Study on Electrical Conductivity of 2-Vinylpyridine-Methyl Methacrylate Copolymer in Presence of Cobalt Acetate

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ABSTRACT: 2-Vinyl pyridine (2VP)-methyl methacrylate (MMA) copolymers with different molar ratios were prepared. Their electrical properties were studied in the presence of $Co(CH_3COO)_2$. It was found that the electrical properties of the copolymer were changed by altering the molar ratio of 2VP : MMA and by varying the concentration of $Co(CH_3COO)_2$. The highest electrical conductivity was found when the 2VP : MMA molar ratio was 1 : 1. © 2001 John Wiley & Sons, Inc. J Appl Polym Sci 80: 2145–2153, 2001

Key words: electrical conductivity; 2-vinyl pyridine; methylmethacrylate; cobalt acetate

INTRODUCTION

Studies were made on the electrical conductivity of oligomers and polymers prepared from aromatic compounds containing nitrogen. Examples of these compounds are phthalocyanine[1–3] (a chelated compound), poly-*N*-vinylcarbazole,[4–9] polypyrrole,[10] and poly(2-vinylpyridine) (P2VP).[11] Chohan et al.[12,13] studied the electrical behavior of some polymeric charge transfer complexes that were prepared by the complexation of P2VP with metal salts. P2VP and its metal-based derivatives were also synthesized and studied by Rafique et al.[14] The dc conductivity of these materials was studied as a function of temperature. However, the electrical conductivity of the copolymer of VP with other monomers was not extensively studied. Awasthi and Srivastava[15] mentioned that 4VP was polymerized with poly(methyl methacrylate) (PMMA) and that a complex compound was obtained.

In this article copolymers of 2VP and MMA were prepared with molar ratios of 1 : 1, 3 : 1, and 1 : 3 in the presence of $Co(CH_3COO)_2$. Interesting results were established when the molar ratio of the two monomers was 1 : 1 in the presence of 1.5 wt % $Co(CH_3COO)_2$ (of the 2VP concentration).

EXPERIMENTAL

Copolymerization of 2VP with MMA

Forty-two grams (0.4 mol, ≈43 mL) of 2VP and 40 g (0.4 mol, ≈42.5 mL) of MMA were dissolved in 100 mL of chloroform, and 1 g of benzoyl peroxide was added. The solution was heated at 60°C for 0.5 h in a round-bottomed flask equipped with a refluxing condenser. The copolymer was precipitated by *n*-hexane, washed with alcohol and a little ether, and dried at 70°C. The copolymer was divided into two equal fractions. The first fraction

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was dissolved in chloroform. A solution of 0.1575 g of $Co(CH_3COO)_2$ in ethyl alcohol (0.75 wt % of the 2VP concentration) was added to the copolymer solution in chloroform, and a thin film was prepared. The second fraction of the copolymer was also dissolved in chloroform to which a saturated solution of 0.315 g of $Co(CH_3COO)_2$ (1.5 wt % of the 2VP concentration) in ethyl alcohol was added, and a thin film was prepared.

In the previous experiment the molar ratio of 2VP : MMA was 1 : 1. This experiment was repeated using a molar ratio of 2VP : MMA ≡ 1 : 3, which was 35 g (≈35.9 mL) of 2VP and 100 g (≈106.4 mL) of MMA. Two weight percent $Co(CH_3COO)_2$ (of the 2VP concentration) was added to the copolymer solution in a saturated solution of $Co(CH_3COO)_2$ in ethyl alcohol, and then a thin film was prepared.

A third experiment was performed using 105 g (≈107.7 mL) of 2VP and 33.333 g (≈35.5 mL) of MMA (i.e., the molar ratio of 2VP : MMA was 3 : 1. Two weight percent $Co(CH_3COO)_2$ (of the 2VP concentration) was added to the copolymer solution, and a thin film was prepared.

Physical Measurements

Samples of the copolymer were prepared as thin films (1-cm diameter and 1-mm thickness). Each sample was tightly adjusted between two copper electrodes of an electric cell. The temperature was measured by a thermocouple placed in the cell near the sample. A thermocouple temperature probe (TP-30) attached to a millivoltmeter was used to measure the temperature. The electric cell was placed in an electric furnace in order to measure the electrical resistivity at different temperatures. The measuring circuit was composed of a high-voltage regulated dc power supply (Gamma), a high impedance electrometer (Keithley 610 C electrometer), and the cell. The relation between the electrical resistivity and the absolute temperature was given by the Arrhenius equation:

$$\log \rho = \log \rho_o + (E_a/RT)$$

where E_a is the activation energy for the electrical conductivity; R is the gas constant, which is approximately equal to 2 col deg^{-1} mol^{-1}; ρ is the electrical resistivity (Ω cm); and ρ_o is the intrinsic resistivity, which is at $T = \infty$.

