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The Elimination of Chromium from Industrial Wastewater by Using Biochar and Zeolite

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ABSTRACT

This study investigates the efficiency of different materials, including, Zeolite (z), Biochar (B) and mix Zeolite/ Biochar (Z/B) for the remediation of industrial contaminated water with Cr. The experiments explore various factors that play an important role in the adsorption process, like initial metal ion concentration, adsorbent dosage, agitation time, and pH value. Results indicate that removal efficiency is higher at lower metal ion concentrations. The adsorption effectiveness of the material is the order: BZ > B > Z for Cr ions. The removal efficiency of BZ was 91% and 63.8%for Cr and increasing adsorbent dosage enhances adsorption percentage, with optimal removal observed at 1 g/L. The study suggests that BZ was the superior material for removing Cr ions, with optimal removal occurring at a mass of a sorbent 1 g/L and an optimum time of 120 minutes. Also, the pH of the solution influenced removal efficiency. with variations for Cr ion concentration Langmuir and Freundlich adsorption isotherms were applied, showing high correlation coefficients for Cr ion adsorption. Overall, the research emphasizes the potential of the tested material in alleviating Cr in contaminated water. Also, the BZ and B and Z exhibit superior performance in removing Cr ion from polluted water.

Keywords: Adsorption, polluted water, remediation, Zeolite, biochar, chromium, Langmuir and Freundlich isotherm

1. Introduction

Many reasons for water pollution as human activities are global nature, causing unpleasant consequences of poisoning, ecosystem disruption, diseases, and death of living different organisms. Due to toxic pollutants containing dyes, organic matter, and toxic heavy metals that are released into water bodies. Tannery wastewater is the largest pollutant among all industrial wastewater. Although it plays a significant contribution to the national economies of numerous countries, it has negative effects on the general environment of the country and high levels in addition, to inability to analyze pollutants in tannery wastewater poses significant environmental and health risks. (Ajala *et al*., 2022)

Chromium is considered one of the 16 most hazardous elements, and due to its carcinogenic and teratogenic characteristics, it poses a significant health risk. Chromium exists in two stable oxidation states of chromium, (III) and (VI). Chromium (III) is essential for human nutrition, especially in glucose metabolism. However, most chromium (VI) compounds are toxic and can lead to lung cancer (Khadija *et al.,* 2021).

Clay minerals are a kind of inorganic materials with specific structural, rheological, and thermal properties. It is naturally hydrophilic due to surface hydroxyl groups (-OH) that readily bind water molecules and have a significant history of binding metals. (Biswas *et al.,* 2020).

Natural zeolite can exchange cations like heavy metals are proplematic due to its ion interaction ability. The chemical composition of natural zeolite affects its cation exchange property, especially its selectivity as natural zeolite has a greater attraction for some heavy metals (Velarde *et al*., 2023)

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Biochar made from agricultural waste has become widely used and known as a versatile material for many purposes containing the complex carbon matrix that can be easily and inexpensively produced by pyrolysis of bio-waste under limited of oxygen- conditions, and is economically sustainable for use in applications environmental, especially in wastewater treatment (Bandara *et al*., 2020; Ashour and Tony (2020). This study showed that natural clay was applied, for Chromium (VI) was removed from the liquid through batch adsorption, and the adsorption efficiency of Cr(VI) removal reached 66% after 90 min equilibrium time. Chanda *et al.* (2022) used the synthesized zeolite for Chromium capture (VI) from solution and found that metal ions adsorption concentration (20–100 mgL^{-1}) for Cr (VI) sorbent dosage (2–10 gL^{-1}) for chromium and maximum adsorption capacity showed that chromium removal was 17% to 23% for a rise in dosing from 2gL^{-1} to 10 gL^{-1}as well as the greatest adsorption capacity of zeolite the sorption data was found to match with Langmuir isotherm, as monolayer adsorption. Biochar is used as a bio-adsorbent to remove toxic metals (Li *et al.,* 2020).

Hence, the purpose is to study the influence of the application biochar (B), Zeolite (Z) and a mix of zeolite and biochar (BZ) for the discarding of Chromium (Cr) from a liquid solution and implemented on an actual sample.

2. Material and Method

This study aims to investigate the application of Biochar, Zeolite, and mixed Zeolite/Biochar for chromium (Cr) removed from an aqueous solution; to achieve this purpose:- (i) study the effect of different materials dosage on the adsorption of Cr at fixed pH and fixed concentration, (ii) evaluate adsorption isotherms of Cr on different materials at fixed pH and fixed dosage for different concentrations of Cr, (iii) studies the effect of time "kinetics adsorption" of Cr at fixed pH and fixed dosage for different materials at different times and (iv) investigate the effect of pH envelops on adsorption of Cron the adsorbent materials.

