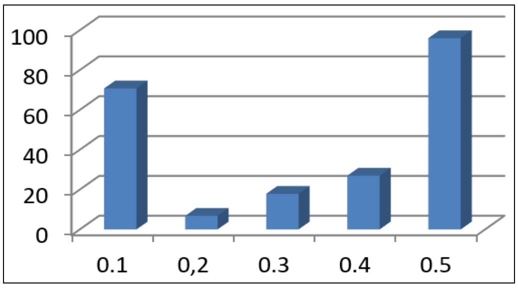

Fig 1: Removal percentage of iron by using various doses of adsorbent (iron)

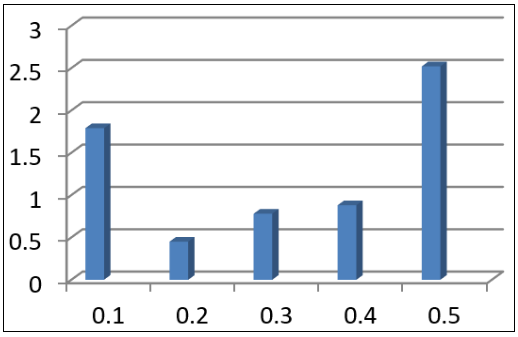

Fig 2: The relationships between qe and doses (iron)

Meanwhile the effect of adsorbent dosage on the adsorption of Copper, the results showed that, the higher effective dose is (0.1g) which recorded percentage of (82.14%), followed by the doses (0.2g and 0.25g) (69.04% and 70.23%), with relative decrease in removal percentages of doses (0.05g and 0.15g), Table (2) and Figures (3 & 4).

Table 2: Effect of adsorbent doses on copper adsorption

Dose (g)	Absorbance	Final concentration	Removal %	q_e (mg/g)
0.05	0.020	3.46	74.9	5.16
0.1	0.015	2.66	80.71	2.82
0.15	0.056	9.27	32.77	0.753
0.2	0.024	4.16	69.78	1.203
0.25	0.022	3.79	71.58	1

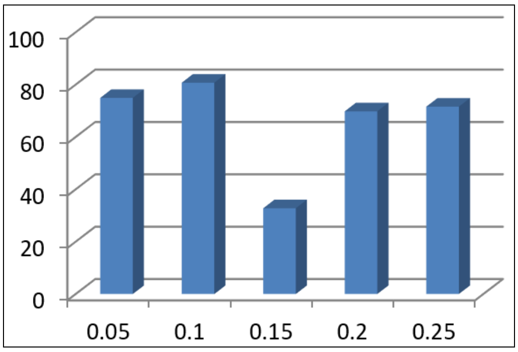

Fig 3: The relationships between Removal and doses (Copper)

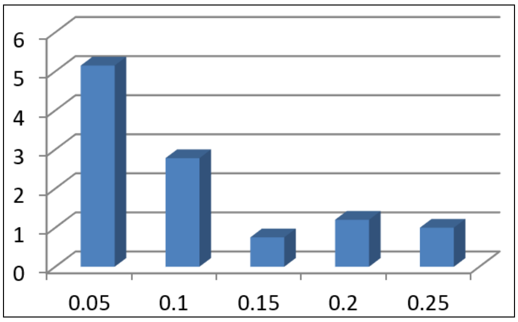

Fig 4: The relationships between q_e and doses (Copper)

On the other side the effect of adsorbent dosage on the adsorption of Nickel, the results showed that, the effect of dose is related to 0.4g and 0,5g (78.116% and 78.116%), its equal, so small dosage (0.4g), followed by doses of 0.2g (77.526%), with relative decrease in the doses 0.1g and 0.3g (74.468% and 70.215%). Table (3) and Figures (5 & 6).

