

# Simultaneous Novel Synthesis of Conducting, Nonconducting, and Crosslinked Polymers by Microwave Initiation

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**ABSTRACT:** A novel synthesis of poly(dibromophenylene oxide) (P), conducting polymer (CP), and/or crosslinked polymer (CLP), and/or radical ion polymers (RIP) was achieved simultaneously from sodium 2,4,6-tribromophenolate by microwave energy in a very short-time interval. The synthesized polymers were characterized via elemental analysis, FTIR,  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR, X-ray diffraction spectroscopy, SEM, DSC, TGA, ESR, GPC, conductivity measurement, and light scattering. It was found that polymerization proceeds through both 1,2- and 1,4-addition at equal rates. The effects of the energy and time on the % conversion and the polymer synthesis were investigated. The optimum condition for synthesis of P (the highest  $M_w$ ,  $2.97 \times 10^5$  g/mol) and CP was 70 W for

5 min in 5 mL water and 100 W for 1 min in 0.5 mL water, having maximum values 23.6% and 27.2%, respectively. In addition, synthesis of CLP and RIP were achieved in 5 mL water at 350 W and 700 W at the end of 1 min, respectively. The direct synthesis of highly conducting polymer, with the conductivity of  $1 \text{ S cm}^{-2}$  was achieved in the absence of applied doping process in a very short time sequence. P, CP, CLP, and RIP had fine granular, sponge-like, dendrite, and coarse surface structures, respectively. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 5427–5435, 2006

**Key words:** poly(dibromophenylene oxide); conducting polymer; crosslinked polymer; radical ion polymer; microwave polymerization

## INTRODUCTION

Conductive polymers (CP) are new class of materials, which exhibit highly reversible redox behavior and unusual combination properties of metals and plastics.<sup>1,2</sup> Since 1977, increasing interest has been devoted to the electrical properties of a new-class organic conducting polymers, which can be either chemically synthesized or electrochemically grafted on electrode.<sup>3</sup> The most useful method for preparing large amounts of CP is chemical polymerization.<sup>4–8</sup> Chemical polymerization (oxidative coupling) is followed by the oxidation of monomers to a radical ion and their coupling to form dications and the repetition of this process generates a polymer. CP represents an important research area with a great potential for commercial and technological applications due to their stability, processability, and low cost. Present and future applications include electromagnetic interference shielding, anticorrosion coating, electrochromic displays, sensors, rechargeable batteries, light emitting diodes,<sup>9</sup> membranes,<sup>10</sup> and enzyme immobilizations.<sup>11</sup>

Crosslinked polymer (CLP) networks have been an interesting subject of research for many years because of their use in fields such as dentistry, microelectronics, or as absorbents in medicine and chemistry, exhibiting different properties depending on their degree of crosslinking. In general, the degree of crosslinking affects the swelling degree, pore size, thermal stability, and mechanical strength of the network.<sup>12</sup>

Poly(dihalophenylene oxide)s (Ps) are polyethers having aromatic groups connected to by an oxygen linkage at the backbone.<sup>13</sup> They can be linear or branched, amorphous or crystalline, and have high or low molecular weight depending on the type and position of the substituent on the starting phenol. The polymerizations of halogenated phenols from transition and inner transition metal complexes with various amine ligands by electroinitiation in solution,<sup>14–28</sup> thermal decomposition in solid state,<sup>29–35</sup> or in solution<sup>36–38</sup> have been investigated thoroughly.

Microwave power irradiation is known as an incredibly rapid and selective, clean and safe, and versatile and energetically convenient heating method, gaining increasing favor for industrial processing of numerous materials: rubbers, ceramics, textiles, minerals, wood, adhesives, some plastics, thermosetting resins, etc.<sup>39</sup> In the field of polymeric materials, in particular, microwave heating has shown a great

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TABLE I  
The Effect of Polymerization Time, Energy, and Amount of Water (w) on the % P, CP, and WL

	1 min, 0.5 ml w	1 min, 1 ml w	1 min, 5 ml w	1.5 min, 0.5 ml w	3 min, 0.5 ml w	5 min, 0.5 ml w	5 min, 1 ml w	5 min, 5 ml w	7 min, 0.5 ml w
70 W									
% P	–	–	–	–	16.2	19.5	20.2	23.6	15.6
% CP	–	–	–	–	20.8	21.0	8.9	–	22.3
% WL	–	–	–	–	41.0	41.2	29.9	25.8	44.1
100 W									
% P	14.1	15.6	15.5	13.7	12.5	–	–	–	–
% CP	27.2	26.4	22.8	23.1	21.8	–	–	–	–
% WL	40.0	29.9	27.5	43.2	43.7	–	–	–	–
350 W									
% P	13.5	14.2	16.7	12.3	11.9	–	–	–	–
% CP	25.6	25.0	20.1 <sup>a</sup>	24.9	22.4	–	–	–	–
% WL	46.9	30.4	28.3	42.8	50.4	–	–	–	–
500 W									
% P	12.0	13.9	15.6	10.8	–	–	–	–	–
% CP	21.9	26.0	25.8	20.4	–	–	–	–	–
% WL	47.1	28.2	29.5	50.8	–	–	–	–	–
700 W									
% P	9.9	10.2	10.5 <sup>b</sup>	6.8	–	–	–	–	–
% CP	19.5	15.1	–	15.4	–	–	–	–	–
% WL	45.6	30.1	30.6	51.0	–	–	–	–	–

<sup>a</sup> CLP synthesized instead of CP.