2.1. Preparation Chromium-contaminated aqueous solution.

The standard solution concentration is 1000 mgL^{-1} (Merck KGaA, 64271 Darmstadt, Germany). This stock solution was carefully diluted to achieve the desired concentrations (10 to 50 mg L^{-1} Cr). This metal was used as pure salt in the form NO_3^-.

2.2. Adsorption experiment

The efficiency of Biochar (B) was evaluated through batch experiments, Zeolite (Z) and mix Zeolite /Biochar (BZ) to remove chromium (Cr) from aqueous solution experiments were conducted using four factors initial metal concentrations, adsorbent dosages, contact time and varying pH values of the solution. Change in absorbance with varying dosage (0.1, 0.2, 0.8, 1 and 2 gL^{-1}) of B, Z, and BZ, they were put into centrifuge tubes containing 50 ml of water contaminant solutions with various Cr ion concentrations, i.e. 10, 20, 30,40 and 50 mgL^{-1} and reaction tube. Cr was introduced to the reaction tube to provide concentrations of Cr50 mgL^{-1}, the total volume of 50 mL containing as well as different contact times (0, 10, 40, 60, 120 and 240) and in this study pH value of 2, 3, 5, 8 and 10 adjusted to 2 or 10 using 0.1 M HCl and 0.1 M NaOH solutions which experiments at (25^{o}C). Next, the samples were shaken carefully. Following batch experiments, each sample was shaken for 3 h and put it in centrifuged at 3000 rpm for half an hour, filtered by supernatants. After shaking samples were collected and the concentrations of the investigated metal ions were determined concentration in the solution using ion-coupled plasma ICPA. The quantity of metal ions adsorbed/ unit mass of the adsorbent is assessed by using the following mass balance equation,

$$q_e = (C_o\text{-}C_e)\ V/M$$

$$\text{Removal (\%)} = C_o - C_e/C_o \times 100$$

Where q_e is the equilibrium adsorption, capacity (mg g^{-1}), C_o and C_e are the initial and equilibrium liquid-phase concentrations of solute in aqueous solution (mgL^{-1}), respectively. V is the volume of liquid phase e, (L) and M is the mass of the sorbent used (g). Adsorption is a process of mass transfer where a substance moves from the liquid state and accumulates on the surface of a solid and becomes bound by using physical and/or chemical interactions.

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2.3. Adsorption Isotherms

Various isotherm models are employed for modeling the equilibrium of adsorption systems. The most models used are (The Langmuir model and the Freundlich model) to describe the mineral adsorption process. The Langmuir model supposed that the adsorption process exhibits at special homogenous adsorbent sites. Also, it is used in many monolayer adsorption processes successfully, while the Freundlich model has the ability to conduct with non–ideal sorption on irregular surfaces and multilayer sorption. Adsorption isotherm studies were carried out in duplicate, with different initial Cr concentrations (from 10 mgL^{-1} to 50 mgL^{-1}) on different adsorbent dosages (0. 1, 0. 2, 0. 4, 0. 8, 1 and 2 g) at a constant pH

2.4. Application of adsorption isotherms equations:

Langmuir adsorption isotherms: - The Langmuir equation was used to interpret the equilibrium adsorption data **(Langmuir, 1918)** the linear form of the Langmuir equation is:

$C/q= 1/ab+ C/b$ ………………………………………………………………………………... (1)

Where:**C**: concentration of Cr in equilibrium solution ($\mu g\ mL^{-1}$), **q**: the amount of Cr adsorbed by clay minerals ($\mu g\ g^{-1}$), **a**: a constant related to the binding energy of the adsorbent with Cr and **b**: Cr adsorption maximum (µg/g).

Equation of Freundlich: Adsorption isotherm in many dilute solutions is formulated by Freundlich, (1926).

as: $X/m= KC^{1/n}$ ……………………………………………………………………………...(2)

Where: **X** = amount of material adsorbed, **m** = amount of adsorbent, **C** = concentration of the equilibrium solution, and **K, n**= are constants. The equation of Freundlich has no theoretical foundation and is empirical. The curve according to Eq. (2) is usually parabolic and exhibits the following characteristic features: There is no single point indicating that the process is completed. There is no region of discontinuity by taking the Logarithms, Eq. (2) changes into

$\log q = \log k + 1/n \log C$ ……………………………………………………………………….. (3)

The log equation gives a ‘straight line Log k is the intercept and 1/n represents the slope or the regression coefficient.

2.5. Analysis methods: -

The pH in the reaction tubes was adjusted using the pH meter model (WTW Series pH 720) according to Estefan *et al.* (2013) the pH was adjusted by using HCl or NaOH. A rotary shaker (GFL 3005 Orbital Shaker) was used to mix the samples. and the centrifuged at 3000 rpm for 30 min; (Beckman Allegra 6R Refrigerated Benchtop Centrifuge), magnetic stirrer (Nouva USA). The prepared samples were filtrated by Nylon Syringe Filter (Diameter was 25 mm and the Pore Size was 0.22 µm), and restored in the refrigerator for analysis. Chromium in aqueous solution was determined using inductively coupled plasma (ICP) Spectrometry (model Ultima 2 JY Plasma) according to "The Environmental Protection Agency, (EPA1991).

