

ELECTROGRAFTING OF POLYACRYLAMIDE ON TO CARBON FIBRE

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[Received: 1987 July; Accepted: 1987 December]

An *insitu* electropolymerization technique is extended to effect graft copolymerization on to carbon fibre matrix. It has been found to be possible to graft polyacrylamide on to carbon fibre through cathodic polymerization. The presence of grafted polymer on to carbon fibre matrix has been confirmed by weight increase of the product, scanning electron micrographs and IR spectral data. Conditions of electropolymerization have been found to influence the course of grafting reactions thus leading to the utility of this process in the modification of carbon fibre matrix for different applications.

Key words: Electrografting, electropolymerization, polyacrylamide

INTRODUCTION

Grafting of a polymer chain on to another is one of the important steps in the modification process of polymer science, as it tends to generate totally different products with the well defined properties for selective applications through the judicious choice of the polymer moieties. Hitherto, such modification has successfully been demonstrated both chemically [1] and photochemically [2]. Effecting a grafting reaction on to the polymer chain through electrochemical process is a novel and new methodology and is expected to be useful when polymer itself is used as electrode. In this investigation, the carbon fibre has served as the basic polymer matrix to demonstrate the electrochemical route, since it is electrically conducting. It has also been found useful in a number of electrochemical applications [3-5] as well as composite systems [6-7]. In both the applications, carbon fibre requires to be modified for effective use and particularly in composite systems, the introduction of another polymer on to the carbon fibre matrix enhances the mechanical properties and prevents possible electrical hazards. With such potentialities of the basic material, the interphase modification of carbon fibre becomes a prerequisite and in this paper the use of electropolymerization is highlighted as a viable methodology of modification process.

EXPERIMENTAL

Fabrication of a carbon fibre electrode

Different types of electrolytic cell arrangements were possible and it was found that both undivided and divided cell could be used for the electropolymerization. Carbon fibre electrode was placed in the central part of the cell and two platinum counter electrodes were placed on either side. Positioning of carbon fibre electrode in the centre at equal distance from the other two platinum electrodes is vital to get the product as uniform coating. The electrolytic bath contained acrylamide and dilute H_2SO_4 as electrolyte. The carbon fibres used as electrode were cut to 10cm length from the tows and tied at both ends with a cotton thread to prevent fibre loss during polymerization, washing extraction and drying. The fibre electrode could be held either between two stainless steel windowed discs with proper insulation or between two teflon spacers together with alligator clip. Both arrangements have required electrical contact with the individual fibre filaments.

Polymerization

Electropolymerization was conducted by impressing a well defined current from a rectifier. Cell current and cell voltage were monitored throughout the polymerization runs.

Product isolation

After polymerization, the carbon fibres were rinsed with distilled water to wash away the monomer acrylamide and electrolyte and then dried under vacuum. Weights of carbon fibre both prior and after electropolymerization were determined. The dried carbon fibre impregnated with polymer was extracted in double distilled water and after drying, carbon fibres were reweighed to calculate the amount of grafted product.

Characterization

Infrared spectra of the extracted sample was run using IR spectrophotometer and the treated and untreated carbon fibres were examined under a scanning electron microscope to detect the presence of grafted polymer.

RESULTS AND DISCUSSION

The crucial step in performing an *insitu* electropolymerization lies in the careful choice of solvent-electrolyte system capable of forming a solution of the monomer with sufficient current conducting properties. In this investigation, water and sulphuric acid have served as solvent-electrolyte system which can dissolve the acrylamide monomer to a large extent with sufficient conductance of solution.

Carbon fibre can be used either as anode or cathode, as the mechanism of the polymerization is dependent on the locus of polymerization. Since the present combination of monomer-electrolyte-solvent system has been found to undergo [8] cathodic polymerization, carbon fibre has been used as cathode in this investigation.

From different electropolymerization runs it has been found that the carbon fibre is a good substrate for electropolymerization.