<sup>b</sup> RIP synthesized instead of P.

potential, still far from being extensively used as it could, for accelerated industrial processing of the entire variety of commercial (both commodity and high performance) plastics, rubbers, thermosetting resins, and related composites. The microwave polymerization technique provides a new way for the production of polymers at high rates and conversions.<sup>40</sup>

In the first study of the polymerization of sodium trichlorophenolate, simultaneous synthesis of poly (dichlorophenylene oxide) and CP were achieved by microwave initiation.<sup>41</sup> As a part of broader study of polymerization of various trihalophenols, we aimed to improve the polymerization technique for sodium tribromophenolate in the simultaneous synthesis of P and CP by using microwave energy in a very short time, studying the effects of heating time, amount of water, energy, and the type of halogen (bromine being better leaving group than the chlorine) on % conversion and structure. In the present study, the simultaneous synthesis of P with white insoluble CLP and the toluene soluble orange-colored radical ion polymer (RIP) with CP were achieved for the first time.

## EXPERIMENTAL

### Materials

Analytical grade 2,4,6-tribromophenol (TBP) (Merck) and reagent grade NaOH (Sigma and Aldrich Chemical) were used in the preparation of sodium 2,4,6-tribromo-

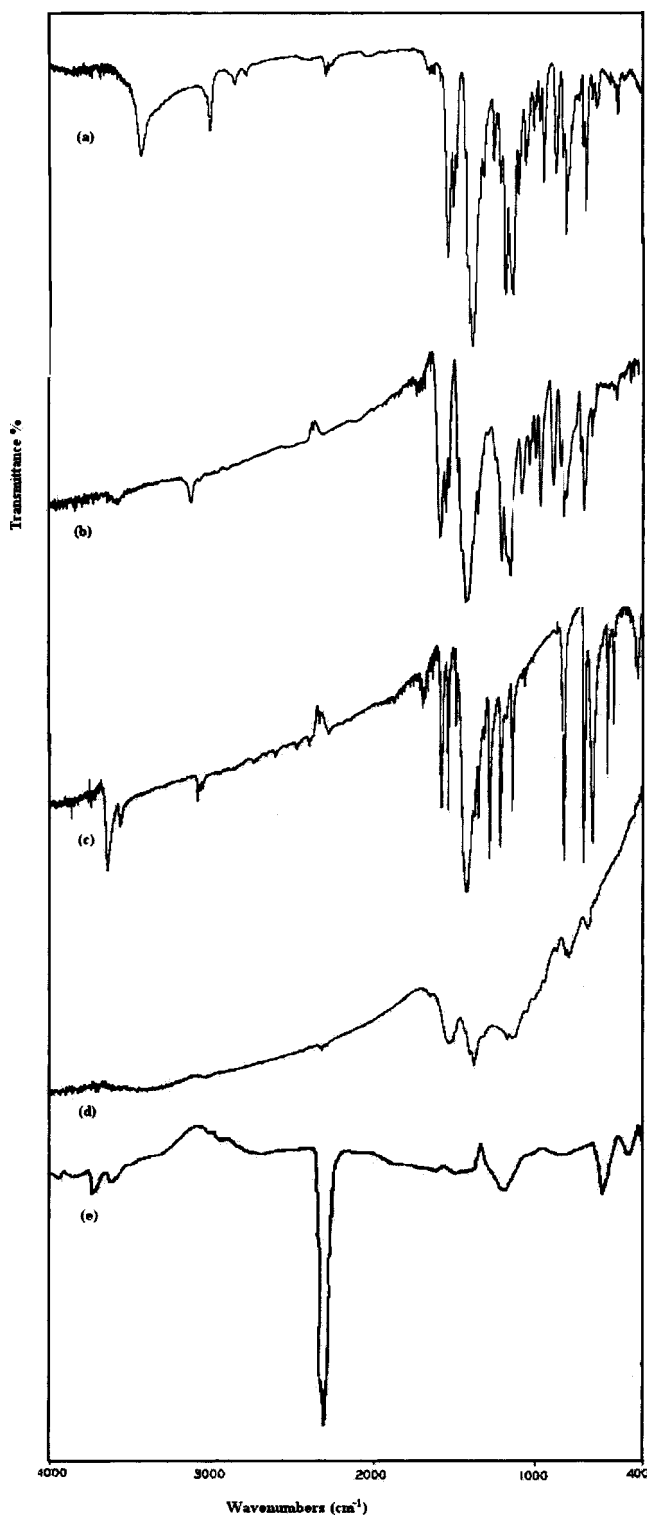
phenolate. Toluene (Merck) was the solvent and ethyl alcohol was the nonsolvent for the polymers. Deuterated chloroform was the solvent for <sup>1</sup>H NMR and <sup>13</sup>C NMR spectrophotometric measurements.

### Polymer synthesis

Microwave initiated polymerizations were performed at several time intervals (1–7 min) in different ranges of microwave energy (70–700 W) and in various amount of water (0.5–5 mL). The decomposition of the grounded TBP and NaOH in deionize water was performed in a Pyrex glass holder, loosely covered with a lid, inserted in a microwave oven, BEKO, working at 2.45 MHz, and having a pulse period of 10 s. The resulting simultaneous products were poured into toluene and the insoluble part CP was removed by filtration, and was washed several times by triple-distilled hot water for purification. The

TABLE II  
Elemental Analysis Results of P, RIP, and CP  
(Experimental Errors ±0.5)

	% C		% H	
	Calc.	Found	Calc.	Found
P	28.82	28.52	0.800	0.936
RIP	29.18	29.30	0.953	0.911
CP	54.6	54.59	1.595	1.399



