



Article

Preparation of Polycarbazole Nanofibers Using an Electric Field and the Investigation of Its Electrical Conductivity

Seyed Hossein Hosseini ^{1,*}, Amir Abbas Kazemi ² and Seyed Arash Hosseini ³¹ Nika Pooyesh Industrial Research Institute, NBP Economic Group, Tehran 1477893855, Iran² Department of Chemistry, Islamic Azad University Saveh Branch, Saveh 1631676757, Iran³ Art, Design and Information Technology Center, George Brown College, Toronto, ON M5R 1M3, Canada

* Correspondence: dr.shhosseini@gmail.com

Abstract: In conventional chemical and electrochemical oxidation methods, it is very difficult to control the active centers, and the average prepared polymers are short and wide. The use of an electric field creates the most stable intermediate form of active centers, as well as permitting a longer half-life. Therefore, this increases the physical resistance and electrical conductivity of the polymer. In this paper, polycarbazole nanofibers were prepared using an electric field, reporting on its influences on the polymerization of carbazole. Therefore, its electrical conductivity and some physical properties were investigated. We observed the nanofibers' shape, increasing electrical conductivity, thermal resistance and a higher molecular weight with the synthesized polycarbazole under an electric field compared to the polymer synthesized in the same conditions in the absence of an electric field. First, we chemically synthesized polycarbazole at different times. Additionally, to find the optimizing conditions, we changed certain parameters, such as the ratio of the obtained molar of initiator to monomer, the oxidant, initiator and solvent, separately, and compared the obtained results. Then, we repeated this reaction in the best conditions and under different electric fields in constant time, allowing us to characterize the shape, mass and conductivity. Next, the polymerization was carried out at the best electric field in different times. Finally, the best time and amount of electric field for polymerization were determined. The electrical conductivity of polycarbazoles was studied with the four-probe method. The conductivity of the films oxidized using FeCl₃ (dry) and protonated with p-toluenesulfonic acid (PTSA) at 3 h was higher than 8.9×10^{-4} S/cm under a 12 KV/m electric field. Additionally, the results showed an enhanced thermal resistance to ageing.

Keywords: conducting polymer; polycarbazole; nanofiber; electric field; polymerization

Citation: Hosseini, S.H.; Kazemi, A.A.; Hosseini, S.A. Preparation of Polycarbazole Nanofibers Using an Electric Field and the Investigation of Its Electrical Conductivity. *Nanomanufacturing* **2023**, *3*, 113–122. <https://doi.org/10.3390/nanomanufacturing3010007>

Academic Editors: Wenjie Zhou,
Sotirios Baskoutas and
Riccardo Frisenda

Received: 19 December 2022

Revised: 24 February 2023

Accepted: 6 March 2023

Published: 17 March 2023



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1. Introduction

Conducting polymers (CPs), such as polyaniline, polypyrrole, polycarbazole and their derivatives, have been widely investigated because of their unique properties, such as electronic and optical properties, flexibility, easy processability, low cost, sensor applications, corrosion resistance, environmental stability, photoluminescence and new applications [1–4]. These polymers have found purpose in a wide range of applications, including as absorbers of electromagnetic waves, in smart materials and the medical industry [5–9]. These compounds are synthesized through the use of different methods, and produce different products, with each product having the potential for different uses. CPs can be prepared with different structures, such as nanofibers, lattices, core-shell and coaxial structures, etc. Each of these structures can have different applications as well [10–12]. Polycarbazole and its derivatives have been extensively used for the last three decades, although interest in these materials briefly decreased. However, the increasing demand for conductive polymers in several applications, including light-emitting diodes (OLEDs), capacitors, memory devices, among others, has renewed the interest in carbazole-based materials [13].

This polymer, like other CPs, can be prepared in different ways and has a very low solubility. They usually have a low molecular weight and little spatial order, so they have limited applications [1,13]. The polymerization of CPs under electric and magnetic fields was first performed by us [14–16]. Polymerization under these fields allows for the spatial order and molecular mass of the polymers to increase, thus, expanding their functional aspects.