Table 3: Effect of adsorbent doses on Nickel adsorption

Dose(g)	Absorbance	Final concentration	Removal%	q_e (mg/g)
0.1	0.039	0.995	74.468	1.451
0.2	0.029	0.876	77.526	0.755
0.3	0.053	1.161	70.215	0.456
0.4	0.027	0.853	78.116	0.380
0.5	0.027	0.853	78.116	0.304

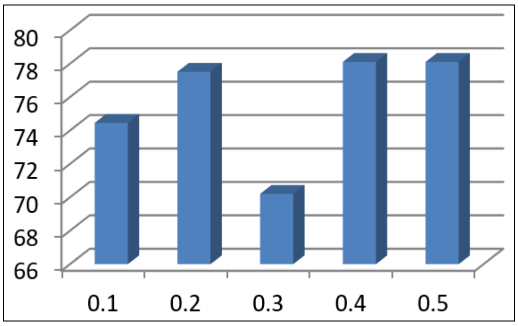

Fig 5: The relationships between Removal and doses for Nickel

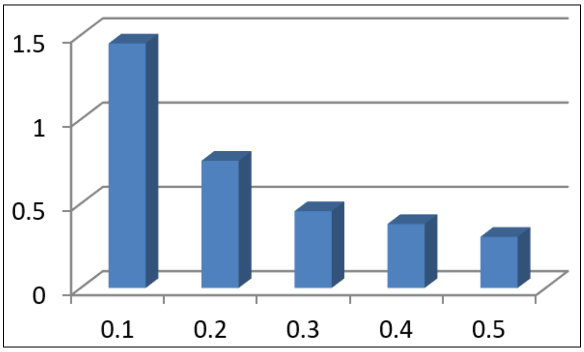

Fig 6: The relationships between q_e and doses for Nickel

Effect of time on the adsorption

There are effects of applied time (15 – 90 min) on the removal percentage and the effect time on the q_e. The removal values were ranged between (97.76 – 99.76 %), the high removal percent was obtained at 60 min (99.76 %), Table (4), Figures (7 & 8).

Effect of time on iron adsorption

Table 4: The effect of time on the removal percentage, q_e and concentration

Time (min)	absorbance	Final concentration	Removal %	q_e (mg /g)
15	0.038	1.173	97.76	2.56
30	0.031	0.964	98.16	2.57
45	0.004	0.155	99.70	2.61
60	0.003	0.125	99.76	1.3
75	0.008	0.275	99.47	2.61
90	0.01	0.335	99.36	2.6

