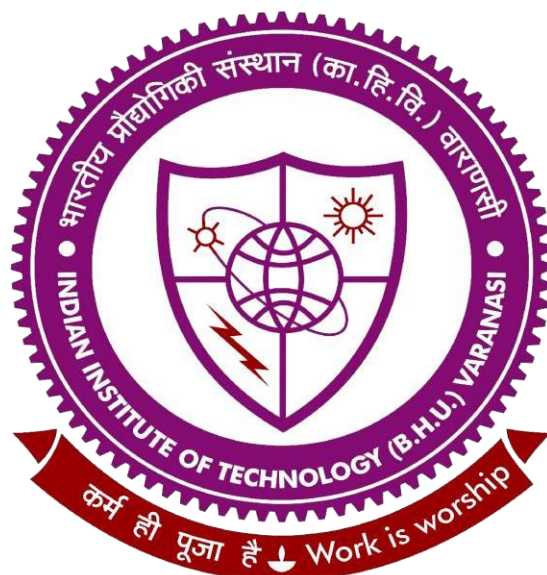


# PROJECT COMPLETION REPORT

## Project Title

**Utilization of low-grade coal for production of high- quality graphene and carbon nanoparticles for energy storage**

(Funded under S&T Grant of MoC)



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**DEPARTMENT OF CHEMISTRY**

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**(BANARAS HINDU UNIVERSITY) VARANASI, INDIA.**

1. Title of the project: **Utilization of low-grade coal for production of high-quality graphene and carbon nano-particles for energy storage.**
2. Project Code: **CMPDI/S&T/CU-59**
3. Date of commencement: **15-10-2022**
4. Approved date of completion (As approved originally) **14-10-2024**
5. Actual date of completion: **14-01-2025**
6. **Objectives as stated in the Proposal**

**I.** To study the formation of different kinds of graphene and carbon nano particles using different grades of coal. Synthesis of soluble versions of graphene Nano sheets with smooth edges and excellent photoluminescence properties.

**II.** Utilization of graphene and carbon nanoparticles synthesized as above for removal and recovery of precious metal ions from acid mine drainage (AMD).

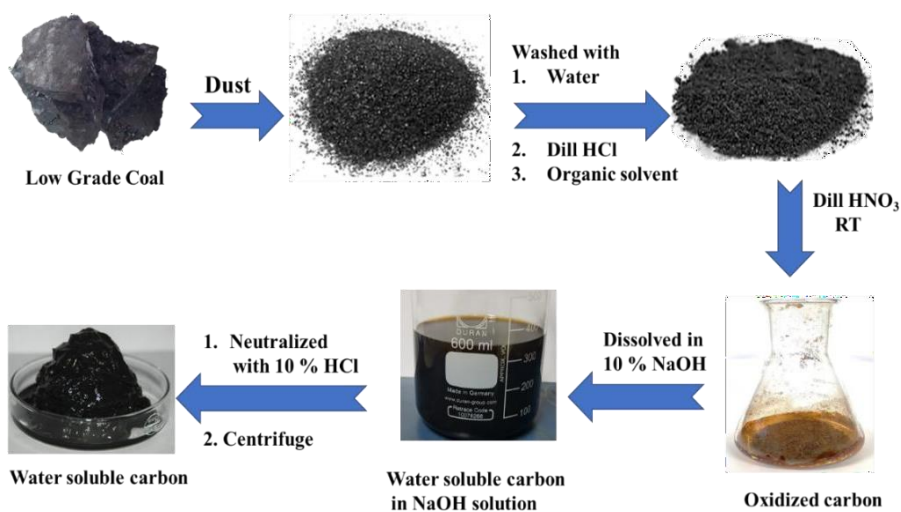
**III.** Utilization of graphene in energy storage devices like super capacitors with high energy and power density.

**Details of the work done during the project run indicating the areas actually covered and as proposed in the original work programme.**

The goal of the proposal was to combine low-grade coal derived high quality graphene, facile synthesis approaches and lessen carbon footprint with aim to achieve energy storage devices with high efficiency. The cost-reduction together with sustainable synthetic approach enabled the large-scale synthesis of graphene, heteroatom doped graphene, carbon nanostructures and their water-soluble version with high yield. During this project, we have used pyrolysis, hydrothermal carbonization or microwave assisted technique for the fabrication of graphene and carbon nanostructures from coal.

**Approach 1:**

The aromatic hydrocarbons and other soluble organic impurities presented in powdered coal were removed by repeatedly washing with organic solvents such as toluene, acetone, and ethanol using Soxhlet extractor. The inorganic oxides and metal impurities were removed by washing with dilute hydrochloric acid and dried in air. The coal powder was treated with dilute nitric acid in portions to avoid excessive heat generation wherein the release of brown fumes started and finally died down. The mixture was allowed to stand overnight at room temperature. Then the mass was dried under a vacuum oven to remove all the excess nitric acid to yield a yellow-brown solid. This material contains oxygen containing functional groups. The sample was further treated with 10% sodium hydroxide solution to get a yellow-brown colored solution. This solution was then slowly neutralized with dilute hydrochloric acid to pH 7 and on standing; a brown colored flaky precipitate appeared. This precipitate was separated out by centrifugation, washed with cold water to free it from sodium chloride formed in the neutralization process and finally dried in air yielding a red brown solid.