Table I Relation between log ρ and $10^3/T$ for 2VP-MMA Copolymer

$T(k = t + 273)$	$\rho = 0.128R$	$\log \rho$	$10^3/T$ (K^{-1})
344	1.87	0.27	2.91
341.5	3.05	0.48	2.93
341.0	6.95	0.84	2.93
340.0	18.24	1.26	2.94
339.0	31.17	1.49	2.95
338.5	32.52	1.51	2.95
338.0	10.4	1.02	2.96
337.0	10.4	1.02	2.97
334.0	9.51	0.98	2.99
332.5	9.51	0.98	3.01
329.0	9.51	0.98	3.04
328.0	9.51	0.98	3.05
327.0	9.51	0.98	3.06
325.0	9.51	0.98	3.07
322.0	32.0	1.51	3.10
319.0	10.31	1.01	3.13
318.0	9.6	0.98	3.14
316.0	8.02	0.90	3.16
314.0	12.27	1.09	3.18
314.0	12.8	1.11	3.18
313.0	10.18	1.01	3.19
312.0	7.54	0.88	3.20
309.5	8.02	0.90	3.23
307.0	8.02	0.90	3.26

The molar ratio was 1 : 1, and the $Co(CH_3COO)_2$ was 0.75 wt % of the 2VP concentration.

The above relation is a straight line. It is valid in the case of semiconductors. The activation energy is calculated from the slope of this straight line.

RESULTS AND DISCUSSION

The 2VP was copolymerized with MMA using a molar ratio of 1 : 1 in the presence of $Co(CH_3COO)_2$. The $Co(CH_3COO)_2$ concentration was 0.75 wt % of the 2VP concentration. The electrical conductivity increased with the temperature as shown in Table I. The log ρ decreased linearly with $1/T$ (Fig. 1) and attained a constant value between 34 and 65°C (i.e., a horizontal line was obtained). This is the typical behavior of semiconductors. The horizontal line between 34 and 65°C means that the activation energy for electrical conductivity $\Delta E_a \rightarrow 0$ at low temperature (i.e., this material can be used in semicon-

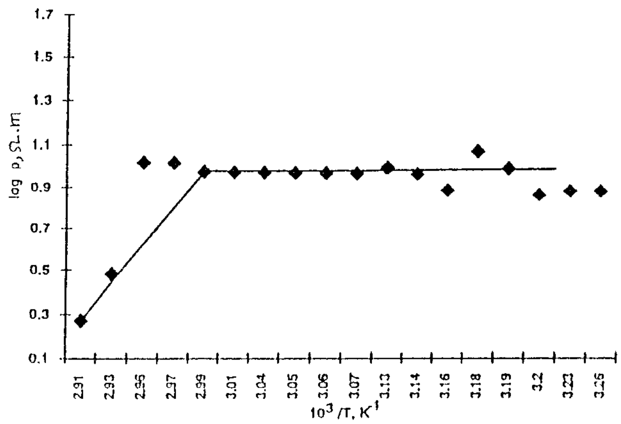


Figure 1 A plot of the log ρ versus 10^3 T for the 2VP-MMA copolymer (molar ratio 1 : 1) + 0.75 wt % $Co(CH_3COO)_2$ (of the 2VPV concn).

Table II Relation between Electrical Current (*I*) and Voltage (*V*) for P2VP-MMA Copolymer

V (mV)	$I \times 10^2$ (mA)
64	6.8
39	4.0
81	7.5
102	9.5
126	11.7
139	12.8
162	14.8
184	16.7
202	18.2
223	20.0
244	21.5

The molar ratio was 1 : 1, and the $Co(CH_3COO)_2$ was 0.75 wt % of the 2VP concentration.

ducting devices at normal temperature with a slight activation energy).

The electrical conductivity (σ) for the 2VP : MMA copolymer was 1.27 Ω^{-1} cm^{-1} at 34°C. The molar ratio was 1 : 1 in the presence of 0.75 wt % $Co(CH_3COO)_2$ (of the 2VP concentration, and the σ was 5.376 Ω^{-1} cm^{-1} at 70°C.