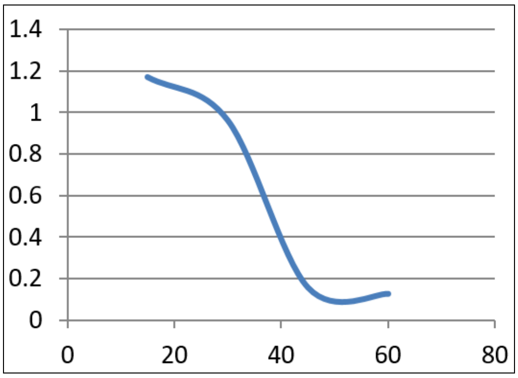

Fig 7: Relationship between time and concentration of iron

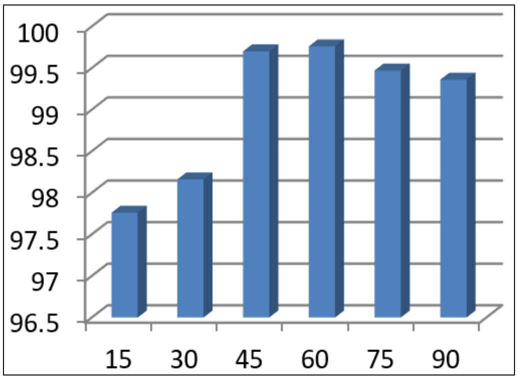

Fig 8: The effect of time on the removal percentage of iron

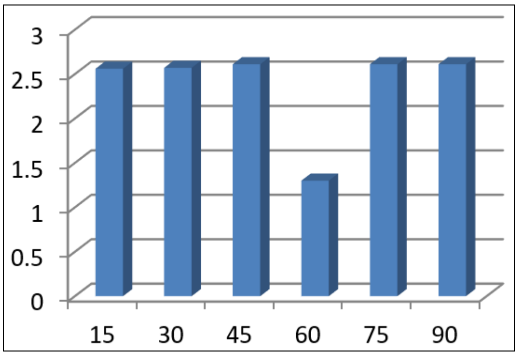

Fig 9: The effect of time on the q_e for iron

Effect the time on copper adsorption

The effect of time (15 - 75 min) on copper adsorbents showed moderate percentages value of (60.84 – 72.51 %), high percentage of removal was obtained ate 15 min then the adsorption process was shown equilibrium at the used time period of adsorption, Table (5) and Figures (3.10, 3.11 & 3.12).

Table 5: Effect the time on copper adsorption

Time (min)	Absorbance	Final concentration	Removal %	q_e (mg)
15	0.022	3.79	72.51	2.5
30	0.024	4.11	70.19	2.42
45	0.031	5.24	62	2.13
60	0.032	5.40	60.84	2.09
75	0.032	5.40	60.84	2.09

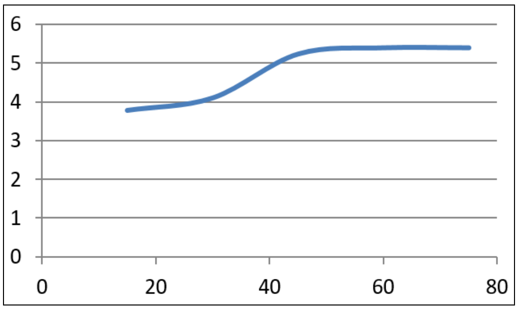

Fig 10: The effect of the time on concentration

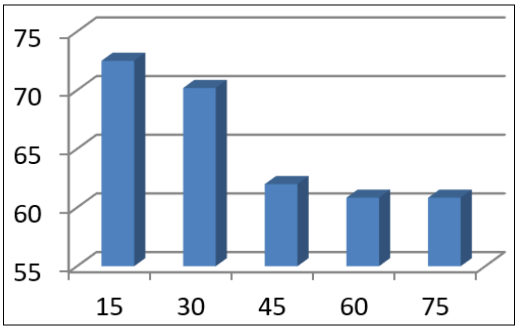

Fig 11: The effect of time on the removal percentage of copper

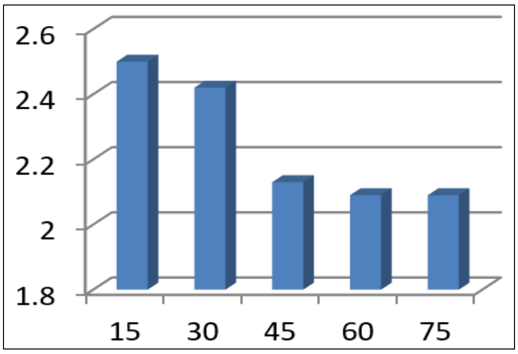

Fig 12: The relationship between time and q_e for copper

Effect the time on Nickel adsorption

The effect of time on the adsorption % showed that, after 60 min the high removal values of 76.65 % was obtained, Table (6) and Figures (3.13-3.14 & 3.15).