Polymer formation was visually seen as a coating within seconds of application of current and no apparent differences were observed between different carbon fibres. As expected, there was a drop in the current during electropolymerization (Fig. 1) as a result of formation of an insulating layer.

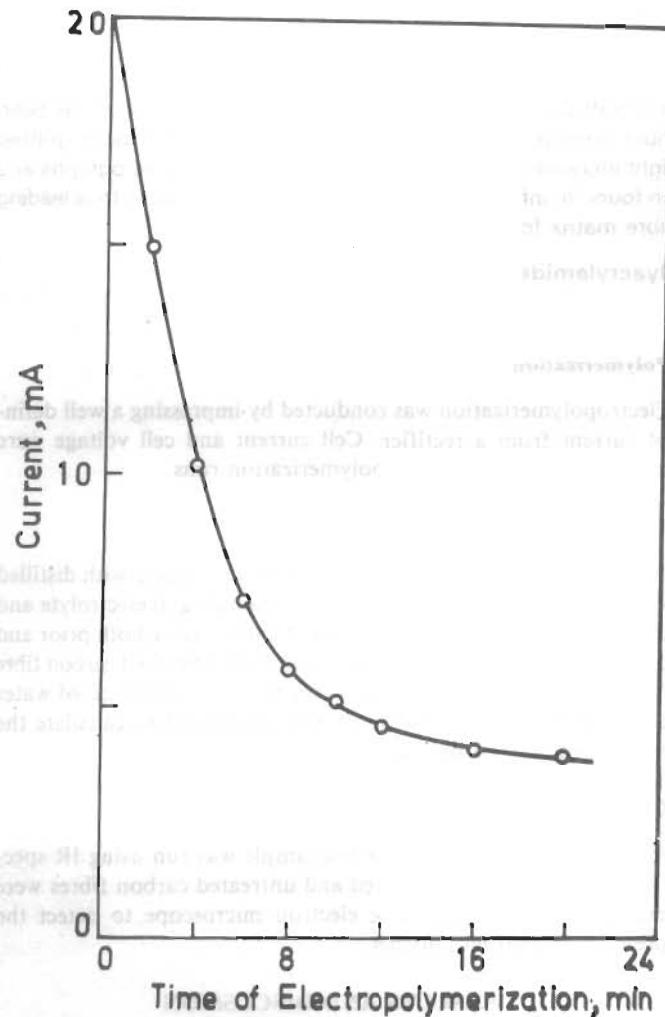


Fig. 1: Variation of current with time during electropolymerization

The presence of polymer has also been identified by the increase in weight of fibres with time (Fig. 2) after electropolymerization.

The increase in weight of carbon fibre has also been realized even after the extraction of fibre with water which indicates the retention of insoluble polymer on the carbon fibre surface. This proves that the polymer is grafted onto the matrix of carbon fibre, as a result of the chemical bonding of the polymer to the fibre. It is further substantiated by scanning electron micrographs of both treated and untreated fibres (Fig. 3).

The presence of unextractable polymer can be seen in the treated fibre which confirms that the polymer is grafted onto the carbon fibre. Fig. 4 shows the increase in yield of grafted product with polymerization time.