The relation between the electrical current (I) and the voltage (V) was found to be linear as shown in Figure 2 (see also Table II). This means that we have an ohmic conduction mechanism (i.e., the relation obeys Ohm's law).

When the $Co(CH_3COO)_2$ concentration was 1.5 wt % of the 2VP concentration and using the same molar ratio for 2VP : MMA (1 : 1), the relation between log ρ and $10^3/T$ does not become more linear. Moreover, we was found that the log ρ increases with temperature (Fig. 3, Table III), which is a property of metals. However, when the electrical resistivity was traced against the temperature (t) a straight line was

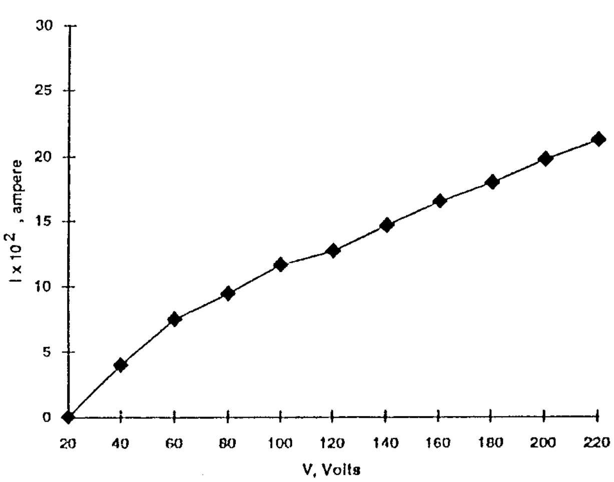


Figure 2 The I versus V for the 2VP-MMA copolymer (molar ratio 1 : 1) + 0.75 wt % $Co(CH_3COO)_2$ (of the 2VP concn).

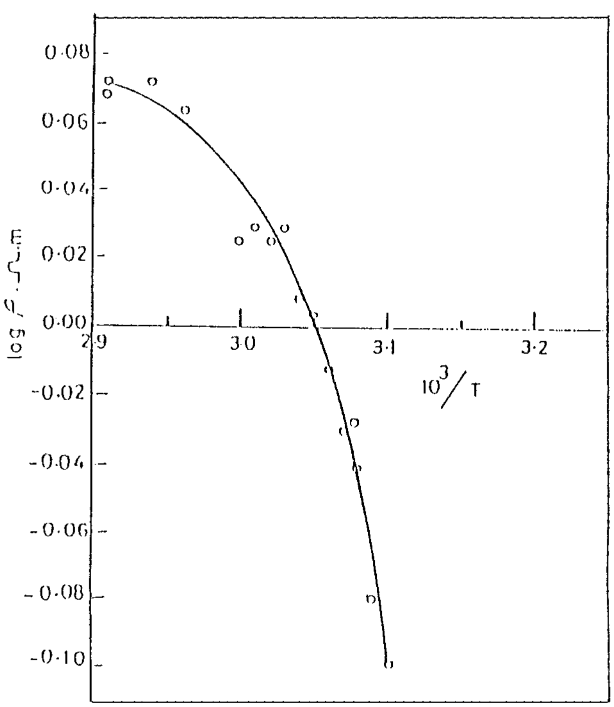


Figure 3 A plot of the log ρ versus $10^3/T$ for the 2VP-MMA copolymer (molar ratio 1 : 1) + 1.5 wt % $Co(CH_3COO)_2$ (of the 2VP concn).

Table III Relation between log ρ and $10^3/T$ for 2VP-MMA Copolymer

T ($k = t + 273$)	$\rho = 0.126R$	log ρ	$10^3/T$ (K^{-1})
344	1.17	0.068	2.91
344	1.18	0.072	2.91
343	1.18	0.072	2.92
340.5	1.18	0.072	2.94
338.0	1.12	0.064	2.96
336.5	1.18	0.072	2.97
336.0	1.18	0.072	2.97
33.0	1.06	0.025	3.00
332.0	1.07	0.029	3.01
330.5	1.06	0.025	3.02
330.0	1.07	0.029	3.03
329.0	1.02	0.0086	3.04
322.0	1.01	0.0043	3.05
326.0	0.97	−0.012	3.06
325.5	0.93	−0.031	3.07
325.0	0.91	−0.041	3.08
3215.0	0.91	−0.041	3.08
324.0	1.06	0.025	3.08
323.0	0.83	−0.081	3.09
322.0	0.77	−0.113	3.10
320.5	0.72	−0.143	3.12
319.5	0.59	−0.229	3.13
319.0	0.75	−0.125	3.13
318.0	0.79	−0.102	3.14
316.5	0.68	−0.167	3.16
315.5	0.89	−0.051	3.17
315.0	0.89	−0.051	3.17
314.5	1.09	0.037	3.18
314.0	0.90	−0.046	3.18
313.0	0.85	−0.071	3.19
313.0	0.98	−0.008	3.19
312.0	1.16	0.064	3.20