Table 6: Effect the time on Nickel adsorption

Time (min)	Absorbance	Final concentration	Removal %	Q_e (mg/g)
15	0.072	1.366	64.95	0.316
30	0.049	1.118	71.44	0.347
45	0.042	1.03	73.55	0.358
60	0.032	0.91	76.65	0.373

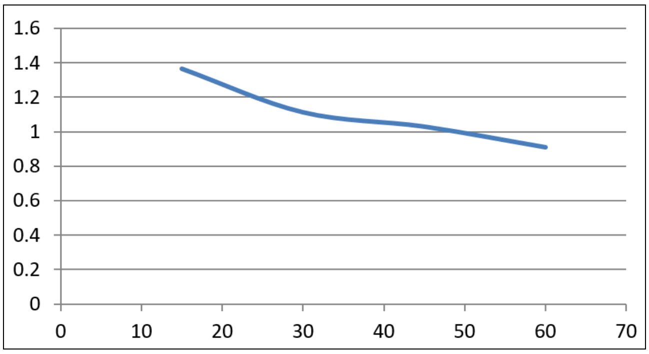

Fig 13: Effect the time on the concentration

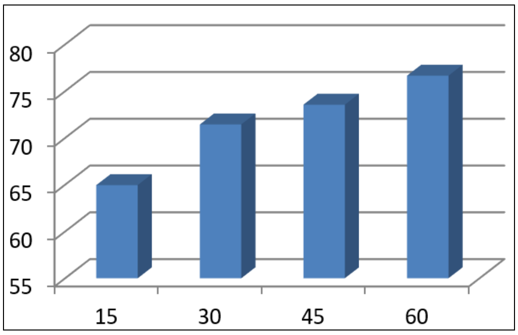

Fig 14: The effect of time on the removal percentage of Nickel

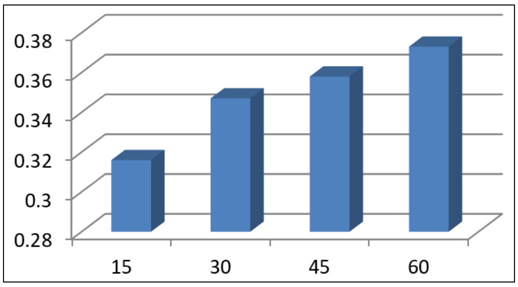

Fig 15: The relationship between time and q_e

Adsorption isotherms (Langmuir and Frindulish)

Langmuir isotherm

The Langmuir equation is the most frequently employed isotherm equation for modeling equilibrium. The linear form of the Langmuir equation is commonly expressed as follows:

$$\frac{Ce}{qe} = \frac{1}{kl} + \left(\frac{al}{kl}\right)Ce$$

A plot of *Ce* versus Ce/qe was linear showing the applicability of Langmuir adsorption isotherm for Alizarin red and Ruthenium red adsorption. (Mamdouh *et al.*,2016) [8].

K_L and a_L are the Langmuir constants related to adsorption capacity and rate of adsorption, respectively, which are calculated from slope and intercept of the plot *Ce* versus Ce/qe. The effect of various times of adsorbent on the adsorption of the studied dyes was monitored. A linear zed of C_e verus C_e/q_e was obtained as shown in Tables (7-9) and Figures (16 -18).

Langmuir isotherms of iron solutions

The effect of time on the adsorption of metal ion solutions selected in this study was recorded in Tables of (7 - 9)and Figures (16 -18).

Table 7: Effect of time (mins) on removal percentage of iron solutions

Time (min)	Final concentration	Removal %	q_e (mg /g)	Ce	Ce/Cq
15	1.173	97.76	2.56	1.173	0.4582
30	0.964	98.16	2.57	0.964	0.3750
45	0.755	99.70	2.61	0.755	0.3145
60	0.525	99.76	2.3	0.525	0.2282
75	0.475	99.47	2.1	0.475	0.2260
90	0.390	99.36	1.8	0.390	0.2160