**Figure 1** FTIR spectra of (a) P, (b) RIP, (c) CLP, (d) CP, and (e) released gases during polymerization.

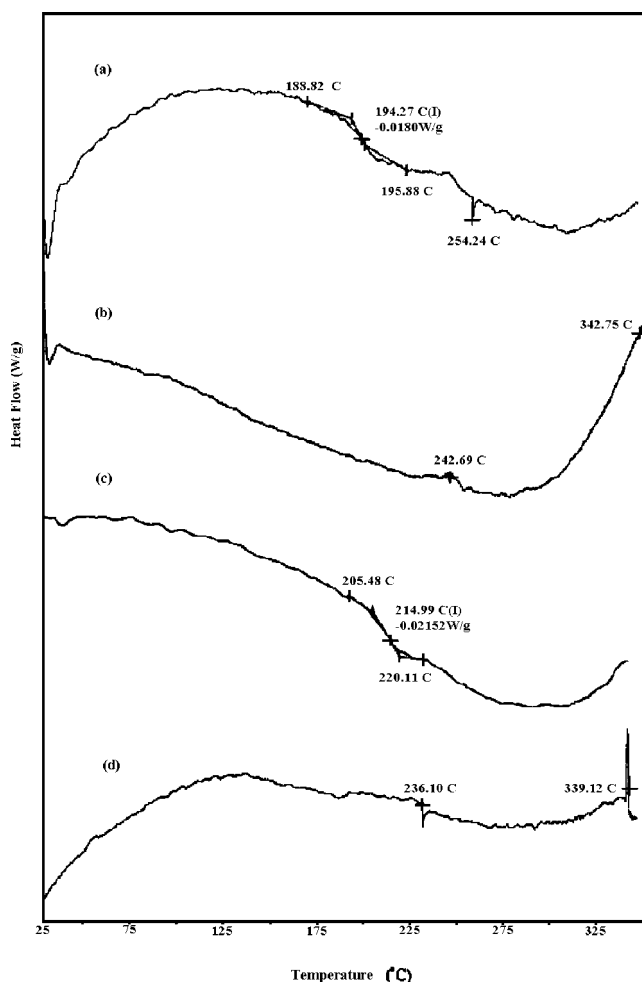
polymer P was precipitated by the addition of toluene solution to ethanol containing few drops of concentrated HCl. The simultaneous synthesis of P and white insoluble crosslinked polymer (CLP) was achieved at 350 W in 5 mL of water at the end of 1 min. The toluene soluble orange-colored RIP was

synthesized with CP simultaneously at 700 W in 5 mL of water. All the recovered precipitates were dried to a constant weight under vacuum.

### Polymer characterization

FTIR spectra of polymers were recorded on a Mattson 1000 model FTIR spectrometer in the 4000–400  $\text{cm}^{-1}$  region by dispersing the sample in KBr pellets.  $^1\text{H}$  NMR and decoupled  $^{13}\text{C}$  NMR spectra of poly(dichlorophenylene oxide) were recorded on a Bruker NMR spectrometer (DPX-400) in deuterated chloroform and TMS as an internal reference. Carbon–hydrogen analysis of samples were carried out with Leco 932 CHNSO elemental analyzer.

Glass transition temperature ( $T_g$ ) was measured by Du Pont Thermal Analyst 2000 DSC 910S Differential Scanning Calorimeter with a scanning rate of  $10^\circ\text{C}/\text{min}$  for 10 mg sample under nitrogen atmosphere. Weight losses of the polymers with the temperature change were determined by PerkinElmer



**Figure 2** DSC thermograms of (a) P, (b) CP, (c) CLP, and (d) RIP.

Pyris TGA. Synthesized polymers were heated from 30 to 850°C with a heating rate of 10°C/min under nitrogen atmosphere. Powder diffraction X-ray spectra of both unwashed and washed conducting polymer were obtained by using a computer controlled automatic Humber-Guinner powder diffractometer with Co K $\alpha$  radiation obtained having a voltage of 30 kV and a current of 7 mA. Molecular weight, radius of gyration, and virial coefficient of P were determined at 37°C by using multiangle laser scattering spectrometer (Malvern 5000). The ALV/CG-3 Goniometer system is designed to perform dynamic and static light scattering simultaneously. ESR spec-

