SODIUM DICLOFENAC ADSORPTION IN AQUEOUS MEDIA BY GRAPHENE OXIDE

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Graphene oxide was synthesized by the Hummers method and characterized by X-ray diffraction, scanning electron microscopy, Fourier transform infrared and ultraviolet-visible spectroscopies'. The obtained material was used as an adsorbent to remove sodium diclofenac from aqueous media. The influence of process parameters (pH, sodium diclofenac concentration, contact duration and adsorbent dose) on graphene oxide efficiency was investigated. A comparative study of the applicability of the Langmuir and Freundlich adsorption models for the description of experimental sodium diclofenac adsorption isotherms was carried out. It was shown that the Freundlich isotherm reflects the adsorption of sodium diclofenac on the graphene oxide from aqueous solutions comparatively better than the Langmuir isotherm.

Keywords: *Graphene oxide, adsorbent, water purification, sodium diclofenak removal, material characterization.*

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Introduction

The presence of various inorganic and organic wastes in the environment poses a threat to both the environment and public health due to long-term consequences associated with their solubility in water, resistance to microorganisms and bioaccumulation in the food chain [1]. In this regard, to the main sources of organic wastes distribution can be included pharmaceuticals widely used as medicines [2]. To date, there are many studies reporting the distribution of pharmaceutical residues in rivers, drinking water, hospital effluents, ground waters and sewage [3]. The main sources of spreading pharmaceuticals to the environment include administration of various medicines in human or animal therapy [4]. Hospital effluents are complex mixtures of compounds, among which a great number of pharmaceuticals and their metabolites can be found [5]. Pharmaceuticals can also reach the groundwater through leaching in agricultural areas, leaking of sewer lines and landfill leachates [6]. In addition, human urine and feces that make their way through municipal or sewage wastewater are considered to be a significant source [6]. When surface water becomes contaminated with these waste compounds, aquatic life can be harmed, even by trace amounts.

The main categories of pharmaceuticals detected in water are antibiotics, analgesics, muscle relaxants, and hormones. In this regard, diclofenac (DCF), a non-steroidal anti-inflammatory drug, is a particularly hazardous water pollutant that can have serious adverse effects on the environment and human health [7, 8]. DCF and its sodium salt are used in human medical care as an analgesic compound in case of inflammatory and painful diseases of rheumatism and arthritis [9, 10]. The continual intake of even little amounts of diclofenac in human body shows several unfavorable effects such as cytotoxicity to liver, kidney and gill cells, renal lesions, platelet dysfunction, convolution, as well as tissue damages [11, 12]. Accordingly, DCF is on the “watch list” of European Directive 2013/39/EU and Commission Decision (EU) 2015/495 [13, 14, 15] regarding priority substances in the water policy, meaning that it should be monitored based on the risk associated with the aquatic environment.

A number of trapping and degradation methods have been developed to remove diclofenac from aqueous solution. Conventional methods for DCF removal consist of ion exchange and adsorption [16]. The recent methods include ozonation, membrane filtration, advanced oxidation processes, electrochemical oxidation, photo catalysis and soil aquifer treatment [17-22]. These methods possess their own advantages and disadvantages in terms of cost, applicability, efficiency, simplicity, toxic byproducts, operability, environmental effect, and other factors [23]. Adsorption is proved to be more efficient and the most economical treatment method for the removal of pharmaceutical contaminants from wastewater effluents [24-28].

At the adsorption processes, one of the significant parameters affecting adsorption efficiency is adsorbent type and structure. Carbonaceous adsorbents are a dominant choice as adsorbent in case of the pharmaceuticals removal from water because of their abundant pore structure and great potential in the removal of pollutants from aqueous solutions. A wide variety of natural sources: olive stones [29], palm shell [30], sawdust [31, 32], walnut wood [33], coconut husk [34], rice husk [35], potato peels [36], etc. can be processed to obtain activated carbon.