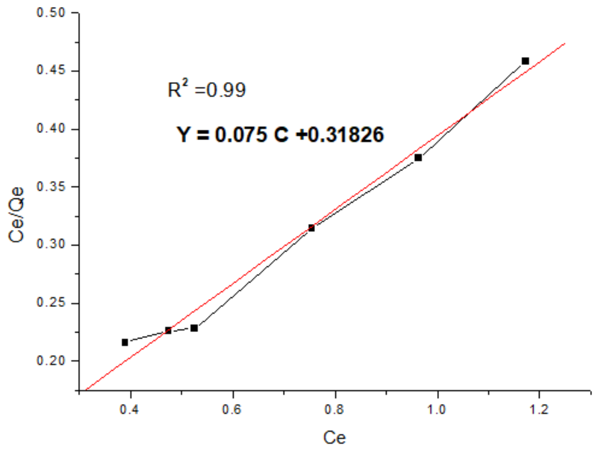

Fig 16: Relationship between Ce & Ce/Qe of iron

Langmuir isotherms of copper solutions

Table 8: Effect of time (mins) on removal percentage of copper solutions

Time (min)	Final concentration	Removal %	q_e (mg)	Ce	Ce/Cq
15	3.79	72.51	2.5	3.79	1.516
30	4.11	70.19	2.42	4.11	1.826
45	5.24	62	2.13	5.24	2.460
60	5.40	60.84	2.09	5.40	2.58
75	5.40	60.84	2.09	5.40	2.58

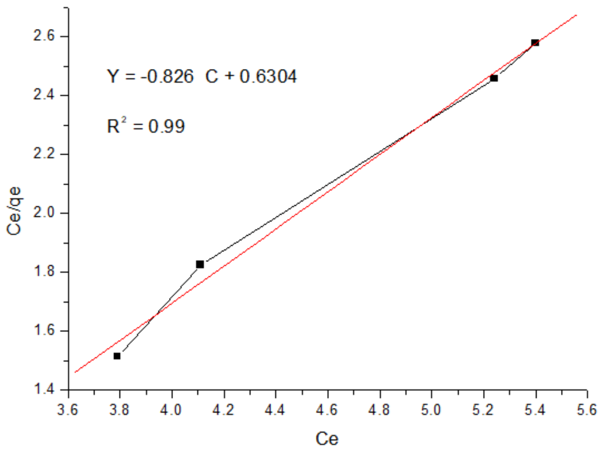

Fig 17: Relationship between Ce & Ce/Qe of copper

Langmuir isotherms of Nickel solutions

Table 9: Effect of time (mins) on removal percentage of Nickel solutions

Time (min)	Final concentration	Removal %	q_e (mg)	Ce	Ce/Cq
15	1.366	64.95	0.13	1.366	4.30
30	1.118	71.44	0.347	1.118	3.22
45	1.03	73.55	0.358	1.03	2.877
60	0.91	76.65	0.373	0.91	2.43

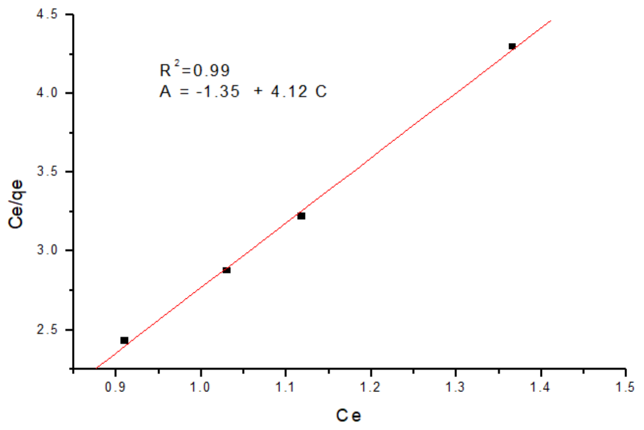

Fig 18: Relationship between Ce & Ce/Qe of Nickel

The excellent fit of the Langmuir model to the sorption data suggests its applicability to the system under investigation (Hanan *et al*., 2019) [7]**.** The Langmuir parameter values, along with their correlation coefficients, were calculated from the intercept and slope of the fitted Langmuir equation, as shown in Table 10.

