

# **Maximizing Poly(3-butylthiophene-2,5-diyl) Electrical Conductivity by Maximizing Transcrystal Length**

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Bachelor of Science

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# Abstract

Polymers are generally considered electrical insulators. Despite this, research in the mid 1970's found that polymers consisting of a conjugated backbone structure could become electrically conductive upon doping.<sup>1</sup> The conjugated polymer analyzed for this project was poly(3-butylthiophene-2,5-diyl) (P3BT). Transcrystals have been found as a way to promote electrical conductivity through mechanisms including  $\pi$  bond atomic orbital overlap and electron mobility.<sup>2</sup> In theory, maximizing transcrystal length would also maximize P3BT electrical conductivity, increasing its applicable use in electronic devices. The goal of this project was to determine a methodological way to maximize P3BT electrical conductivity by producing the longest transcrystal length possible. This was attempted through two objectives. The first objective was to determine what solvent combination and solvent evaporation temperature (24°C and 26°C) would yield in the longest transcrystal length possible. The solvents investigated included carbon disulfide (CS<sub>2</sub>), 1,2-dichlorobenzene (DCB), liquid 1,2,4-trichlorobenzene (L-TCB), and solid 1,3,5-trichlorobenzene (S-TCB). The second objective was to determine if increasing transcrystal length would increase electrical conductivity. Three solutions of 2 wt.% P3BT were produced with the following solvent combinations: solution #1 (7wt.% L-TCB, 91wt.% CS<sub>2</sub>), solution #2 (7wt.% L-TCB, 91wt.% DCB), and solution #3 (7wt.% S-TCB, 91wt.% DCB). Transcrystals were nucleated from a carbon nanotube (CNT) aerogel fiber via a 24-hour controlled CS<sub>2</sub> solvent evaporation. Polarized optical microscopy (POM) and scanning electron microscopy (SEM) were used to capture the transcrystal lengths and morphology of each condition analyzed. It became evident through the POM images that solution used had the largest impact on transcrystal length, while temperature had a smaller yet significant impact. Solution #1 produced the longest average transcrystal length of 44.8 $\mu$ m. This was followed by solution #2 and #3, with respective transcrystal lengths of 24.1 $\mu$ m and 1.6 $\mu$ m. A solvent evaporation temperature at 24°C yielded slightly longer transcrystals than a solvent evaporation temperature at 26°C, with respective lengths of 26.2 $\mu$ m and 20.8 $\mu$ m. A correlation between transcrystal length and electrical conductivity remain inconclusive due to a limitation with the four-point probe testing apparatus and its ability to analyze transcrystals in a micrometer scale. Transcrystal formation proved unpredictable, demonstrated through several attempts to nucleate a transcrystal under the same conditions. Overall, a methodological way to produce the longest transcrystal length possible has been established.

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# 1. Introduction

## 1.1 Background

Polymers are commonly known for being electrical insulators; however, this notion changed in 1977 when three scientists- Shirakawa, MacDiarmid, and Heeger- found that doping polyacetylene with halogenated solvents yielded a ten-million-fold increase in electrical conductivity.<sup>3</sup> This led to the option of producing polymeric based electronic devices that are cheap, lightweight, and flexible compared to metallic based devices. Applications benefiting from such properties include antistatic packaging, organic light-emitting diodes (O-LEDs), and organic solar cells. A limitation is that polymers require doping and are only semi-conductive at best, limiting its usage to low-voltage technology. Increasing a polymers electrical conductivity could increase its applicable use.

## 1.2 Problem Statement

The purpose of the following project was to establish a methodological way to increase polymer electrical conductivity, achieved through two distinct objectives. The first objective was to determine what combination of solvents (as listed below) in a 2 wt.% poly(3-butylthiophene-2,5-diyl) (P3BT) solution and solvent evaporation temperature (24°C or 26°C) yielded the longest transcrystal length possible. The second objective was to determine if producing longer transcrystals correlated with an increase in electrical conductivity. If so, then the conditions that yielded the longest transcrystals could be implemented as a known way to maximize electrical conductivity of P3BT.

### Solvents Investigated

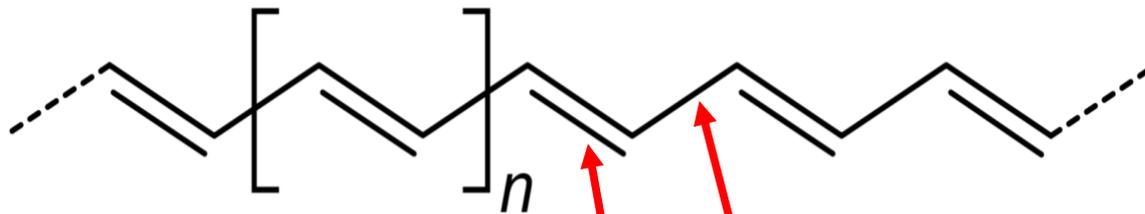
1. 91 wt.% Carbon Disulfide with 7 wt.% liquid 1,2,4-Trichlorobenzene
2. 91 wt.% 1,2-Dichlorobenzene with 7 wt. % liquid 1,2,4-Trichlorobenzene
3. 91 wt.% 1,2-Dichlorobenzene with 7 wt.% solid 1,3,5-Trichlorobenzene

