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Article

Functionalized graphene oxide nanoparticles for improved saltwater treatment

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SUMMARY

With the increasing severity of water droughts and water shortages caused by climate change, it becomes more important than ever to find accessible and reliable sources of freshwater. One way to do this is through the removal of salt from saltwater. Graphene oxide (GO) nanoparticles can be functionalized, allowing for saltwater absorption and water purification. Our objective was to synthesize, characterize, and determine the ability of different functional groups on GO nanoparticles to purify saltwater. We hypothesized that functionalized GO nanoparticles, due to their increased surface area and optimal surface energy, could increase the adsorption of salt from saltwater for freshwater generation. GO was synthesized and then was functionalized with environmentally-friendly chitosan and silver (Ag). Chitosan was selected for its high hydrophilicity which aids in dispersing the GO nanoparticles in water and Ag for its ability to counter biofouling. The size of the nanoparticles was measured to be around 350 nm, thus, providing for greater surface area for water purification than larger traditional particles. Results showed that the functionalized GO nanoparticles removed salt from saltwater providing an inexpensive, environmentally-friendly, and effective way to generate freshwater.

INTRODUCTION

Saltwater desalination involves the complex use of several filters or filtration methods to produce freshwater from saltwater. Increasing the efficiency of the desalination process is critical to alleviate the stress caused by freshwater scarcity around the world (1). Such a new process and new materials would allow for an alternative and reliable source of freshwater. The two primary processes used for water desalination include water distillation and reverse osmosis. Reverse osmosis utilizes a semi-permeable membrane and high pressure to separate salt from water (2). To increase the efficacy of this process, which currently accounts for 80% of the world's desalination capacity, it is critical to find less expensive and easier methods (3-5). With this goal in mind, researchers have considered the use of graphene oxide (GO) as a primary material for the treatment of water due to its low cost, environmentally-friendly carbon-based chemistry, and availability (6). Several different uses have been envisioned for GO in water purification, from membrane-use to an absorbent in/on a water filter (3). Due to the greater surface area to volume ratio compared to micron GO particles, GO nanoparticles have attracted a lot of attention especially if they can made hydrophilic to optimally interact with water (3).

One method of water purification that has been previously investigated uses GO nanoparticles added directly to water to bind to impurities and be removed through filtration (7). GO nanoparticles generally have a large surface area which allows for a greater region to remove salt than larger particles (3). Moreover, functionalizing GO with specific environmentally-friendly chemical groups can increase the amount of negative charge and hydrophilicity of the GO nanoparticles to thus increase salt absorption (7). Zahed and others have been successful in functionalizing GO with chitosan to remove salt from saltwater (7). However, further improvements can be made concerning how the GO is made and functionalizing the GO nanoparticles with additional chemistries (such as silver (Ag)) to improve hydrophilicity and water purification.

Multiwalled carbon nanotubes (MWCNTs) are another form of carbon which have attracted great attention in science and engineering due to their high purity, exceptional strength, light weight, and high conductivity (6). Due to their growing popularity in science and engineering, MWCNTs were chosen for fabricating functionalized GO nanoparticles in the present study (8,9). Importantly, MWCNTs can be found in the soot of burning wood, further adding to the ability to easily find such materials in the environment that can form GO for inexpensive and environmentally-friendly water purification (8,9).

Considering the above, we hypothesized that functionalized GO nanoparticles, due to their increased surface area and optimal surface energy, can remove salt from saltwater for freshwater generation. The aim of this study was to develop an alternative method to synthesize GO nanoparticles, and test whether such functionalized GO nanoparticles possess salt adsorption abilities. Specifically, GO was functionalized with a combination of both chitosan and Ag. The Zahed process was used for this purpose, involving the use of methanol to form the functionalized GO. Importantly, here, an environmentally-friendly process was designed to synthesize Ag nanoparticles out of tea leaves and sunlight due to focus of this study on cleaning salt water. One does not want to harm the environment through the synthesis of nanoparticles used for environmental water purifying purposes. We hypothesized that functionalizing GO nanoparticles with both chitosan and Ag will further enhance salt adsorption.

RESULTS

Characterization of Silver (Ag) Nanoparticles

As with any study formulating new materials, our first objective was to prove that Ag nanoparticles can be synthesized from commonly found inexpensive environmentally-friendly materials using low energy, such as by brewing tea leaves and a silver nitrate solution in sunlight.

