The first conductive polymers were discovered in 1975. Then, they have attracted considerable attention due to their interesting electrical conductivity [1]. Nowadays, the studies related to conductive polymers have gained a new acceleration due to the rapid developments in science and technology. In most of these studies, it is aimed to give to the polymeric materials some properties of semiconductors. Some properties which do not naturally exist in semiconductors are trying to be imparted to conductive polymers synthetically [2]. Since conductive polymer compounds, which are the product of polymerization, have a wide area in polymer technology, comprehensive studies have been carried out to date [3,4]. Among these conductive polymers, phenothiazine based polymers has been extensively studied for their applications in various electronic and electrochemical devices [5,6]. They are good electron-donor and hole transport materials, since the phenothiazine rings have electron-rich sulfur and nitrogen heteroatoms. However, many of their features are still known to be limited. On the other hand, to date, various methods have been used for the synthesis of conductive polymers. Chemical, enzymatic, oxidative, and electrochemical polymerization methods are used effectively for the conversion of monomers carrying groups such as thiophene, pyrrole, and carbazole to conductive polymers. Electrochemical polymerization is one of the most important polymerization methods of recent years. The first electrochemical synthesis is the production of PANi polymer by Letheby [7]. Later this method was used by Szaways [8]. Since then, conductive polymers such as polypyrrole (PPy), polythiophene (PTh), polypurane (PF), polyphenylene (PP), and polycarbazole (PCz) with π-electron conjugate structure have been synthesized for use in many applications [9]. Electrochemical polymerization is highly advantageous over other polymerization methods because of its superior properties such as the reaction being carried out at room temperature, controlling the film thickness by varying the applied voltage or current, the homogeneity of the films, and not requiring purification [10]. Here, it is aimed to synthesize phenothiazine copolymers by constant potential electrolysis. For this, the monomer was prepared as described in the literature [11]. The structure of the synthesized monomers was illuminated by FT-IR, UV-Vis, 1H- and 13C-NMR techniques. Characterizations of copolymers were carried out by TG-DTA, CV, and GPC. In the final stage of the research, the solid-state degradation kinetics of phenothiazine based conductive polymers were calculated using TG-DTG data under non-isothermal conditions. Accordingly, to our knowledge, this study is the first report to exa-
To determine the electrochemical properties of resulting polymers, cyclic volt-ammograms were taken using CH Instruments 660 C Electrochemical Analyzer (CH Instruments, Texas, USA). The copolymers were coated on platinum or ITO. A platinum wire was used as a counter electrode. The non-aqueous Ag/Ag+ was determined as a reference electrode. After the coating of resulting copolymers was on ITO/glass surface via electro-oxidative polymerization, the films were rinsed with ACN. The thermal properties of synthesized polymers were determined by using Thermogravimetry-Differential Thermal Analysis (TG-DTA Perkin Elmer Diamond Thermal Analysis System) [12,13]. The measurements were carried out in an N₂ atmosphere between 20-1000°C, at the heating rate of 5, 10, 15, and 20°C/min.

**Synthesis of 3,7-dibromo-10H-phenothiazine**

A solution of 10 mmol (1.99 g) phenothiazine in 20 mL THF was placed in a 100 mL two-necked flask in an ice bath. To the resulting yellow solution, a solution of 22 mmol (3.91 g) of NBS dis-solved in 45 mL of THF was added dropwise for 1 hour. The dark green solution formed after the drop was cooled to room temperature and stirred for a further 24 hours. After the solvent removal, the residue was purified over silica gel. A light green product was obtained (2.50 g, yield: 70%) [14]. The reaction scheme for the synthesis of 3, 7-dibromo-10H-phenothiazine is given in Fig. 1.