In recent years, the unique properties of graphene, carbon nanotubes, carbon nitride, and graphene oxides—new forms of carbon nanomaterials—have revolutionized various fields of nanotechnology: electronics, optics, medicine, chemical synthesis, and catalysis. These materials also have generated significant attraction for the removal of inorganic pollutants, organic wastes, pharmaceuticals etc. from aqueous media. It was shown that Single-Walled Carbon Nanotubes have more efficiency for the removal of pharmaceuticals than Multi-Walled Carbon Nano Tubes [37, 38, 39]. This is probably because the exposed surface area is larger in Single-Walled Carbon Nanotubes and the pores are more attainable. Graphene oxide (GO), with its abundant functional groups such as carboxyl (-COOH), hydroxyl (-OH), and epoxy (-C-O-C-) groups on its surface, has recently been considered a promising adsorbent. Only two studies used graphene oxide as an adsorbent for diclofenak removal. As it was noted in [40], pure diclofenak sorption reached equilibrium within 24 hours and at this point diclofenak removal was 50%. After sonication of the graphene oxide solution, the maximum diclofenak removal reached 75%. In the second study [41], the adsorption results of sodium diclofenak showed that the maximum adsorption capacity was 128.74 mg g^{-1} at 25°C, and the removal percentage was 74% in 300 minutes.

Modern materials science also has every reason to consider graphitic carbon nitride as a promising universal "green" adsorbent [42]. The porous microstructure of graphitic carbon nitride and various types of interactions that can occur on its surface provide the possibility of highly effective adsorption processes by a complex mechanism, including pore filling, surface complexation, acid-base interaction, formation of hydrogen bonds, π-π conjugation, electrostatic attraction, and hydrophobic effects.

The search carbon nitride as adsorbents has been intensively conducted by us over the last years. The efficiency of exfoliated graphitic carbon nitride in the adsorption of Fe^{2+}, Fe^{3+}, and Cu^{2+} ions was studied [43, 44]. Carbon nitride-based magnetic nanocomposites Fe_3O_4/g-C_3N_4 and $CuFe_2O_4/C_3N_4$ have been proposed as effective, environmentally friendly adsorbents for the treatment of aqueous solutions contaminated with phosphates [45, 46]. It is known that exfoliated graphitic carbon nitride also acts as an effective photocatalyst for degrading pharmaceutical pollutants, including diclofenac, under UV-visible light [47].

We are currently studying the adsorption efficiency of carbon nitride, GO, and their hybrid composites for the removal of pharmaceuticals from wastewater. In this work, given the significant discrepancies in the results of the above studies [40, 41], we re-evaluated the suitability of GO for the adsorption of diclofenac sodium (SDCF) from aqueous media. The factors, affecting adsorption performance, such as solution pH, contact time, initial adsorbate concentration, and adsorbent dosage were analyzed with data obtained from batch experiments. The experimental data were evaluated by known isotherm models to understand the adsorption mechanism.

Experimental part

Materials

Sodium diclofenac (SDCF), (Sodium 2-[2-[(2,6-dichlorophenyl)amino] phenyl acetate, Merck), sulfuric acid (H_2SO_4, KimyaLab Türkiye), sodium hydroxide (NaOH, Sigma–Aldrich), ammonia solution (28% NH_4OH, Merck), potassium pyrosulfate ($K_2S_2O_8$, Sigma–Aldrich), potassium permanganate ($KMnO_4$, Sigma–Aldrich), phosphorus pentoxide. (P_2O_5, Merck), hydrogen peroxide (30% H_2O_2, KimyaLab Türkiye), hydrochloric acid (HCl, KimyaLab Türkiye), Graphene oxide (GO) used as adsorbent in this study was produced by oxidation from commercially available graphite by modified Hummers method [48, 49]. The powdered graphite (20 g) was put into an 80°C solution of concentrated H_2SO_4 (30 ml), $K_2S_2O_8$ (10 g), and P_2O_5 (10 g). The resultant dark blue mixture was then carefully diluted with distilled water, filtered, and washed on the filter until the pH became neutral. The obtained product was dried in air at ambient temperature overnight, then was put into cold (0°C) concentrated H_2SO_4 (460 ml). $KMnO_4$ (60 g) was added gradually with stirring and cooling, so that the temperature of the mixture was not allowed to reach higher 20°C. The reaction was terminated by the addition of distilled water and 30% H_2O_2 solution. The resulting mixture was filtered and washed with 1:10 HCl solution in order to remove residual metal ions.

Instruments for the study of the properties of the samples

The structural identity and phase composition of the samples were studied by X-ray diffraction (XRD) on a D2 PHASER Bruker diffractometer (CuKα emitter, nickel filter) in the 2θ angle range of 10÷70°. The infrared spectra of the samples were recorded using a Nicolet iS 10 FT IR spectrometer, Thermo Fisher Scientific, USA, in the frequency range of 400-4000 sm^{-1}. UV visible spectroscopic studies of the samples were carried out on a Specord 210 Plus UV-Vis spec trophotometer in the wavelength range of 190-700 nm. The surface morphology and microstructure of graphite oxide were analyzed by scanning electron microscopy (SEM) (SEM-EDX on a JEOL JSM 6610LV in LEI mode) at an accelerating voltage of 15 kV and a working distance of 4.5 mm. The pH values of the solutions were determined using a pH/mV/conductivity/TDS/temperature Meter 86505.

