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SYNTHESIS OF POLY(VINYLENE SULPHIDE)

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Abstract Pure and high molecular poly(vinylene sulphide) was synthesized from 1,2-dichloroethylene and sodium sulphide anhydride using dimethyl sulfoxide as a solvent.

INTRODUCTION

Poly(vinylene sulphide), $\{CH=CH-S\}_x$, (PVS) is the simplest sulphur containing unsaturated polymer. We have reported a preparation method of this novel polymer[1]. We have recently succeeded to prepare higher and more pure PVS by using water-free materials and dimethyl sulfoxide(DMSO) as a solvent.

PREPARATION OF PVS

PVS was prepared by the condensation polymerization of sodium sulphide anhydride and 1,2-dichloroethylene as follows,

$$ClCH=CHCl + Na_2S \longrightarrow \{CH=CH-S\}_x + 2NaCl$$

Dimethyl sulfoxide(DMSO) was used as a solvent. DMSO(90ml) and Na_2S(15.2g) were added to a reacter equipped with a powerful stirrer under an argon atmosphere. The sodium sulphide solution was stirred vigorously at 20~50°C, and then trans- or cis-1,2-dichloroethylene(19.3g) was added. After 12~15 hours for cis-1,2-dichloroethylene and 70~100 hours for trans-1,2-dichloroethylene, the reaction mixture was filtered and the filtrate poured into a large quantity of methanol. Precipitation of PVS is given in powder form which has light brown colour and it can be obtained as a film by heat pressing at above 100°C.

The structure of PVS was examined by ^{13}C-n.m.r. and i.r. spectra; ^{13}C-n.m.r. data are shown in Figure 1 and i.r. data are shown in Figure 2.

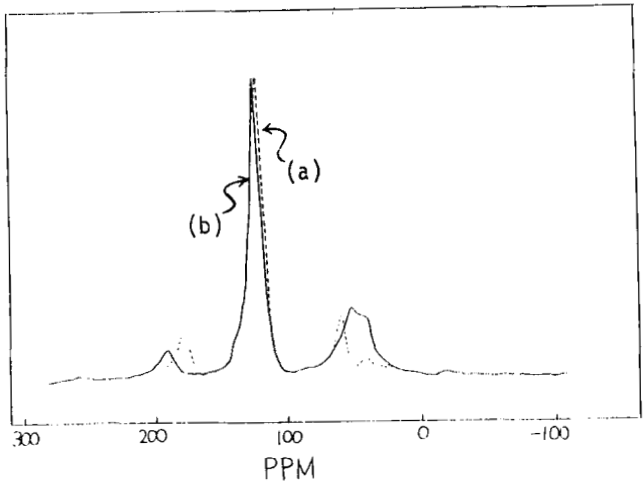

FIGURE 1 ^{13}C n.m.r. spectrum: (a) cis-1,2-dichloroethylene based PVS; (b) trans-1,2-dichloroethylene based PVS.

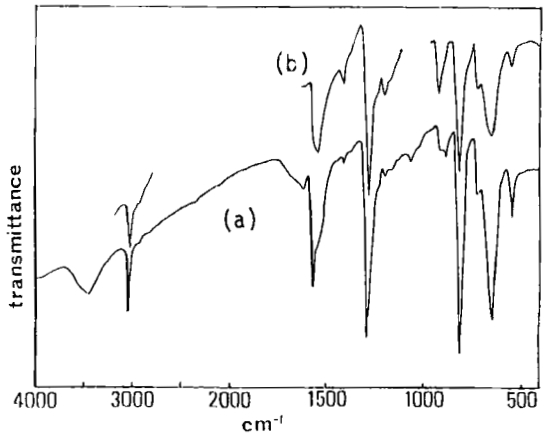

FIGURE 2 i.r. spectrum: (a) cis-1,2-dichloroethylene based PVS; (b) trans-1,2-dichloroethylene based PVS.

The sharp signal at ca. 121.9 ppm for cis-1,2-dichloroethylene based PVS and 122.2 ppm for trans-1,2-dichloroethylene based PVS in ^{13}C-n.m.r. spectrum indicates the presence of the alkenic carbon atom. The i.r. spectrum exhibits absorption bands at 3024, 1540, 910, 800, 660 cm^{-1}. The band at 3024 cm^{-1} is due to the olefinic C-H stretching. A band centered at 1540 cm^{-1} is characteristic of the C=C double bond positioned between two sulphur atoms, such as S-CH=CH-S.[2] A sharp band at 800 cm^{-1} is also characteristic of S-CH=CH structure. The two bands at 910 and 660 cm^{-1} can be assigned to the olefinic C-H deformation vibration out of plane in trans and cis conformation, respectively.

The elemental analysis is also supporting the structure of PVS. The result is shown in Table 1.

TABLE I Elemental analysis of PVS.

cis- based PVS	$C_{2.0}H_{2.00}S_{0.97}Cl_{0.032}$
trans- based PVS	$C_{2.0}H_{2.16}S_{1.02}Cl_{0.006}$

The molecular ends are thought to be chlorine atoms and from these experimental formula molecular weight can be calculated as 2400 ~ 3300 for cis-1,2-dichloroethylene based PVS and 10000~14000 for trans-1,2-dichloroethylene based PVS respectively.

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