## 2. Literature Review

### 2.1 Conductive Polymer Mechanism

The mechanism making a polymer electrically conductive is its conjugated backbone structure, consisting of  $\sigma$  and  $\pi$  bonds arranged in an alternating single and double bond pattern.<sup>2</sup> This is demonstrated with polyacetylene, which is the polymer that Shirakawa, MacDiarmid, and Heeger found to be electrically conductive (Figure 1a). Both  $\sigma$  and  $\pi$  bonds are covalent; however,  $\sigma$  bonds are significantly stronger than  $\pi$  bonds due to the structural arrangement each bond type is in.  $\sigma$  bonds are arranged laterally along the conjugated backbone plane. This results in an atomic orbital overlap, giving it its strength.  $\pi$  bonds are arranged perpendicular to the backbone plane with one of its atomic orbital lobes sharing an orbital lobe with adjacent backbone chains.<sup>3</sup> This results in a comparatively weaker bond type that can become delocalized upon doping (Figure 1b).

a)



b)

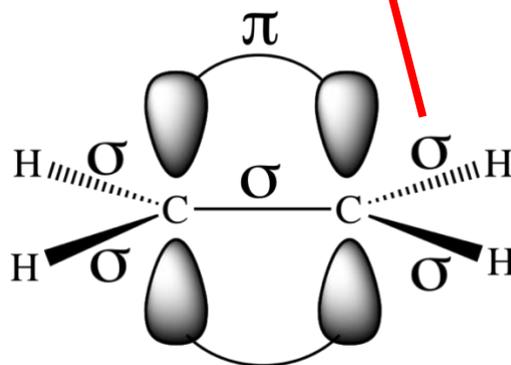


Figure 1: (a) Molecular Structure diagram of polyacetylene, displaying its conjugated backbone structure.<sup>4</sup> (b) Molecular structure diagram displaying how  $\sigma$ -bonds and  $\pi$ -bond are configured in a conjugated polymer.<sup>5</sup>

When  $\pi$  bonds are delocalized, a cloud of electrons known as electron deformation is formed. This can be compared to the electrically conductive “sea of electrons” mechanism found in

metals. Electron deformation is in a lower state of entropy due to a high electron concentration relative to the rest of the backbone chain. Following the second law of Thermodynamics, the electrons are shifted along the conjugated backbone chain to a higher state of entropy in a process known as electron mobility.<sup>6</sup> Increasing electron mobility has a direct correlation on electrical conductivity as shown with Equation 1. In this equation,  $\sigma$  is electrical conductivity (S/cm),  $\eta$  is electron density ( $\text{m}^3$ ),  $e$  is charge (C), and  $\mu_e$  is electron mobility ( $\text{m}^2/\text{Vs}$ ).

$$\sigma = \eta e \mu_e \quad (\text{Equation 1})$$

## 2.2 Transcrystals

While electron mobility could be increased through the amount of dopant applied, a crystalline arrangement could be used to promote electron mobility as well.<sup>7</sup> Directed alignment of the conjugated backbone causes the  $\pi$  bonds to overlap with adjacent backbone chains (Figure 2). This  $\pi$ - $\pi$  bond overlap produces a pathway for the electrons to flow through the polymer. Maximizing this pathway could increase electron mobility, and thus electrical conductivity.

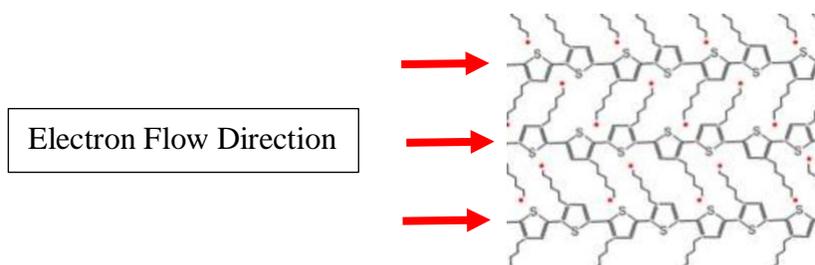
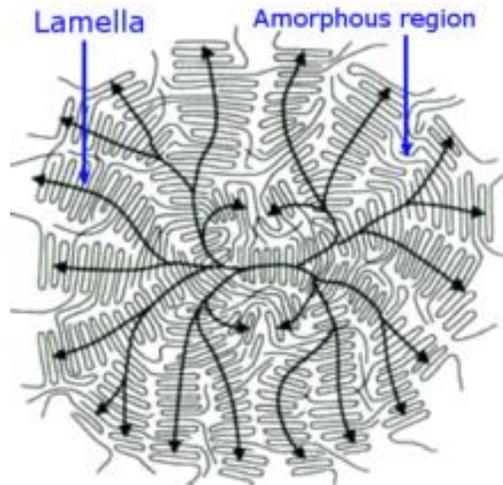


Figure 2: Aligned P3BT chains with arrows pointing at the direction of electron mobility.<sup>8</sup>

Unfortunately, polymers typically nucleate radially into a primarily amorphous spherulite (Figure 3a).<sup>9</sup> A demonstrated way to yield a polymer with a higher degree of crystallinity is through transcrystal growth, which indicates that the polymer has nucleated from the surface of a carbon nanotube (CNT) fiber (Figure 3b).<sup>10</sup> Increasing the transcrystal region would increase the pathway for electrons to flow through. Overall, maximizing transcrystal length should maximize P3BT electrical conductivity.