The last study conducted by us showed that the molecular weight and electric conductivity of the polymers increased under an electric field compared to those from similar conditions with the exception of the absence of an electric field. A number of mechanisms for electric field effects in chemical reactions have been established, well documented and received a proper theoretical analysis. The active center in conducting polymers is the radical cation. On the other hand, carbazole creates several active positions, of which only one form is more stable, and produces a uniform, flat polymer with a greater length and molecular mass. In the presence of an electric field, the three and six or c and c' forms of the carbazole radical cation are much more stable, thereby increasing the molecular mass and electrical conductivity of the polymer. Figure 1a shows the polar intermediate of a monomer and Figure 1b shows the orientation of a polar monomer in an electric field. A polar oxidative carbazole can rotate toward positive and negative electric fields.

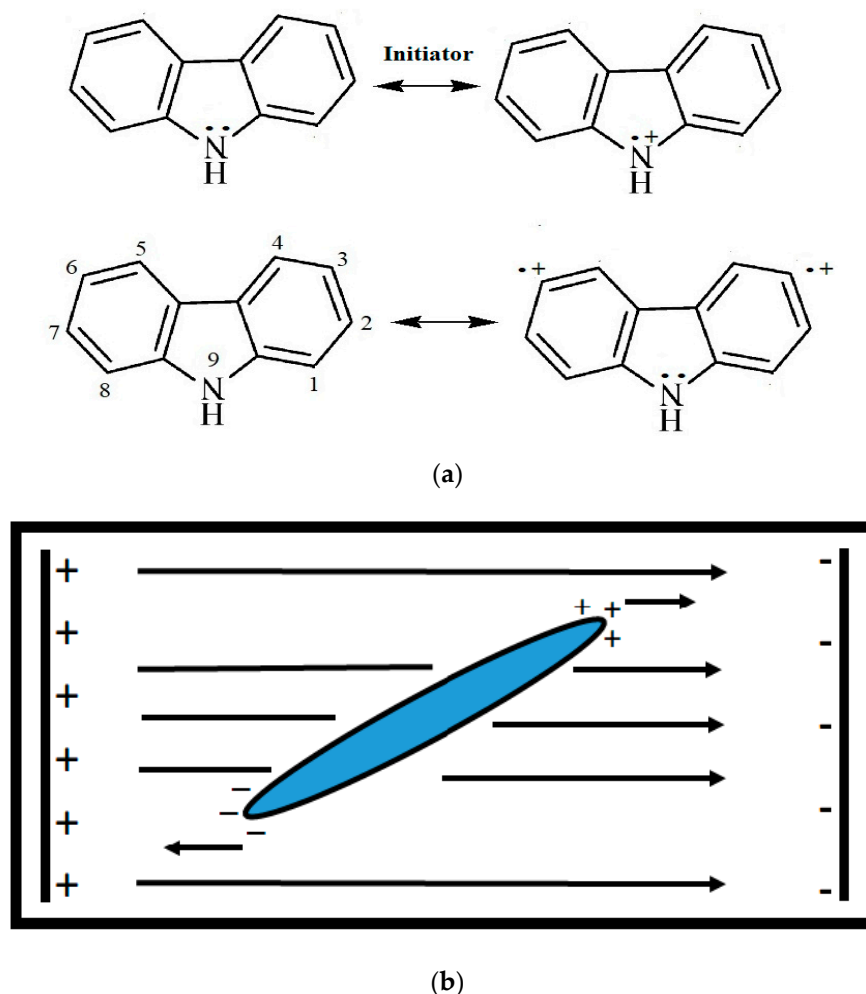


Figure 1. (a) Polar intermediate of monomer and (b) orientation of a polar monomer in electric field.

Orientation techniques are commonly used on conducting polymers to improve their conductivity or to induce the anisotropy of conductivity. In this research work, we perform polymerization in a constant electric field. Due to the fact that the carbazole monomer can increase the polarity after oxidation through the initiator, they would orient under the

influence of the field and create the most regular polymer chains. We describe studies on the dependency of the electrical conductivity and morphology of polycarbazole to using an electric field when used in a field effect experimental configuration. Additionally, we report on further studies on different conditions of the polycarbazole synthesis. The structure of products was evaluated with FT-IR, SEM and using thermogravimetric analysis (TGA) techniques.