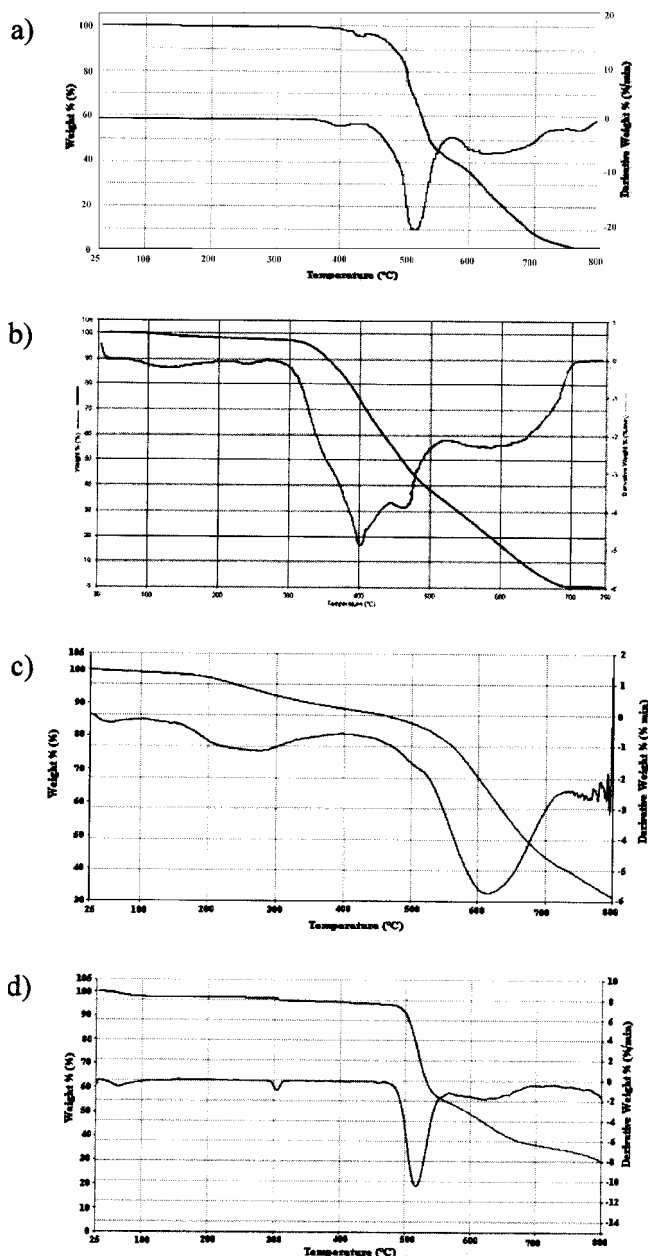


Figure 3 TGA thermograms of (a) P, (b) RIP, (c) CP, and (d) CLP.

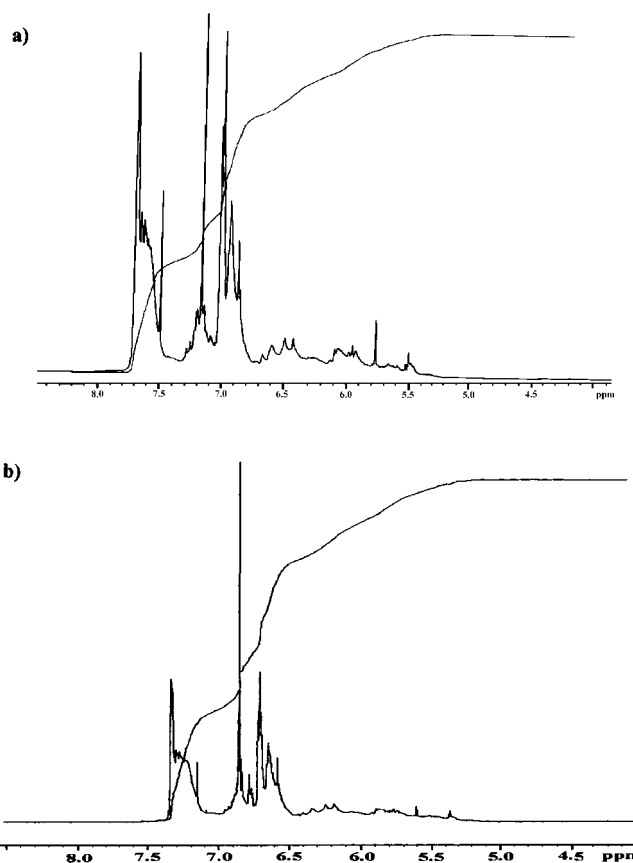


Figure 4  $^1\text{H}$  NMR spectrum of (a) P and (b) RIP.

trum of the products was recorded by Bruker Xepre ELEXSY-580 spectrometer in quartz cell at room temperature where diphenylpicrylhydrazyl was the reference.  $M_w/M_n$  values are measured with PL-GPC 220 gel permeation chromatography in THF. Conductivity of unwashed and washed CP was measured by four-probe technique.

## RESULTS AND DISCUSSION

The effect of polymerization time, energy, and amount of water on the % conversions and % weight losses were listed in Table I. At 70 W in 0.5 mL water, the

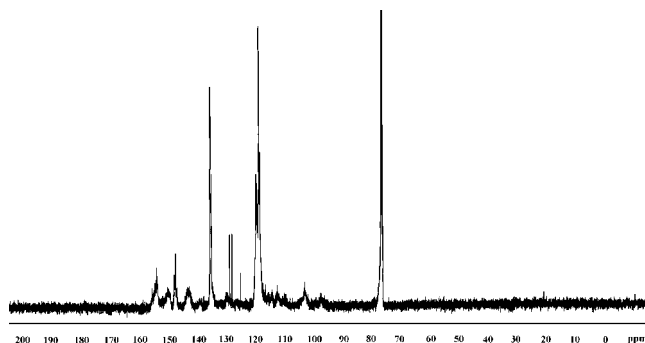


Figure 5 Proton-decoupled  $^{13}\text{C}$  NMR spectrum of P.