a)



b)

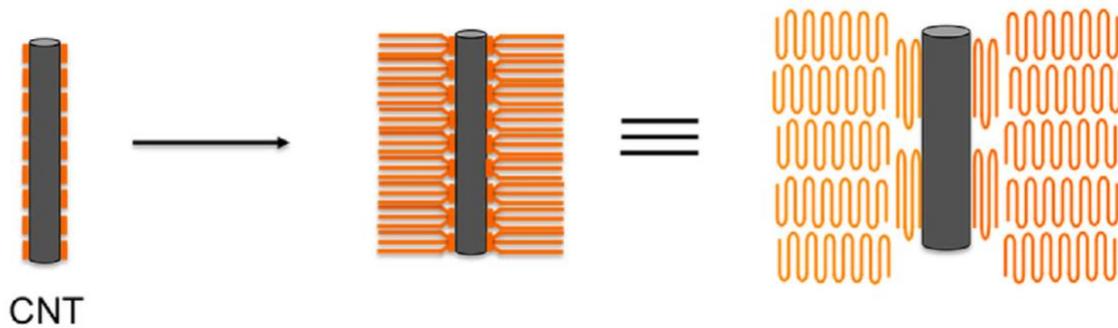


Figure 3: (a) Highly amorphous spherulite formation displaying minimal degrees of alignment.<sup>11</sup> (b) Conjugated polymer nucleating perpendicular to the CNT fiber, resulting in an aligned conjugated backbone structure.<sup>12</sup>

### 2.3 Poly(3-butylthiophene-2,5-diyl)

Poly(3-butylthiophene-2,5-diyl) (P3BT) is a polythiophene conjugated polymer that can become electrically conductive upon oxidative p-type doping.<sup>13</sup> In other words, electrons are removed from the atomic orbitals, increasing empty space for electron mobility to occur. A polythiophene is composed of a halogen and a butyl side chain, as shown with P3BT (Figure 4). The halogen contains the conjugated backbone structure with the mechanism that promotes electrical conductivity while the butyl side chain hinders electrical conductivity. Despite this, the butyl side chain length is short relative to other polythiophenes and electrical conductivity is still observed. Polythiophenes in general are stiff relative to other polymers as a result of its halogenated backbone structure, increasing the difficulty to solubilize on a larger scale. Regardless, a polythiophene such as P3BT can be used to produce organic light emitting diodes (O-LEDs) and

other light emitting applications. This is possible as a result of its halogenated structure promoting electroluminescence upon doping.<sup>11</sup>

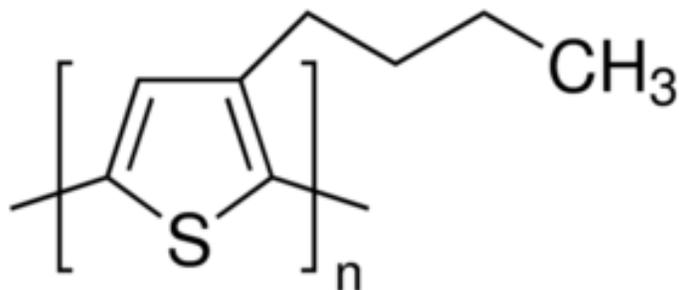


Figure 4: Poly(3-butylthiophene-2,5-diyl) molecular structure displaying a conjugated halogen and butyl side chain.<sup>13</sup>

## 2.4 Polymers vs. Metals

While benefits have been noted for electrically conductive polymers, usage has been limited to low-voltage technology. This stems from a limitation in electrical conductivity due the short-range order at which transcrystals can form, typically micrometers in length.<sup>7</sup> Metals are electrically conductive from the “sea of electrons” mechanism it has, resulting in electrical conductivity values that are magnitudes higher than polymers. For instance, polyacetylene has been demonstrated to produce the highest electrical conductivity of any conductive polymer produced so far, with a value of  $10^6$  S/cm. This is magnitudes smaller than copper, a common metallic electrical conductor, which has displayed electrical conductivity values near  $10^9$  S/cm.<sup>14</sup> Increasing a polymer’s electrical conductivity to that of an electrically conductive metal could increase its applicable use. Research is ongoing to achieve this goal.

### 3. Experimental Procedures

#### 3.1 Solution Preparation

To determine what combination of solvents yielded the longest transcystals possible, three P3BT solutions were prepared (Figure 5). Each solution produced contained 2 wt.% P3BT (0.04g), 91 wt.% (0.14) CS<sub>2</sub> or DCB, and 7 wt.% L-TCB or S-TCB.