2. Experimental Section

2.1. Materials and Instrumentals

The carbazole monomer was purchased from the company Merck and purified with the use of xylene. Other chemicals, such as ceric ammonium nitrate (CAN), FeCl_3 (dry), KMnO_4 , $\text{Fe}(\text{ClO}_4)_3$, p-toluenesulfonic acid (PTSA), tetrahydrofuran (THF), dimethylformamide (DMF), dimethylsulfoxide (DMSO), acetone and other reagents, were also purchased from the company Merck and purified per standard procedure before use.

Electrical conductivity was measured with a four-probe device (homemade, ASTM Standards, F 43–93). The electric field device that was applied was by Hipotronics S.O. No. 004390-00, with an HV power supply, model 830.50, made in the USA. A Fourier-transform-infrared spectrometer, FT-IR, (Bruker) was used for the spectral measurements of the polymer and reported (sh = sharp; w = weak; m = medium; b = broad). The thermal properties (thermogravimetric analysis (TGA)) of the polymer were assessed with an STA 625-PL from Thermal Science and a heating rate of $10\text{ }^\circ\text{C min}^{-1}$. Scanning electron microscopy (SEM) was employed to determine the type of surface morphology of the polymer. A Cambridge S-360 SEM device was used for this purpose.

2.2. Preparation of Polycarbazole in the Absence of an Electric Field

First, the project team followed the same instructions to oxidize $59 \times 10^{-5}\text{ M}$ carbazole with $29 \times 10^{-4}\text{ M}$ from FeCl_3 (dry), KMnO_4 , $\text{Fe}(\text{ClO}_4)_3$ and $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$ (CAN) as different initiators and DBSA (1M) as dopant in organic media separately. Carbazole (0.1 g) and 2 mL DBSA were dissolved in 5 mL of acetone in a 10 mL volumetric flask. Initiators was dissolved in 3 mL acetone, then was added (Table 1). The polycarbazole precipitate was collected on a filter and then washed with acetone. The polycarbazole powders were dried in air and then in vacuum at $60\text{ }^\circ\text{C}$. Then, their weights and electrical conductivities were measured with the four-probe method [10,11].

Table 1. Electrical conductivity of produced polycarbazole with different initiators and PTSA (1M) as the dopant in acetone, in absence and presence of 12 KV/m electric field after 3 h.

Absence of Electric Field			Under E = 12 KV/m		
Initiators	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta \times 10^{-6}\text{ S/cm}$)	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta_E \times 10^{-4}\text{ S/cm}$)	$r = \delta_E/\delta$
CAN	0.079	5.5	0.041	2.8	50.9
KMnO_4	0.063	2.3×10^{-7}	0.028	5.2×10^{-6}	22.6
FeCl_3 (dry)	0.093	7	0.045	8.9	127.1
$\text{Fe}(\text{ClO}_4)_3$	0.021	7.8	0.084	3.1	39.7

Then, to identify the best molar ratio of initiator to monomer, the values 1/1, 1/3, 1/5 and 1/7, using the best initiator, were examined (Table 2). The polycarbazole powders were prepared in a similar manner with the best molar ratio of initiator to monomer using tetrahydrofuran (THF), dimethylformamide (DMF), dimethylsulfoxide (DMSO) and acetone as the solvents. These solvents had different dielectric constants (Table 3). Additionally, by using the best obtained initiator and solvent, the polycarbazole was prepared over different times (1, 2, 3, 4 and 5 h) at room temperature ($\sim 18\text{--}24\text{ }^\circ\text{C}$) in a similar manner (Table 4).

Table 2. Electrical conductivity of polycarbazole produced with different molar ratios of FeCl_3 (dry) initiator to monomer, with PTSA (1M) as the dopant in acetone and the absence of an electric field after 3 h.

Sample	Molar Ratio of Initiator to Monomer	Color Change Time (min)	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta \times 10^{-6} \text{ S/cm}$)
1	1/1	5	0.004	2.3
3	1/3	5	0.039	6.6
4	1/5	5	0.093	6.9
5	1/7	5	0.071	7.1

Table 3. Electrical conductivity of polycarbazole produced with FeCl_3 (dry) and PTSA (1M) as the initiator and dopant (1/5 mol ratio) in different solvents, in the absence and presence of a 12 KV/m electric field after 3 h.