Adsorption experiments

A stock solution with a concentration of 500 mg l^{-1} was prepared by dissolving SDCF in bidistilled water. Working solutions of the required concentrations were prepared on the basis of the initial solution. Batch adsorption experiments were performed to explore the effect of adsorption parameters such as solution

pH, contact time, initial adsorbate concentration, and adsorbent dosage were analyzed using data obtained from batch experiments. Adsorption experiments were performed according to the following scheme: 0.01g of the GO (with the exception of experiments investigating the effect of GO dose) was mixed on a shaker with an aqueous solution (30 ml) of SDCF at the desired concentration until equilibrium was reached, depending on its initial concentration (5—100 mg l^{-1}). The influence of pH was studied in the pH range from 2 to 10, adjusting the value using 0.1 M NaOH and 0.1 M HCl solutions. After adsorption, the solution was centrifuged and the concentration of residual SDCF was analyzed. SDCF content was determined by a spectrophotometric method using a calibration graph (absorbance data were recorded at 276 nm). The degree of adsorption (R, %) and ad sorption capacity (Q_e, mg g^{-1}) was determined using equations (1) and (2), respectively:

$$R = \frac{C_0 - C_t}{C_0} * 100\% \quad (1)$$

$$Q_e = \frac{(C_0 - Ce)\,V}{m} \quad (2)$$

where C_0 and C_e are the initial and equilibrium concentrations of SDCF in the solution (mg l^{-1}), **m** is the mass of the dry **GO** (g), and **V** is the volume of the sample (ml).

Results and Discussion

Structural characteristics of adsorbent

XRD pattern of graphitic oxide obtained by oxidation of graphite is shown in Fig.1. During oxidation high intensity peak around 26.7° corresponding to the pure graphitic structure (002) disappears [50] and an intensive peak at 10-11° appears after oxidation, which is corresponding to the (001) diffraction peak of GO. The increased d-spacing of GO sheets is due to the presence of abundant oxygen-containing functional groups on both sides of the graphene sheet causing an atomic-scale roughness on the graphene leaves [51].

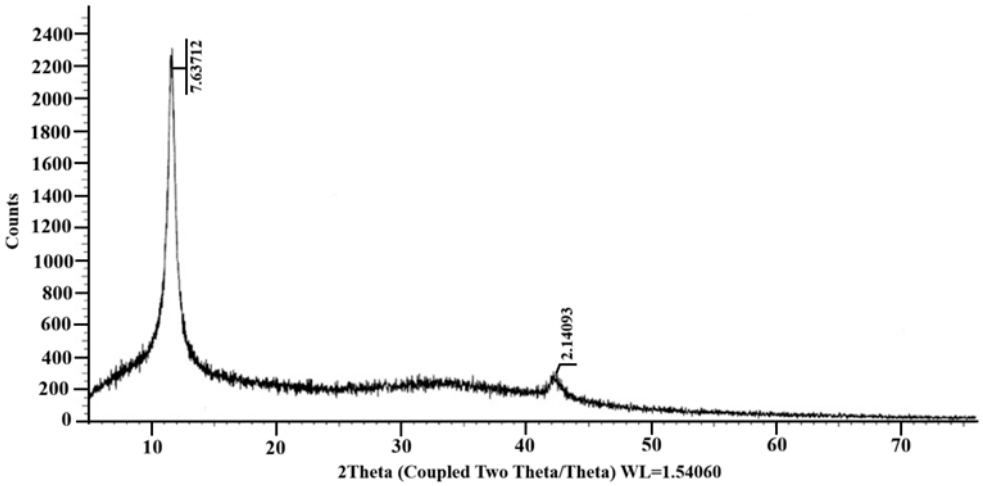

Fig.1. X-ray diffraction spectra of graphene oxide.