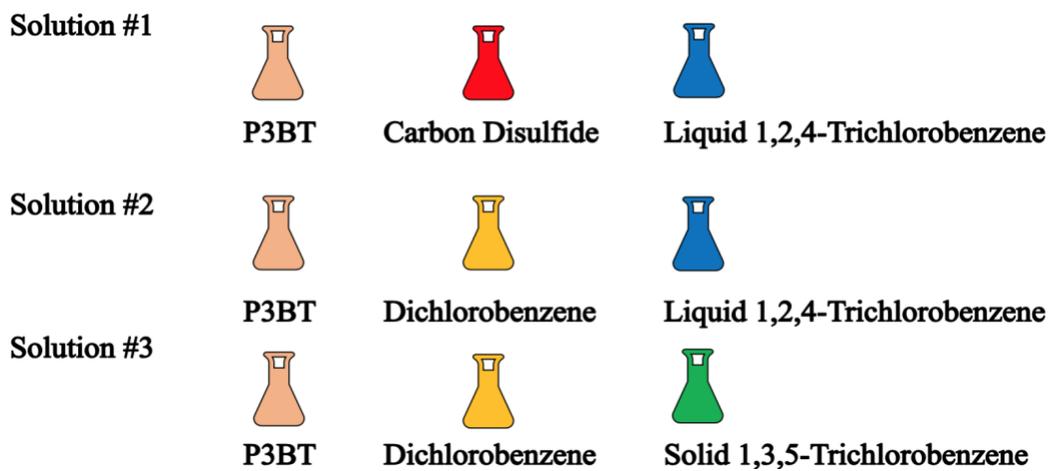


Figure 5: A diagram of the three solutions produced to test the solvent combination effects on transcystal growth.

P3BT and the respective solvents for each solution were combined in a glass beaker. The beaker was placed on a scale which was used to ensure the appropriate amount of P3BT and solvents were added. P3BT and S-TCB were administered into the beaker with tweezers while CS<sub>2</sub>, DCB, and L-TCB were added with a glass pipet (Figure 6).

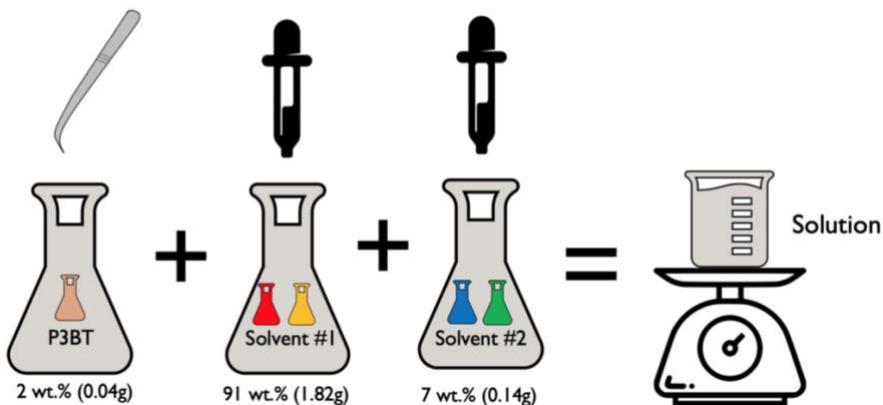


Figure 6: Diagram demonstrating layout required to prepare each solution.

The combined weight of P3BT and solvents for each solution was 2 grams. Once P3BT and the solvents were in place, Teflon tape was wrapped around the outer rim of each beaker and capped. The Teflon tape sealed any potential leaks, preventing the volatile solvents from evaporating. To aid in the solution ingredients diffusing into a homogenous mixture, the beakers containing solution were placed in an oven at 50°C for 10 minutes. Subsequently, each solution was refrigerated to minimize the rate of crystallization until a transcystal template was prepared.

### 3.2 Transcrystal Template

A transcystal template was prepared by first scoring a glass substrate so that the approximate dimensions were 2.5cm x 2.5cm. This provided a sufficient area to nucleate transcystals and still fit in the solvent evaporation jar. Impurities that could impede transcystal growth were removed by soaking the glass substrates in acetone and water. A CNT aerogel bundle was obtained, and tweezers were used to pluck a single fiber, subsequently densifying it in methanol to provide a smooth surface area to promote transcystal nucleation. Methanol was then used as an adhesive between the glass substrate and fiber interface. This was achieved by pipetting 2-3 drops of methanol onto the substrate prior to adding the fiber. The final step was pipetting 2-3 drops of the solution being investigated (Figure 7). Overall, six transcystal templates were prepared with each solution being applied to two templates. This was done so that each solution could be tested at a solvent evaporation temperature of 24°C and 26°C.



Figure 7: Diagram showing the layout for producing each transcystal template.

### 3.3 Solvent Evaporation Jar

A solvent evaporation jar apparatus was setup in a fume hood. The setup began by first placing a jar into a silica oil bath on a hot plate. The silica oil bath was used to ensure a uniform heat distribution in the jar, and the hot plate was used to achieve the solvent evaporation temperature. A smaller jar was placed in the larger jar, to be used as a platform for the transcrystal template. A glass pipet was used to administer 10 mL of the evaporating solvent, CS<sub>2</sub>, into the base of the solvent evaporation jar (Figure 8). The jar was loosely capped with a 180° turn and the hot plate was turned to the solvent evaporation temperature being investigated (24°C or 26°C). A thermometer was placed in the silica oil bath to gauge the hot plate temperature, and the reaction was left to run over a 24-hour period. It was after this time that all of the CS<sub>2</sub> would evaporate, leaving transcrystal formation.

