J. de D. López-González, A. Martín-Rodríguez, A. Martín-Pozuelo and F. Rodríguez Reinoso.

Departamento de Química Inorgánica. Facultad de Ciencias. Universidad de Granada. (Spain).

## INTRODUCTION

During the process of formation of graphite oxide from any graphite, there is an alteration of the graphite structure, leading to a separation of the graphite lamellae, the original basal spacing in graphite (3,35 Å) changing to about 6,3 Å in graphite oxide. The basal spacing in graphite oxide will depend on the degree of oxidation of the original graphite and on the amount of water retained between the lamellae. The mechanism of graphite oxide formation leads to the fact that graphite oxide develops most of its surface area in internal regions of its structure. From the adsorption point of view, the interlamellae regions play the role of submicropores (1) as it happens with many other adsorbents (2). These internal regions of graphite oxide are not accesible to non-polar molecules adsorbed at very low temperatures (3), since the interlamellae spacing must increase in order to facilitate the adsorption of these molecules. However this increase in the basal spacing may take place when retaining polar molecules (4).

In this work, the adsorbates used have been ethylene-glycol, methanol and ethanol, and the adsorption temperature 25°C. The graphite oxides (prepared by the Staudenmaier method (5)), have been obtained from different graphites: natural based graphite (N), artificial graphite (A) and pyrolytic graphite (P).

## RESULTS

## 1. Adsorption of ethylene-glycol

The adsorption of ethylene-glycol has been studied in a conventional gravimetric system using silica springs balances. The samples studied have been graphite oxides from the different kinds of graphite mentioned above, attacked from 1 to 5 days.

The adsorption of ethylene-glycol by the different samples has been studied first at relative pressure p/p =1, followed by the desorption at 10<sup>-3</sup> torr. The adsorption and desorption runs have been repeated several times, and it has been found that both processes are not reproducible although the differences are small in the samples from graphites N and A (both poly crystallines), as it can be seen in Fig. 1 for the oxide from natural based graphite. However, the differences are very large for those graphite oxides from pyrolitic

graphite.

On the other hand, the irreversibility is much larger for the adsorption runs of a given graphite oxide, than for the corresponding desorption runs as it can be deduced from Fig. 2, in the case of graphite oxide from pyrolytic graphite.

From the experimental results of adsorption of ethylene-glycol, and considering 22  $\mbox{\Bar{A}}^2$  (4) as the cross-sectional area of the adsorbate in this case, the interlaminar surface areas of the graphite oxides have been calculated giving values from 1500 to 1650  $\mbox{\Bar{m}}^2$ . which are of the same order of the theoretical interlaminar surface area of graphite oxide obtained following different experimental methods (4).

In any case, and according to the interlaminar surface area determined by the retention of ethylene-glycol on samples attacked during 5 days, the order of such area is A > P > N.

2. Adsorption of methanol and ethanol

The adsorption of these alcohols has been followed in a gravimetric system with mercury cutt-offs and using silica spring balances. In this case samples of graphite oxide studied are those resulting from a 5 days attack on the different kinds of graphites. The adsorption isotherms of methanol on the three graphite oxides can be seen in Figure 3 in which, for simplicity, desorption isotherms are not shown. The adsorption of ethanol follows the same pattern as methanol.

The adsorption of methanol and ethanol shows also the same pattern as the adsorption of ethylene-glycol in the sense that the order of adsorption is A>P>N. In all cases the calculated monolayer of methanol or ethanol adsorbed ranges from 20 to 30 mg  $\cdot$  g<sup>-1</sup>.

The desorption after saturation of methenol and ethanol leads, in all cases, to hysteresis loops indicating that the desorption process is taking place in a different way to the adsorption of the adsorbate. The oxide from pyrolytic graphites gives a large hysteresis loop but those from artificial and natural based graphites are small. This indicates that in the former oxides there is a large retention of adsorbate after desorption.

At the present moment the electron diffraction of the different oxides and the X-ray diffraction study of the graphite oxideadsorbate complexes are been carried out

## REFERENCES

- (1) FRIPIAT, J.; CLAUSSIDON, J. and JELLY, A.; "Chimie Physique des Phenomenes de Surface". Masson & Cia. Paris (1971).
- (2) GREGG, S.J.; "Surface Chemistry of Solids". Chapman and Hall, London (1965).
- (3) LOPEZ-GONZALEZ, J.D.; BAÑARES MUÑOZ, M.A. AND RAMIREZ SAENZ, A.; Química e Industria, <u>17</u>, 4 (1971).
- (4) MARTIN RODRIGUEZ, A.; LOPEZ-GONZALEZ, J. D. and DOMINGUEZ VEGA, F.; Anales de Química, <u>69</u>, 909 (1973).
- (5) STAUDENMAIER, L.; Ber., <u>31</u>, 1481 (1898); <u>37</u>, 1394 (1899).

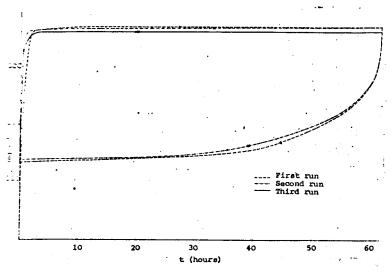


Fig. 1

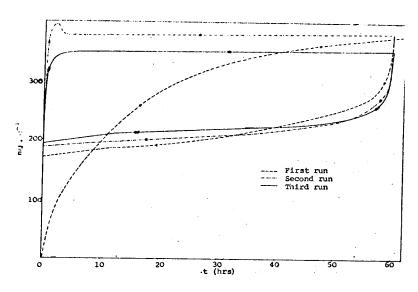


Fig. 2

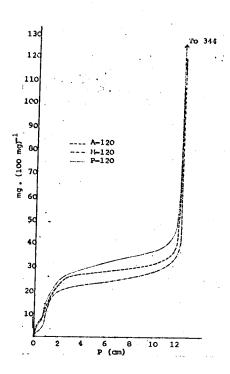


Fig. 3