

ELECTROSYNTHESIS OF POLYVINYL ACETATE*M CHANDRASEKARAN, S PITCHUMANI and V KRISHNAN*

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An electrochemical route for the preparation of polyvinyl acetate is found feasible wherein the polymerization proceeds through electro-reduction. The use of different cathodes such as lead, lead amalgam and mercury has given different yields which are also influenced by other electropolymerization conditions. The use of biphasic system is found to be not helpful in isolating the polymer in appreciable yield.

Key words:- Polyvinyl acetate, electropolymerization, reduction

INTRODUCTION

Polyvinyl acetate is one of the important raw materials for many of the industrial applications such as surface coating [1], adhesives [2] and binders [3]. Although chemical method of synthesising this polymer is well known, active interest has been shown in working out electrochemical route for the synthesis of polyvinyl acetate [4,5]. An electropolymerization route has already been reported [6] and the extension of this methodology using cheaper electrolytes has been reported in this paper.

EXPERIMENTAL**Materials**

Vinyl acetate monomer was distilled prior to polymerization. H_2SO_4 has been used as such. Lead, graphite and mercury were used without any pretreatment or purification. Lead amalgam electrode was prepared by electrochemically depositing mercury over lead [7].

Electrosynthesis

The polymerization was conducted in an undivided two electrode cell assembly wherein lead was used as anode and lead, or lead amalgam or mercury was employed as cathode. The cell containing monomer and electrolyte was impressed with a calculated quantity of current which initiated the polymerization at $303 \pm 1K$ under N_2 atmosphere. The product was isolated directly around the cathode and washed with suitable solvent to remove occluded monomer. In biphasic system, benzene was added slowly to the cell to dissolve the polymer formed around cathode. The product was identified through IR spectral analysis.

RESULTS AND DISCUSSION

It has been found that polyvinyl acetate is readily formed around the cathode. The use of lead and graphite does not yield polymer whereas the use of mercury and lead amalgam cathodes facilitate the formation of polyvinyl acetate. At lead-amalgam cathode, the polymer formed has been found to be sticking around the cathode. It is found that at 1:7 ratio of monomer to electrolyte, polyvinyl acetate is formed in appreciable yield. The use of higher acid con-

centration facilitates the formation of acetaldehyde as a byproduct along with polyvinyl acetate and consequently reducing the yields of polymer considerably. However good yields are obtained when higher current densities are used (C.D. = 0.2 - 0.4 $kA.m^{-2}$). The maximum obtainable yield in this preparation is 50%. The reduction in yield can be accounted for the fact that polyvinyl acetate formed readily sticks to the electrode surface due to its tacky nature. This has been further realized by the increase in cell voltage.

When more and more polymer is formed around the cathode it becomes ineffective in helping the polymerization process to proceed continuously. An ideal synthetic recipe in these conditions for the preparation of polyvinyl acetate is the following:

At a current density of 0.4 $kA.m^{-2}$, a ratio of 1:7 for monomer to electrolyte yields a maximum 50% of polyvinyl acetate. The polymerization has a current efficiency of 50% with cell voltage remaining at 5 to 5.5V. The temperature of the electrolytic bath during the course of polymerization increased to 323K which warrants the use of ice cold water circulation through the electrolyte.

The product is identified by the absence of characteristic IR bands due $-C = \bar{C}$ at 1650 cm^{-1} which indicates the polymerization has proceeded through electrochemical activation of the double bond of the vinyl acetate monomer.

In order to avoid "spoiling" of cathode due to the adherence of the resultant polyvinyl acetate, the use of biphasic system by adding benzene during polymerization has been attempted. Although the polymer formation is seen in such experiments, the yield is considerably lower than that obtained in the previous experiments reported above. This may probably be due to the fact that unreacted monomer gets into the solvent thereby creating non-availability of the monomer for further polymerization.

Fig. 1 shows the determination of intrinsic viscosity from which the molecular weight of polyvinyl acetate thus prepared is deduced as 5.24×10^4 which is in agreement to the value got in conventional solution-polymerization of vinyl acetate.

CONCLUSION

An electrochemical route for the preparation of polyvinyl acetate using a cheaper electrolyte is found feasible and the ratio of monomer to electrolyte decides the yield of polyvinyl acetate.

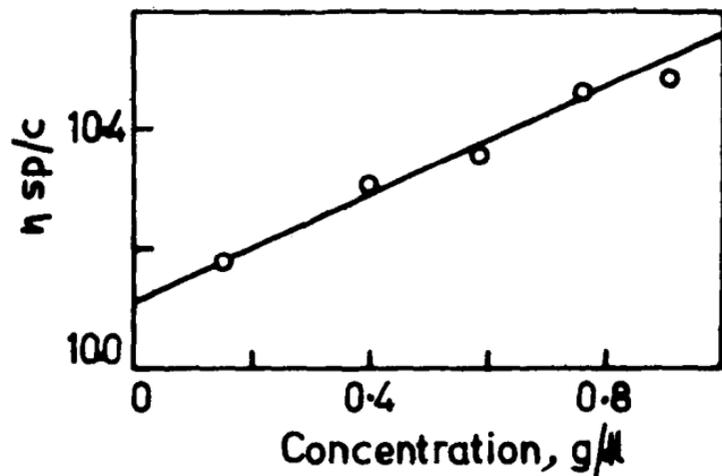


Fig.1: Determination of intrinsic viscosity (η) for polyvinyl acetate

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