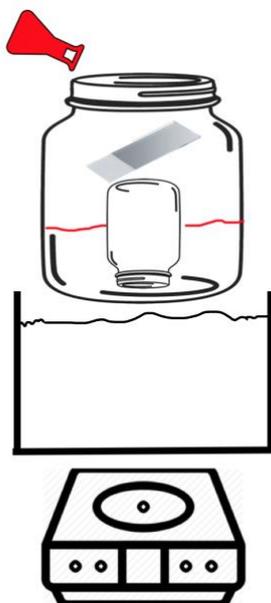


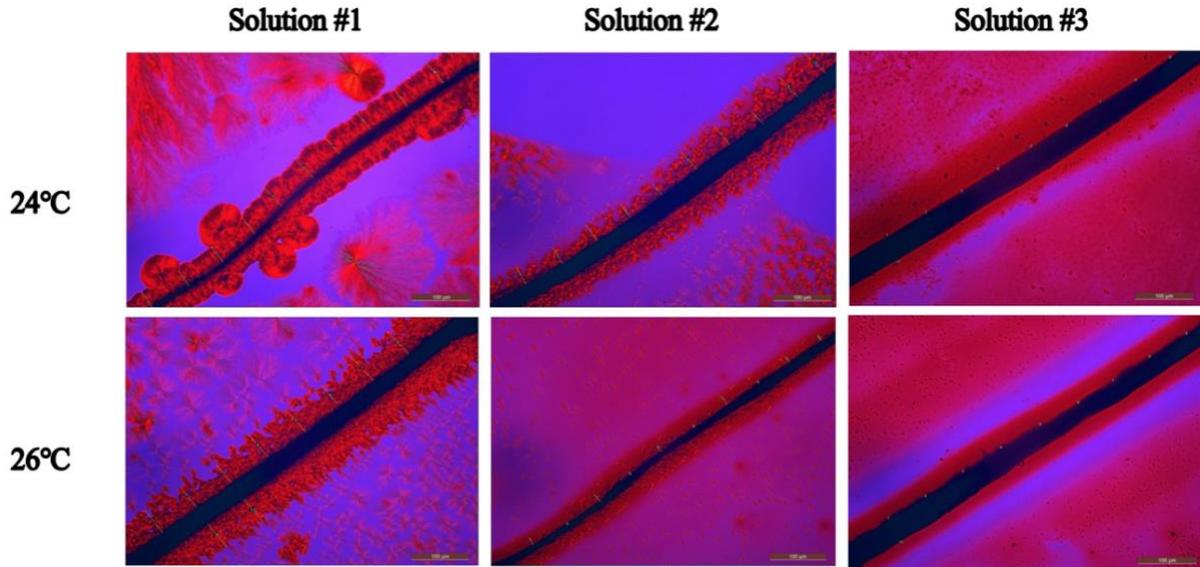
Figure 8: Extruded diagram of solvent evaporation jar apparatus displaying each individual part.

### 3.4 Microscopy Characterization

Following transcrystal formation, polarized optical microscopy (POM) images were obtained for each transcrystal sample produced (Figure 9a). Using these images, ImageJ was used to place ten measuring bars in 120µm increments along the CNT aerogel fiber, measuring the adjacent transcrystal length. Each of the transcrystal samples analyzed under the SEM were sputter coated

with a gold coating to be analyzed by the scanning electron microscope (SEM). The SEM was used in low-vacuum mode as a secondary measure to reveal the adjacent spherulite formation in solution #1 and lack thereof in solution #6 (Figure 9b).

a)



b)

**Solution #1 at 24°C      Solution #3 at 26 °C**

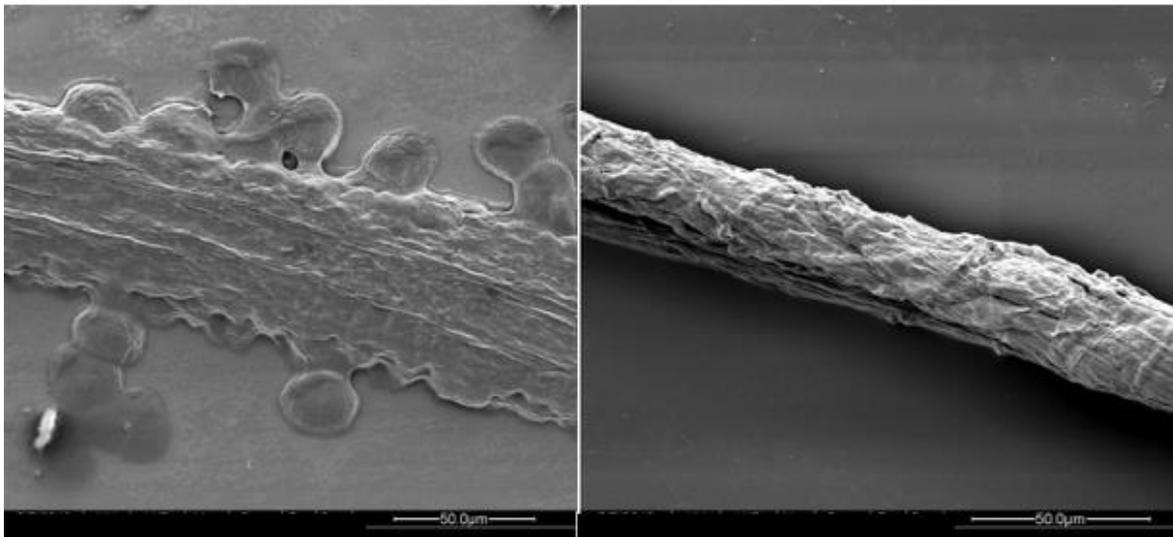


Figure 9:(a) Polarized Optical Microscopy (POM) images used to measure transcrystal length of each sample. (b) Scanning electron images (SEM) obtained to demonstrate solution #1 producing adjacent spherulites that could impinge transcrystal growth while solution #6 contained no spherulites that could cause impingement.