Table 10: The Langmuir parameters for the adsorption of metal using sea grasses

Metal	aL	Kl	r^2
Fe	0.075	0.318	0.99
Cu	0.826	0.6304	0.99
Ni	4.2	-1.3	0.99

The high r2 values for both studied dyes suggest successful adsorption onto the seagrass adsorbent. The Langmuir isotherm's key characteristic, the dimensionless separation factor RL, can be expressed as follows:

$$\mathbf{RL} = \frac{1}{1 + al\,.Ci}$$

Where, Ci = initial dye concentration and al=Langmuir constant.

The types of equilibrium isotherms are related to the RL values for RL > 1 Unfavorable, RL =1 Linear, 0< RL < 1 Favorable, RL=0 Irreversible. The current research demonstrated that the RL values for the selected dyes and sea grasses were between 0 and 1, suggesting successful adsorption (Table 11). The results showed that the sea grasses activated carbon gave favorable results, where the RL values of selected metal solutions were ranged between (0 - 1), 0.930, 0.547 and 0.19 for iron, copper and Nickel metal solutions, respectively.

Table 11: The RL values of adsorption metals by active carbon of sea grasses

Metal	RL
Fe	0.930
Cu	0.547
Ni	0.19

Freundlich adsorption isotherm

The Freundlich isotherm model, a pioneering equation in adsorption studies, empirically describes non-ideal sorption processes involving heterogeneous surfaces. This model posits that an adsorbent's surface comprises diverse adsorption sites with varying adsorption potentials, each capable of adsorbing molecules independently**.**

Langmuir equation. It is given by the following nonlinear equation below:

$$\mathbf{q=KC}$$

KF, a system constant linked to bonding energy, can be defined as the adsorption or distribution coefficient. It quantifies the amount of dye adsorbed by the adsorbent per unit equilibrium concentration**.** The adsorption intensity of the dye onto the adsorbent, or surface heterogeneity, is inversely proportional to the value of 1/n. A more heterogeneous surface is indicated by a value closer to zero. A normal Freundlich isotherm is characterized by a 1/n value below 1, while a value above 1 signifies cooperative adsorption (Enas *et al*., 2019) [4].

The above equation can be linearized in the logarithmic form the following equation and the Freundlich constants can be determined:

$$log\ qe = log\ K_F + \frac{1}{n} logCe$$

A plot of log Ce versus log qe was linear, where kF is measure of adsorption capacity and (n) is adsorption intensity. 1/n values indicate the type of isotherm to be irreversible (1/n = 0), favorable (0 < 1/n < 1), unfavorable (1/n > 1). The values of 1/n and kF can be calculated from the slope and intercept respectively, Table (12).

Table 12: Freundlich isotherms for metals adsorption

Metal solution	1/N	KF
Iron	0.630	-0.826
Copper	1.419	-0.626
Nickel	1.42	0.440

The results of Freundlich of the present study were illustrated in Table (13 -15) and represented in Figures of (19 - 21).

Table 13: The values of freundlich isotherm of iron solutions

Time (min)	log C_e	Log q_e
15	0.0692	-0.3389
30	-0.0159	-0.4259
45	-0.1220	-0.5023
60	-0. 2798	-0.6416
75	-0.3233	-0.657
90	-0.856	-0.699

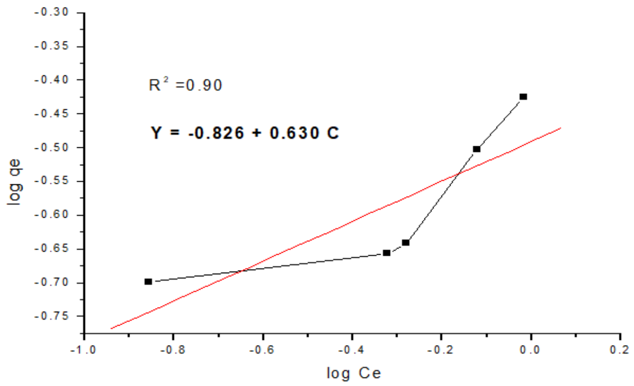

Fig 19: Freundlich adsorption isotherm for iron adsorption

Table 14: The values of Freundlich isotherm for copper solutions

Time (min)	log C_e	Log q_e
15	0.5786	0.1806
30	0.613	0.2615
45	0.7193	0.3909
60	0.7323	0.4116
75	0.7323	0.4116