**Synthesis of 3,7-di-2-thienyl-10H-phenothiazine**

In a 100 mL flask, 4 mmol (1.42 g) of 3, 7-dibromo-10H-phenothiazine was dissolved in a boiling ethoxyethanol-water (40: 4, 44 mL) mixture. 20 mmol (2.76 g) of K₂CO₃ was added to the resulting green solution and stirred at 130°C for half an hour. The dark green solution formed after the drop was cooled to room temperature and stirred for a further 24 hours. After the solvent removal, the residue was purified over silica gel. A light green product was obtained (2.50 g, yield: 70%) [14]. The reaction scheme for the synthesis of 3, 7-dibromo-10H-phenothiazine is given in Fig. 1.
of 2-thiophene boronic acid and 0.06 mmol (70 mg) [Pd (PPh)₃]₄ as catalyst was added and the mixture was refluxed at 130 °C for 2 hours. After two hours, the mixture was cooled down to room temperature and precipitated with the distilled water. The precipitation was carried out by stirring the mixture continuously with a magnetic stirrer. The precipitate was filtered and washed with an ethanol-acetone (1:1 v/v) solvent mixture. The resulting light yellow product was dried in a vacuum oven at 40°C for 6 hours (1.16 g, yield: 81%) [15]. The reaction scheme for the synthesis of 3, 7-di-2-thienyl-10H-phenothiazine is given in Fig. 2.

**Electrochemical Characterization**

Data from voltammograms were used to determine the electrochemical properties of the resulting copolymers on the ITO surface [16]. Electrochemical measurements were performed in a quartz cell using non-aqueous Ag/Ag⁺ as the reference electrode, platinum wire as the counter electrode, and ITO as the working electrode. 0.1 M TBAPF₆ was used as a support electrode.

**Electrochemical Copolymerization of TF Monomer with Thiophene and EDOT**

To determine the electrochemical properties of synthesized TF monomer by using cyclic voltamograms, CH Instruments 660 C Electrochemical Analyzer was used. The resulting copolymers were obtained by cyclic voltamograms repeated at a scanning rate of 50 mV/s in 0.1 M TBAPF₆/ACN. For this, 20 mg TF monomer and 0.9 ml thiophene (d: 1.05 g/cm³) were dissolved in 9 ml 0.1 M TBAPF₆/ACN and 1 ml BF₃.OET₂ support electrolyte solution. Copolymerization was then performed on the ITO surface at a scanning speed of 50 mV/s, with a potential range of (±1.8)-(-0.4) V. In the case of copolymerization of TF monomer with EDOT, 20 mg of TF monomer and 0.7 ml of 3,4-ethylene dioxythiophene (d:1.33 g/cm³, EDOT) were dissolved in 9 ml of 0.1 M TBAPF₆/ACN and 1 ml of BF₃.OET₂ support electrolyte solution. Copolymerization was performed by the electrochemical method to the ITO surface at a scanning speed of 50 mV/s, in the potential range (-1.6)-(+1.6) V. Synthetic pathway for electrochemical copolymerizations of the TF monomer with thiophene or EDOT is given in Fig. 3. The films of the resulting copolymer were coated onto the ITO surface by electro oxidative polymerization and then washed with ACN.
RESULTS AND DISCUSSION

Structural Characterization

3,7-di-2-thienyl-10H-phenothiazine used in electro-polymerization was synthesized from 3,7-dibromo-10H-phenothiazine according to standard literature procedures [11]. The chemical structure of each compound was confirmed by FT-IR, ¹H-NMR, and ¹³C-NMR spectra. Significant changes were also observed in the FT-IR spectra of each compound relative to the starting material pheno-thiazine. The C-Br bending peak of 3,7-dibromo-10H-phenothiazine was observed at 649 cm⁻¹ in the FTIR spectrum. Also, the N-H tensile peak of the thiomorpholine ring was determined at 3337 cm⁻¹, and the C-N peaks at 1311 cm⁻¹. In the ¹H-NMR spectrum, the hydrogen atom in the thiomorpholine ring showed a single signal at 8.85 ppm. Thus, both the FT-IR and ¹H-NMR spectra of this compound showed that the Bromine did not bind at the N-position. Also, the aromatic proton signal at 6.59 ppm (1H, binary) and 7.11 ppm (1H, single) was observed. On the other hand, it was deter-mined that the Hb proton at 7.14 ppm split into the doublet of the doublet due to the remote group interaction with the Ha proton. In the ¹³C-NMR spectrum, characteristic resonance peaks of six dif-ferent carbon atoms between 113-141 ppm were determined. All these results confirm that the -NH group on phenothiazine is protected and that the halogenation reaction takes place via the aromatic ring. However, in the FTIR spectrum of 3,7-di-2-thienyl-10H-phenothiazine, the C-Br peak at 649 cm⁻¹ was lost and the C-S peak in the thiomorpholine ring was observed at 798 cm⁻¹. The N-H and C-N peaks in the thiomorpholine ring were also observed, respectively, at 3334 cm⁻¹ and 1298 cm⁻¹. In the ¹H-NMR spectrum, the N-H bond in the thiomorpholine ring was exhibited as a single peak at 8.96 ppm. Also, this compound gave peaks at 7.49 ppm (1H, doublet), 7.42 ppm (1H, doublet), 7.33 ppm (1H, singlet), 7.28 ppm (1H, doublett), 7.13 ppm (1H, triplet) and 6.75 ppm (1H, doublet). However, in the ¹³C-NMR spectrum, this compound showed resonance signals of ten different car-bon atoms between 115-143 ppm.