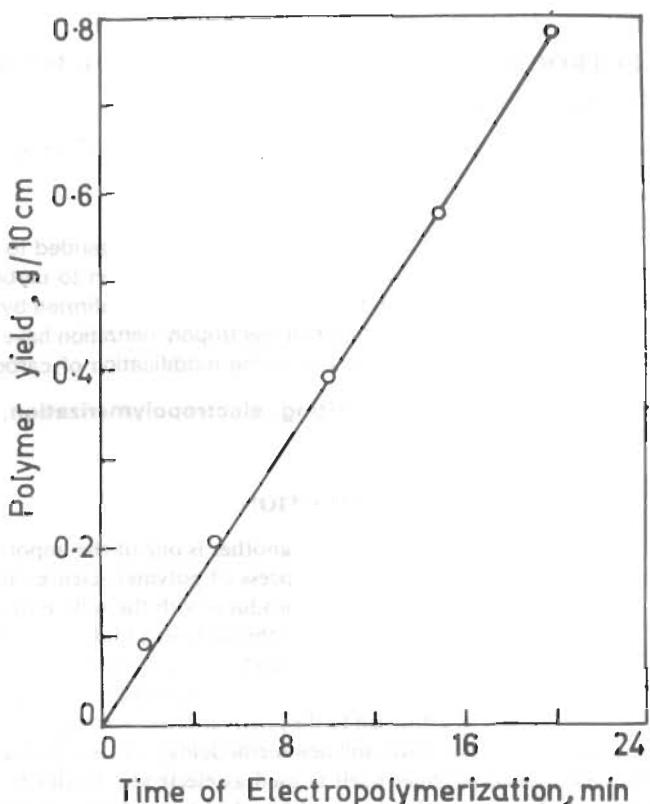


Fig. 2.: Yield of polymer as a function of time during electropolymerization

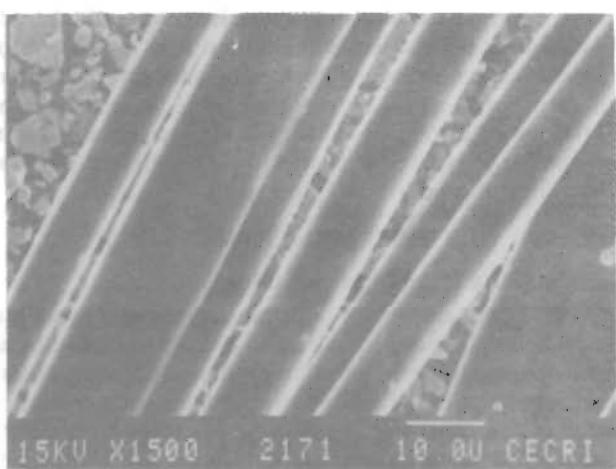


Fig. 3: SEM photographs of carbon fibres — Untreated

The linearity observed in Figs. 2 and 4 shows the possibility of growing a polymer layer of controlled thickness. It also indicates that grafting reaction is faster, as product formation occurs in minutes. The polymer product formed has been confirmed to be polyacrylamide by observing the characteristic IR absorption bands