The spectra obtained by Fourier transform IR spectroscopy shown in Fig. 2 indicate the presence of rich oxygen-containing functional groups on the GO surface [52]: a wide absorption band at 3185–3648 cm^{-1} can be assigned to the O–H stretching mode of hydroxyl groups; peaks at 160.34, 1714.99 cm^{-1} usually assigned to C=O stretching vibrations of carboxyl ketones, or aldehydes groups; broad bands at 1053, 1227 cm^{-1} have been assigned to C–O stretching in acids, alcohols, ethers.

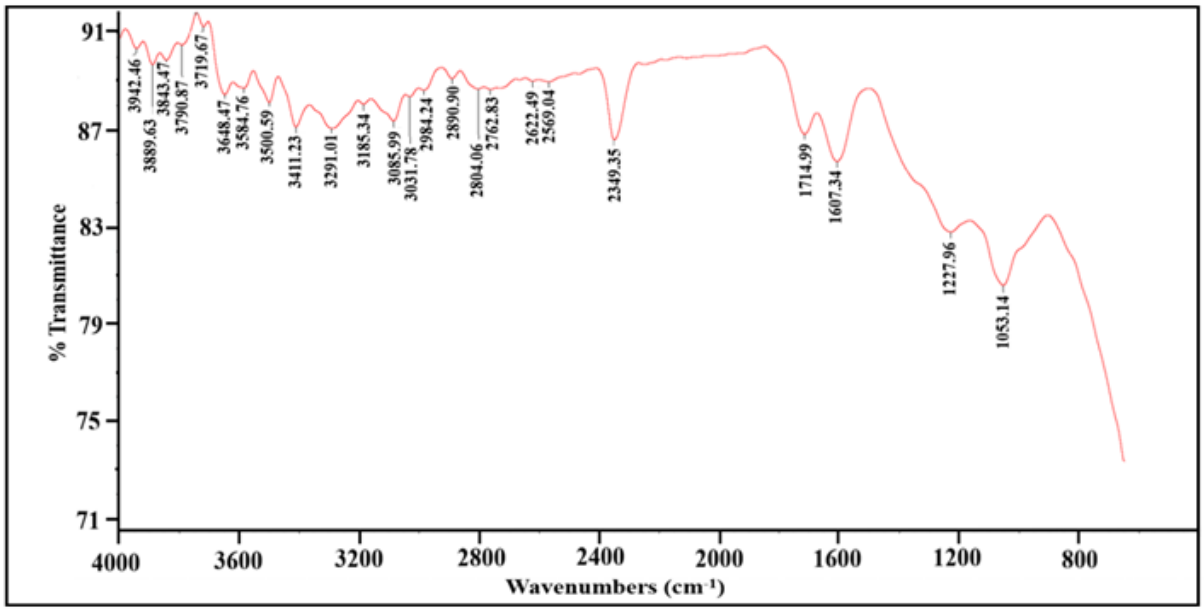

Fig.2. IR spectra of graphene oxide.

SEM was utilized to investigate the morphology of the as-prepared samples. Fig. 3 shows the typical SEM images of GO obtained by a modified Hummers method, SEM images revealed that the reduced GO material consists of randomly aggregated and crumpled folds closely associated with each other and forming a disordered surface.

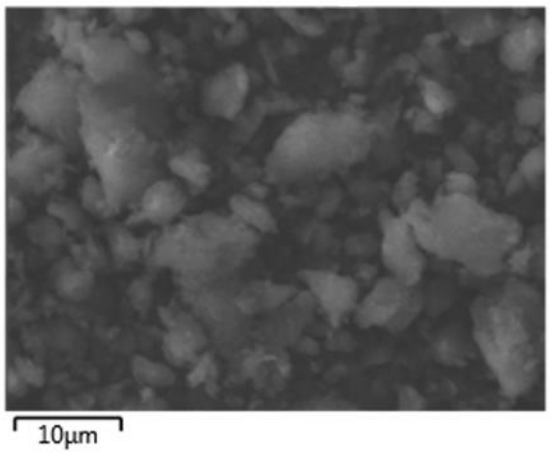

Fig. 3. SEM image of GO obtained Hummers method.

Adsorption properties of GO

The efficiency and selectivity of adsorption on GO depend on its textural characteristics. According to the recent studies GO consists of oxidized graphene sheets (or 'graphene oxide sheets') [53], having a large number of oxygen-containing hydroxyl, epoxide, carboxyl and carbonyl groups on the layers. These oxygen functionalities render the graphene oxide layers hydrophilic which readily forms stable colloidal suspensions of sheets in water [40, 41]. So, graphene oxide layers like many other compounds containing various chemically active groups are able protonated and deprotonated in aqueous suspensions which play an important role in the interactions of GO with SDCF contaminated water molecules can intercalate into the interlayer galleries. A suitable ultrasonic treatment can produce stable dispersions of very thin graphene oxide sheets in aqueous media.

