TUNING GRAPHENE PROPERTIES BY A MULTI-STEP THERMAL REDUCTION PROCESS

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The chemical methods for producing graphene materials, via the formation of graphite oxide which must be subsequently exfoliated and reduced to obtain the final graphene, are among the most valuable due to their simplicity and easy scalability. The characteristics of the graphene materials obtained (rGO), which will determine their applicability, are greatly affected by the experimental conditions.[1,2] In any case, the use of single-step thermal treatment does not allow the properties of the materials obtained to be tuned as once the final temperature has been fixed, the resulting properties (C/O ratio, BET surface area, processability into suitable electroders, etc.) are also fixed.

The aim of this work is to design a route for the preparation of rGOs of enhanced BET surface area as well as maximizing their suitability as electrodes in energy devices. For that, a GO obtained by a modified Hummers method was thermally exfoliated/reduced at 700 and 1000ºC by two single step procedures (ramp and flash pyrolisis) and by a novel multi-step procedure. The ramp pyrolisis in a single step gave rise to rGOs with low BET surface areas (~200 m2g-1) which are easily conformed into stable electrodes (Figure 1a). In contrast, by flash pyrolisis in a single step, high BET surface areas were obtained (~500 m2g-1) but it is not possible to conform them into stable electrodes (Figure 1b).

By using the multi-step procedure developed herein it is possible to prepare rGOs that have an increased BET surface area (compared to that of the material prepared by the single-step ramp-heated material) and that are easily conformed into stable electrodes for electrochemical energy storage devices (Figure 1c). SEM images of the rGOs indicate the formation of tridimensional structures derived from the expansion of the graphite oxide on heating, which is the factor responsible for the development of porosity in these graphene materials. In the case of the flash-pyrolized sample (Figure 2a) cavities with sizes more in the range of mesopores are observed, contributing to an increment in the BET surface area and to the loss of suitability as electrodes. These structures are not so abundant in the ramp-heated sample, which explains its lower BET surface area (Figure 2b). An intermediate situation is obtained by the multi-step procedure, which explains the high BET surface area and good suitability as electrode of these materials.

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References

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[2] Botas C et al. Carbon **50** (2012) 275.

Figures

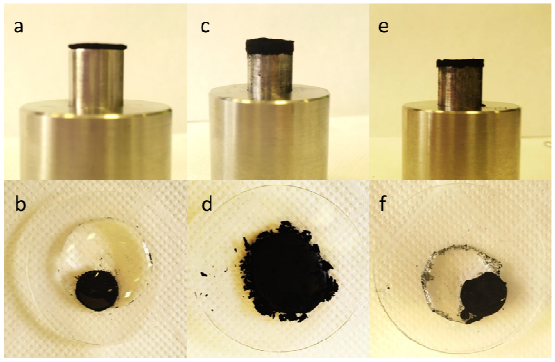

Figure 1: Electrodes from rGO obtained by ramp single steps: ramp (a,b) and flash (c,d) and multi-step (e,f)

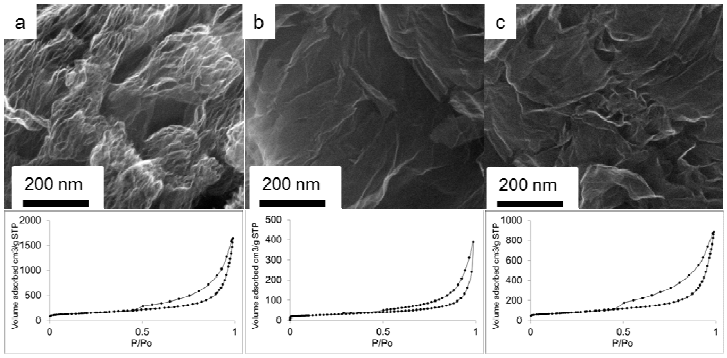

Figure2: SEM image (up) and BET curves (down) of rGO obtained by ramp single steps: ramp (a) and flash (b) and multi-step (c)