2.6. Real Samples of contaminated waters with heavy metals

Real polluted wastewater samples from the Harby tannery factory in Rubiki (Badr City), by using auto-samples in more than one polyethylene container of capacity one liter, these containers were washed first with tap water and then distilled water than by sample before putting the sample in it.

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Table 1: Content of chemical properties and heavy metal tannery in wastewater

Parameter				Value			
EC				1.42			
pH				7.8			
Soluble cations and anions							
Na^+	K^+	Ca^{++}	Mg^+	Cl^-	SO_4^{--}	CO_3^{--}	HCO_3^{--}
6.4	0.62	3.4	2.5	8.23	4.27	----	0.42
Heavy metals							
Cd	**Pb**	**Ni**	**Cr**	**Fe**	**Co**	**Zn**	**Cu**
0.023	0.047	0.027	0.89	0.045	0.043	0.015	0.063

3.Result and Discussions

3.1. The effect of the initial metal concentration of synthetic Chromium on its removal by different materials

The experiment was carried out to different metal concentrations from 10 to 50 mgL^{-1} and a shaking time of 240 min, the adsorbent doses were 0. 1 mgL^{-1} of materials Zeolite clay mineral (Z), Biochar (B) and mixed Zeolite / Biochar (BZ). The effect of initial Cr concentrations in the solution on adsorption is shown in Table (2). The amount of Cr removed from the initial metal concentration of 10 mgL^{-1} to 50 mgL^{-1} at the equilibrium time resulted shown the removal efficiency percentage at low concentration of Cr being more than at high concentration which became (59.400 to 49.936, 71.00 to 59.294 and 91.0 to 63.8) % respectively, by using, Z, B, and BZ from above results could be interpreted due to the swelling of clay minerals into several times of its original volume and disband in sol rise to so large surface area as well as hence ameliorates capacity of adsorb large amounts of metals and explained the reality at depressed concentrations, which taken adsorption positions more fast and then boost of concentration led to saturation of sorbent surface. From the above results the effect of adsorption by clay minerals on the adsorption of Cr can be arranged as follows: - BZ>B> Z a similar result showed to Asanu *et al.* (2022). the adsorption capacity of natural zeolite and it was found that adsorption dose 2 gL^{-1}, contact time 75 minutes, initial Cr (VI) concentration = 10 mgL^{-1}, and solution pH 1:5 are the optimal conditions to achieve 93.57% Cr (VI) which confirmed that zeolite is an economically and promising material for removing Cr (VI) from liquid solution.

3.2. Adsorption isotherm of Cr on clay minerals.

3.2.1. Langmuir adsorption isotherm.

The Langmuir isotherms obtained by plotting the 1/qe versus $1/C_e$ values and correlation at Fig. (1) coefficient R^2 were (0.93and 0.95), (C_e/q_e= 0.0151C_e+0.6027 and C_e/q_e=0.0209 C_e+ 0.0115) found to be for added synthetic Cr solution to Z, and BZ materials respectively, the results proved that Z, BZ, respectively favorable for adsorption of Cr under the experimental condition used, but correlation coefficients R^2 were (0.80) (C_e/q_e= 0.0148C_e+ 0.2485) synthetic Cr solution by using B for Cr in the concentration range studied**.** These results revealed biochar was not favorable for the adsorption of Cr under the experimental condition used.

3.2.2. Freundlich adsorption isotherm.

The Freundlich isotherm is obtained by plotting ln q_e versus ln C_e values in Fig. (1) Cr ions The R^2 was found (0.99, 0.95 and 0.93) to be indicating that the Freundlich model was able to for Cr by, zeolite, biocar, and BZ respectively and the equations ln qe=0.2891+ 0.8032ln Ce, ln qe = 0.592+ 0.6788ln Ce, ln qe = 0.9606+ 0.3823ln Ce and The values K_f and n were found to be (0.302, 0.139, 0. and 0.89) and (1.5, 1.37, and 1.22) for Cr ion. Thus, the Freundlich equation is the relationship that describes the adsorption phenomena and is useful as a means of data description due to the isotherm assumes that adsorbent surface sites have a spectrum of different sites having different binding energies and can adequately describe the relationship between all different materials.

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Table 2: Impact of initial Cr concentration on removal efficiency of Cr removal capacity, mg g^{-1} by clay minerals.

