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Microemulsions as an alternative approach for analytical determination of graphene oxide's reduction degree

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Abstract

A novel method for quantitative determination of reduction degree of graphene oxide (GO) that combines simplicity and rapidity with analytical performance is presented. This approach relies on the effect of the analyte to the thermodinamic stabilization of microemulsions (MEs). Such phenomenon was expressed by the minimum volume fraction of amphipile required (in this case, the analyte of interest GO) to form ME.

Key words: *Microemulsion, graphene oxide, amphiphile*

Introduction

Analytical platforms for rapid tests are part of an important current research field that aims to perform in situ measurements. Currently employed techniques are often complex and expensive to assess the degree of reduction of GO suspensions such as XPS (X- Ray Photoelectron Spectroscopy) and Raman Spectroscopy. Technology presented in this work is attractive because is cheap, fast, portable, bypassing any qualified operators.

The microemulsification (MEC) phenomenon is related to thermodynamic stabilization of dispersions or emulsion, to obtain ME. This effect was expressed by the minimum volume fraction of amphiphile (AP) required to form ME, which can be measured simply by visual inspection (direct observation)[1]. This is possible because the dispersions change from cloudy to transparent during the ME formation in similar way to titration (*Figure 1*). For the formation of the ME, in this case, the GO plays the role of AP compound, and the turning point will be directly proportional to the degree of reduction of GO and its influence as surfactant compound in the emulsion between the aqueous phase and the organic solvent.

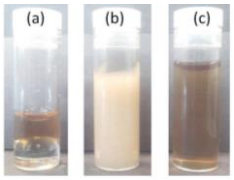

Figure 1. Procedure for obtaining the ME. In (a) biphasic mixture with an aqueous GO suspension (upper) and organic (lower) phase before vigorous stirring ; in (b) emulsion formed after vigorous shaking and (c) ME translucent obtained after the turning point .

Recently, Jaemyung and coworkers[2] demonstrated the use of GO as surfactant compound in the formation of oil/water emulsions, and dispersing hydrophobic compounds in the aqueous media, due to its amphiphilic character. GO hydrophilic propertie comes from the presence of oxygenated groups formed during its synthesis by chemical oxidation of graphite, followed by exfoliation. On the other hand, its hydrophobic character comes from the presence of sp^2 carbon domains at the remaining basal plane of graphite, which ensures its amphiphilic character. This feature, combined with the possibility of restoration of conductivity and control the hydrophobicity of this material through its reduction, make this very attractive material.

Results and Discussion

Initially, assays were carried out in the absence of GO in order to obtain the blank for comparison. The diagram constructed for the composition of the obtained ME is shown in *Figure 2.* MEC experiments were conducted by adding ethanol to W-O mixtures with the aid of micropipettes.

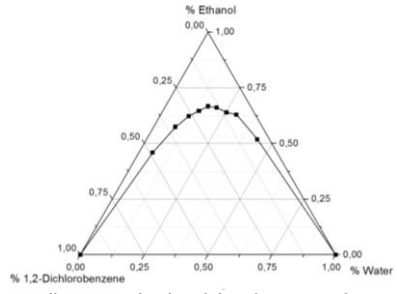

Figure 2. Ternary diagram obtained for the ME. The experimental points show the turning point for water-1,2-dichlorobenzene-ethanol composition, in% v / v.

MEC relies on the effect of the analyte content over the Gibbs free energy of dispersions by modifying the surface area and/or interfacial tension. Mathematically, this effect was expressed by the minimum volume fraction de AP necessary to form ME. As a further step, it was selected one point of the curve (*Figure 2*) with a well known composition of the aqueous phase containing GO, organic phase and also ethanol, which was used as a co-surfactant to help controlling the surface tension of the obtained ME. Preliminary results have shown that the proportion of GO is related to the final composition of the ME obtained, indicating the possibility of assessing the degree of reduction of GO. *Figure 1c* shows the first ME in the presence of GO, in the proportion of 50% to 50 % organic phase GO (1.16 mg mL^{-1}) in the aqueous phase.

Conclusions

A new strategy for quantitative determination of the reduction degree of GO is reported in this work. The MEC represents an important contribution for the development of rapid test platforms taking into account rapidity, and simplicity and no sophisticated instrumentation or equipment is required. To overcome the problem of the organic solvent, it intended to carry out further tests with oleic acid as a substituent.

Acknowledgement

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