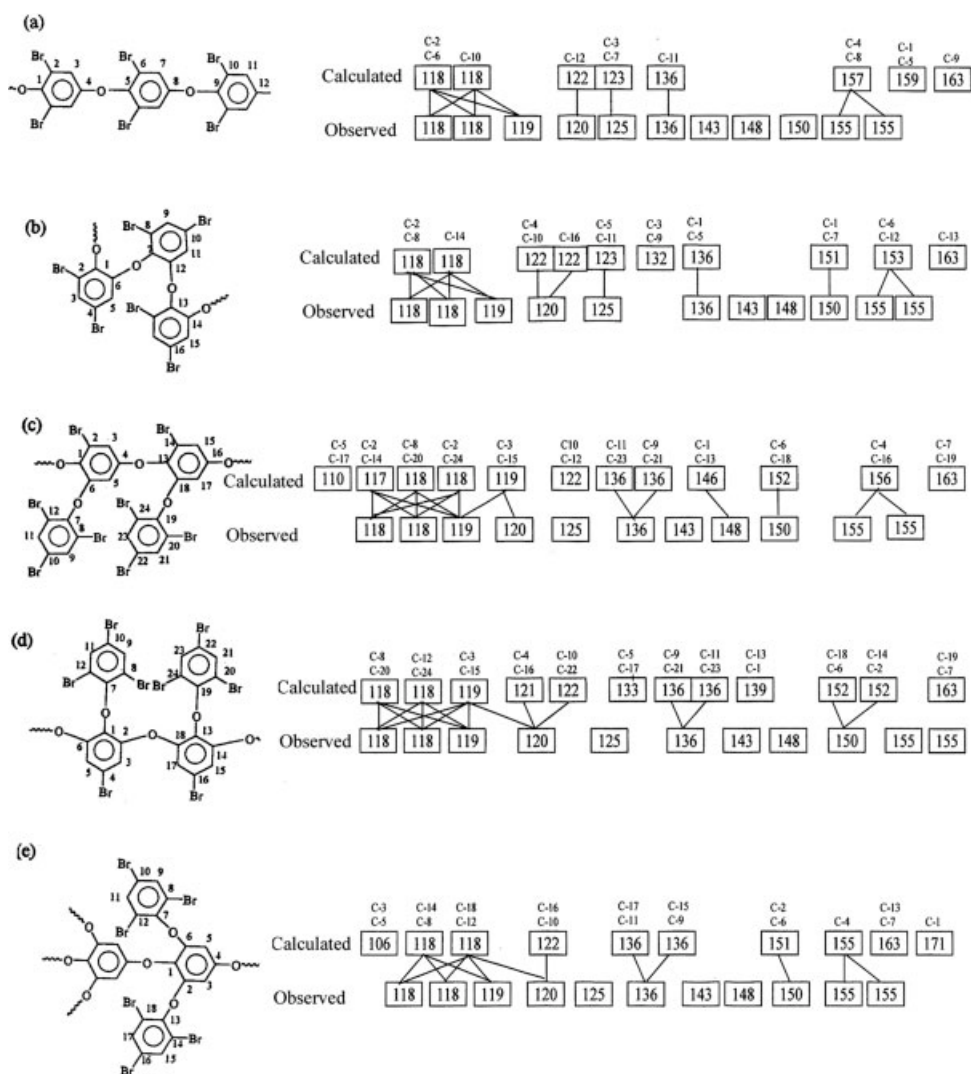


Figure 6  $^{13}\text{C}$  NMR proton shift data of P.

% conversion of P showed an increasing trend up to 19.5% followed by a decrease to 15.6% at the end of 7 min. As the water content increased, the % conversion of P increased while CP decreased significantly up to 5 mL water. At 70 W, the synthesis of P and CP were achieved at the end of 3 min whereas in the range of 100–700 W, the syntheses were in between 1 and 3 min. In addition, in 5 mL water, only P was synthesized at the end of 5 min whereas CLP an RIP syntheses were achieved at 350 and 700 W, respectively, at the end of 1 min. In the range of 100–700 W, generally % conversion slightly increased for P and whereas %WL<sup>41</sup> decreased for CP as the time increased from 1 to 3 min. On the other hand, % conversion increased slightly for P, while decreased slightly for CP and sharply for WL as the amount of water was increased (0.5–5.0 mL). Hence, the optimum condition for P and CP was 70 W for 5 min in