### 3.5 Electrical Conductivity Test

In an attempt to establish a correlation between electrical conductivity and transcystal length, a four-point probe electrical conductivity test was administered on each sample. In this test, four probes were lowered onto the surface of each transcystal sample and varying voltages ranging from 1 to 10 mV were applied (Figure 10). The output for this testing apparatus was current. To convert this value into electrical conductivity, the current output was divided by the corresponding voltage to obtain a value in Siemens. This value in Siemens was divided by the length between each probe, 0.1588cm, outputting a conductivity value in S/cm.

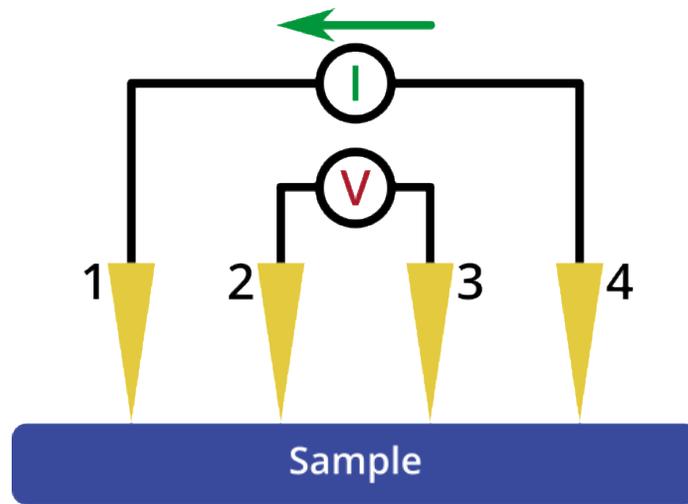


Figure 10: Diagram displaying how the four-point probe testing apparatus interacted with the sample being tested.<sup>14</sup>

## 4. Results

### 4.1 Conditions for Nucleating Longest Transcrystals Possible

The average transcrystal lengths obtained from the POM images indicated that varying the combination of solvents and solvent evaporation temperature impacted transcrystal length, although to varying degrees (Table I). A main effects plot generated from Minitab indicated that the effect of solvent evaporation temperature on transcrystal length was minimal compared to the combination of solvents used (Figure 11). Solution #1 yielded the longest transcrystals, followed by solution #2 and #3. An increase in solvent evaporation temperature from 24°C to 26°C produced transcrystals that were slightly shorter in length.

Table I: Mean Average Transcrystal Lengths for each Sample Produced

Temp (°C)	Fitted Mean (μm)	Temp (°C)*Solution	Fitted Mean (μm)
24	26.2	24*1	38.8
26	20.8	24*2	37.6
Solution	Fitted Mean (μm)	24*3	2.1
1	44.8	26*1	50.7
2	24.1	26*2	10.7
3	1.6	26*3	1.1

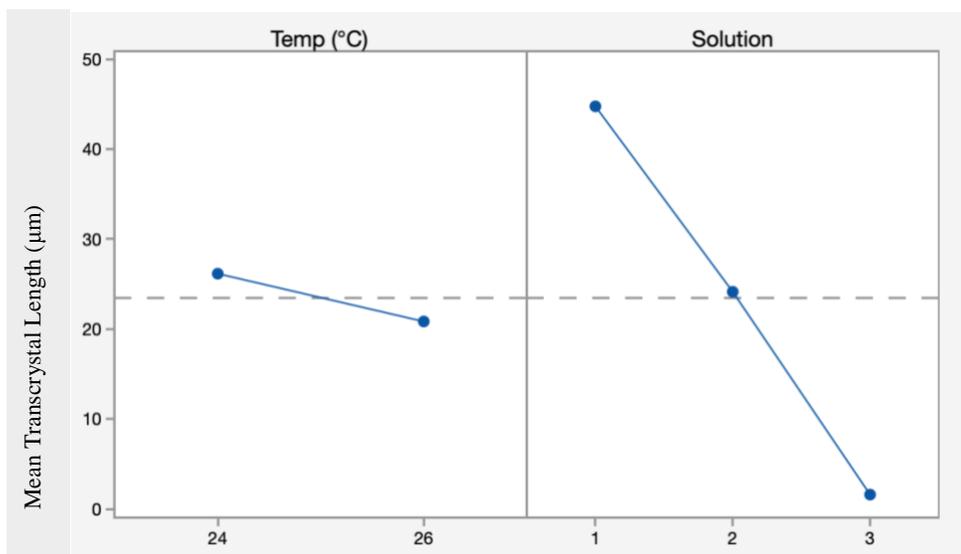


Figure 11: Main Effects plot generated from Minitab displaying how temperature and solution affect transcrystal length.

An interaction plot was generated to understand how each solution interacted with varying solvent evaporation temperatures, and the trend was inconsistent (Figure 12). While the main effects plot displayed a decrease in transcrystal length with increasing temperature, solution #1 had the opposite effect. Temperature also affected each solution to varying degrees with solution #1 being moderately affected, solution #2 being greatly affected, and solution #3 being minimally affected. It seems the main effects plot on solvent evaporation temperature was influenced by the effects of temperature on solution #2.