Adsorption materials	C_0	C_e	q_e	Ce/qe	ln q_e	ln C_e	Removal %
	10	4.060	5.940	0.684	0.774	0.609	59.400
	20	8.775	11.225	0.782	1.050	0.943	56.125
Zeolite	30	13.943	16.057	0.868	1.206	1.144	53.523
	40	18.687	21.313	0.877	1.329	1.272	53.283
	50	25.032	24.968	1.003	1.397	1.398	49.936
Langmuir isothermal	$R^{2=}$ 9378 Ce/qe= 0.0151Ce+0.6027						
Freundlich isotherm	R^2=0. 9976 ln q_e = 0.2891+ 0.8032ln C_e k_f=-0.302 n=1.50						
	10	2.900	7.100	0.408	0.851	0.462	71.000
	20	5.300	14.700	0.361	1.167	0.724	73.500
Bichar	30	10.630	19.370	0.549	1.287	1.027	64.567
	40	15.717	24.283	0.647	1.385	1.196	60.708
	50	20.353	29.647	0.687	1.472	1.309	59.294
Langmuir isothermal	$R^{2=}$0.8066 Ce/qe= 0.0148Ce+ 0.2485						
Freundlich isotherm	$R^{2=}$0.9554 ln q_e = 0.592+ 0.6788ln $_{Ce}$ k_f=-0.139 n=1.37						
	10	0.900	9.100	0.099	0.959	-0.046	91.000
	20	4.300	15.700	0.274	1.196	0.633	78.500
Mix Zeolite/biochar	30	11.200	18.800	0.596	1.274	1.049	62.667
	40	14.000	26.000	0.538	1.415	1.146	65.000
	50	18.100	31.900	0.567	1.504	1.258	63.800
Langmuir isothermal	R^2=0.6768 C_e/q_e= 0.0202C_e+ 0.0043						
Freundlich isotherm	R^2=0.9319 ln q_e = 0.9606+ 0.3823ln Ce k_f=-0.089 n=1.22						