Absence of Electric Field			Under E = 12 KV/m		
Solvents	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta \times 10^{-6} \text{ S/cm}$)	Weight of Produced Polymer (g)	Electrical Conductivity (S/cm)	$r = \delta_E/\delta$
Acetone	0.093	6.9	0.045	8.9×10^{-4}	128.9
THF	0.063	6.1	0.072	6.5×10^{-4}	106.5
DMSO	0.018	1.5	0.093	5.6×10^{-5}	37.3
DMF	0.048	2.4	0.08	6.4×10^{-5}	26.6

Table 4. Electrical conductivity of polycarbazole produced with FeCl_3 (dry) (PTSA 1M in acetone) as the initiator (1/5 mol ratio) at different times, in the absence and presence of a 12 KV/m electric field.

Absence of Electric Field			Under E = 12 KV/m		
Time of Polymerization (h)	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta \times 10^{-6} \text{ S/cm}$)	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta_E \times 10^{-4} \text{ S/cm}$)	$r = \delta_E/\delta$
1	0.022	1.1	0.023	4.6×10^{-5}	42.2
2	0.028	3.2	0.038	2.7	85.1
3	0.036	6.9	0.046	8.9	128.9
4	0.039	7.1	0.045	3.5	49.2
5	0.039	7.3	0.045	1.2	16.4

FT-IR (KBr): 3344.9 (b), 1700 (w), 1599–1450 (sh), 1403.21 (m), 1325–1188 (m), 1117–1097.73 (sh), 883–729 (w), 685.94 (m) and 655–473 (w) cm^{-1} .

2.3. Preparation of Polycarbazole under an Electric Field

Under the best conditions, carbazole polymerization was performed under different electric fields (5, 10, 15, 20, 25 and 30 KV/m) in 3 h at room temperature (Table 4). Here, to find the best electric field level, the polymerization was conducted under 11, 12, 13 and 14 KV/m (Table 5). Then, their weights and electrical conductivity were measured. However, the polycarbazoles were prepared using the same prior reaction condition but over various time spans (1, 2, 3, 4 and 5 h) in the best electric field (Table 4). Finally, we carried out the polymerization again under the best obtained results in the various initiators and solvents, respectively (Tables 1 and 3).

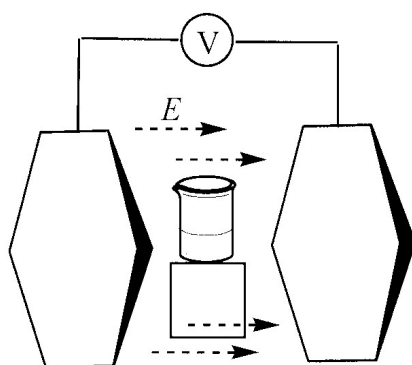
Table 5. Electrical conductivity of polycarbazole produced with FeCl_3 (dry) (PTSA 1M in acetone) as the initiator (1/5 mol ratio) in different electric fields after 3 h.

Sample	Electric Field (KV/m)	Weight of Produced Polymer (g)	Electrical Conductivity ($\delta_E \times 10^{-4}/\text{cm}$)
1	5	0.069	8.4×10^{-5}
2	10	0.078	1.4
3	11	0.043	4.5
4	12	0.045	8.9
5	13	0.049	7.3
6	14	0.046	5.4
7	15	0.027	4.9
8	20	0.018	1.2
9	25	0.013	8.4×10^{-5}

FT-IR (KBr): 3412 (m), 3047 (w), 1599 (sh), 1492 (w), 1449 (sh), 1325 (sh), 1236 (sh), 1064 (w), 927 (m), 747 (sh), 725 (sh), 573 (sh) and 443 (sh) cm^{-1} .

3. Results and Discussion

The polymerization scheme under the electric field is shown in Figure 2. The distances and conditions of the experiments are mentioned in the text.