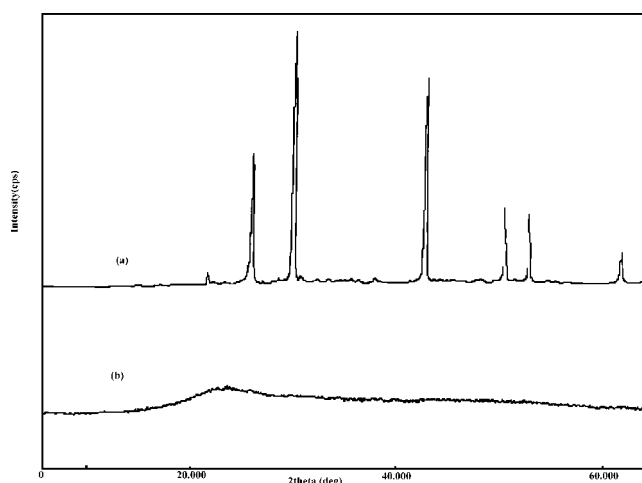
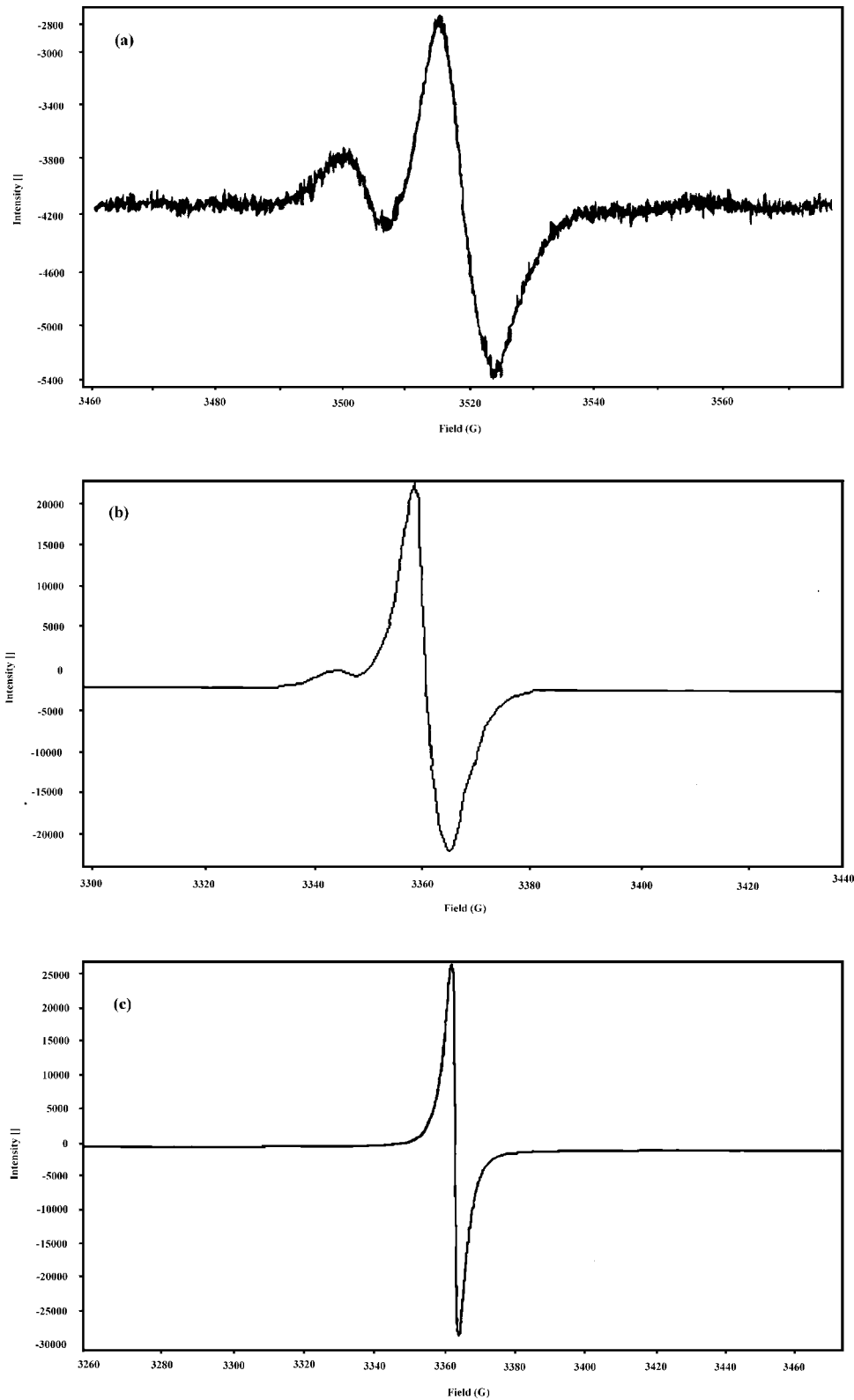


Figure 7 X-ray powder diffraction spectra of (a) unwashed CP and (b) washed CP.

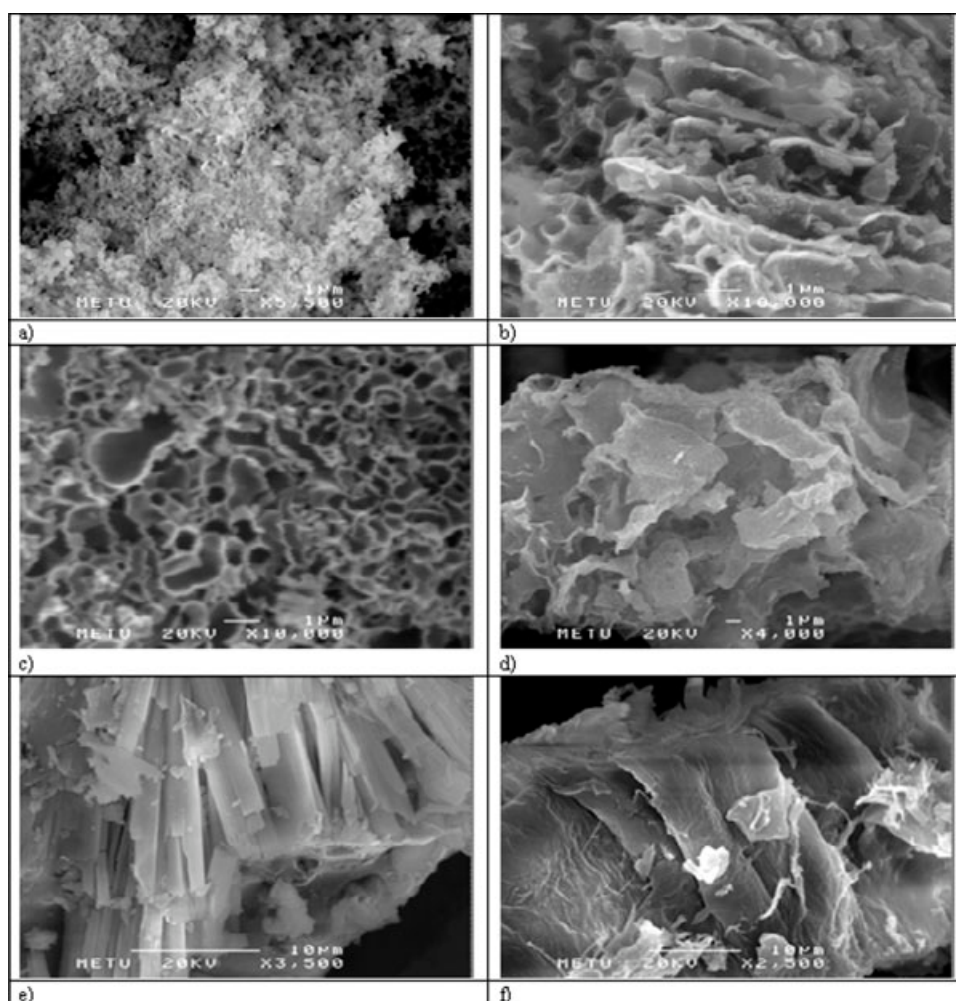


**Figure 8** ESR spectrum of (a) RIP, (b) CLP, and (c) CP at room temperature.

5 mL water and 100 W for 1 min in 0.5 mL water, having maximum values 23.6% and 27.2%, respectively. The %WL generally increased slightly as the time and energy increased and sharply decreased as

the amount of water increased reaching to the maximum value at 50.8% at the end of 1.5 min at 500 W.