Optical and Electrochemical Properties

UV-vis spectra of PTFT and PTFE copolymers were taken from the ITO surface and given in Fig. 4. The UV-vis spectrum of the TF monomer exhibited a maximum absorption (λmax) at 364 nm. This maximum peak is attributed to π- π* transitions in the aromatic ring. The UV-vis spectrum of the PTFT copolymer showed a peak at 397 nm shifting to 33 nm red when compared to the TF monomer. This is related to the higher conjugation of PTFT than the TF monomer. On the other hand, PTFE showed two peak absorption peaks at 460 nm and 643 nm due to high conjugation compared to both TF and PTFT copolymer. The lowest spectral energy edge bands of the absorption spectra of FT and PTFT copolymer were measured as 481 and 494 nm, respectively. Accordingly, the optical band gaps were 2.57 eV for TF monomer and 2.5 eV for PTFT. In the case of PTFE, the lowest spectral energy edge bands λmax₁ and λmax₂ were determined to be 567 and 783 nm, respectively. Accordingly, the optical band gap for PTFE was calculated as 2.18 eV for λmax₁ and 1.58 eV for λmax₂.

On the other hand, PTFT and PTFE copolymers were formed by electrochemical cyclic voltammetry at a scanning rate of 50 mV/s. The first cyclic voltammograms for TF, PTFT, and PTFE co-polymers are given in Fig. 5.

Fig. 5 show the first cyclic voltammograms of PTFE and PTFT at a scanning speed of 50 mV/s. According to Fig. 5, the first oligomeric products were taken in the range of -1.4-1.6 V for PTFT and -0.4-1.6 V for PTFE. The oxidation peak of PTFE was observed at 0.51 V. Its reduction peak was at -0.39 V. Also, the oxidation peak and reduction of PTFT was exhibited at 0.82 V and 0.44 V, respectively. Accordingly, each voltammogram showed a single oxidation and reduc-tion peak with a reversible character. Functioned thiophene compounds such as thiophene or 3,4-ethylene dioxythiophene (EDOT) can be very easily oxidized. The radicals formed as a result of this oxidation can be joined together to form a polymerization reaction easily on the ITO elect-rode. In here PTFE and PTFT copolymers were obtained by cyclic Voltammetry. Voltamgram collection was continued until no electroactive species could be recovered from the monomer. The copolymeriza-tion reaction of TF monomer with EDOT was carried out between -1.4-1.6 V. In the volt-amgram of PTFE, the oxidation peak at 0.51 V shifted
to 0.88 V and the reduction peak at -0.39 V shifted to 0.62 V. On the other hand, the copolymerization reaction of TF monomer with thiophene was carried out between -0.4-1.8 V. Here, the oxidation potential shifted from 0.82 V to 1.27 V and the reduction potential shifted from 0.44 V to 0.62 V. The sequential voltammograms obtained by consequent the shift of the reaction to the high potentials by binding of the active species showed that the copolymerization reactions took place [17]. Cyclic voltammograms for electroactive PTFE and PTFT copolymers deposited on ITO are given in Fig. 6.

Furthermore, the dependence of the scanning speed versus the current density of copolymers de-posted on ITO in the 0.1 M TBAPF6/ACN support solution is shown in Figs 7 and 8. Figs 7 and 8 showed a linear relationship between current density and scanning speed. This event was at-tributed to the non-diffusion process.