Effect of pH of the medium

The most important factor in the adsorption process is the pH factor, since it affects not only the ionized form of the target pollutant molecule in the solution, but also the form of the surface charge of the adsorbent [54]. Fig. 4 shows the change in the adsorption capacity of GO in the pH range of 3-10. The maximum value of the adsorption capacity for GO is clearly visible at pH values of 2.5-4 (97-98%), which decreases with increasing pH. However, in such a highly acidic environment, the solution does not remain stable and loses its transparency. This can be attributed to SDCF precipitation from the solution [41]. Although the sorption rate gradually decreases until the pH of the solution reaches 5-6, the solution becomes transparent. At pH 5.5 the sorption rate (R) was determined to be 90% and the sorption capacity (Qe) was 137.9 mg/g (Figure 4). A similar experiment conducted on a sample of GO obtained after exfoliation by the ultrasonic treatment showed an sorption rate of 92% and a sorption capacity of 141.2 mg/g, which does not differ significantly from previous results.Under alkaline conditions, the sorption rate of DCF decreases sharply. Based on the results of the experiments, the optimal pH value was determined to be 5-6.

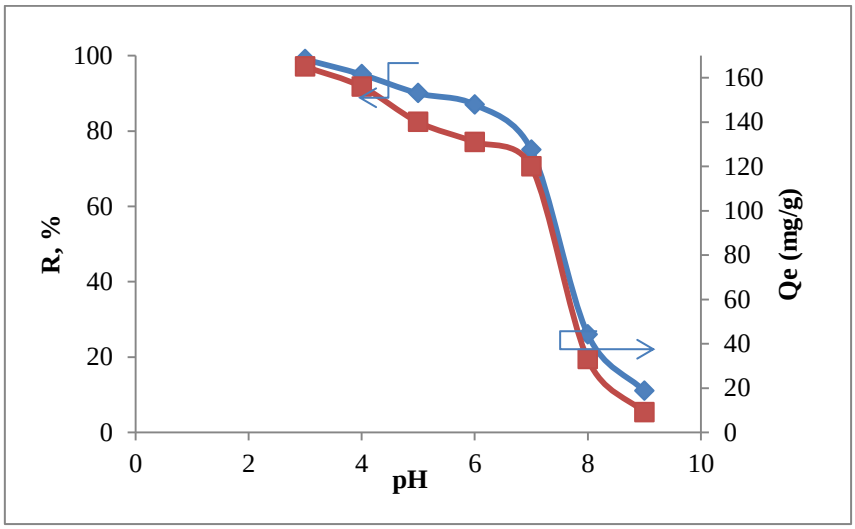

Fig. 4. Adsorption of SDCF on GO as a function of pH.
Solution volume - 30 ml; adsorbent -10 mg; temperature = 20°C; contact time = 2 h

The main data required for modeling adsorption isotherms are the time required for the concentration of ions in the solution (Ce) to reach equilibrium and the mass of adsorbate adsorbed per unit mass of fixed sorbent (Qe) at the corresponding Ce values. The effect of contact time on the adsorption capacity of SDCF on GO is shown in Fig. 5. The solution volume was 30 ml, pH 5.5, and an adsorbent dosage of 10 mg. The adsorption rate of SDCF reached equilibrium after about 60 min at room temperature. The adsorption rate increase during this time and reached 90 %. This result can be attributed to the presence of a large number of free centers on the GO surface during the initial period of adsorption, which become saturated with time.

The effect of the GO dose on the adsorption of SDCF was studied by adding a known amount of sample (5-20 mg) to separate flasks. The results are shown in Fig. 6. The adsorption rate increased with increasing GO dose, which can be explained by the presence of more adsorption sites on the surface of the larger sample. As can be seen from Fig.6, the adsorption capacity decreased sharply with increasing adsorbent dose. A mass of 10 mg GO was chosen for further experiments.