The weight-average molecular weight ( $M_w$ ) of the P (in 5 mL water, at 70 W) and RIP was determined



**Figure 9** SEM micrographs of (a) P at 70 W, 5 min, (b) CP at 70 W, 5 min, (c) CP at 100 W, (d) and (e) CP at 350 W, and (f) CP at 700 W, 1 min in 0.5 mL water.

as  $2.97 \times 10^5$  g/mol and  $1.2 \times 10^4$  g/mol, respectively, by the light scattering. The highest molecular weight among the synthesized P was achieved. The radius of gyration and the second virial coefficient for P determined as  $2.285 \times 10^2$  nm and  $2.125 \times 10^{-7}$  mol dm<sup>3</sup>/g<sup>2</sup>, and for RIP  $3.619 \times 10^1$  nm and  $4.119 \times 10^{-7}$  mol dm<sup>3</sup>/g<sup>2</sup>, respectively. The observed  $M_w/M_n$  values for P and RIP are 1.026 and 1.363, respectively, by GPC.

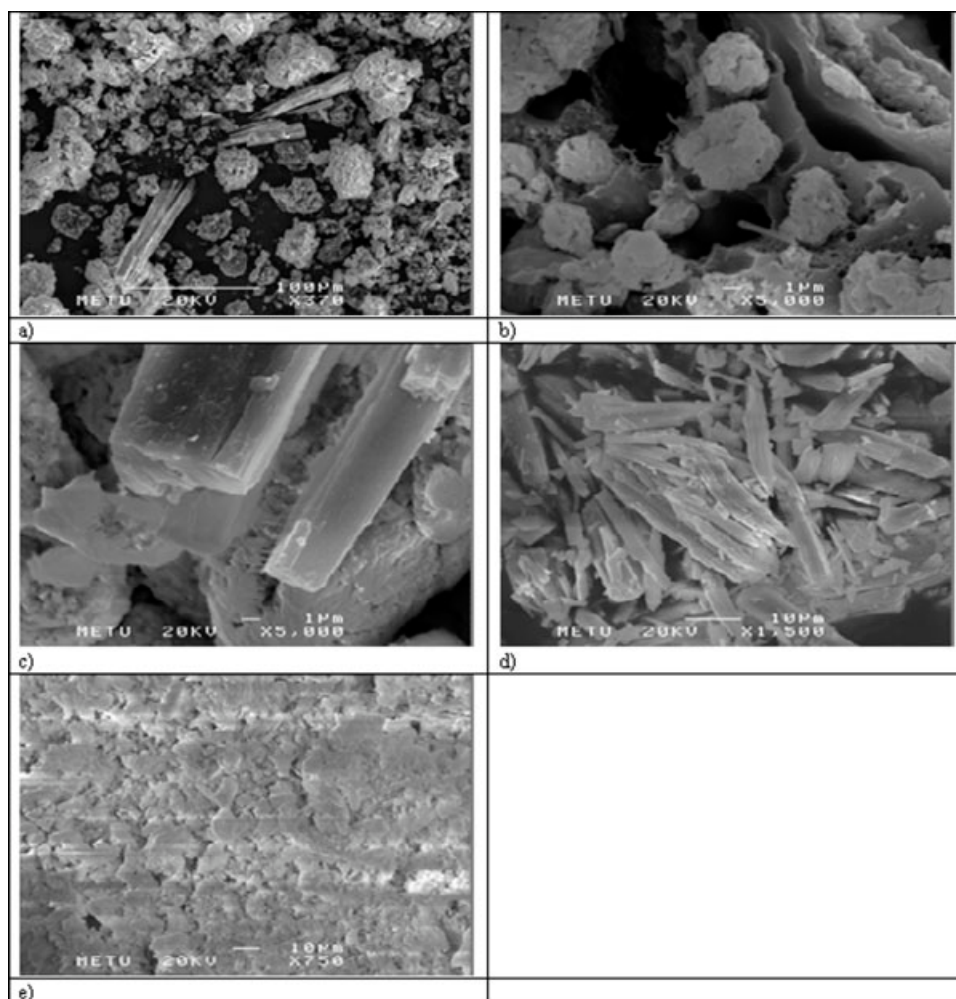
Elemental analysis results of the polymers correlate perfectly with the written stoichiometries as tabulated in Table II.

FTIR spectrum of P and RIP exhibits the characteristic absorptions at 850 cm<sup>-1</sup> (out of plane C—H bending), 955–1040 cm<sup>-1</sup> (C—O—C stretching), 1140–1210 cm<sup>-1</sup> (C—O stretching), 1440 and 1580 cm<sup>-1</sup> (C=C ring stretching), 3079 cm<sup>-1</sup> (aromatic C—H stretching), and 3500 cm<sup>-1</sup> (the phenolic end group) [Fig. 1(a,b)]. FTIR spectrum of CLP [Fig. 1(c)] was similar to P, except 955–1040 cm<sup>-1</sup> (C—O—C stretching) was shifted to 1050–1200 cm<sup>-1</sup>. FTIR

spectrum of CP exhibits the peaks at 1460–1600 cm<sup>-1</sup> (C=C stretching of both benzenoid and quinoid structures), 1710 cm<sup>-1</sup> (C=O stretching), 1110–1280 cm<sup>-1</sup> (C—O stretching), 3050 cm<sup>-1</sup> (aromatic C—H stretching), and 730–760 cm<sup>-1</sup> (C—Br stretching) [Fig. 1(d)]. The FTIR spectrum of the evolved gas collected in a glass cell during polymerization exhibits the peak at 2450 cm<sup>-1</sup> (CO<sub>2</sub>) and 1270 cm<sup>-1</sup> (C=O stretching) [Fig. 1(e)].