The molar ratio was 1 : 1, and the $Co(CH_3COO)_2$ was 1.5 wt % of the 2VP concentration.

obtained (Fig. 4, Table IV); it obeys the same law as that in metals,

$$\rho_t = \rho_0(1 + \alpha t)$$

where ρ_0 is the resistivity at 0°C and αt is the coefficient of the electrical resistance curve.

From the curve in Figure 4 ρ_0 was found by extrapolation to 0°C, and then α was calculated using the slope of the curve. From the above relationship we have

$\Delta\rho = \alpha\rho_0\Delta t$, and

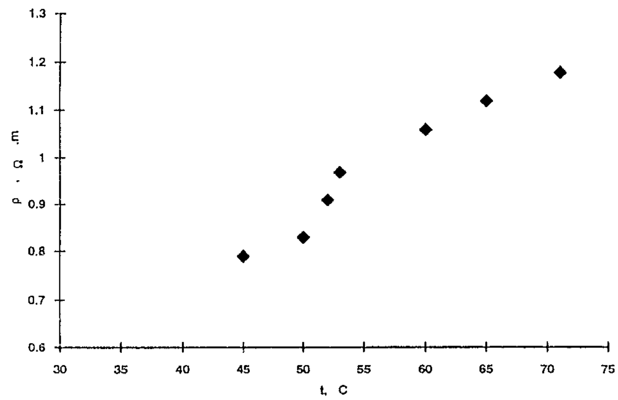


Figure 4 The temperature dependence of the electrical resistance ρ for the 2VP-MMA copolymer (molar ratio 1 : 1) + 1.5 wt % $Co(CH_3COO)_3$ (of the 2VP concn).

$$\alpha = \left(\frac{\Delta\rho}{\Delta t}\right)/\rho_0 = \frac{\text{slope of curve}}{\rho_0} = \frac{0.0155}{0.08} = 0.194$$

Here we notice that the α for the copolymer was 50 times greater than that of metals ($\alpha M \approx 4 \times 10^{-3}$). This means that the conductivity of the copolymer was highly affected by the $Co(CH_3COO)_2$ concentration.

The relation between I and V was linear (Fig. 5, Table V), which means that the conduction was ohmic. However, when the molar ratio of 2VP : MMA was 3 : 1 in the presence of 2 wt % $Co(CH_3COO)_2$ (of the 2VP concentration), $\sigma = 2.3 \times 10^{-8}$ at 65°C and 1.25×10^{-10} at 40°C (Table VI). The log ρ versus $10^3/T$ was linear (Fig. 6). The relation between I and V was also linear (Fig. 7, Table VII) when the 2VP : MMA molar ratio was 1 : 3 in the presence of 2 wt % $Co(CH_3COO)_2$

Table IV Relation between Electrical Resistivity and Temperature for 2VP-MMA Copolymer

t (°C)	ρ (Ωm)
71	1.18
65	1.12
60	1.06
53	0.97
52	0.91
50	0.83
45	0.79

The $Co(CH_3COO)_2$ was 1.5% of the 2VP concentration, and the molar ratio of the two monomers was 1 : 1.

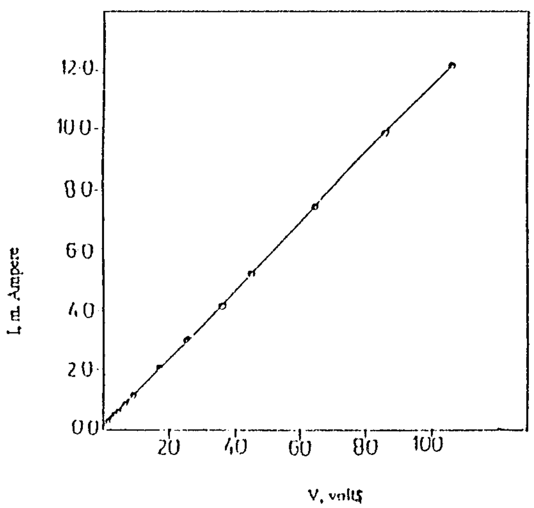


Figure 5 The plot of I versus V for the 2VP-MMA copolymer (molar ratio 1 : 1) + 1.5 wt % $Co(CH_3COO)_2$ (of the 2VP concn).