**Figure 2.** The polymerization scheme under the electric field.

3.1. Spectrometric Studies

Figure 3 shows the FT-IR spectrum of the polycarbazole prepared under the best conditions (in acetone, FeCl_3 and for 3 h) and electric field (12 KV/m). The peaks of significance in this spectrum were 3412 cm^{-1} (N-H stretching H-bonding), 3047 cm^{-1} (C-H aromatic stretching), 1599 cm^{-1} (C=C stretching aromatic), $1492, 1449 \text{ cm}^{-1}$ (C=N stretching aromatics for different benzoic quinoic forms), 1325 cm^{-1} (C-C stretching different aromatics), 1236 cm^{-1} (C-N stretching aromatics) and $725, 747 \text{ cm}^{-1}$ (C-C stretching different aromatic internal polymeric bonding forms). The FT-IR spectrum of the polycarbazole prepared in the same conditions and in the absence of an electric field was recorded in the Experimental Section. There was no noticeable differences except for the sharpening of the stretching vibrations of the aromatic ring, which was due to the increased electrical conductivity of the polymer.

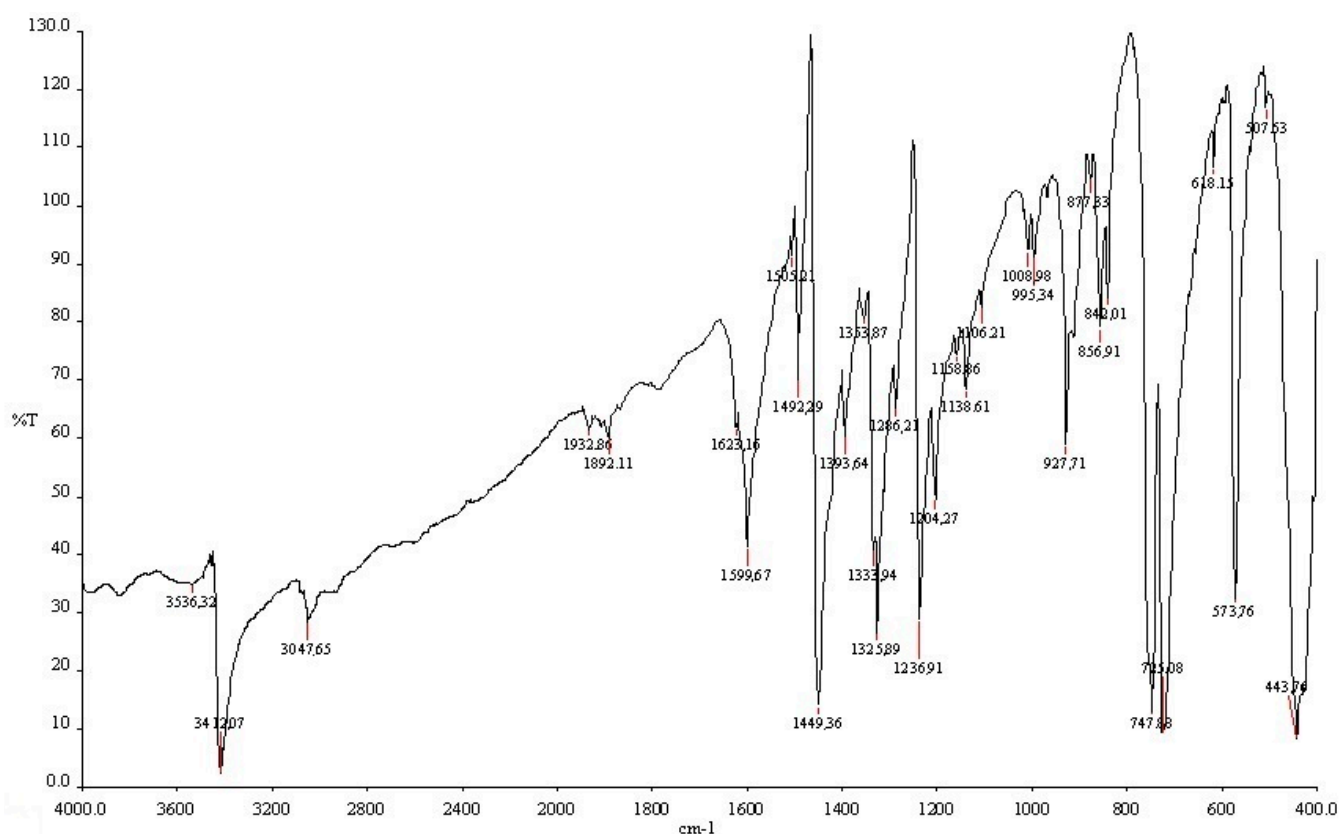


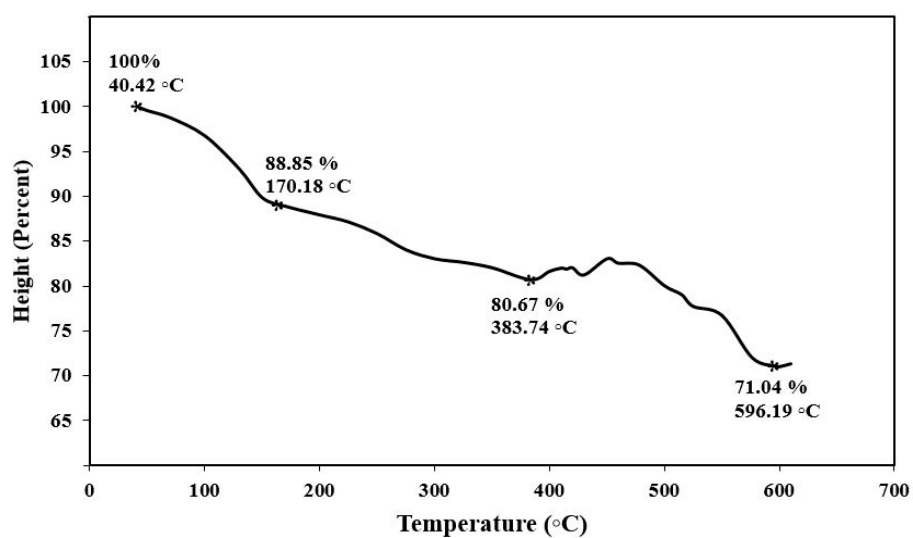