It was determined that there is a linear relationship between scanning velocities and current in-tensives in Figs 7 and 8, which shows the scanning velocity dependence in copolymerization re-actions. GPC was used to calculate the average molar masses of the soluble portions of the PTFE and PTFT. The results are given in Table 1. According to GPC analysis, polymers have low PDI values. Low PDI indicates a controlled polymerization reaction [18].

| Copolymers | M_n (gmol⁻¹) | M_w (gmol⁻¹) | PDI |
|------------|-------------|-------------|-----|
| PTFE       | 22782       | 32158       | 1.43|
| PTFT       | 19380       | 29113       | 1.38|

**Table 1. GPC analysis of PTFE and PTFT**

**Kinetic Evaluation**

Non-isothermal methods are often used for the evaluation of mechanisms and kinetics of solid-state reactions. Most of these thermo analytic methods are based on the Arrhenius equation.

\[
k = A \exp\left(-\frac{E}{RT}\right)
\]

and rate expression occurs

\[
da / dt = kf(a)
\]

Here, \( f(a) \) is the kinetic function. \( \alpha \) is the conversion fraction. If \( f(a) = (1-\alpha)^n \) and \( dT / dt = \beta \) occur equally at constant temperature, Eq. 2 can be written as follows.

\[
g(a) = \int_{0}^{1} \frac{1}{(1-n)^n} \, da = \frac{\beta}{\beta - \alpha} \exp\left(-\frac{E}{RT}\right) dT
\]

Here; \( n \) is the degree of reaction. Eq. 4 can be written for the special case \( n = 1 \). On the other hand, Eq. 5 can be written for \( n \neq 1 \) values.

\[
\int_{0}^{1} \frac{1}{(1-n)^n} \, da = -\ln(1-a)
\]

\[
\int_{0}^{1} \frac{1}{(1-n)^n} \, da = \frac{1-(1-a)^{1-n}}{1-n}
\]

Many techniques are using different approximations to solve Eq. 3. The following isoconver-sional methods were used to calculate kinetic parameters related to the solid-state degradation of copolymers.

**Tang Method**

When an appropriate approach is used for the solution of Eq. 2 and the logarithm of both sides is taken, the equation given below can be obtained [19].

\[
\ln \left( \frac{g(a)}{T_{1001450}} \right) = \ln \left( \frac{AR}{Rg(a)} \right) + 3.635041 \left( \frac{1.001450E}{RT} \right) - 1.894661 \ln E
\]
Here, the activation energy (Ea) can be found from the slopes of the Arrhenius plots at different heating rates.

**Kissenger-Akahira-Sunose Method**

This method is an integral isoconversional method and Ea is calculated in similar ways as in other methods. According to the Kissenger-Akahira-Sunose method, an equation is obtained as follows [20,21].

\[ \ln \left( \frac{g(\alpha)}{T^2} \right) = \ln \left( \frac{AE}{Eg(\alpha)} \right) - \frac{E}{RT} \quad (7) \]

**Kissinger Method**

Ea can be calculated without using any reaction mechanism, by using the following equation [20].

\[ \ln \left( \frac{E}{RT} \right) = \left[ \frac{\ln(1 - a_{\text{max}})}{\beta_{\text{max}}} \right] - \frac{E}{RT_{\text{max}}} \quad (8) \]

where \( \beta, T_{\text{max}}, A, a_{\text{max}} \) and \( n \) are the heating rate, temperature, pre-exponential factor, the maximum conversion fraction, and reaction order. Ea can be calculated from the graph of \( \ln (\beta/T_{\text{max}}^2) \) versus 1000 / \( T_{\text{max}} \).

