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Adsorption of nitroaniline onto high surface area nanographene

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ABSTRACT

High surface area nanographenes (HSANGs) were used for the adsorption and removal of *p*-nitroaniline from a model solution and wastewater sample. HSANGs were characterized using TEM, STM, and surface area analysis, and the results revealed that HSANGs exist as short stacks of transparent platelet-like graphene sheets with an average thickness of 2.0 nm and a BET specific surface area of 677.5 $m^2 g^{-1}$. The results showed that most of the NA was removed from the solution within a few minutes at ambient conditions. The adsorption was analyzed kinetically and the results showed the suitability of the pseudo-second-order model for describing the adsorption kinetics. The thermodynamics study showed that the adsorption process was exothermic in nature, product favored, and associated with an increase in entropy due to the adsorption of NA to the HSANGs surface. The HSANGs exhibited a tremendous adsorption ability to remove NA from a model solution and a wastewater sample.
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Introduction

Since the creation of Earth, this century is the most critical because of the extensive pollution due to the anthropogenic activities, which affect the lives and fate of humans on the planet. Among different types of pollution, aquatic pollution is considered the most crucial because it limits the amount of fresh water required for the existence of the living organisms on Earth. Hence, most of the international organizations and societies work to solve this persisting problem either by legislation or through the introduction of new limits and regulations. The scientific community feels responsibility toward this matter through its extensive research to highlight and explore the effects of different pollutants and how to lessen or remove them from the environment. One of the most persistent classes of pollutants in the environment is organic pollutants. There are different methods used to treat polluted water from organic pollutants [1–6], but most of these methods usually suffer from drawbacks such as high cost, secondary pollutants, regeneration problems, sludge generation, long retention times, and low efficiency. On the other hand, adsorption of organic pollutants from polluted water is considered a promising way for environmental remediation due to the ease of application, low cost, and ability to regenerate both adsorbents and pollutants [7–11]. One of the main concerns of research scientists is the search for new types of adsorbents characterized by high adsorption capacities and a strong affinity toward the organic pollutants.

Nanographenes (NGs) are fascinating new carbon materials that have attracted the attention of scientists of materials science in the recent years. They are composed of a one-atom-thick, two-dimensional (2D) layer of sp^2-bonded carbon. Nanographenes also exhibit extraordinary mechanical, electrical, thermal, and optical properties as well as a high specific surface area [12]. These extraordinary properties allow graphene to be used in different applications [13–17]. Furthermore, graphene has been used in environmental remediation in the photocatalytic degradation of different pollutants [18–22], and as an excellent adsorbent for different pollutants [23–27] due to its large, delocalized π-electron system, which can form strong interactions with other pollutants as well as their ability to be functionalized. Although, it has been reported that graphene have very strong adsorption capability for different pollutants, the removal of organic compounds such as nitroaniline using NG is still scarce in the literature [23–26]. Further investigations on the adsorption/removal of nitroaniline, as an example of organic pollutants, by the new and promising adsorbent, high surface area NG, are needed for the remediation of the aqueous environment.

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In this study; for the first time, high surface area nanographenes (HSANGs) were used for the removal of *p*-nitroaniline (NA) as an example of organic pollutants from an aqueous solution. The physical characterization for the NGs was first studied to explore their morphological properties. The effect of different environmental conditions, solution pH, temperature, and adsorption time, which affect the removal process, were investigated. Additionally, the adsorption process was studied kinetically and thermodynamically to predict the adsorption rate in order to understand the adsorption behavior, the mechanism of adsorption, and its spontaneity by calculating different thermodynamic parameters.

Materials and methods

Materials

High surface area nano graphenes (HSANGs) were obtained from XG Science, USA. xGnP® and were used as received. All chemicals used in this study were obtained from Sigma-Aldrich (analytical grade), and all solutions were prepared using deionized water.

Characterization techniques

A transmission electron microscope (TEM; type JEOL JEM-1230, operating at 120 kV, attached to a CCD camera) and a Scanning tunneling microscope (Agilent 5500) were used to characterize the HSANGs' morphological structure. The specific surface areas of the HSANGs were determined from nitrogen adsorption/desorption isotherm measurements at 77 K, using a model NOVA 3200e automated gas sorption system (Quantachrome, USA).