Figure 3. FT-IR spectrum of polycarbazole prepared under the best conditions (acetone, FeCl_3 , 3 h), under an electric field (12 KV/m).

3.2. Thermal Properties Studies

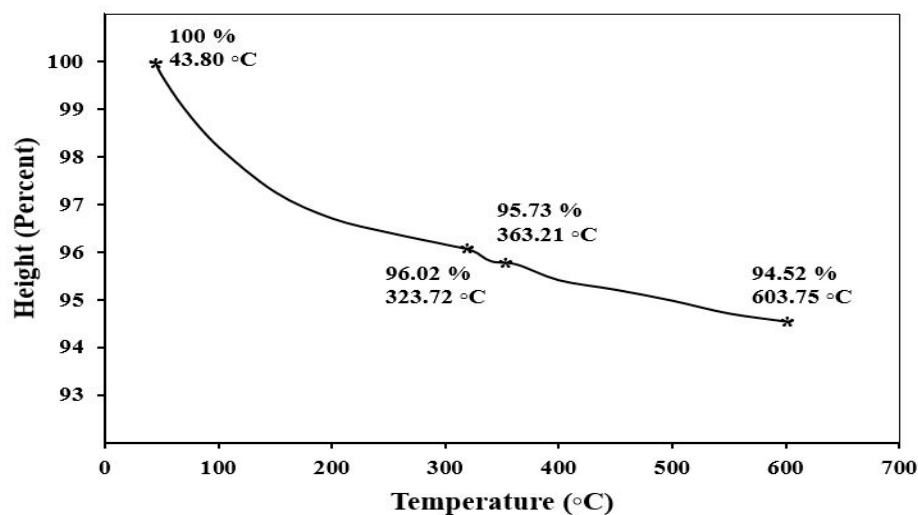
Figure 4a,b show the TGA thermograms of the polycarbazole before and after being exposed to an electric field, respectively. A comparison of the figures showed that the polycarbazole produced under an electric field was more stable than the polycarbazole produced in the absence of an electric field. In Figure 4b, we could see that the polymer was stable up to 323.72 °C and lost some weight (4.3%) in the region from 43 °C to 363.21 °C, which relates to the evaporation of water, the solvent and most likely some of the oligomers of the polycarbazole, which began to be destroyed. Additionally, the polymer was even stable up to 600 °C, with only 1% of weight being lost, while, as shown in Figure 4a, the thermogram had a more varying and reduced weight. This evidence came as a result of the considerable increase in molecular mass and spatial order, as well as the reduction in the amount of obtained oligomers.