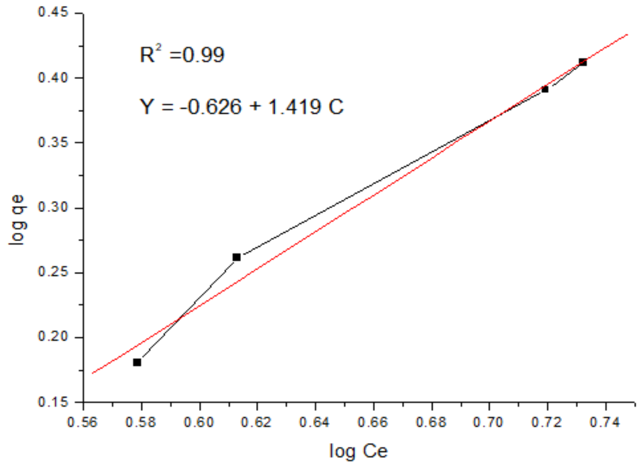

Fig 20: Freundlich adsorption isotherm for copper adsorption

Table 15: The values of Freundlich isotherm for Nickel solutions by sea grasses

Time (min)	log C_e	Log q_e
15	0.1354	0.6356
30	0.04844	0.5079
45	0.0128	0.4589
60	-0.0409	0.3856

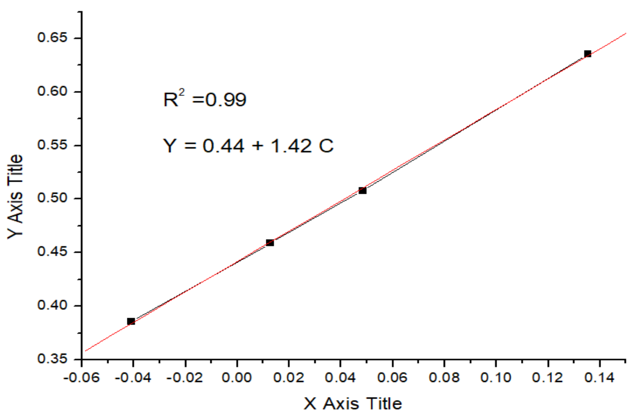

Fig 21: Freundlich adsorption isotherm for Nickel adsorption

According to Freundlish isotherms obtained in this study the seas grasses active carbon gave favorable adsorption for copper and iron metals.

Kinetics of adsorption

According to the values obtained from the isotherms in this study, for studied metals. The adsorption process follow the first order reaction. The Kinetics of the adsorption was conducted by the values which recorded according to the Effect of time on adsorption of metals. The values were shown in Figures (22 -24), and Tables (16 -18).

Table 16: Effect of time on iron adsorption

Time (min)	Concentration(ppm)
15	1.173
30	0.964
45	0.155
60	0.125
75	0.275
90	0.335

Table 17: Effect of time on copper adsorption

Time (min)	Concentration(ppm)
15	3.79
30	4.11
45	5.24
60	5.40
75	5.40
90	3.79

Table 18: Effect of time on Nickel adsorption

Time (min)	Concentration(ppm)
15	1.366
30	1.118
45	1.03
60	0.91

The adsorption rates are measured by determining the concentration of the metals as a function of time. (C_t is the concentration of dyes at different times) versus time (min),Tables (19-21) Figures (22 and 24). The adsorption rates were calculated from the slopes (slope = -K/2.303), (Enas *et al*., 2019) [4], the results were given in Table (19).