(of the 2VP concentration); $\sigma = 5.8 \times 10^{-10}\ \Omega^{-1}\ cm^{-1}$ at 74°C and $3.3 \times 10^{-12}\ \Omega^{-1}\ cm^{-1}$ at 40°C (Table VIII), and log ρ versus $10^3/T$ was linear (Fig. 8).

The relation between I and V was also linear (Table IX, Fig. 9). When a relation was made

Table V **Relation between I and V for 2VP-MMA Copolymer**

V (mV)	I (mA)
1.9	0.22
2.0	0.23
2.3	0.27
2.4	0.28
2.7	0.31
3.0	0.34
3.5	0.40
5.0	0.57
7.1	0.81
9.0	1.03
17.5	2.01
26.5	3.04
36.2	4.15
45.5	5.21
64.8	7.4
86.5	9.9
107.0	12.26

The molar ratio was 1 : 1, and the $Co(CH_3COO)_2$ was 1.5 wt % of the 2VP concentration.

Table VI **Relation between log ρ and $10^3/T$ for 2VP-MMA Copolymer**

T ($k = t + 273$)	$\rho = 6.7R$	log ρ	$10^3/T$ (K^{-1})
337.5	4322.5×10^6	6.63	2.96
337.0	4785.7×10^6	9.68	2.97
335.0	6483.8×10^6	9.81	2.98
334.0	7730.7×10^6	9.88	2.99
333.0	9136.4×10^6	9.96	3.00
331.0	11823.5×10^6	10.07	3.02
330.0	13400×10^6	10.13	3.03
329.0	17478×10^6	10.24	3.04
328.0	20100×10^6	10.30	3.05
326.5	25125×10^6	10.40	3.06
325.5	31904.7×10^6	10.50	3.07
324.5	38653.8×10^6	10.59	3.08
323.0	50250×10^6	10.70	3.09
320.5	77307.7×10^6	10.89	3.12
319.0	100500×10^6	11.00	3.13
318.0	134000×10^6	11.13	3.14
316.0	191428×10^6	11.28	3.16
314.0	251250×10^6	11.40	3.18
313.0	335000×10^6	11.52	3.19
310.5	502500×10^6	11.70	3.22
308.0	804000×10^6	11.90	3.25

The molar ratio was 3 : 1, and the $Co(CH_3COO)_2$ was 2 wt % of the 2VP concentration.

between log ρ and $10^3/T$ for 2VP-MMA copolymer using the molar ratio of 3 : 1 and in the absence of $Co(CH_3COO)_2$, ρ did not change extensively at a wide range of temperatures, ($\sigma = 1.8 \times 10^{-7}$ at

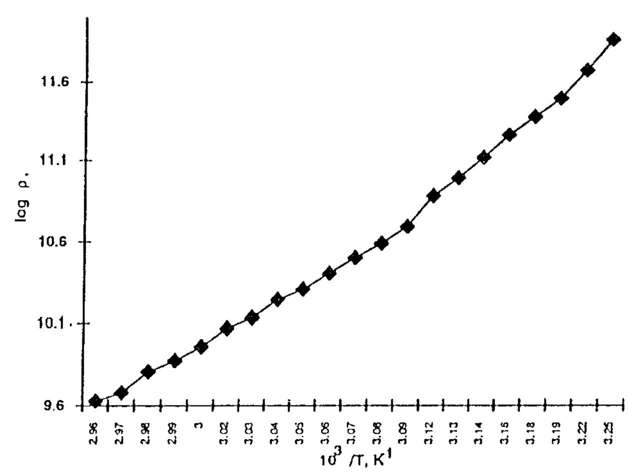


Figure 6 The log ρ versus $10^3/T$ for the 2VP-MMA copolymer (molar ratio 3 : 1) + 2 wt % $Co(CH_3COO)_2$ (of the 2VP concn).