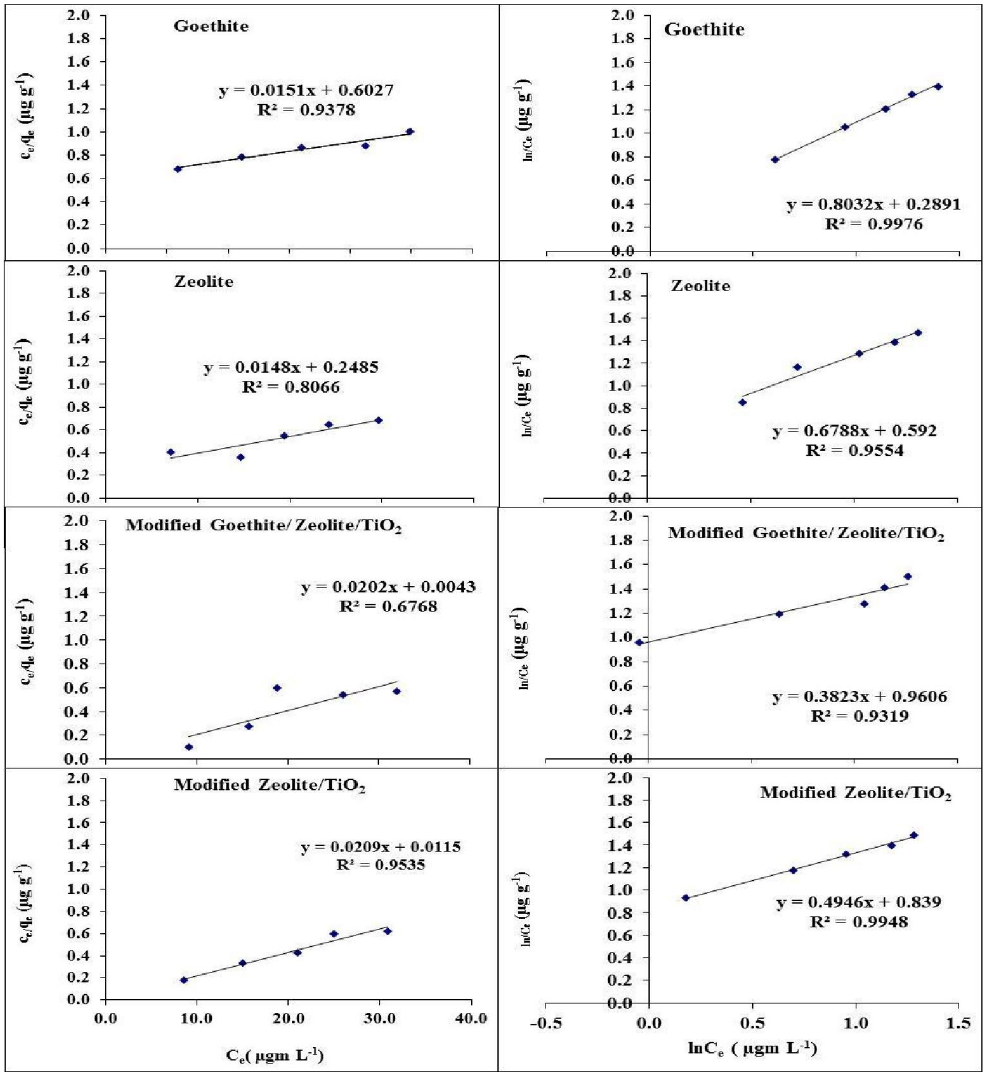

Fig. 1: Adsorption isotherms (Langmuir - Freundlich) for Cr

3.3. Effect of dosage on efficiency removal percentage Cr from synthetic solution.

As shown in Figure (2) the Cr adsorption is greatly influenced by the adsorption dose of the material used. The data revealed that the adsorption elimination efficiency of Cr was higher 57, 76 and 91.3 % at 1 gL^{-1} while the lowest removal efficiency of 25, 54, and 65 %was recorded at 0.1 gL^{-1} with Cr by using, zeolite, biochar and BZ respectively; thus, the amount of was selected as 1 gL^{-1} for further experiments. Generally, the removal efficiency of Cr by clay minerals can be arranged as follows: BZ> B > Z for Cr ion was most effectively removed at sorbent weight 1 gL^{-1} and their removal increased as the weight of sorbent increased. Metal elimination efficiency increased with increasing clay minerals content. The rise of % elimination is debit to the increase of the ready sorption surface and adsorption sites. At the same time, the adsorption capacity per unity mass of clay minerals reduces considerably with a boost in sorbent dose for Cr ion, these results harmonically with Chanda *et al.* (2022)

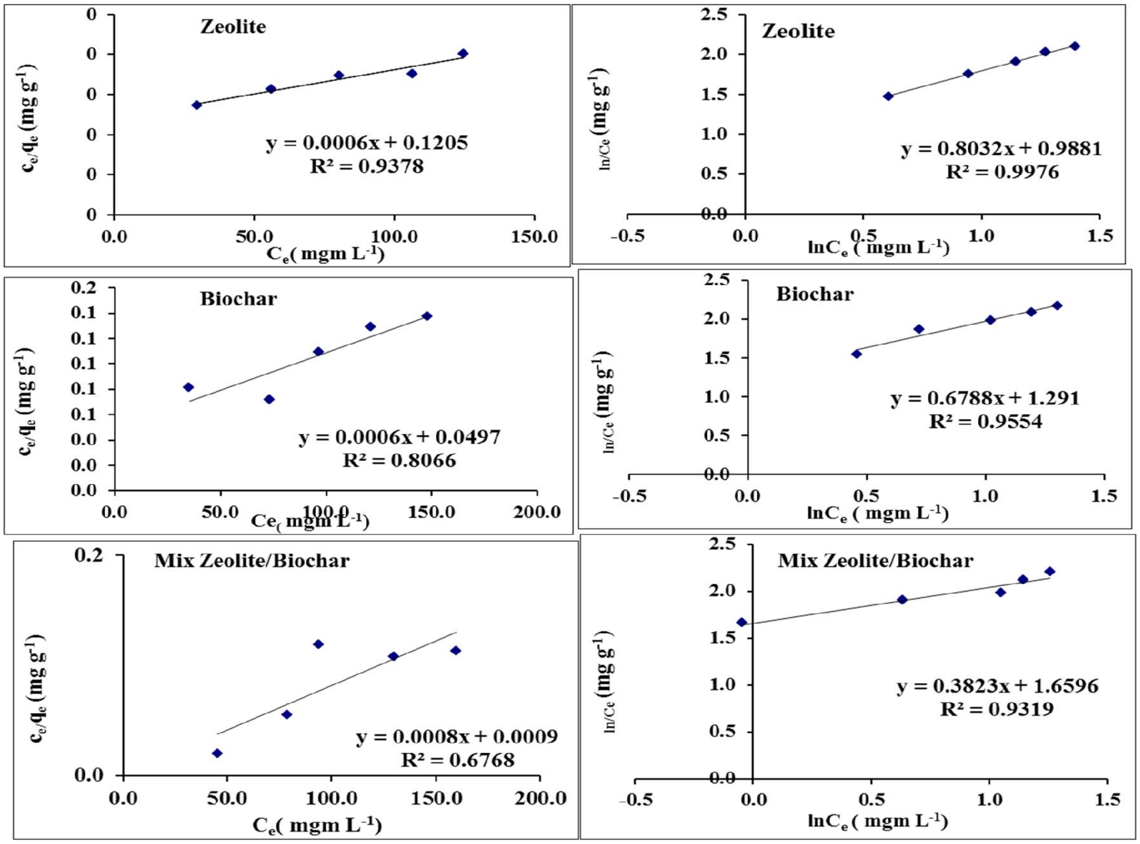

Fig. 2: Adsorption isotherms (Langmuir - Freundlich) for CrKinetics of Cr adsorption.