The DSC thermograms of P and CLP have glass transition temperature 194.27°C and 214.99°C, respectively, indicating high rigidity [Fig. 2(a,c)]. The glass transition temperature was not observed for CP and RIP [Fig. 2(b,d)].

In the TGA thermogram, the P and RIP was stable up to nearly 300 and 360°C, respectively, and then ~ 80% of sample was lost when the temperature reached to 600°C [Fig. 3(a)]. However, in case of CP and CLP, the weight loss started from 500°C, indicating a higher thermal stability and still having residues less than 27 and 30% beyond 800°C, which



**Figure 10** SEM micrographs of (a, b, and c) CP at 70 W, 5 min in 0.5 mL water, (d) CLP at 350 W, and (e) RIP at 700 W, 1 min in 5 mL water.

could be due to the loss of the pendant groups [Fig. 3(b,c)].

The  $^1\text{H}$  NMR spectrum of P and RIP was characterized by the small peak at 7.0 ppm due to the protons of 2,6-dibromo-1,4-phenylene oxide (1,4-addition), the intense peak at 7.4 ppm due to the protons of 2,4-dibromo-1,6-phenylene oxide unit (1,2-addition), and the boarder peaks at higher field due to the presence of 1,2- and 1,4-additon on the same monomeric unit (Fig. 4).  $^{13}\text{C}$  NMR decoupled spectrum of P is displayed in Figure 5. The theoretical  $^{13}\text{C}$  NMR chemical shift data for five possible addition products were calculated by using appropriate tables.<sup>42</sup>  $^{13}\text{C}$  NMR shift data showed that P correlated well with the structure [Fig. 6(c,d)] indicating 1,2- and 1,4-addition at almost equal rate.

The powder diffraction X-ray spectra of unwashed CP contains three strongest line of  $d$ -spacing of NaBr (by-product of the polymer synthesis) [Fig. 7(a)] and washed CP, having a broad line, indicates an amorphous polymer [Fig. 7(b)].

ESR spectrum of microwave initiated RIP, CLP, and CP products revealed the signals with  $g$  values of 2.00552, 2.00549, and 2.00294, respectively [Fig. 8(a-c)], which were very close to  $g$  values of free electron.

Analysis of the surface morphologies of all type polymers was done by scanning electron microscope, (Figs. 9 and 10). In Figure 9(a), P had fine granular structure, significantly different from the CP having sponge-like structures [Fig. 9(b-d)] and tubular structures [Fig. 9(e-f)] were observed as the applied energy increased. However, as the amount water increased (0.5 to 1.0 mL) at 70 W, the coexistence of spongy and dendrite structures of CP and CLP was detected [Fig. 10(a)]. In Figure 10(b and c), structures of CP and CLP were more clearly seen when magnified 5000 times. At 350 W in 1 min, as the amount of water increased (0.5 to 5.0 mL), only dendrite structures of CLP were observed [Fig. 10(d)]. By the application of 700 W for 1 min in 5.0 mL water solution orange-colored coarse surface of RIP was detected [Fig. 10(e)]. The X-ray micro-



analysis system detected the existence of O, Br, and C on all of P, CP, CLP, and RIP whereas Na was detected on the unwashed CP.

The electrical conductivities of washed and unwashed CP were measured as 0.1 and 1 S cm<sup>-2</sup>, respectively whereas RIP and P were insulators.

### CONCLUSIONS

For the first time, synthesis of P and CP and/or CLP and/or RIP was achieved simultaneously from sodium 2,4,6-tribromophenolate by microwave energy in a very short-time interval. The % conversions of TBP are higher than TCP for P whereas lower for CP. P synthesis proceeds through 1,2- and 1,4-addition at almost equal rate leading to a branched polymer. The highest  $M_w$  value of P ( $2.97 \times 10^5$  g/mol), synthesized at 70 W in 5 mL at the end of 5 min, was achieved till now whereas the molecular weight of P from TCP was  $1.8 \times 10^4$  g/mol induction period for the polymerization of P is very short, less than 3 min at 70 W and 1 min at higher energies when compared with the other methods of synthesis in solution and in solid state. The optimum conditions for % conversion of P and CP were 70 W for 5 min in 5 mL water and 100 W for 1 min in 0.5 mL water, having maximum values of 23.6% and 27.2%, respectively. The %WL generally increased slightly as the time and energy increased and sharply decreased as the amount of water increased reaching the maximum value at 50.8% at the end of 1.5 min at 500 W. The direct synthesis of highly conducting polymer with the higher conductivity value of 1 S cm<sup>-2</sup>, compared to CP of TCP, was achieved in the absence of applied doping process in a very short time sequence. High  $T_g$  value of P and CLP indicated high rigidity, and ESR spectrum of microwave initiated RIP, CLP, and CP products revealed the signals very close to  $g$  values of free electron.

Analysis of the surface morphologies of all type polymers indicated fine granular, sponge-like, dendrite, and coarse surface structures for P, CP, CLP, and RIP, respectively.

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