**Flynn-Wall-Ozawa Method**

This is an integral method. In this technique, \( A, f(\alpha) \), and \( E_a \) are independent of both conversion fraction and the temperature. This technique uses the following logarithmic equation [22,23].

\[ \log \beta = \log \left( \frac{AE}{R} \right) - \log \beta + \log \left( \frac{E}{RT} \right) \quad (9) \]

Using Doyle approximation and assuming \( E/RT > 20 \) for the integral function, the above equation can be simplified as follows

\[ \log \beta = \log \left( \frac{AE}{R} \right) - \log g(\alpha) - 2.315 - 0.4567 \frac{E}{RT} \quad (10) \]

**Friedman Method**

The Friedman method uses Eq. 11 based on the Arrhenius equation for thermal decomposition kinetics [24].

\[ \ln \left( \frac{\text{d} \alpha}{\text{d}t} \right) = \ln(\alpha) + \ln(1 - n) - \frac{E}{RT} \quad (11) \]

In the above equations, \( \alpha, g(\alpha), \beta, T_{\alpha}, A, \) and \( R \) are the degree of reaction, integral function, heating rate, temperature, pre-exponential factor, and gas constant, respectively. A line is obtained by plotting \( 1/T \) versus \( \ln (\text{d} \alpha / \text{d}t) \) and \( E/R \) is determined from the slope of this line.

**Thermal Decomposition Kinetic**

The decomposition kinetics of the copolymers were performed by the TG technique. Kinetic parameters were calculated using multiple heating rate methods. The TG curves obtained in the dynamic nitrogen atmosphere of the copolymers were given in Fig. 9. TG experiments were carried out in nitrogen of 50 mL min\(^{-1}\) gas at heating rates of 5, 10, 15, and 20 \(^{\circ}\)C min\(^{-1}\). Analyses were performed by using 8-10 mg samples. All TG curves showed that copolymers degrade in one step.

The first decomposition temperature (\( T_{\alpha_{\text{on}}} \)) in addition to the temperatures at which 20% and 50% mass losses occurred were found from the TG thermograms. Furthermore, DTG curves were used to determine temperatures (\( T_{\alpha_{\text{max}}} \)) corresponding to the maximum decomposition rate. DTA curves were used to determine the exothermic and endothermic nature of decomposition. According to the TG curve of PTFE copolymer at a heating rate of 5 \(^{\circ}\)C/min, the initial decomposition temperature was 104 \(^{\circ}\)C. 20% and 50% mass loss was observed at 245 \(^{\circ}\)C and 328 \(^{\circ}\)C, respectively. The amount of residue at 1000\(^{\circ}\)C was calculated to be about

![Figure 9. TG / DTG and DTA thermograms of PTFE (a) and PTFT (b) at different heating rates](image-url)
11%. This compound was degraded in one step and \( T_{\text{max}} \) was found as 303 °C. During the decomposition of the PTFE copolymer, the presence of an endothermic peak at 318 °C was observed in the DTA curve. According to the TG curve obtained at a heating rate of 5 oC/min of PTFE copolymers the initial decomposition temperature was determined as 231 oC. The temperatures at which 20% and 50% mass loss were observed were 283 °C and 347 °C, respectively. The amount of residue at 1000°C was calculated to be approximately 8%. This compound was determined to be degraded in one step. From the DTG curve, \( T_{\text{max}} \) was calculated as 381 °C. During the decomposition of PTFT copolymers, the presence of an endothermic peak at 399 °C was observed in the DTA curve.

### Kinetic Studies

There are many techniques for the solution of Eq. 3. In this study, several techniques were used to investigate the kinetic parameters of copolymers. These techniques are FWO, KAS, Tang, and Kissinger methods. For these methods, the TG curves at multiple heating rates are used. For the calculation the kinetic parameters of copolymers, firstly Kissinger method and isothermal methods were used. According to the Kissinger method, \( E_a \) related to the thermal decomposition kinetics of PTFE and PTFT was found to as 75.23 kJ mol\(^{-1}\) and 83.47 kJ mol\(^{-1}\), respectively. According to equation [6], \( E_a \) can be found from the graph of \( 1000 / T \) versus \( \ln (\beta / T^{1.894661}) \). The average value of \( E_a \) for PTFE and PTFT was found to as 75.8 kJmol\(^{-1}\) and 93.8 kJmol\(^{-1}\), respectively. The values obtained for copolymers were given in Table 2. KAS is another method used in this study. In the conversion fractions given \( \alpha = 0.05-0.95 \), \( E_a \) determined from the slope of the graph of \( \ln (\beta / T^3) \) vs sus 1000/T was calculated as 74.7 and 92.8 kJmol\(^{-1}\) for PTFE and PTFT, respectively. The result is very close to the value obtained by the Tang method. Another method used in this study is FWO. At a constant conversion, the slope of the line obtained from log \( \beta \) vs 1000/T is equal to 0.456 E/RT. \( E_a \) can be found from here. In Fig. 15, the plots of ln \( \beta \) versus 1000/T at a different conversion value are plotted, and the values found here are 75.7 and 93.1 kJmol\(^{-1}\) for PTFE and PTFT, respectively. As a result, the \( E_a \) obtained by all three methods for copolymers were very close to each other. Finally, \( E_a \) for PTFE and PTFT using the Friedman method was determined as 78.8 and 97.9 kJmol\(^{-1}\), respectively.