3.4. Impact of time contact on Cr removal efficiency of artificial sol by different materials.

The excitation time also plays a critical factor that influences the adsorption efficiency. Samples were taken at different times between 0 to 240 min and the remaining concentrations were 10 mgL^{-1} with 1 gL^{-1} of adsorbent different materials for Cr ions to evaluate the excitation time impact on adsorption efficiency. Fig. (3) Observed that the elevated adsorption efficiency of Cr was found at 120 min. However, the efficiency of adsorption was not remarkably decreased at excitation times lower than 120 min by using clay minerals, further, the adsorption rate was slow motion leading smooth and continuous saturation curve and the start of rapid adsorption may be due to an increase in the number of unoccupied sites ready at the premier phase. Then, low adsorption would follow as the ready adsorption site progressively reduces. At all, when adsorption includes adsorption surface and the time at which no adsorption takes place it has been fixed as equilibrium time, which was used for further experiments. Figure (3) indicates high efficiency for Cr adsorption can be obtained by different materials determined at 120 min where (36.1, 45, and 42) % of Cr removal efficiency is obtained, on the other side, the efficiency of adsorption at the end of 240 min were decreased the values were , 36, 44.5 and 41%)) regard to use zeolite, BZ, and B respectively. These results agree with Ashour and Tony (2020)

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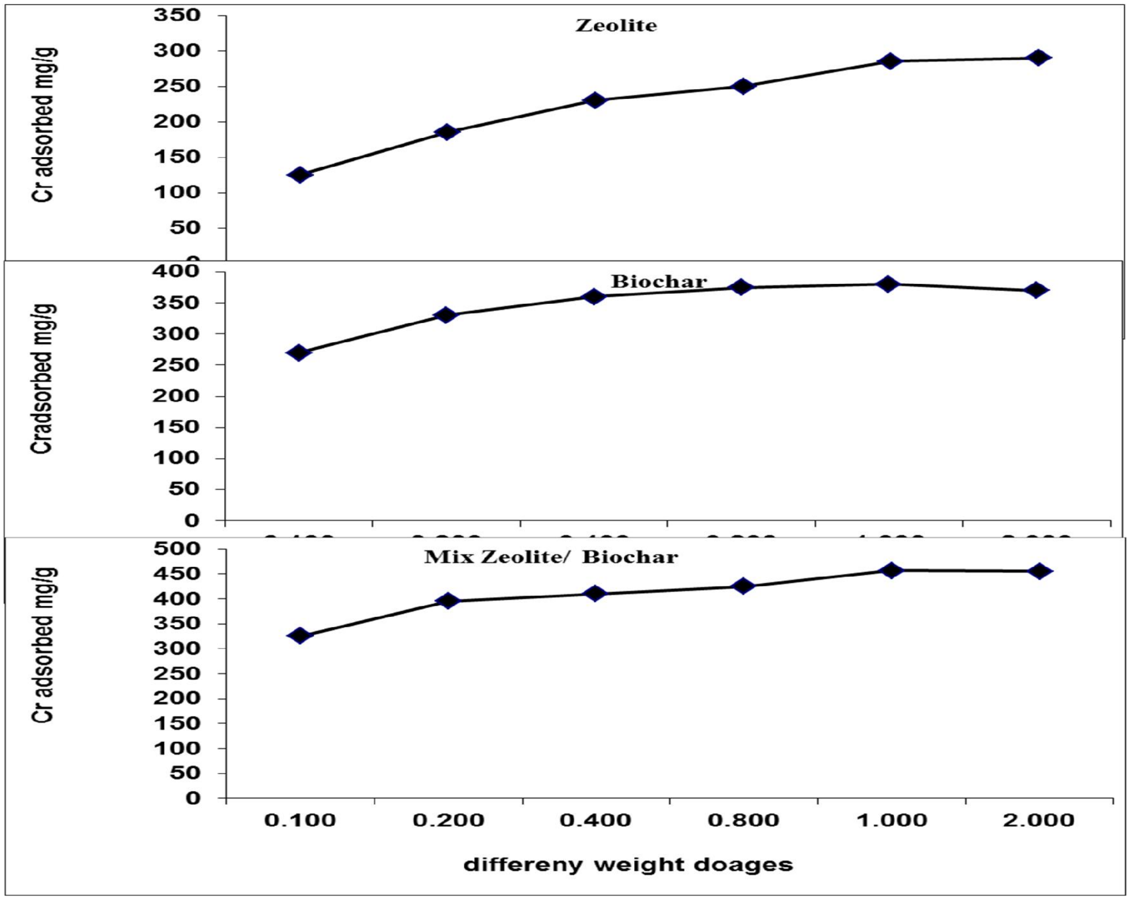

Fig.3: Impact of different doses materials on Cr ion efficiency removal.

