STUDIES ON PREPARATION AND APPLICATION OF GRAPHENE BASED COMPOSITES FOR WATER PURIFICATION

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INTRODUCTION

One of the most socially relevant aspects of nanotechnology is in the field of environmental remediation. Diverse applications of nanomaterials in decontamination of air, water and soil are intensely pursued in the recent past. One of the fascinating new additions into the carbon family is graphene, the one-atom thick sheets of carbon. High thermal and electrical conductivity, electronic properties, quantum hall effect, and application in drug delivery and DNA sensing, have been investigated in the recent past. In this report we show that chemically synthesized graphene, as well as graphene oxide, can be anchored onto the surfaces of river sand to make effective adsorbents that remove heavy metal ions, pesticides, and natural dyes. Such materials show higher adsorption capacity in comparison to activated carbon when equal masses of carbon are compared. When used as a stationary adsorbent material in a flowing water stream, it is necessary to anchor the nanoscale adsorbent onto inexpensive and reliable substrates. Utilization of such technologies in people-oriented applications requires the materials to be affordable. Among the simplest of natural sources of carbon are sugars, which upon dehydrogenation get converted completely into elemental carbon, leaving only water to escape.

OBJECTIVES

1. Preparation of Graphene Coated sand by surface coating of graphene on sand via thermal treatment.
2. To Study the physical structure and chemical properties of the Graphene Coated Sand.
3. Details on purification performance of the graphene-based composites for three major classes of pollutants - metal ions, organic dyes, oils and Bacterial contaminants.
4. To Study Regeneration capabilities for the Graphene coated sand.
5. To Study for viability in large scale application and its Future prospective
METHODOLOGY

MATERIALS USED:
- River sand (~0.2 mm particle size).
- Sugar (edible).
- Sulphuric acid and acetone were procured from local suppliers.
- Deionised water for washing.
- Tap water for testing.

PREPARATION OF THE COMPOSITE

![Preparation of the graphene composite](image)

Figure 1: Preparation of the graphene composite

Common sugar (sucrose crystals) was used as the carbon source. At first, the sugar was dissolved in water and then, the solution was mixed with requisite amount of sand (river sand). Calculated amounts of sugar and sand were taken to make different loading ratios. In each case, the mixture was dried at ~95 °C in a hot air oven for about 6 h with constant stirring. The sugar coated sand was then placed in a silica crucible and heated in a furnace in N\textsubscript{2} atmosphere. The furnace temperature was programmed to ensure complete graphitization. The temperature of 750 ± 5 °C was chosen as the final temperature after optimization through several experiments. The black sample was named graphene sand composite (GSC). For activation, 5 g of the composite was treated with 10 mL of concentrated sulphuric acid and kept undisturbed at room temperature for 30 min. washing with concentrated sulphuric acid leads the composite with higher adsorption sites. The mixture was then filtered and dried at 120°C and stored in air-tight containers.

ADSORPTION EXPERIMENTS

Time-dependent adsorption capacity of the as-synthesized composite was investigated in a batch reactor of 250 mL capacity at a temperature of 30°C on Rotary Shaker. The working volume and the adsorbent dose were maintained as 100 mL and 5 g, respectively. Hard water was used for removal of hardness with GSC. The solid–liquid separation was done by filtration. The filtrate was analysed for TDS. All the experiments were conducted in duplicate and the samples were analysed immediately. To measure the adsorption capacity, a fixed-bed column was operated under down flow mode at a feed flow rate of 1.19 cm\textsuperscript{3}/cm\textsuperscript{2}/min and feed concentration of 1 mg/L. The column was made by packing GSC to different depth in a transparent glass tube with a length of 50 cm and an internal diameter of 8 mm. The performance of the column was evaluated as a function of time at...
room temperature. The experiments were conducted for methylene blue at 30°C. Four parameters including initial concentration, flow rate, bed depth and pH were studied in the column tests. The effluent of Methylene blue solution was sampled at regular intervals of 15 min and the samples of MB was withdrawn and was analysed by UV-visible spectrophotometer at 667 nm.

RESULTS AND CONCLUSION

Adsorbent Characterization

1. BJH Pore Size Distribution Desorption
   - Surface Area = 26.320 m²/g
   - Pore Volume = 0.03 cc/g
   - Pore Diameter = 2.142nm

2. Multi-Point BET
   - BET Surface Area = 16.35 m²/g

3. SEM analysis

The graphenic morphology from the SEM images reveals thin sheets of carbon are protruding outward. Thicknesses of these sheets are in nanometre regime. The physical appearance of the sand changes completely upon carbon loading. The deposition of GC and its growth is visible in SEM images.

BATCH AND COLUMN ADSORPTION

The figure 3 shows TDS vs. Time for reduction of TDS, it indicates that after batch adsorption experiments with different amounts of samples say 0.5g, 1g, 2g to remove total hardness of water with 320 ppm and 565 ppm. It was seen that, for 2g of sample, it removes maximum hardness of the tap water than other quantity of samples. The adsorption capacity of the GSC for tap water is observed at 220 minute and the maximum adsorption rate is 314 ppm. Similarly plots for different amount of samples for different concentration of water were done and is mentioned above. Performance of the material was tested in the column experiment and results are shown in figure 3. The detailed results are shown in the project report.

Figure 3: TDS vs Time for reduction of TDS using 1g/100 ml
Figure 3: Reduction of TDS vs Height of the bed

REMOVAL OF DYE (Methylene blue)

Table 1: Removal of dye from column packed with GSC at different time.

<table>
<thead>
<tr>
<th>Time(min)</th>
<th>% Removal of Dye</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>87.7</td>
</tr>
<tr>
<td>10</td>
<td>86.67</td>
</tr>
<tr>
<td>15</td>
<td>85.56</td>
</tr>
</tbody>
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The above table indicates that the maximum removal of coloured matter is evident from the decolourisation of the filtrate when the column was run with methylene blue. This observation ensures the maximum removal of dye. This adsorbent can also be tested for removing undesirable chemicals, biological contaminants, suspended solids and gases from contaminated water. Thus the above method proves new inexpensive and eco-friendly method for water purification. The large adsorption capacity, green methodology and the availability of the materials across the world enables it to be used in different parts of the world.

FUTURE SCOPES

Further modification and entrapment of GSC with various carbon sources may provide efficient and smart materials/technologies for the treatment of waste water containing various pollutants. Owing to specific advantages in terms of simplicity of the process, design of column adsorber and cost estimation may be done for applying in large scale implementations. Small scale applications may include an addition of small column adsorber in line with RO membrane to reduce the load (hardness) on membrane and increase its life.