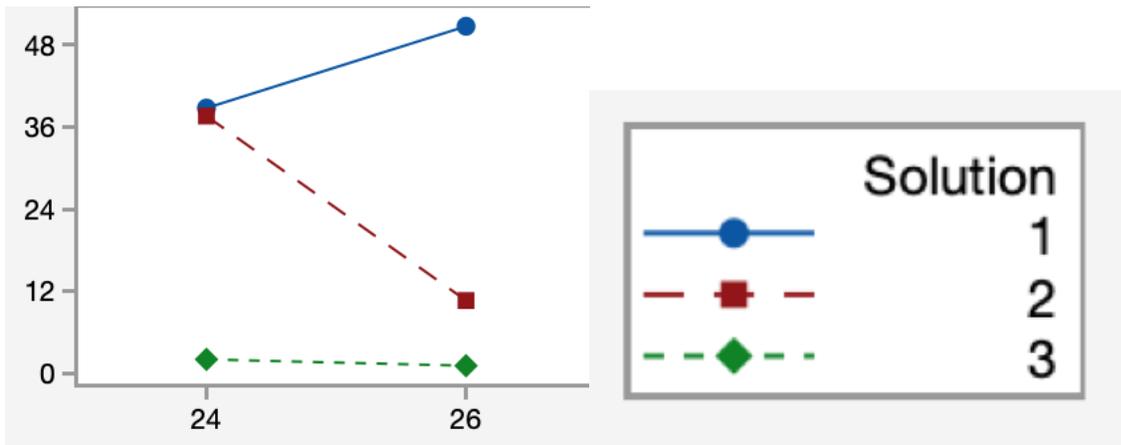


Figure 12: Interaction plot generated from Minitab displaying how each solution was affected to different degrees by solvent evaporation temperature.

A two-way analysis of variance (ANOVA) test indicated that the effects of solution and solvent evaporation temperature on transcrystal length were statistically significant with 95% confidence. This was apparent by the near zero p-values obtained for each parameter investigated (Table II).

Table II. Two-way Analysis of Variance for Solvent Evaporation Temperature and Solution on Transcrystal Length

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Temp	1	423.7	423.66	14.79	0.0003
Solution	2	18646.4	9323.22	325.37	<0.0001
Temp*Solution	2	3930.5	1965.24	68.59	<0.0001
Error	54	1547.3	28.65		
Total	59	24547.9			

## 4.2 Transcrystal Length on Electrical Conductivity

The four-point probe conductivity test data was used to generate an interval plot (Figure 13). The conductivity values were almost nonexistent and varying transcrystal lengths did not yield a noticeable trend on electrical conductivity. A one-way ANOVA analysis indicated that the following data was random and statistically insignificant. This was demonstrated with a high p-value of 0.9119 (Table III).

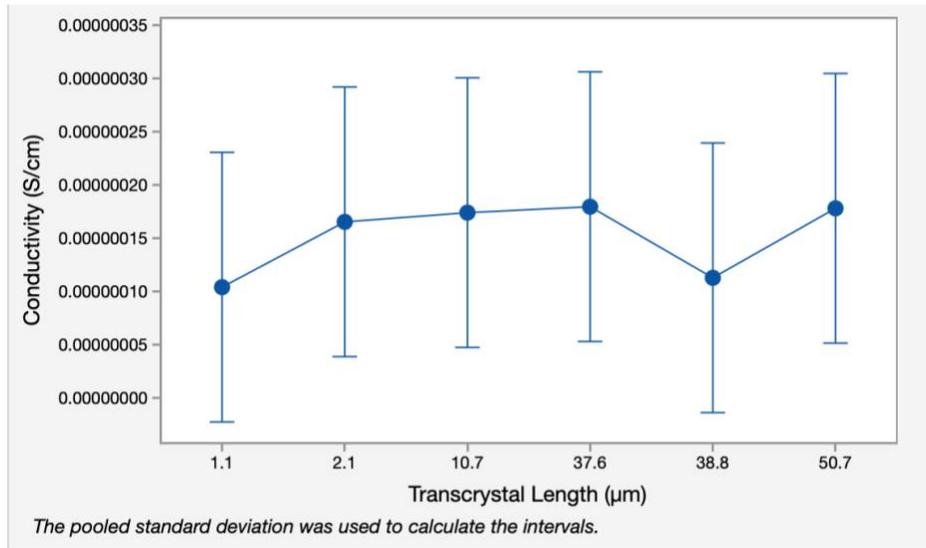


Figure 13: Interval plot generated from Minitab displaying insignificant data on the effects transcrystal length on electrical conductivity.