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Dynamic Light Scattering (DLS) scans of the Ag nanoparticles showed sizes ranging from around 150 nm to 900 nm (**Table 1**). The Ag particles were synthesized according to the work of Parlinska-Wojtan *et al.* (9). The larger size is likely due to the agglomeration of the silver nanoparticles in the solution. Further, utilizing Energy Dispersive Spectroscopy (EDS), the nanoparticles were indeed shown to be Ag (**Figure 1**). A significant quantity of aluminum (Al) was also detected which could be seen as a contaminant from the tea leaves.

Characterization of Graphene Oxide Nanoparticles

DLS scans were obtained for all three repetitions of GO nanoparticle synthesis showing very little batch-to-batch variation in size with an average size slightly above 1000 nm **(Table 1).** While the average lies above 1000 nm, the range allows for sizes below 1000 nm fitting their description as nanoparticles. The lack of variation in the size of each repeated synthesis method suggests that GO nanoparticles can be reliably formed from MWCNTs using the Hummer's method as described in the materials and methods section. Modification of the Hummer's progress, particularly the use of MWCNTs instead of graphite as the carbon source, could have potentially generated a different chemistry, thus, it was important to verify the chemistry of the GO nanoparticles as well. The results from EDS confirmed the presence of large quantities of oxygen and carbon, suggesting that the process was successful in breaking down the MWCNTs into graphene layers and then oxidizing them to form GO nanoparticles due to the high carbon to low oxygen ratio (**Figure 2**).

Salt Adsorption

The salt absorbance measurements showed that as the concentration of salt increased in the water, salt absorbance by the GO functionalized nanoparticles increased (**Figure 3**). Moreover, by using different functionalized GO nanoparticles, we found that further increases in the quantity of salt added, increased the qe value (a measure of the quantity of the original salt that was absorbed) (**Figure 4**). The correlation between the points plotted was also high, with an R^2 value of 0.9494, indicating that we created functionalized GO nanoparticles that effectively removed salt from saltwater. However, the increased amount of salt following the experiment (compared to the initial amount added) could be attributed to an inability of the filter paper to filter all the graphene oxide, allowing some to pass through and be interpreted as salt weight as further discussed below. However, all materials were assessed in the same way, thus, this amount should be similar between different experimental conditions. None-the-less, this should be the focus of future experiments.

Nanoparticle	DLS Nanoparticle Size Average (nm)	DLS Nanoparticle Size Range (nm)
Ag	324.2	150 to 900
GO (trial 1)	N/A	720 to 1670
GO (trial 2)	N/A	1110 to 1790
GO (trial 3)	N/A	880 to 1710
GO (trial 1-3)		Average: 1020

Table 1: Ag and GO nanoparticles of interest to the present study have nanometer sizes. DLS results show size measurements of each of the three different synthesis trials of GO and the original Ag nanoparticle solution.

DISCUSSION

Results of the present study showed the correct fabrication of the proposed nanoparticles using environmentally-friendly processes, and, most importantly, demonstrated that the presently fabricated functionalized GO nanoparticles removed salt from saltwater. The results of this study showed that by functionalizing GO nanoparticles with chitosan and Ag, the GO nanoparticles retained their salt adsorption capability. Such results are critical in developing easy processes that can purify saltwater into pure water for consumption and use.

The most interesting developments uncovered by this research were both the possibility of using MWCNTs instead of graphene to create GO nanoparticles as well as the alteration to the Hummer's synthesis method which could reduce the time necessary to produce GO while still retaining properties to purify water. The Hummer synthesis focuses on a combination of acids in order to break down carbon and allow for the formation of graphene oxide. In particular, the use of a 200°C oven reduced the time necessary for the proper production of GO by approximately 3 hours.

However, despite these promising results, some flaws

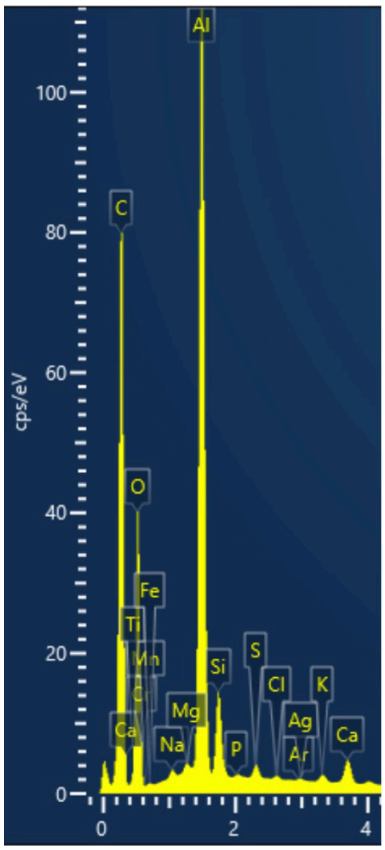

Figure 1: Ag nanoparticles synthesized from silver nitrate and tea leaf solution containing Ag. EDS results demonstrate that Ag along with other ingredients from the tea leaves were present in the Ag nanoparticles. The X axis indicates Kev values which correspond to different atomic signatures.