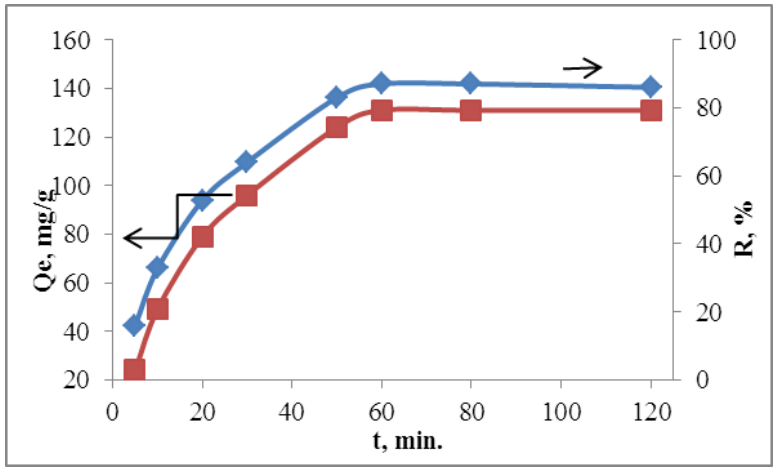

Fig. 5. Effect of contact duration on the adsorption parameters R and Qe; pH 5.5; solution volume - 30 ml; adsorbent -10 mg.

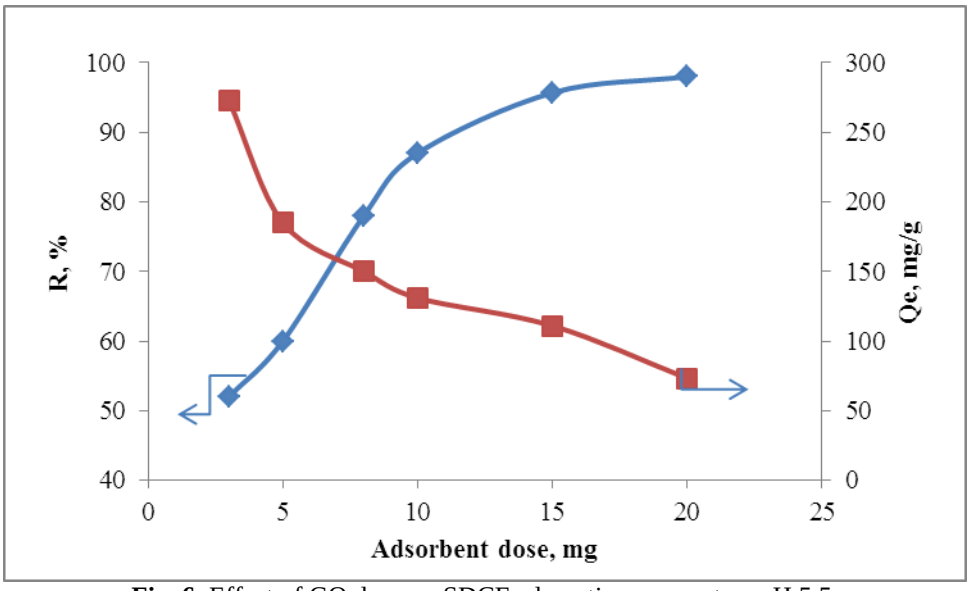

Fig. 6. Effect of GO dose on SDCF adsorption parameters pH 5.5; solution volume is 30 ml.

An experimental adsorption isotherm demonstrating the relationship between the equilibrium concentration of SDCF on the surface of the sorbent under normal conditions and its equilibrium concentration in the liquid phase was studied by varying the concentration of SDCF in solution over 10 mg of GO in a medium with pH 5. 5 in the range of 7 - 45 mg/l (Fig. 7).

The mechanism of the sorption process of SDGF was analyzed using Langmuir and Freundlich isotherm models.

The Langmuir model is valid for sorption on a monolayer surface with a certain number of identical reaction sites and is described by the equation

$$Ce/Qe = 1/bQm + Ce/Qm.$$

In this equation, b (mg^{-1}) is the Langmuir constant, Ce (mg. l^{-1}) is the equilibrium concentration, Qe (mg. g^{-1}) is the sorption volume capacity of the adsorbent, and Qm (mg. g^{-1}) is the single-layer capacity. The linear plot of Ce versus Ce/Qe is a straight line with a slope of 1/Qm and an intercept of 1/bQm (Fig.8).

