

## **Tuning the carboxylic groups and increasing the number of defects in graphene oxide nano sheets by changing the oxidation duration of modified Hummers method**

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Graphene Oxide (GO) is a 2-D monolayer/ few stacked layers material synthesized from graphite by introducing oxygen-containing functional groups (carboxylic, hydroxyl, and epoxy) to the graphene sheet. GO has both SP<sub>2</sub> and SP<sub>3</sub> hybridized carbon atoms, which are decorated by oxygen-containing functional groups. GO is extensively investigated in the translational research rather than fundamental research due to the broad range of applications in the synthesis of energy storage devices as fuel cells. The Modified Hummers method is the most frequently used method for the synthesis of GO. In this work, we have used the oxidation duration of the modified Hummers' method to tune the carboxylic groups that are attached to the GO sheets with a higher number of defects because the more COOH groups with higher number of defects could enhance the activity of the GO. Here, grinded vain graphite, KMnO<sub>4</sub> (an oxidizing agent), and H<sub>2</sub>O<sub>2</sub> were used as basic precursors and three distinct GO materials were synthesized by varying the oxidation durations as 24, 48, and 72 hours for GO-1, GO-2, and GO-3. SEM, PXRD, FTIR, and RAMAN techniques were used to examine the qualitative and quantitative variations in COOH bonds in all three materials. According to the observed SEM images, the sheet morphology is visible in all three materials. But, the GO-3 sample exhibits a cloud-like structure with sheet morphology. It suggests that changing the oxidation time affected the number of defects in the materials. Next, the PXRD spectrums were observed to analyze interlayer spacing of GO sheets. Characteristic peaks of PXRD were obtained between 10° and 12°, but the d-spacing value increased with the oxidation duration of the materials. According to the observed FTIR spectrums, the peak intensities relevant to the carboxylic bonds at 1756cm<sup>-1</sup> enhanced with the oxidation duration while all the other peaks remained the same. Stronger vibration relevant to the COOH indicates the production of COOH bonds and GO-3 contains more COOH bonds rather than other two materials. The Raman analysis shows that the intensity of the G and D peaks has grown with the length of oxidation. With the rise in oxidation duration, the FWHM (full width half maximum) of band G, which indicates the presence of SP<sub>3</sub> bonding in the materials have also increased. Similarly, the oxidation duration affected the nature of the D band which confirmed the increment of number of defects with oxidation duration. In conclusion, the oxidation duration of the modified Hummers' method directly influences the formation of COOH bonds, and the number of COOH bonds increase with oxidation duration.

**Keywords:** Graphene oxide, Carboxylic tuning, Modified Hummers method, Defects rich GO

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