Table III: One-way Analysis of Variance for Transcrystal Length Effects on Electrical Conductivity

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Transcrystal Length	5	0.00000000	0.00000000	0.30	0.9119
Error	54	0.00000000	0.00000000		
Total	59	0.00000000			

## 5. Discussion

### 5.1 Solvent Combination Effects on Transcrystal Growth

The results indicated that solution #1 yielded the longest transcrystals possible, followed by solution #2 and #3. A variation of 96.4% in transcrystal length between solutions indicated that the combination of solvents used had the largest impact on transcrystal length. In the transcrystal solutions produced, the solvents acted as orientation templates, aligning the P3BT polymer chains into a crystalline formation prior to evaporation in a process called epitaxial growth.<sup>15</sup> In addition, each solvent contributed a different vapor pressure to the overall solution as a way to control the solvent evaporation rate. CS<sub>2</sub> and DCB increased the solutions overall solvent evaporation rate with higher vapor pressures of 48.1 kPa and 48.0 kPa, respectively. L-TCB and S-TCB decreased the evaporation rate with lower vapor pressures of 0.1 kPa and 0.03 kPa, respectively.<sup>16</sup> It seems that vapor pressure had a direct influence on transcrystal length since the average vapor pressures from solvents in solution #1 was the highest, followed by solution #2 and #3. Increasing vapor pressure was effectively increasing P3BT molecule mobility, allowing for the transcrystals to nucleate a further distance. Perhaps maximizing the combined solvent vapor pressure while maintaining control over the evaporation rate would maximize P3BT transcrystal growth. While vapor pressure demonstrated the largest influence on transcrystal length, solvent evaporation temperature had a smaller, yet statistically significant impact.

### 5.2 Solvent Evaporation Temperature Effects on Transcrystal Growth

Theoretically, increasing solvent evaporation temperature from 24°C to 26°C would increase the kinetic rate at which transcrystals could nucleate a further distance. This is shown following the Arrhenius equation, where  $k$  = kinetic rate (1/s),  $A$  = constant for each chemical reaction,  $E_a$  = activation energy (J/mol),  $R$  = 8.314 (J/(°C\*mol)), and  $T$  = temperature (°C).

$$k = Ae^{-\frac{E_a}{RT}} \quad (\text{Equation 2})$$

While solution #1 followed this theory, transcrystal lengths for solution #2 and solution #3 decreased with increasing temperature. A reason for this could be that transcrystal density had a larger influence over transcrystal growth. At a slower solvent evaporation rate, less transcrystal nuclei would have been generated, resulting in increased room for the nuclei that were generated to form longer transcrystals. In contrast, a faster solvent evaporation rate would have generated more nuclei, causing an impedance that would have caused limited transcrystal growth.<sup>17</sup> Since the effects of solvent evaporation temperature on transcrystal length were minimal, it should be considered that transcrystals nucleated were micrometers in length. The nature of this size means that external factors, such as uneven CNT fiber surface area, could have impacted the overall trend. It also did take several attempts to produce transcrystals following the same standard operating procedure under the same conditions, indicating that transcrystal growth was unpredictable. Despite obtaining a weak trend, it was statistically confirmed that a solvent evaporation temperature at 24°C yielded transcrystals 20.6% longer than transcrystals nucleated with a solvent evaporation temperature at 26°C.

### 5.3 Microscopy Analysis

POM images were captured for each sample to obtain the respective average transcrystal lengths with measuring bars using ImageJ (Figure 9a). SEM images were captured to reveal that solution #1 at 24°C produced neighboring spherulites in addition to the longer transcrystals nucleated. This is in contrast to solution #6 at 26°C, which nucleated the shortest transcrystals from the samples produced and lacked spherulites (Figure 9b). The generation of spherulites could have impeded and decreased transcrystal growth. Regardless, any impedance from spherulites were minimal since the average transcrystal length produced from solution #1 at 24°C was still 97% longer than the average transcrystal length produced from solution #3 at 26°C.

### 5.4 Electrical Conductivity Analysis

It became apparent from the one-way ANOVA test that the data gathered for the effects of transcrystal length on electrical conductivity was insignificant. It seems the electrical conductivity values obtained for P3BT yielded an electrical conductivity value in the range of  $10^{-7}$  S/cm, which is magnitudes smaller than previously observed electrical conductivity values

of  $10^3 \text{ S/cm}$ .<sup>13</sup> The difficulty in obtaining measurements could have stemmed from the transcrystals being too small for the four-point probe testing apparatus to register. Since the data gathered was statistically insignificant, a correlation between transcrystal length and electrical conductivity was not established. Perhaps a four-point probe test designed for measuring microelectronic devices could be used in future research to determine if any potential correlation exists.

## 6. Conclusions

Determining what combination of solvents and variation in solvent evaporation temperature proved successful in producing various transcrystal lengths. Determining a correlation between transcrystal length and electrical conductivity was limited with the accuracy of the four-point probe conductivity test on a micrometer scale. Despite this, data and observations (listed below) could aid future researchers that hope to maximize P3BT electrical conductivity through transcrystal growth.

1. The combination of solvents investigated had the largest influence on transcrystal length with a variation of 96.4% between solutions. Solution #1 produced the longest transcrystals, followed by solution #2 and #3, respectively.
2. The solvent evaporation temperature conditions analyzed (24°C and 26°C) had a minimal yet statistically significant effect on transcrystal growth. Transcrystals nucleated at 24°C were 20.6% longer than transcrystals nucleated at 26°C.
3. A correlation between transcrystal length and electrical conductivity remain inconclusive as a result of the four-point probe conductivity test being incompatible with the transcrystal micrometer scale.
4. Transcrystal growth is unpredictable, as demonstrated by several attempts of following the same standard operating procedure under the same conditions to produce one transcrystal for each respective sample.

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