Fig. 10 shows the Arrhenius type curves in the range of \( \alpha = 0.05-0.95 \). As given in Fig. 10, the minimum \( E_a \) values required for copolymers to start degradation is 64.3 and 94.6 kJmol\(^{-1}\) for PTFE and PTFT, respectively. \( E_a \) required for the degradation step of PTFE and PTFT copolymer are very close to each other within the limits of 0.2 < \( \alpha < 0.9 \). When the

| Decomposition Steps | PTFE | PTFT |
|--------------------|------|------|
| \( \alpha \)       | Tang | KAS  | FWO | FR | Tang | KAS  | FWO | FR |
| 0.05               | 65.8 | 64.3 | 65.9 | 68.9 | 92.6 | 94.6 | 94.6 | 100.5 |
| 0.1                | 69.8 | 68.9 | 70.1 | 72.5 | 94.7 | 93.4 | 94.6 | 97.8 |
| 0.2                | 72.3 | 71.1 | 72.4 | 75.8 | 93.2 | 92.6 | 92.4 | 97.5 |
| 0.3                | 76.8 | 75.6 | 77.1 | 79.8 | 92.7 | 91.6 | 92.9 | 96.7 |
| 0.4                | 75.7 | 74.5 | 76.4 | 78.9 | 91.4 | 90.7 | 91.4 | 96.9 |
| 0.5                | 76.4 | 75.4 | 75.6 | 79.7 | 92.3 | 91.5 | 91.6 | 96.5 |
| 0.6                | 76.5 | 75.8 | 75.9 | 78.6 | 91.4 | 90.8 | 92.3 | 95.7 |
| 0.7                | 76.9 | 75.4 | 75.2 | 79.4 | 92.3 | 91.7 | 91.5 | 96.4 |
| 0.8                | 77.1 | 76.7 | 76.3 | 80.2 | 92.5 | 91.4 | 90.8 | 96.2 |
| 0.9                | 82.3 | 81.3 | 83.5 | 84.3 | 96.8 | 95.3 | 94.7 | 99.6 |
| 0.95               | 85.2 | 83.2 | 85.2 | 88.9 | 98.9 | 97.8 | 98.8 | 103.6 |

Table 2. \( E_a \) values obtained by Tang, KAS, FWO, and Friedman methods
mass loss reaches 90%, co-polymers have required energy of 90 kJmol$^{-1}$.

**CONCLUSION**

Here, a phenothiazine monomer whose preparation was previously presented in the literature was again synthesized and its PTFE and PTFT were prepared by the electropolymerization method. Characterization of the phenothiazine-based monomer was performed by UV-Vis, FT-IR, $^1$H-NMR, and $^{13}$C-NMR spectra. Also, the average molecular mass of PTFE and PTFT by the GPC was found as approximately 13850 g mol$^{-1}$ and 22782 g mol$^{-1}$, respectively. In the following study, the solid-state decomposition kinetics of PTFE and PTFT were evaluated by using thermal curves. The kinetic parameters of the decomposition were determined using Kissinger, Tang, Kas, FWO, and Friedman methods based on multiple heating rates. Accordingly, the mean values of $E_a$ obtained by the Tang method for PTFE and PTFT were 75.8 and 93.8 kJmol$^{-1}$, respectively. The obtained co-polymers can be used in various applications such as organic field-effect transistors [25], chemiluminescence [26] and light-emitting diodes [27] due to their the electro-optical properties.

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