Adsorption experiment

Adsorption experiments were performed to determine the effect of time and temperature on the adsorption of NA by HSANGs under different environmental conditions. The experimental procedures were performed as follows: (1) a series of solutions of various NA concentrations were prepared; (2) the initial pH was measured, and a defined amount of the HSANGs was then added to the solutions; (3) these solutions were agitated on a magnetic stirrer for a certain period of time, at room temperature; (4) at defined points in time, a certain volume of the solution was removed and immediately filtered to collect the supernatant; and (5) the residual MB concentration in the supernatant using an UV/Vis instrument at 370 nm. The amount of NA adsorbed was determined by measuring the difference in the concentrations of the samples that were obtained at two consecutive time intervals over the course of the adsorption experiment. The adsorption capacity of the HSANGs (q_t, mg g^{-1}), which represents the amount of NA adsorbed per amount of HSANGs was calculated using a mass–balance relationship:

$$q = \frac{(C_0 - C_t)V}{m}, \quad (1)$$

where C_0 and C_t are the concentrations of NA in solution (g L^{-1}) at time $t = 0$ and t, respectively. V is the volume of the solution (L), and m is the mass of the dry adsorbent used (g). The kinetic curves obtained were analyzed using various-order kinetic equations to obtain the parameters for understanding the adsorption process.

Results and discussion

Characterization of HSANGs

Fig. 1 shows the transmission electron microscope images of the HSANGs. The images show that graphene consisting of short stacks of transparent graphene sheets have a platelet shape, a layered structure with a smooth surface, and many wrinkles. This was confirmed by the scanning tunneling microscope (STM) measurements. Fig. 2 shows the typical STM images of HSANGs. It is clear from the figure that graphene is present in the form of sheets with different sectional areas that are less than a micron and with an average thickness of 2.0 nm. Nitrogen adsorption/desorption isotherms for HSANGs were determined using N_2 at 77 K, and the results are presented in Fig. 3. The N_2 adsorption/desorption isotherms could be classified as type IV isotherms with H3 type hysteresis loops according to the original IUPAC classification similar to a mesoporous-plate-like industrial adsorbents. The BET specific surface area was found to be 677.5 m^2 g^{-1}, with a BJH adsorption/desorption cumulative volume of pores (between 1.000 and 3000.000 Å width) equal to 0.902795 cm^3/g, and a BJH adsorption/desorption average pore width (4 V/A) equal to 70.483 Å.

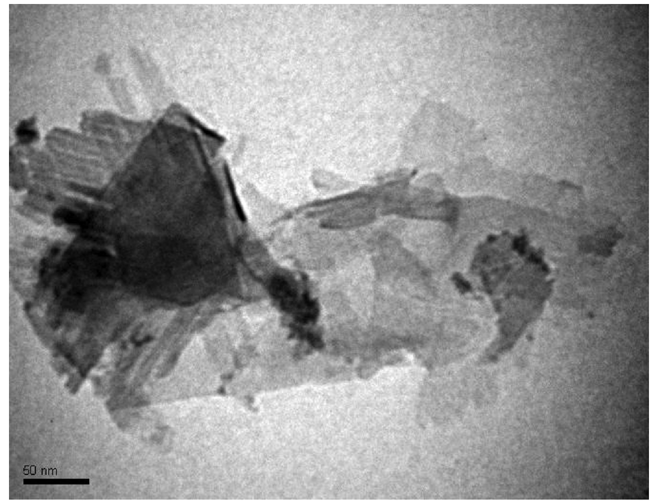

Fig. 1. Transmittance electron microscope image for HSANGs used for NA adsorption from solution.

Adsorption study

The effect of adsorption parameters

Generally, the adsorption process of any pollutants from an aqueous solution using a solid adsorbent depends on different operational and environmental factors such as adsorbent mass, solution pH, temperature, and the contact time between the pollutant and the adsorbent. The effects of these factors on the removal of NA by HSANGs from an aqueous solution were investigated. Fig. 4 shows the effect of the HSANGs mass on the removal of NA. The percentage of NA removed from the aqueous solution increased gradually as the HSANGs mass increased, until it reached almost 100% when 30 mg of HSANGs were applied. The amount of adsorbed NA increased along with the HSANGs mass, mainly due to the availability of more active sites available for adsorption. Consequently, for the rest of the experiment, 15.0 mg HSANGs corresponding to 76.9% allowed for a clear observation to the effect of other parameters. The effect of time on the adsorption of NA from an aqueous solution by HSANGs was studied. As Fig. 5 shows, the percentage of NA removed from the aqueous solution increased significantly from 58.4% after 1 min to 71.0% within 30.0 min. After 30.0 min, almost the entire adsorption reached equilibrium with an amount of removed NA equal to 75.7%. A contact time of 120 min was used for the further studies. The effect of the solution temperature on the adsorption and removal of NA by HSANGs was explored at 283, 293, 308, and 323 K. The results

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