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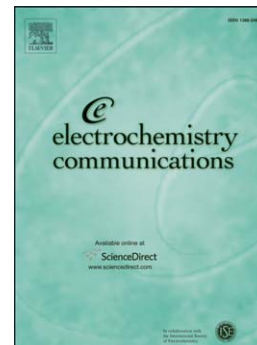
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Surface modification by electrochemical reduction of alkyldiazonium salts

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ABSTRACT

Since alkyldiazonium salts are highly unstable, their grafting is not as easy as that of aryldiazonium salts. However, it is possible to graft alkyl chains on copper electrodes by the *in situ* reaction of alkyl amines dissolved in aqueous perchloric acid with nitrite ions electrogenerated from nitrate ions.

Key words: alkyldiazonium salts, copper surface, electrochemical reduction, sodium nitrate.

1. Introduction

Aryl diazonium salts are involved, for example, in the important Sandmeyer and Meerwein reactions; more recently they have been used as a source of aryl radicals to modify the surface of various materials [1-3]. They can be prepared from aryl amines by a variety of methods either in acidic aqueous solutions with NaNO_2 , or in aprotic media by using isopentyl nitrite or nitrosonium ions. For surface modification experiments, they can be isolated or prepared *in situ* [4] and reduced either in aprotic solvents or aqueous solutions (from acidic to basic). The amines can be prepared directly in the grafting solution by electrochemical reduction of the nitro derivative [5]. The nitrite reagent can also be prepared directly in the grafting solution by electrochemical reduction of nitrate on a copper surface [6].

Conversely, alkyl diazonium salts are “extremely unstable even in solution” and are “useless for preparative purposes” [7]. Once formed, they are transformed immediately into carbocations and nitrogen and the reaction products are usually a mixture containing alcohols, alkenes, alkyl dimers and N-nitrosoamines [8-11]. However, under specific conditions, $\text{CF}_3\text{CH}_2\text{N}_2^+$ was obtained by adding trifluorodiazethane to fluorosulfonic acid (FSO_3H) in deuterated chloroform (confirmed by NMR measurements at $-60\text{ }^\circ\text{C}$) [12-13].

The high instability of alkyldiazonium salts is the main reason why the electrografting of alkyl groups on surfaces by reduction of alkyldiazonium salts has not been achieved until now [14]. Herein, we demonstrate the possibility of attaching alkyl groups on a copper surface by

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