

CHAPTER - 10

CONCLUSIONS AND SCOPE

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10.1 CONCLUSIONS

The present research work on the fluoride based graphite intercalation compounds with special reference to solvent effects has definitely led to some interesting conclusions. These were presented at the end of each individual units of investigations (Chapters 4-9) earlier. However, it would be worthwhile to present a broad outline of the overall conclusions of the present work, here.

In aqueous HF solutions, glassy carbon is very stable. It does not undergo any significant damage in the cyclic voltammetric time scale. Fluoride intercalation also does not proceed on this surface. The electrode contains some redox surface functions. Fresh oxidation of the glassy carbon surface occurs beyond 2.0V. The surface oxidation in these potential ranges are independent of HF concentration and are also highly irreversible in nature (Chapter 4).

Compared to the above behaviour of Glassy Carbon, graphite electrodes are much more active in HF solutions. Graphite electrodes are surely prone to chemical attack to some extent in strong HF solutions. This is confirmed by the dependence of redox peak currents in the middle potential region on the HF concentration. Fresh graphite oxidation is generally initiated around 1.4V itself on graphite. Fluoride intercalation also is initiated around this potential

region. With increasing HF concentration, the intercalation predominates over graphite oxide formation as indicated by increasing proportion of deintercalation charges. Cyclic voltammetric as well as double-potential step chronoamperometric experiments support this view. However, oxygen evolution is still a strong competitive process for graphite intercalation even in 20.0M HF. Surface degradation is also higher in this medium (Chapter 5).

Methanol-water mixture seems to be a better solvent system for intercalation/deintercalation process. Methanol adsorption on the electrode surface seems to prevent surface oxidation and oxygen evolution, thus favouring higher fluoride intercalation. The surface modification is also found to be more uniform in this medium (Chapter 6). DMF-water mixed solvent system prevents the surface oxidation of graphite to a significant extent. But this system also retards fluoride intercalation significantly (Chapter 7). Dioxane-water solvent system seems to favour oxygen evolution considerably. This leads to very poor overall charge recovery in this solvent system (Chapter 7).

In strong HF solutions, other anions present in the solvent system at much lower concentration levels can

also co-intercalate during the fluoride intercalation process. In the case of ferricyanide, such co-intercalation leads to two new redox peaks on either side of the diffusion controlled redox peaks of the ferricyanide species present in solution. These peaks may be connected with the protonation/deprotonation equilibria of intercalated species or with the formation of entirely new compounds like prussian-blue. Iodide and bromide ions also co-intercalate along with fluoride into graphite lattice. But the intercalated species also show redox responses in the potential region corresponding to the redox species in solution. Comparison of the co-intercalation behaviour of these three anions also suggest that the efficiency of co-intercalation increases with decreasing solvation of the ionic species involved (Chapter 8).

The stability of the graphite electrode during intercalation/deintercalation cycles in 15.0M HF aqueous-methanolic solutions can be further improved by forming a thin polypyrrole layer on graphite surface. If the intercalation/deintercalation time is kept low, the charge recovery remains above 80% even after 10 charge/discharge cycles. Forming a polypyrrole layer on a fluoride intercalated graphite electrode however did not have any favourable impact.

10.2 SCOPE FOR FURTHER WORK

The present work mainly opens up a new dimension in the field of electrochemical intercalation studies, namely, the solvent effect. The systematic investigations presented here clearly shows that solvent can play a significant role both in stabilising the intercalating surface and in improving the intercalation efficiency. In the fluoride intercalation of graphite itself only three mixed solvents namely methanol-water, DMF-water and dioxane-water systems were comparatively evaluated in this work. There is considerable scope for studying other solvent systems. It may also be worthwhile to study electrochemical intercalation in complete absence of water. This can be done for example by directly mixing anhydrous HF and the solvent of choice in required proportions. If, for example, methanol influences intercalation mainly through adsorption, presence of trace levels of methanol in anhydrous HF itself, can bring about such changes.

Extending the solvent effect studies to other intercalants like sulphate, nitrate and fluorosulfonate species is an obvious possibility. By judicious choice of the solvent, fairly efficient intercalation systems with considerable long-term stability may certainly be developed

in future. In this sense it may be worthwhile to reinvestigate many conventional systems in addition to taking up newer systems.

The new directions taken up in Chapters 8 and 9 are indeed very interesting. In these lines, the results presented here are quite preliminary in nature. Co-intercalation of redox anionic species along with the main intercalant for example opens up an entirely new potential field. If these electrodes where redox currents at different lower potential regions can match with intercalation/deintercalation currents at more anodic potential regions, and the stability of the whole system can be improved further, one can envisage many new applications for such systems. A number of such cointercalation systems can be visualised and investigated in considerable detail.

Forming a conducting polymer based protective layer on graphite for intercalation is another new potential area where considerable scope exists for further research. A variety of conducting polymer films can be prepared and their ability to protect the surface without blocking intercalating anions may be investigated. The very nature of the conducting polymer film on these electrodes and the ion-transport mechanisms through these films themselves are questions of considerable interest.

So, in conclusion, it may be said that there is a beginning at every end. The present investigation has led to some interesting conclusions. At the same time it has raised some more interesting questions and opens up scope for further investigations.