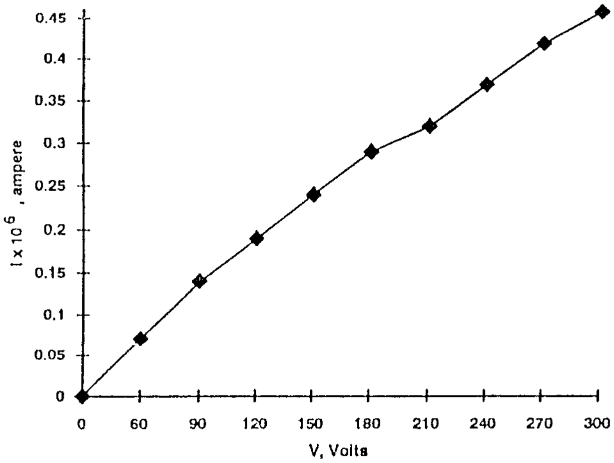


Figure 7 The *I* versus *V* for the 2VP-MMA copolymer (molar ratio 3 : 1) + 2 wt % $Co(CH_3COO)_2$ (of the 2VP concn).

50°C and 1.8×10^{-7} at 56°C; Table X); and the relation of *I* versus *V* was linear (Table XI, Fig. 10).

The IR spectrum of the 2VP-MMA copolymer when the molar ratio was 1 : 1 and in the presence of 1.5 wt % $Co(CH_3COO)_2$ (of the 2VP concentration) was studied, and there were two characteristic bands found at 1715 and 1676 cm^{-1}. These bands were absent in the 2VP spectrum. The band at 1715 cm^{-1} was due to cyclic lactam fused to another ring,[16] and the band at 1676 cm^{-1} was either due to an imide cyclic α-β unsaturated six-membered ring or a urea cyclic six-membered ring. The first proposal means that the carbon atom of the carbonyl group in MMA is linked to the nitrogen atom in 2VP as shown in the structure below. The second proposal means that the nitrogen atoms in two adjacent 2VP rings are linked to the carbon atom of the MMA carbonyl group (i.e., a complex compound was formed). Accordingly, the increase in the electrical conductivity of the copolymer was due to the fact that the nitrogen in 2VP carried a positive charge when it was linked to the carbon of the MMA carbonyl group, while the oxygen of the carbonyl group carried a negative charge. The presence of a transitional element such as CO in $Co(CH_3COO)_2$ increases this ionization.

Table VII Relation between *I* and *V* for 2VP-MMA Copolymer

V (V)	$I \times 10^6$ (amp)
300	0.46
270	0.42
240	0.37
210	0.32
180	0.29
150	0.24
120	0.19
90	0.14
60	0.98
30	0.04

The molar ratio was 3 : 1, and the $Co(CH_3COO)_2$ was 2 wt % of the 2VP concentration.

Table VIII Relation between log ρ and $10^3/T$ for 2VP-MMA Copolymer

log ρ	$10^3/T$ (K^{-1})	T ($k = t + 273$)	$\rho = 0.108R$
11.23	2.88	347	1.7×10^{11}
11.30	2.89	346	2.01×10^{11}
11.34	2.894	345.5	2.18×10^{11}
11.40	2.90	344.5	2.53×10^{11}
11.54	2.91	343.0	3.48×10^{11}
11.69	2.93	341.5	4.91×10^{11}
11.78	2.937	340.5	6.11×10^{11}
11.86	2.94	339.5	7.19×10^{11}
11.96	2.95	339.0	9.25×10^{11}
12.40	2.96	338.0	10.9×10^{11}
12.19	2.97	336.5	15.8×10^{11}
12.25	2.98	336.0	17.99×10^{11}
12.31	2.98	335.5	20.46×10^{11}
12.37	2.99	334.5	23.64×10^{11}
12.51	3.00	333.0	32.4×10^{11}
12.61	3.01	332.0	41.0×10^{11}
12.64	3.02	331.5	43.78×10^{11}
12.69	3.02	331.0	49.46×10^{11}
12.73	3.026	330.5	54×10^{11}
12.85	3.04	329.0	70.43×10^{11}
12.92	3.05	328.0	84.15×10^{11}
13.03	3.06	327.0	108×10^{11}
13.11	3.07	325.5	129.6×10^{11}
13.20	3.08	324.0	162.0×10^{11}
13.25	3.09	323.0	180.0×10^{11}
13.29	3.10	322.0	196.36×10^{11}
13.32	3.12	320.5	210.39×10^{11}
13.38	3.14	318.0	240.0×10^{11}
13.45	3.16	316.0	281.7×10^{11}
13.45	3.17	315.5	281.7×10^{11}
13.46	3.18	314.5	291.89×10^{11}
13.48	3.19	313.0	305.6×10^{11}
13.51	3.20	312.0	324.0×10^{11}

The molar ratio was 1 : 3, and the $Co(CH_3COO)_2$ was 2 wt % of the 2VP concentration.