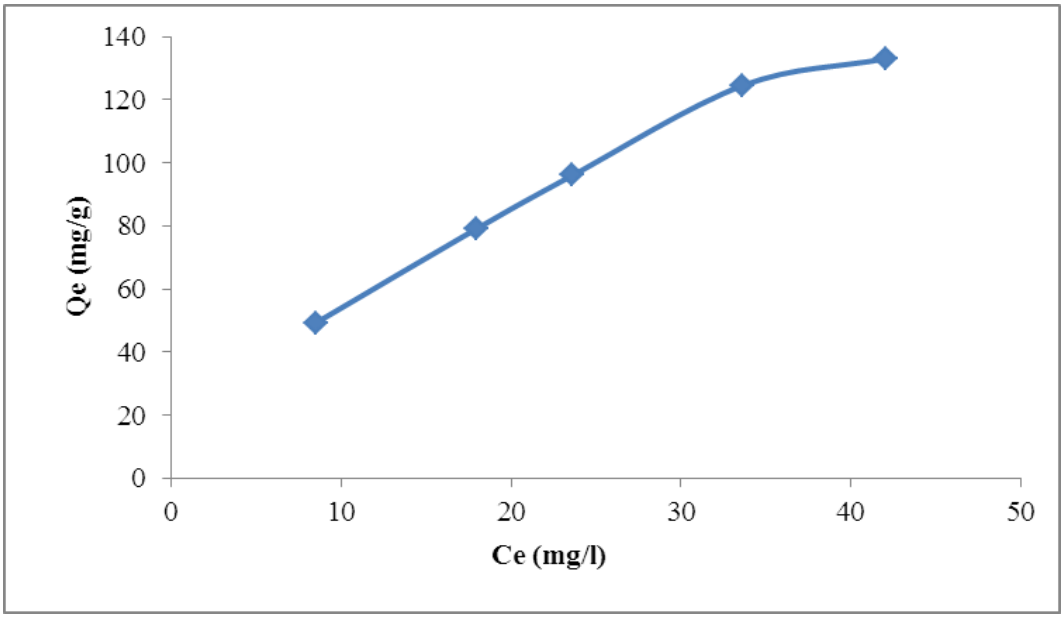

Fig. 7. Isotherm of adsorption of SDCF in aqueous medium.
pH 5.5, Adsorbent -10 mg

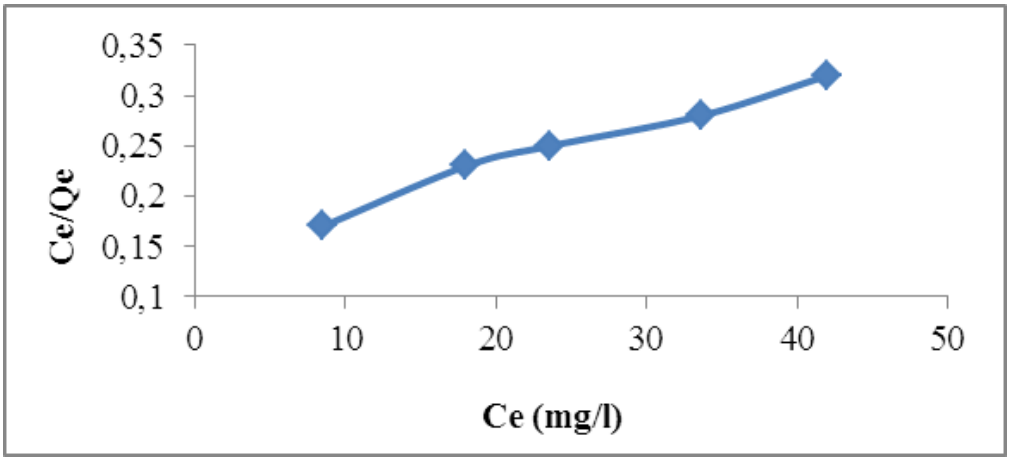

Fig. 8. SDFC adsorption isotherm in the coordinates of the linear form of the Langmuir equation

The Freundlich isotherm model is described by the equation

$$\mathbf{lnQe = lnKF + (1/n)\ lnCe.}$$

In this equation, KF (mg/g) and n are the Freundlich constants, which characterize the adsorption capacity and adsorption intensity of the adsorbent, respectively. The Freundlich equation can be expressed in linear form as logarithms and constants (Fig.9).

The obtained parameters of the models are summarized in Table 1. The Langmuir model has a correlation coefficient (R2) of 0.9782, while the Freundlich model has a correlation coefficient of 0.9835. According to the obtained results, the Freundlich model, which indicates the adsorption of SDCF on the heterogeneous surface of GO, is more likely.

