Monitoring chemical reduction of graphene oxide using pulsed amperometric waveform in ion chromatography

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Hydrazine is commonly used as a reducing agent for the synthesis of reduced graphene oxide (rGO) [1-3], which has attracted researchers across many fields, as chemically reduced rGO exhibits unique properties (e.g., flexible conductor [4], anomalous quantum Hall effect, high charge carrier mobility [5], and high impermeability to gases [3]) and offer numerous applications (e.g., supercapacitors [1], high-strength low-weight nanocomposites [4], and photovoltaic devices to name a few [6]. An exact measurement of consumption of reductant (i.e., hydrazine) during reduction of GO and the point of complete reduction are essential, as such measurement provides a better insight into GO reduction kinetics (i.e., determining reaction rate and order). This is also important as the exact mechanism of GO reduction is still unknown [3]. The consumption of hydrazine during the reduction of GO was commonly measured with UV-Vis absorption spectroscopy. However, the existing mechanisms [7-9] was reported with high detection range (micro or millimole), requirement specific colorant reagent in indirect spectrophotometry, complex derivatization process in chromatographic procedures [7], dependence of GO functional groups on small range of UV $\lambda_{max}$ from 230 nm to 270 nm [8], and significant interference from excess ammonia in spectrophotometry [9]. In this study, we overcame the associated drawbacks of existing mechanisms [7-9] by using pulsed amperometric detection (PAD) technique for the first time and accurately measured consumption of reductant and reaction end point. The reducing agent concentration was analysed at regular intervals (5- and 10-min) through the ion chromatographic platform to a miniaturised EC detection system for the determination of complete reduction of GO ca. 30 min. Significantly, the monitoring of reductant consumption during GO reduction can offer various advantages, such as ease of quantifying the different stages of rGO, unequivocal determination of the reduction endpoint, percent yield, and new insights into the GO chemistry.

References