Table 19: The relationship between time(min) and log C_e of iron

Time (min)	Log Ce
15	0.0692
30	-0.0159
45	-0.1220
60	-0. 2798
75	-0.3233
90	-0.856

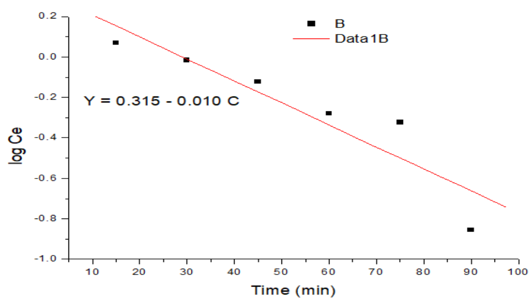

Fig 22: The relationship between Time(min) and Log C_e of iron

Table 20: The relationship between time(min) and log C_e copper solutions

Time (min)	Log Ce
15	0.5786
30	0.613
45	0.7193
60	0.7323
75	0.7323
90	0.5786

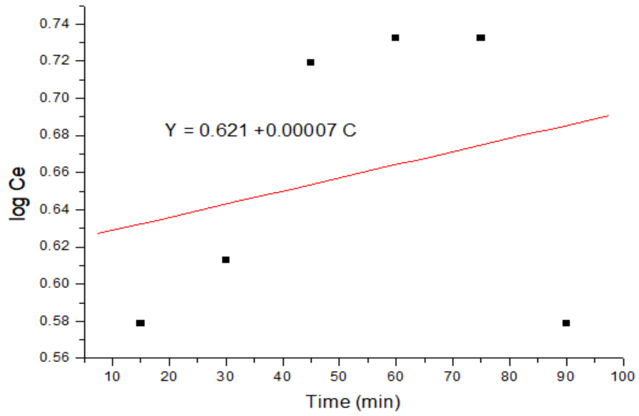

Fig 23: The relation between Time (min) and Log C_e. of copper

Table 21: The relationship between time(min) and log C_e Nickel

Time (min)	Log Ce
15	0.1354
30	0.04844
45	0.0128
60	- 0.0409

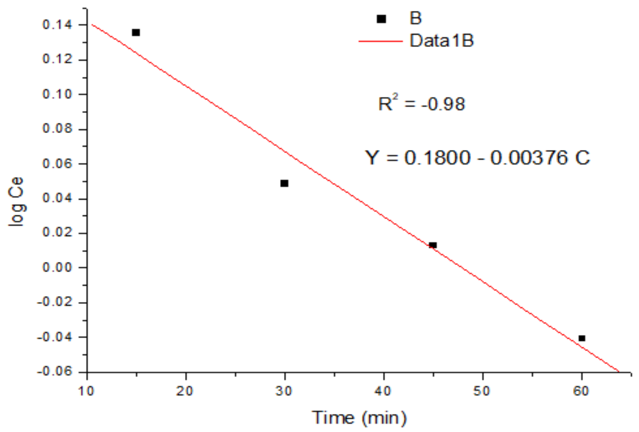

Fig 24: The relationship between time(min) and log C_e and Nickel

From the obtained the figures which showed relationship between time and concentrations, the order rate of adsorption of metals selected in this study by sea grasses activated carbon follows first order, Table (22).

Table 22: Adsorption rates of metal solutions activated carbon of sea grasses

Metal	Rate of reaction (K)
Fe	0.023
Cu	0.00016
Ni	0.0085

Conclusion

The aim of this research is evaluate the efficiency of low cost adsorbents of activated carbon obtained from sea grasses. The observations and conclusions can be summarizing as follows:Adsorption efficiency of metals onto activated carbon increase in contact time till reached equilibrium. Increase in adsorbent dose led to 98 % removal while decrease in adsorption capacity.

The adsorption isotherm studies showed that the best Langmuir isotherm was for adsorption of metals. However, the best Freundlich isotherm was obeyed for studied metals.

Recommendation

According to the results which obtained in this study, the sea grasses can be converted into activated carbon and used as a low cost and abundant materials for removal of some metals. This is give indication to use it for the treatment of some samples as water in field of economic and environmental.

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