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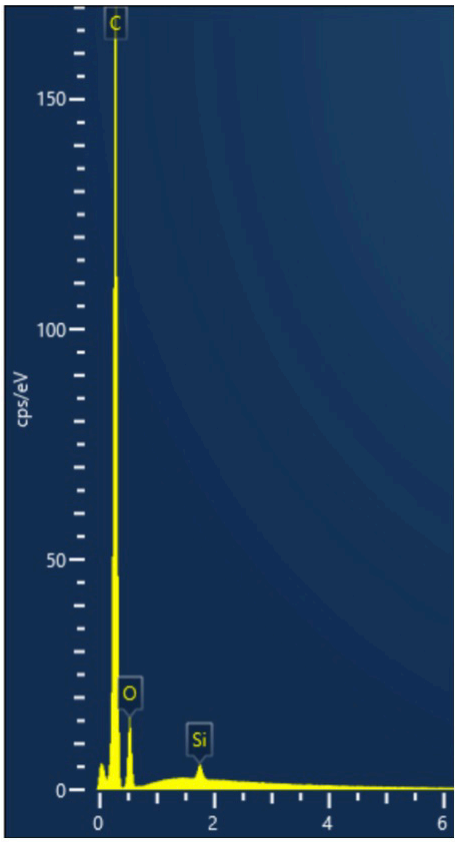

Figure 2: GO nanoparticles synthesized from MWCNTs contain carbon and oxygen. EDS results demonstrate that carbon and oxygen were present in the GO nanoparticles. The X axis indicates Kev values which correspond to different atomic signatures.

within this study were apparent. For example, the initial value for absorbance was negative, indicating that during the filtration process a significant quantity of GO functionalized nanoparticles made it through the filter. The high correlation also suggests that a consistent amount of GO nanoparticles made it through the filter paper thus confounding measurements. Future studies should also assess the role of chitosan and Ag in killing bacteria which may be present in unpurified saltwater.

It further remains to be seen whether the nanoparticles formulated here can be used on a larger scale. The high amount of time and expense necessary to produce small quantities of GO, especially when synthesized from MWCNTs, and removing the nanoparticles after removing salt, requires more research to overcome.

Additional further research avenues also exist to improve the present GO functionalized nanoparticles. First, it is possible that using more electronegative functional groups, such as hyperbranched polyamide-amine, an even better salt absorbance rate could be reached. Second, a proper extraction method to remove the GO nanoparticles out of the purified water would be necessary before widespread adoption of this strategy. Third, researchers could facilitate the filtration of the GO nanoparticles via centrifuging to reduce clumping and larger GO particle sizes could more easily be filtered out. Finally, research could be conducted to determine salt adsorption to the functionalized GO nanoparticles using atomic adsorption spectroscopy or other means than the simple filtrate weight differences as used here.

In summary, this work demonstrated the removal of salt from saltwater using novel environmentally-friendly functionalized GO nanoparticles. Such nanoparticles and approaches are crucial in purifying saltwater to meet global freshwater needs.

MATERIALS AND METHODS

Nanoparticle Synthesis

MWCNTs, sulfuric acid, phosphoric acid, hydrochloric acid, hydrogen peroxide, potassium permanganate, deionized water, sodium chloride, chitosan, and silver nitrate were used in the below experiments and were all obtained from Sigma-Aldrich. Tazo tea bags were obtained from CVS.

The method used to synthesize silver nanoparticles was based on the work of Parlinska-Wojtan *et al.* (9). First, 20 ml of water was boiled with 4 g of Tazo tea. The tea bags were included to properly reduce the Ag particles to produce Ag-NPs and as a capping agent to ensure a smaller nanometer size for the particles. The mixture was then passed through a filter paper to remove the unreacted tea components resulting in a solution with about half of the original volume. An equal volume of silver nitrate in a 0.15 M solution was added to the solution. The new solution was left in a dark and dry space for an hour to allow for the formation of a solute. The new solution was then repeatedly centrifuged at 5000 rpm for 4 min to separate out the supernatant from the pelleted Ag-NPs.