**Figure 1.** Schematic representation of synthesis procedure of water-soluble carbon nano structures from low-grade coal.

### **Approach 2:**

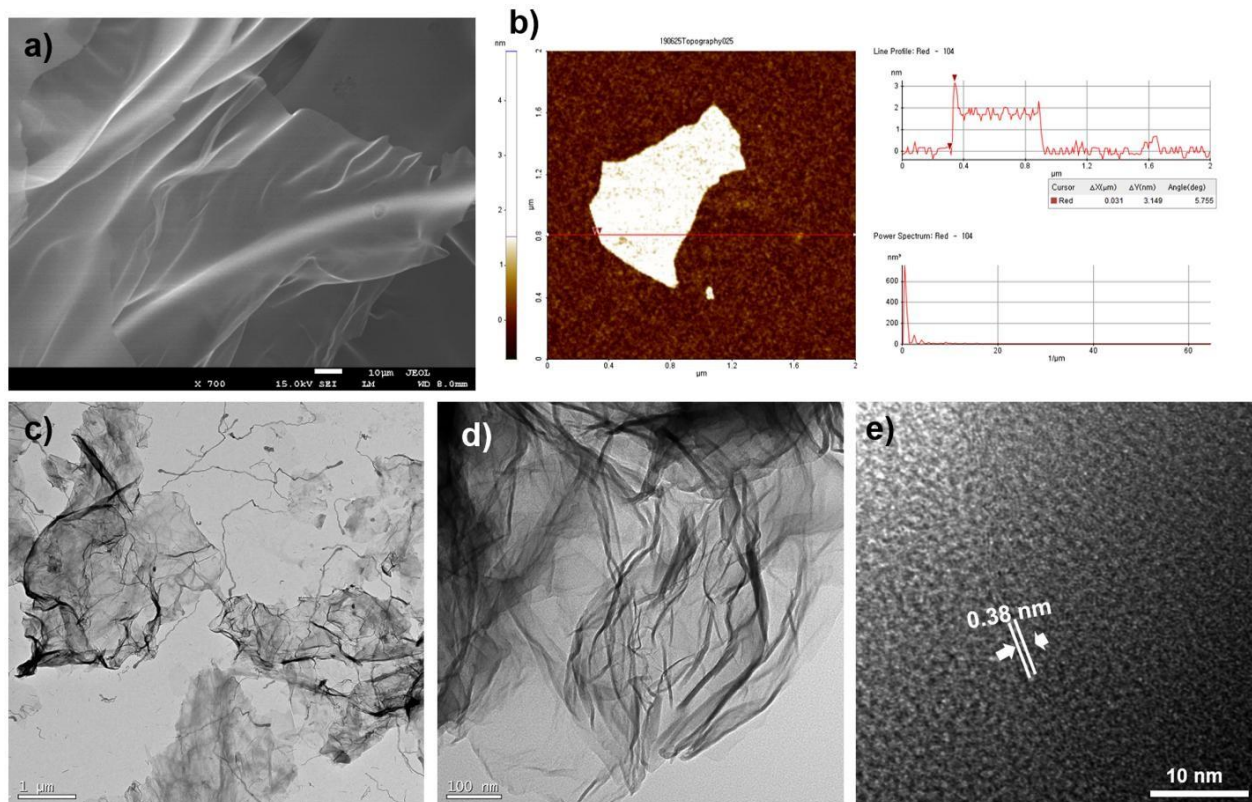
Raw coal powder was washed with dil. Hydrochloric acid to eliminate metallic and inorganic impurities without altering the coal's structure. The acid was removed by continuous washing with DI water. The coal is then annealed at 1100 °C for 2 h in an inert atmosphere to remove volatile impurities and promote graphitization. Furthermore, we synthesized the different samples by varying the quality of coal and the elemental composition of synthesized graphene is shown in **Table 1**.

**Table 1.** Elemental composition of synthesized graphene.

<b>Batch</b>	<b>C (%)</b>	<b>O (%)</b>	<b>N (%)</b>
<b>1</b>	80.24	16.87	2.89
<b>2</b>	88.01	7.50	4.49
<b>3</b>	82.58	16.09	1.33
<b>Average</b>	<b>83.7</b>	<b>13.48</b>	<b>2.9</b>

### **Approach 3:**

Low-grade coal was finely grinded and then sieved with a 90 µm mesh, followed by annealing at 1200 °C for 2 h in a muffle furnace. One gram of the resulting coal powder was treated with 8 g of KOH dissolved in a minimal amount of water for activation. This mixture was then subjected to hydrothermal treatment in an autoclave at 180 °C for 12 hours. The resulting sample was washed with HCl and DI water until reached a neutral pH. Finally, high quality graphene was obtained after freeze-drying for 48 hours. The morphological characterization of as-synthesized graphene is shown in **Figure 2**.



**Figure 2.** Morphological characterization of graphene synthesized from coal. **(a)** SEM image. **(b)** AFM image with line-profile. **(c)** Low resolution TEM image. **(d)** HRTEM image and **(e)** HR- TEM image with lattice fringes.