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RESEARCH ARTICLE

Electrosynthesis of Epoxy Resins via Superoxide Anion

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Abstract

Epoxy resins are a class of reactive pre polymers and polymers which contain epoxide groups. Inspite of their high costs the epoxy resins find many important applications. The resins are used in both molding and laminating technique for making glass fiber-reinforced articles which are having better mechanical strength chemical resistance and electrical insulating properties. They also use in casting, potting encapsulating and embedment in the electrical and tooling industries. The general important uses are industrial flooring adhesive foams, highway surfacing patching material and stabilizers for vinyl resins. Epoxy resins may be reacted either with them- selves through catalytic homo polymerization or with a wide range of co-reactant like amino acid ,phenol, alcohols and thiols. These co-reactants are referred to as curatives. When linear epoxy resins react with suitable curative to form three dimensional cross linked thermo state structure. Epoxidation of unsaturated alcohol is carried out with the help of superoxide. The electrochemical generation of super-oxide anion and subsequent electro oxidation in situ is found to be advantageous and easy over the conventional technique. The work extended further in persuasive oxygen reduction studies on non-metallic surface such as different types of carbon and graphite with a view to understand the reaction mechanism and possible application to fuel cell technology.

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Introduction

A fuel cell operates electrochemically or more literally "Chemico- electrically . A fuel cell is a device that convert the chemical energy from a fuel into electricity through a chemical reaction with oxygen or another oxidizing agent. This cell is actually a reactor it consist of two electronic conductor electrodes separated by an ionic conducting electrolyte with provision for the continuous movement of fuel oxidant and reaction product in to and out of the cell. The fuel is carbon rod and electrolyte tetra methyl ammonium halide , perchlorate, Borates and oxidant is superoxide. Electrochemical investigation in aprotic solvents have established that molecular oxygen is reduced by a reversible one electron process to superoxide ion. Superoxide is a nucleophilic agent and its reducing power is roughly equivalent to dithionite ion. On the other hand, its property to act as oxidizing agent is much more equivocal. The superoxide anion in aprotic solvent has been generated by the electrolytic reduction of molecular oxygen at mercury pool cathode. The ease of formation plays an important role in polymer synthesis. We synthesized different types of epoxy resin using superoxide.

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Methodology

The investigation in this work have been carried out by polymerization of epoxy group in the presence of superoxide. The electro generated superoxide act as an oxidant in the monomer electrolysis and moves towards the carbon electrode conducting electricity.

Materials/Chemical Used:-
Acetonitrile (A.R.Grade), glycerols, resorcinol, epichlorohydrin, Tetra methyl ammonium halides, - perchlorate, Borates ,disphenol, pthalic etc.

Cell Assembly
A beaker (500 ml) was used as the undivided cell. Mercury pool was used as cathode and carbon rod was as anode. The electrolyte consist of 150 ml acetonitrile. Solution which was 2.5 mili molar wrt substrate and 0.05M wrt tetra methyl ammonium bromide. A mixture of glycerol (1 mole) and epichlorohydrin (4 mole) is electrolyse in the presence of sodium hydroxide .Air was bubbled and mixture was magnetically stirred throughout the reaction. Electrolysis was carried out by passing a constant current (100,200, and 300 mA) from a regulated D.C. power supply. When reaction is completed the resin is washed with water to remove sodium chloride and dried to remove water .Both end of the polymer molecules have epoxide units while there are hydroxyl group along the chain. We also used glycerols and resorcinoles instead of glycols. The epoxy resins obtained through these reactions are either highly viscous liquids or solids with high melting point. The cured resins are stable at 150-200^0 C depending on cross linking agent used. They are resistant to chemical attack and are flexible and strongly adhesive . The electro analytical techniques direct current and alternating current polarography cyclic voltametry ,chronopotentiometry,constant potential macro electrolysis have also applied for the study of kinetics and mechanism of superoxide generation.

Proposed Mechanism
The validity of the fact that the molecular oxygen is reduced in. aprotic solvents to be the stable superoxide ion ($.O_2^-$) has been established by the study of different electrode processes by reversible one electron process.

$$O_2 + e^- \text{----------} \rightarrow .O_2^- \quad -0.85\ V \quad V/S \quad SCE$$
$$\leftarrow \text{--------}$$

$$2C + 3.O_2^- \text{----------} \rightarrow 2CO_3^{-2} + 4e^-$$

During this monomer electrolysis the electron is accepted by the monomer and give radical anion and finally the desired polymer.
We can prepared different types of epoxy resins when we used disphenol, glycole, glycerols and resorcinol with epichlorohydrin..

EPICHLOROHYDRIN + X ----------→ EPOXY RESINS

X= BISPHENOL OR GLYCOLE OR GLYCEROLS OR RESORCINOL

Cyclic acid anhydride e.g. pthalic employed as curing agents for epoxy resins. They form esters epoxy..These resins have better thermal stability and good electrical insulation and chemical resistance

TABLE

Cell undivided ,cathode mercury pool,Anode carbon rod , Electrolyte acetonitrile containing substrate and tetramethyl ammonium borate at temperature 25^{o}C

Substrate	Current used mA	Current density mA cm^{-2}	cell voltage V	% Yield
disphenol	100	2.84	13-16	46%
and epich-	200	5.67	16-20	68%
-lorhydrine	300	8.51	20-26	53%
(1:4 Molar Ratio)				
Glycol and	100	2.84	12-15	36%
Epichloro-	200	5.67	14-18	58%
-hydrine	300	8.51	19.22	43%
(1:4 molar Ratio)				
Resorcinol	100	2.84	12-18	38%
and epichl-	200	5.67	15-20	63%
orohydrine	300	8.51	20-25	52%
(1:4 molar Ratio)				

Conclusion and Significance

The application of electrochemical reduction of molecular oxygen to super-oxide anion generated in situ has been utilized in the synthesis of epoxy resins. Epoxy resins finds a large number of uses, because of their remarkable chemical resistance and good adhesion concentration of the principal constituents in many of the fibre reinforced plastic is an epoxy polymer.

The cured resins are stable at 150-200^{o}C depending on cross linking agents used . They are resistance to chemical attack and flexible and strongly adhesive. They are used as surface coatings and yield as excellent enamel after esterification which is used for floors, walls, tanks, domestic equipment etc.

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