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Investigation of Electrode Metals and Supporting Electrolytes in the Electrochemical Polymerization of Acrylamide

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Abstract **Original Research Article**

The electrochemical polymerization of acrylamide in N, N-dimethyl formamide was carried out using electrode of different metals and with different supporting electrolytes. Maximum polymer yield was obtained when Zn metal was used as an electrode. Similarly, among the different tetraalkylammonium or phosphonium halide used as the supporting electrolyte, the yield was maximum with tetrabutylammoniumhexachloroantimonate. The effects of electrode metals and nature of supporting electrolytes on the polymer yield was investigated.

Keywords: Acrylamide, Electrode metal, Supporting electrolyte, Electrochemical polymerization.

Introduction

Both the electrode material and the supporting electrolyte are critical parameters in electrochemical polymerization. Their careful selection and optimization are essential to control the polymerization process and obtain polymer films with desired properties for specific application. The electrode material significantly affects the electrochemical process through several factors such as electron transfer kinetics[1-2], nucleation and growth of polymer films[3], electrocatalysis[4]. Similarly, supporting electrolyte also plays a vital role in electrochemical polymerization by increasing solution conductivity, facilitating ion transport, doping[5] and affecting the electrochemical double layer[6].

Materials and Experimental Methods

Materials

N,N-dimethylformamide (DMF) was purified by fractional distillation. Acrylamide (AA) and supporting electrolytes were of analytical grade and used without further purification.

Polymerization Method

The electrochemical polymerization of AA in DMF with different supporting electrolytes such as $(CH_3)_4NCl$, $(C_4H_9)_4NI$, $(C_4H_9)_4NSbCl_6$ etc. was carried out in an H-type sintered cell using different metal electrode like Pt, Zn, Cu, Al, etc. During electrolysis, the locus of polymerization was in the cathode

compartment. The polymerization initially furnishes a homogeneous medium and becomes heterogeneous with the increase of polymer conversion. The polymer yields were determined gravimetrically after precipitation in acetone.

In order to investigate the effect of electrode materials on the polymer yields, the polymerization have been carried out at different metal electrodes and the results are summarized in the table 1. Under the same experimental conditions, superior polymer yields were obtained at Zn than the Pt electrode. Following polymerization rate sequence is obtained for different metal electrodes

Zn > Fe > Pb > Cu > Al > Pt.

Table 1. Effect of different electrode metals on the polymerization of acrylamide (AA) (2.02 mol. L^{-1}) in the Dimethylformamide (DMF) solution of Tetrabutylammonium iodide (TBAI) (0.271 mol. L^{-1}) at 20mA and 25°C.

Electrode Metal	Polymer Yield (%)	Colour of Catholyte
Zn	70.00	Yellow
Fe	65.50	Yellow
Pb	62.16	Colourless
Cu	60.30	Blue
Al	55.35	Colourless
Pt	52.15	Yellow -orange

The adsorption and catalytic activities of the electrode metals, nature of impurities, hydrogen or oxygen overvoltage, the physical state of the electrode may significantly affect the polymerization process. It is also assumed that the electrode surface of the reactive electrodes may change structure during the electrolysis. The electrode materials may react chemically with AA and cations of the corresponding metals may be discharged. Consequently, negative potential of the cathode would counterpart the dissolution of the metal. The absorption and /or catalytic activity of the electrode may also account for the observed differences in the rates.

Similarly, supporting electrolyte has also a significant role in the electrochemical polymerization. It furnishes the conducting medium for passage of electric current. In many cases, it also provides catalytic species which may be free radical or ionic in nature. It also gives necessary counter ions associated with growing active chains and thus the propagation reaction may be significantly affected by the variation of counter ions. In many cases,

supporting electrolyte may also act as a terminating agents[7,8]. Therefore, some supporting electrolytes in regard to their suitability in the electrochemical polymerization of AA had been tested and pertinent results are summarized in Table 2.

Table 2. Effect of different supporting electrolytes (0.271 mol L^{-1}) on the polymerization of acrylamide (AA) (2.82 mol L^{-1}) in Dimethylformamide (DMF) at Pt electrodes. Electrolysis time = 120 min; Catholyte vol. = 12.5 mL at 25 °C.

Supporting Electrolyte	Polymer Yield (%)
$(CH_3)_4NCl$	42.60
$(C_4H_9)_4NI$	52.15
$(C_2H_5)_4NSbCl_5$	55.50
$(C_4H_9)_4NSbCl_6$	60.30
$(CH_3)_4NNO_3$	50.00
$(C_4H_9)_4PI$	35.40

Collins et. al. [9] examined the influence of tetraalkylammonium and tetraarylphosphonium cations on the rate of polymerization in the catholyte. A higher yield with phosphonium salt was obtained in the same electrolysis time even though there was only one-tenth as much as phosphonium salt as the ammonium salt. Contrary to this, in the present work, slightly higher polymer yield was obtained with tetrabutyl salts. On the basis of an intimate ion pair model and the cathodic potentials of the salt, the following propagation mechanism has been advanced.

Propagation with alkylammonium salts

$AA^- (C_4H_9)_4N^+ + e^- \rightarrow$ No cathodic reduction of cation

(reduction potential : - 2.4 V vs SCE) - (i)

$AA^- (C_4H_9)_4N^+ + AA \rightarrow AA_{(n+1)} (C_4H_9)_4N^+$ -(ii)

Propagation with phosphonium salts

$AA_n^- P(C_6H_5)_4 + AA^{\bullet} \rightarrow AA^-_n + P(C_4H_5)_3 + C_4H_5^{\bullet}$

(reduction potential : - 1.20 V vs Hg pool) -(iii)

$AA^- P(C_6H_5)_4 + AA^{\bullet} \rightarrow AA^-_{n\,H} P(C_6H_5)_4^+$ -(iv)

$AA^- + AA \rightarrow AA^-_{(n+1)}$ -(v)

The reduction of the cation in reaction (iii) results in the formation of polymeric free ion as in reaction (v), which might have been responsible for the observed large increase in the overall rate of the electrochemical polymerization with the aromatic phosphonium salt. No such free ion appears to be generated

electrically in the case of alkyl quarternary phosphonium cations.

Conclusions

Electrode metals and supporting electrolyte have the active role in electrochemical polymerization. Adsorption and catalytic activities of the electrode metal significantly affect the polymerization process. Similarly, the supporting electrolyte not only makes the solution electrically conducting but it may provide catalytic species for polymerization.

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