The synthesis of the GO nanoparticles followed the modified Hummers process with the exception that MWCNTs served as the carbon source instead of graphite(10). For this, 27 ml of sulfuric acid was mixed with 3 ml of phosphoric acid. 225 grams of MWCNTs were then added followed by 1.32 grams of potassium permanganate. The solution was covered and left to stir for 6 hours, at which point 10 ml of H_2O_2 was added and the solution was left to stir until cooled back to room temperature. 10 ml of HCl and 30 ml of distilled water was added. The solution was repeatedly centrifuged at 4900 rpm and decanted each time. Once the solution no longer produced a supernatant, the solution was placed in an

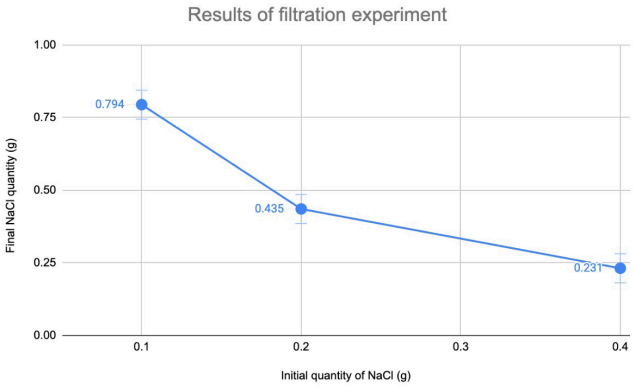

Figure 3: Increased removal of salt (NaCl) from saltwater by the functionalized GO nanoparticles. This graph shows the final quantity of salt compared to the original salt quantity in water.

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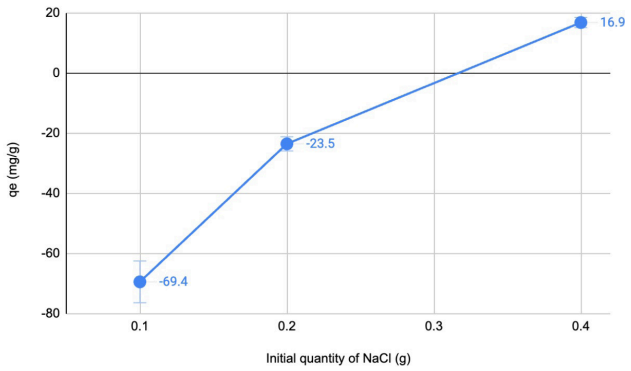

Figure 4: Graph of the absorbance of GO nanoparticles compared to initial salt (NaCl) quantity in water. qe is a measure of the quantity of original salt that was absorbed. The R^2 value for this graph is 0.9494 showing a direct relationship between removal of salt from saltwater using the functionalized GO nanoparticles.

oven for 2 hours to dry at 200 degrees Celsius. This process was repeated three times to confirm consistency. For the third run, the stirring time following the addition of potassium permanganate was reduced to a little over an hour to test if this would have an impact on the formation of GO.

To form GO functionalized with Ag and chitosan, the Zahed process was used (6). Specifically, 3 grams of GO were added to 50 ml of methanol, as well as 0.75 grams of Ag and 1.5 grams of chitosan. The solutions were then left to stir for 20 hours at room temperature.

Nanoparticle Characterization

The nanoparticles selected for this study were characterized for size using DLS and for chemistry by EDS.

Functionalized GO Nanoparticle Purification of Salt from Saltwater

The functionalized GO nanoparticles were tested for their ability to remove salt from saltwater. For this, first, saltwater solutions at concentrations of 0.1, 0.2, and 0.4 g of salt/100 ml of water were created. Once the salt and GO-Ag-chitosan solutions were prepared, a third of the GO solution was added to each salt solution. These new solutions were then passed through filter paper to remove the absorbent. This step was repeated another time to better remove the salt. The filtered solutions were then boiled until all liquid seemed to have evaporated and then was weighed. The difference in weight between the initial quantity of salt added, and the final weight of the flask was taken to be the quantity of salt which was absorbed.

To determine the absorbance of salt by the nanoparticles, the amount of salt added to the solution was compared to the quantity of precipitate formed when the filtered solution was evaporated. The difference in mass was used as the quantity absorbed. The absorbance was then determined from the equation:

$$q = (Co - Cf)V/w$$

where q is the absorbance (measured in mg/g), Co is the initial concentration (measured in mg/ml), Cf is the final concentration, V is the volume (measured in ml), and w is the mass.

It was assumed that all the filter precipitate was salt and the distribution within the solution was homogeneous, thus, ensuring that the weight used was consistent and can be calculated for each experiment. However, it is important to note that the existence of a larger amount of mass at the end of the experiment might suggest that some of the mass that appeared after the experiment is not NaCl but instead is some other contaminant (such as GO which may have passed through the filter paper). However, since all experiments were completed exactly the same, such contaminants would be the same for each experimental condition, thus, not influencing the trends observed here.

All experiments were completed in triplicate and statistical differences were determined using student *t*-tests.

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