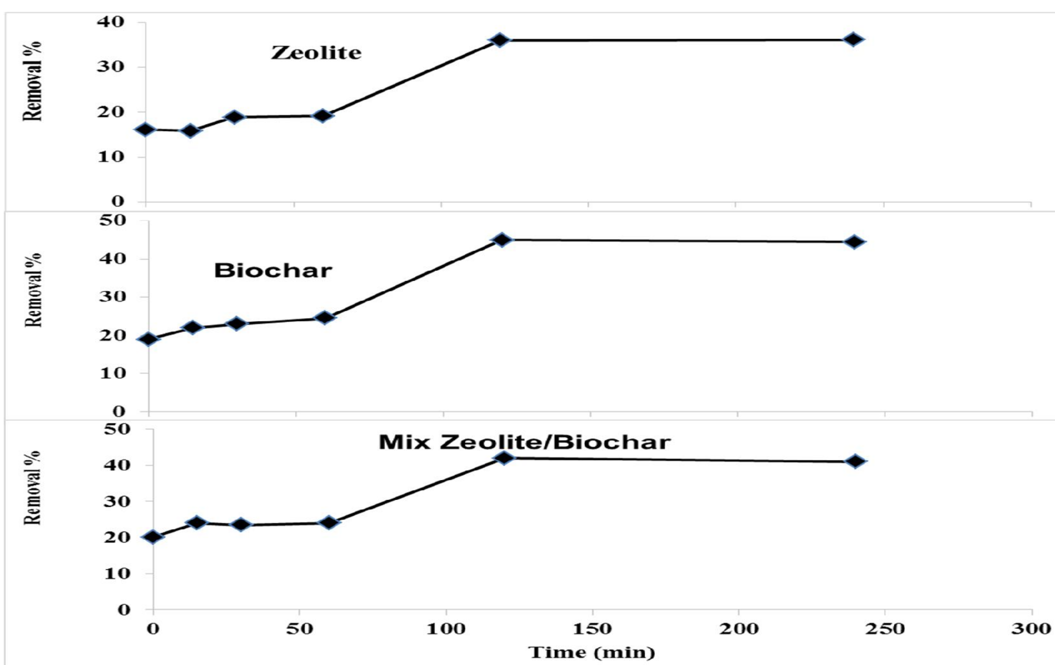

Fig.4: Impact of contact time on Cr removal by using different materials.

3.4. Influence of pH on Cr adsorption:

pH of the aqueous phase is strongly effective on the adsorption process as it impacts on the quantity and intensity of the surface charge of the adsorbent, the ionization degree and the speciation of the metal in the solution.

In the present study, five different pH values (2, 3, 5, 8, and 10) were used to study its contribution to Cr removal's efficiency, and the data is depicted in Fig (4).

Removal efficiencies at pH 3.5 and 7.8 of the zeolite were 59% and 20%. Meanwhile, at pH 4.6 and 7.4 biochar was 76% and 27%. On the other hand, adsorption efficiency at pH 3 and 6.3 were 91.80% and 47 % for Cr with BZ where the capacity of adsorption increases with decreasing value of pH. The sorption effect is high at decreasing pH since predominant chromium types at most in monovalent $HCrO_4^-$ formula, which is then gradually converted to divalent CrO_4^{2-} and $Cr_2O_7^{2-}$ as pH increases. The free energy of $HCrO4^-$ sorption is smaller than that ofCrO_4^{2-} and $Cr_2O_7^{2-}$, and then, $HCrO_4^-$is widely more easily sorbed than CrO_4^{2-} and $Cr_2O_7^{2-}$at the same concentration. With increasing pH, the composite materials surface turns out to be more and more deprotonated then the positive charge quantity of the surface is highly decreased, so that the ability of Chromium decreases. Thus, the amounts of chromium sorption at low pH are greater than that at high pH Nasanjargal el.al., (2021). This is mostly due to the pH-dependent speciation of the Cr ion and the charge of surface on the adsorbent.