3.3. Scanning Electron Microscopy Studies

Figure 5a,b show the SEM images of the polycarbazole synthesized in the absence and presence of an electric field, respectively. Figure 5a shows the disorder and amorphous shape of the polycarbazole chains that grew on the form of the bulk polymerization. However, Figure 5b shows the SEM of the polycarbazole synthesized in the presence of an electric field. Figure 5b shows that the polycarbazole had relatively high regular and fibrous surfaces. This aspect was caused through the growth of more polymer chains with the gathering and development of order in the polymer morphology under an electric field. As observed, the preparation of polycarbazole under normal conditions produced an amorphous polymer with a low molecular weight, while the polymers prepared under the electric field produced high-molecular-weight polymers and nanofibers.



(a)



(b)

Figure 4. TGA thermograms of polycarbazole prepared in (a) the absence and (b) presence of an electrical field.

3.4. Electrical Conductivity Studies

The obtained polymer had a high purity, because the excess initiator was readily washed with acetone. The purity was confirmed through an elemental analysis. The conductivity of all the samples was measured at room temperature with a four-probe method on pellets compressed at 700 MPa, 13 mm in diameter and 1–1.5 mm thick [6–8,11,12].

The carbazole was polymerized using four different initiators, including FeCl_3 (dry), CAN, KMnO_4 and $\text{Fe}(\text{ClO}_4)_3$; the electrical conductivities of the produced polycarbazoles are shown in Table 1. The best experimental molar ratio (monomer/initiator) was defined as 1/5. We conducted all the experimental work in the same ratio. Then, in the presence of the best initiator, the polymerization time was changed to between 1 and 5 h, with the best polymerization time being 3 h.

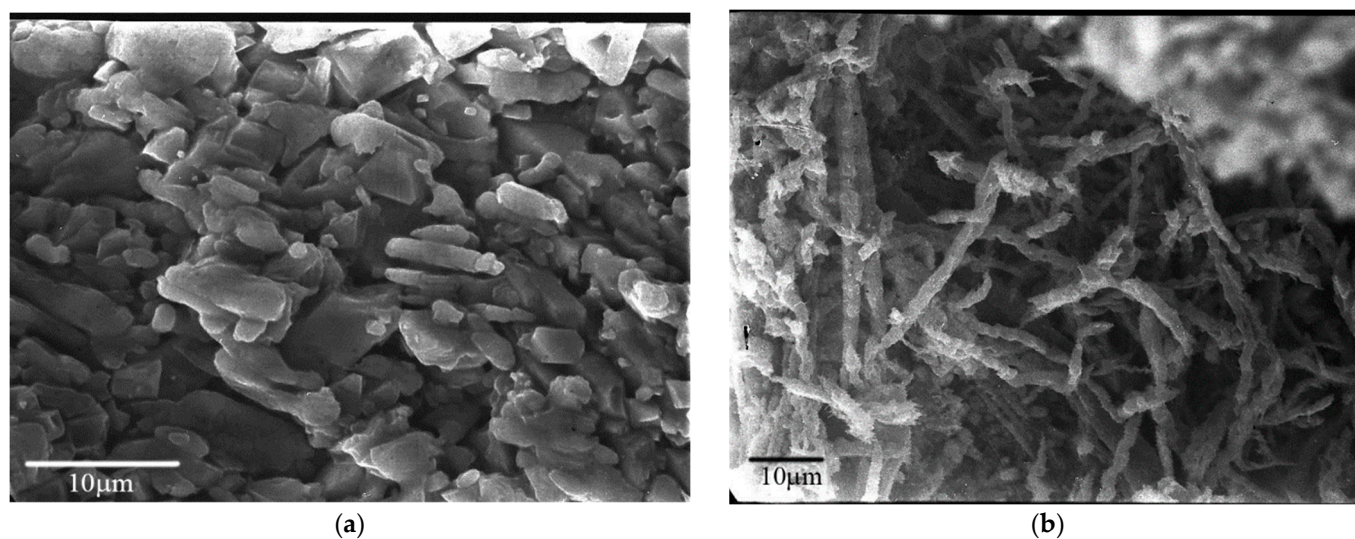


Figure 5. SEM images of polycarbazole prepared in the absence (a) and presence of an electrical field (b).