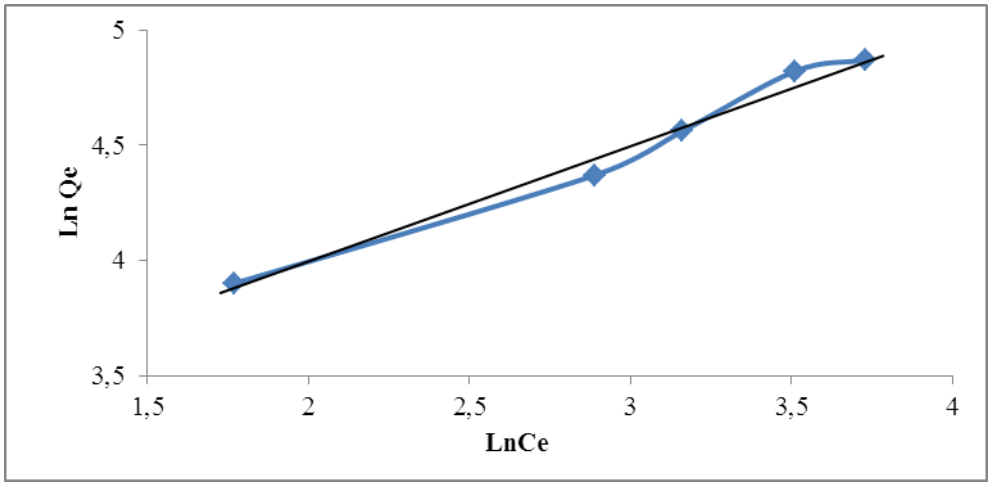

Figure 9. Adsorption isotherm of SDCF in the coordinates of the linear form of the Freundlich equation

Table 1. Parameters of the Langmuir and Freundlich models for the adsorption of SDCF on GO

Langmuir model			
SST_{maks}, mq/q	**K_L, l/mq**	**R_L**	**R^2**
238.1	0.0293	0.29÷0.78	0.9782
Freundlich model			
1/n	**K_F, mq/q**	**n**	**R^2**
0.51	19.45	1.961	0.9835

Conclusions

Graphene oxide was synthesized by the Hummers method and was used as an adsorbent to remove sodium diclofenac from aqueous media. It was found that the graphene oxide is highly effective in removing sodium diclofenac. At pH 5.5 the sorption rate was determined to be 90% and the sorption capacity was 137.9 mg/g. It was shown that the sorption efficiency of sodium diclofenac using graphene oxide synthesized by the Hummers method directly, compared to its exfoliation analogue, does not differ significantly. The sorption isotherm of sodium diclofenac on the graphene oxide can be modeled by both the Langmuir adsorption isotherm and the Freundlich adsorption isotherm based on the correlation coefficients. However, the Freundlich R2 value (R2 = 0.9835) is higher than the Langmuir correlation coefficient (R2 = 0.9782). Therefore, the Freundlich isotherm reflects the adsorption of sodium diclofenac on the graphene oxide from aqueous solutions comparatively better than the Langmuir isotherm. Thus, direct use of graphene oxide synthesized by the Hummers method can be an effective and environmentally friendly adsorbent for the treatment of aqueous solutions contaminated with sodium diclofenac.

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NATRİUM DİKLOFENAKIN SULU MÜHİTDƏ QRAFEN OKSİDİ İLƏ ADSORBSİYASI

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Qrafen oksidi Hummers metodu ilə sintez edilmiş və rentgen difraksiyası, skan edici elektron mikroskopiyası, Furye çevirmə infraqırmızı və ultrabənövşəyi görünən spektroskopiya ilə xarakterizə edilmişdir. Əldə edilən material, natrium diklofenakı sulu mühitdən sorbsiyası üçün adsorbent kimi istifadə edilmişdir. Proses parametrlərinin (pH, natrium diklofenak qatılığı, təmas müddəti və adsorbent dozası) qrafen oksidinin sorbsiya səmərəliliyinə təsiri araşdırılmışdır. Langmuir və Freundlix adsorbsiya modellərinin eksperimental natrium diklofenak adsorbsiya izotermlərinin təsviri üçün tətbiqinin müqayisəli tədqiqi aparılmışdır.

Açar sözlər: *Qrafen oksidi; adsorbent; suyun təmizlənməsi; natrium diklofenakın sorbsiyası; materialın xarakteristikası*