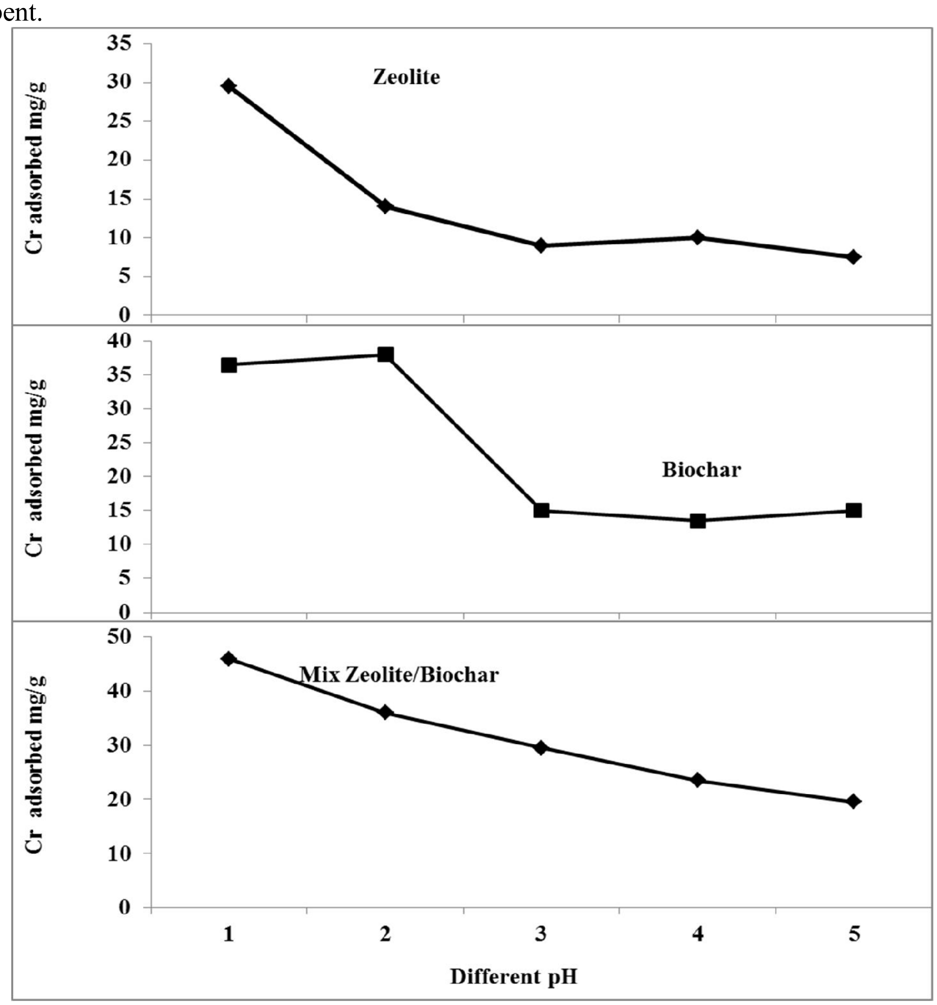

Fig.5: Impact of Adsorption envelopes of Cr by using different materials.

3.5. Treatment of natural polluted water

As shown in Table (2), all the tested heavy metals in the industrial water contaminated sample Cd, Pb, Ni, Cr, Fe, Co, Zn, and Cu were below the threshold level of their corresponding values, Meanwhile, Cr was above the limit > 0.1 ppm (FAO, 1995), consequently it is pose hazards for food cycle. The adsorption isotherm study of the industrial Cr -contaminated water was carried out based upon the previous pre-adsorption experiment results, it was evaluated at equilibrium time (120 min), the adsorbent weight material was (1gm), and the pH of the solution wasn't exceeded than 4.

The results (Fig 6) showed that the highest removal efficiency values were (63, 72 and 85) % respectively for (Zeolite, Biochar and mix Zeolite\Biochar) from the previous results, it can be noted that the removal of chromium ions by different materials from naturally polluted water can be arranged as follows: mix Zeolite\Biochar>Biochar>Zeolite.

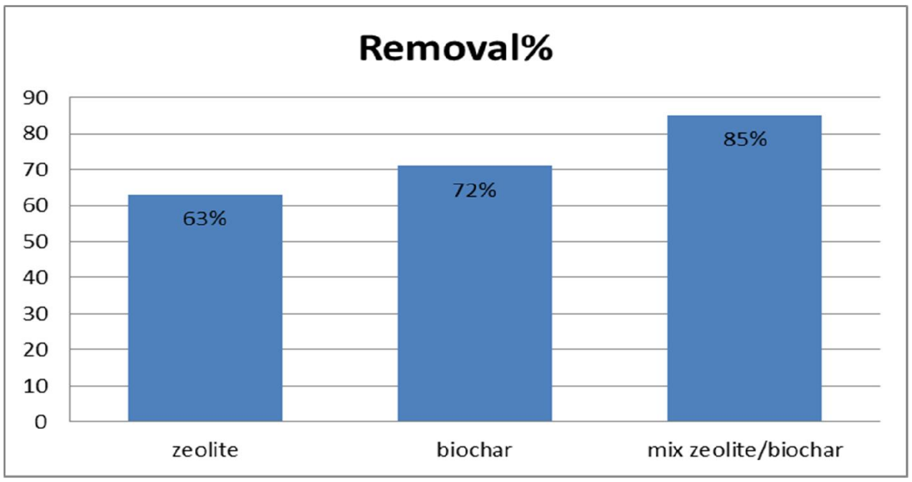

Fig. 6: Impact of different adsorbent materials on wastewater for removal efficiency

4. Conclusion

Previous data showed that using all natural materials under study had a significant effect on chemical pollutants concentration of tannery wastewater.

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