In the next step, we repeated the best polymerization conditions in the presence of different electric fields. In all experiments, we focused on the obtained mass of the polymer, m , the electrical conductivity of the obtained polymer in the absence of an electric field, σ , the electrical conductivity of the obtained polymer in the presence of an electric field, σ_E , and their ratio $r = \sigma_E / \sigma$.

To identify the best molar ratio of FeCl_3 (dry) initiator to monomer, the values 1/1, 1/3, 1/5 and 1/7 were examined using the PTSA solution as the dopant, and the best electrical conduction was $\sigma = 6.9 \times 10^{-6} \text{ S/cm}$ for the 1/5 ratio (Table 2). In other experiments, FeCl_3 (dry) was used as the initiator and different solvents with a molar initiator to monomer ratio of 1/5 were used at a fixed time of 3 h, the results of which are reported in Table 3.

To obtain the best reaction time, the polymerization was performed at a molar ratio of 1/5 at times ranging between 1 and 5 h. The best result was obtained at 3 h with $\sigma = 6.9 \times 10^{-6} \text{ S/cm}$ of electrical conductivity. In this condition, the highest obtained product mass was $m = 0.036 \text{ g}$. According to the results in Table 4, increasing the reaction time beyond 3 h had no effect on the mass of the product, but reduced the electrical conductivity. Here, the optimized conditions were employed under a uniform electric field with different intensities, including 5, 10, 11, 12, 13, 14, 15, 20, 25 and 30 KV/m. According to Table 5, the most effective electric field intensity was 12 KV/m, producing a mass of 0.045 g and an electrical conductivity of $\sigma_E = 8.9 \times 10^{-4} \text{ S/cm}$.

According to the results shown in the above tables and the comparison of the presence and absence of an electric field, it was clear that the presence of an electric field had a great effect on reducing the polymerization time, increasing the electrical conductivity and naturally increasing the mass and molecular order of the resulting polymer. The effect of the presence of the field on the regular shape of the polymer could also be clearly seen in the SEM images.

4. Conclusions

The preparation of polycarbazole through conventional methods usually gives polymers with a low molecular weight and low spatial array. Therefore, it minimizes the practical aspects of this type of polymer, compared to other conductive polymers of the same family. By using this method of producing polycarbazole with a regular spatial arrangement, good mechanical properties and higher electrical conductivity, the efficiency of such a polymer can be increased. In this work, the electroactive monomers were arranged by using the electric field, in which the head to tail connections were increased. Therefore, the obtained polymer had the longest chain length, the highest electrical conductivity, the

best space order and more appropriate physical resistance in comparison with the polymer obtained in the absence of an electric field. Other results also showed the electric field effect of an enhanced thermal resistance of up to 600 °C. The results of the experiments showed that the electrical conductivity of the polycarbazole produced in different electric fields and the most constant time (3 h) increasing with the increase in the electric field intensity. However, after approximately 12 KV/m, the electrical conductivity decreased steadily.

Additionally, the results of the experiments showed that the electrical conductivity of the polycarbazole produced in a constant electric field intensity of approximately 12 KV/m increased with the increase in time. However, after approximately 3 h, the electrical conductivity also decreased steadily.

On the other hand, considering the results of applying the various solvents and initiators, the best initiator was FeCl₃ (dry), the best solvent was acetone and the best dopant was p-toluenesulfonic acid (PTSA). It seemed that by increasing the time and intensity of the electric field after passing a specific amount, the destructive processes in the polymer chain started as a result of increasing the length of the polymer chain and dipolar moment forces. Additionally, due to polymeric confusion in the reaction solution, the polymers involved defective reactions, such as producing secondary branches on the polymer chain. Finally, the conductivity measurements of the polymers showed that the electric conductivity of the polymers increased under the electric field approximately 120 times, respectively. Among the limitations of this method was the size of the carbazole monomer and the processability of the resulting polymer. Although the latter was present in all conducting polymers, the processability of polycarbazole could be increased through functionalizing the monomer. We hope that its applications also increase in the future.

Author Contributions: S.H.H.: Corresponding author. A.A.K.: Cooperation in the electric field devices, co-author, Programming, software development; designing computer programs; implementation of the computer code and supporting algorithms; testing of existing code components. S.A.H.: Conducting a research and investigation process, specifically performing the experiments, or data/evidence collection. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

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