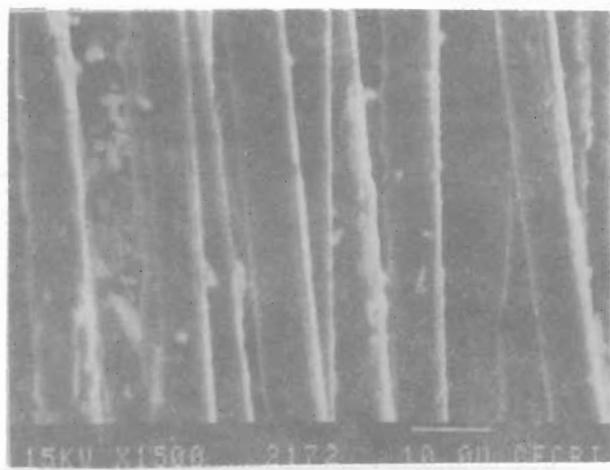


Fig.3: SEM photographs of carbon fibres — Treated

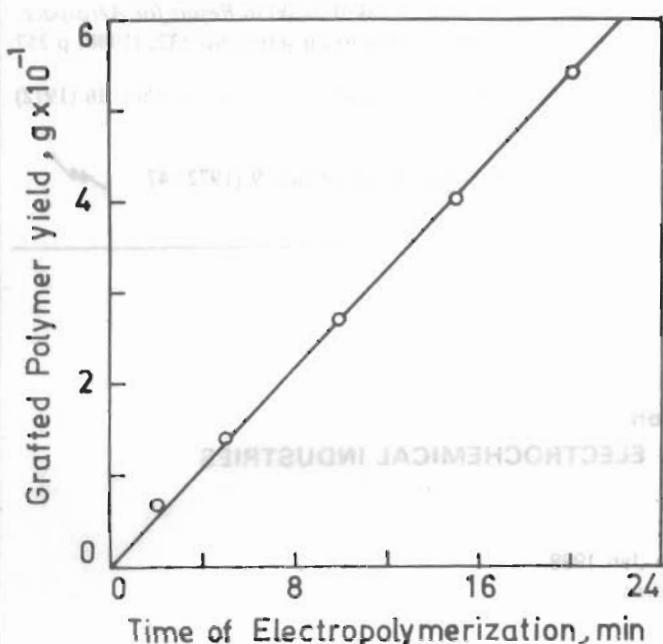


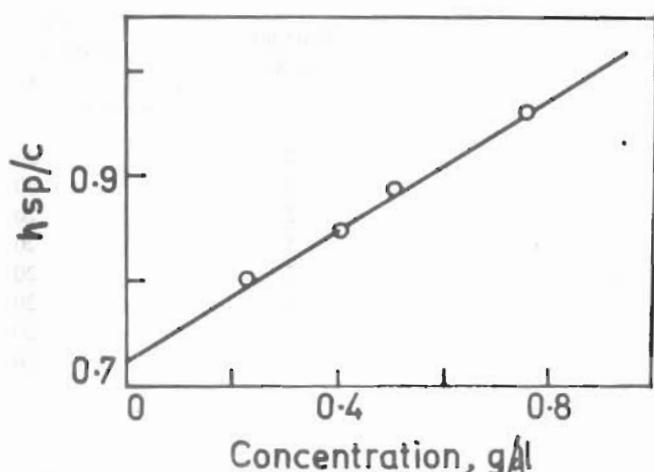
Fig.4: Yield of grafted polymer as a function of time during electropolymerization

at 3400 cm^{-1} , 1600 cm^{-1} and 1100 cm^{-1} .

Table I shows the influence of conditions of electropolymerization on the yield and grafting conversion efficiency. Within the range studied, it has been found that increases of current as well as monomer concentration increases the yield of product and consequently grafting conversion efficiency.

Fig.5 shows a plot of η_{sp}/C vs C (where η_{sp} is specific viscosity and C is concentration) from which the molecular weight of

polyacrylamide formed is deduced as 9.25×10^4 viscometrically.

Fig.5: Determination of intrinsic viscosity (η) of polyacrylamide

It is not unexpected that grafting should take place on carbon fibres, exposed to the electropolymerization medium containing monomer during the generation of reactive species by electron transfer at the electrode surface. Although the mechanism of grafting was not investigated, the presence of organic functional groups such as -COOH and -OH on carbon fibres is capable of forming free radical sites, for example, by chain transfer. Initiation of free radical polymerization or termination by combination of growing polymer radicals at these sites can lead to the observed polymer grafting.

The uniformity of coating observed in this investigation can be due to the possible adsorption of organic monomers on to the electrode surface and its better wettability from aqueous system due to the higher surface tension of water.

CONCLUSION

The use of *in situ* electropolymerization for carrying out graft copolymerization has been demonstrated on to carbon fibre with polyacrylamide. The grafted polymer product has been identified through weight increase, scanning electron micrographs and IR spectral data. The conditions of electropolymerization has also been found to influence the course of grafting reactions.

Acknowledgement: Thanks are due to Dr. O P Bhal of NPL, New Delhi for the gift sample of carbon fibre.

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TABLE-I: Electropolymerization of acrylamide on carbon fibres at 30°C

Concentration monomer (M)	Current (mA)	Time of polymerization (min)	Conversion (%)	Grafting efficiency %
0.05	2	20	42.5	68.3
0.05	5	20	47.6	69.5
0.1	2	20	50.3	70.4
0.1	5	20	55.8	71.8
0.15	2	20	57.2	70.2
0.15	5	20	62.4	70.6
0.2	2	20	62.8	70.1
0.2	5	20	65.9	70.9

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