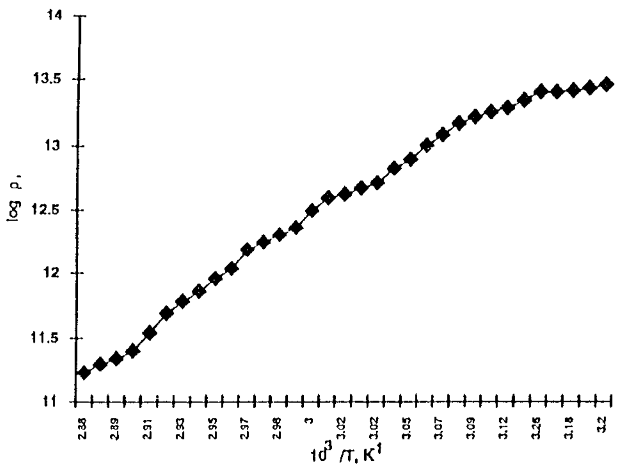


Figure 8 The log ρ versus $10^3/T$ for the 2VP-MMA copolymer (molar ratio 1 : 3) + 2 wt % $Co(CH_3COO)_2$ (of the 2VP concn).

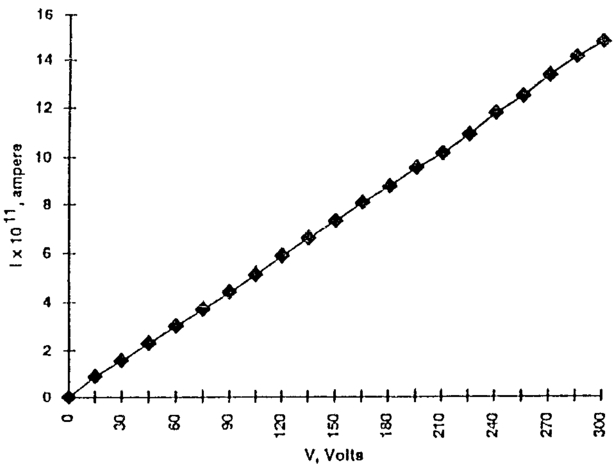
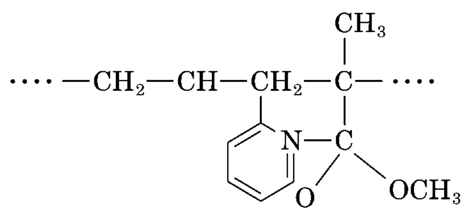
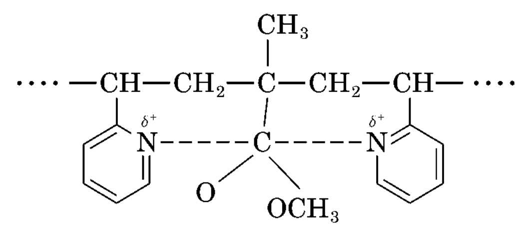


Figure 9 The I versus V for the 2VP-MMA copolymer (molar ratio 1 : 3) + 2 wt % $Co(CH_3COO)_2$ (of the 2VP concn).



Cyclic lactam fused to another ring



Imide cyclic unsaturated six-membered ring or urea cyclic six-membered ring

Table IX Relation between *I* and *V* for 2VP-MMA Copolymer

V (V)	$I \times 10^{11}$ (amp)
15	0.87
30	1.57
45	2.30
60	3.00
75	3.70
90	4.43
105	5.15
120	5.90
135	6.60
150	7.30
165	8.10
180	8.80
195	9.60
210	10.2
225	11.0
240	11.9
255	12.6
270	13.5
285	14.3
300	15.0

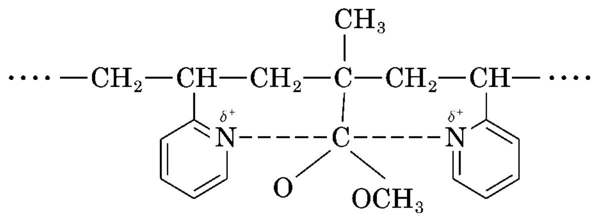
The molar ratio was 1 : 3, and the $Co(CH_3COO)_2$ was 2 wt % of the 2VP concentration.

Table X Relation between log ρ and $10^3/T$ for 2VP-MMA Copolymer

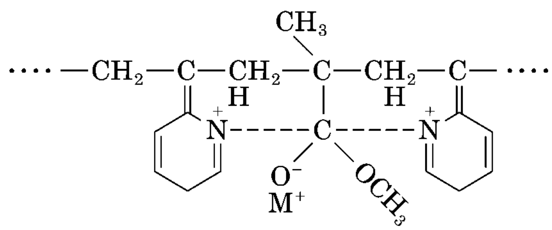
T ($k = t + 273$)	$\rho = 8.07R$	log ρ	$10^3/T$ (K^{-1})
318.0	1152.8×10^6	9.06	3.14
319.0	931.1×10^6	8.96	3.13
320.5	807×10^6	8.91	3.12
322.0	637×10^6	8.80	3.10
323.0	576.4×10^6	8.76	3.09
324.0	403.5×10^6	8.61	3.08
325.5	366.8×10^6	8.56	3.07
326.5	336.2×10^6	8.53	3.06
328.0	281.5×10^6	8.45	3.05
330.5	242.1×10^6	8.38	3.02
333.0	237.3×10^6	8.37	3.00
335.5	230.6×10^6	8.36	2.98
337.0	228.4×10^6	8.36	2.97
334.0	247×10^6	8.39	2.99
332.0	356×10^6	8.55	3.01
330.5	440.2×10^6	8.64	3.02
329.0	537.9×10^6	8.73	3.04
328.0	756.5×10^6	8.88	3.05
327.0	1008.7×10^6	9.00	3.06

The molar ratio was 3 : 1, and the $Co(CH_3COO)_2$ was 2 wt % of the 2VP concentration.

Also, we may predict the equilibrium



(a)



(b) Imide cyclic α-β unsaturated six-membered ring

This ionization leads to the activation of the copolymer chain; and when an electric field was applied through the polymer chain, these charges became mobile, leading to an increase in the electrical conductivity of the copolymer. Part (b) of the above structure shows the possibility of the formation of quinoid structures that further activate the copolymer chain. This investigation was enforced by the increase of the electrical conductivity of the copolymer by the increase in the cobalt acetate concentration.

Table XI Relation between *I* and *V* for 2VP-MMA Copolymer

V (V)	$I \times 10^6$ (amp)
15	0.04
30	0.09
60	0.16
90	0.24
120	0.32
150	0.36
180	0.44
210	0.50
240	0.56
270	0.60
300	0.64

The molar ratio was 3 : 1 without $Co(CH_3COO)_2$.

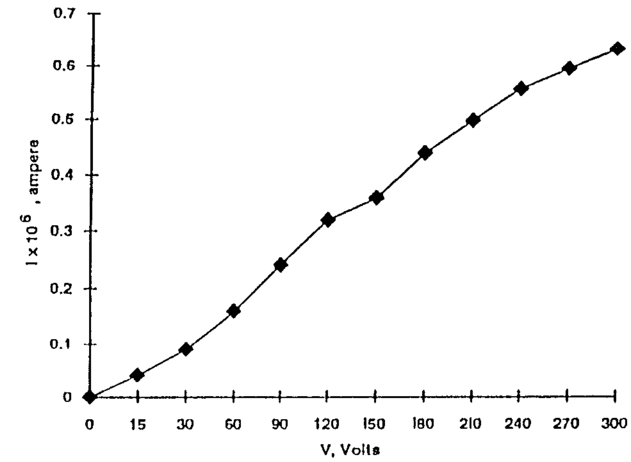


Figure 10 The *I* versus *V* for the 2VP-MMA copolymer (molar ratio 3 : 1).

The previous discussion about the creation of a complex when the molar ratio of 2VP : MMA was 1 : 1 was strengthened by the result obtained by Awasthi and Srivastava[15] who elucidated that a complex was formed when 4VP was polymerized with PMMA. The maximum complexation occurred when the template : polymer ratio was 1 : 1. 2VP can also form a complex when it is copolymerized with MMA as was shown by IR spectroscopy and due to the increase in the conductivity of the copolymer when the molar ratio of 2VP : MMA was 1 : 1 and in the presence of $Co(CH_3COO)_2$. The higher the electrical conductivity the higher is the concentration of $Co(CH_3COO)_2$. The complexation that occurred when 2VP and MMA were copolymerized at a molar ratio 1 : 1 did not occur when the molar ratio deviated from this value (e.g., when the molar ratio was 3 : 1 or 1 : 3).

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