



MARMARA UNIVERSITY  
FACULTY OF ENGINEERING



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**DESIGN AND SYNTHESIS OF FLAME-RETARDANT PCL  
ELECTROSPUN NANOFIBERS**

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**GRADUATION PROJECT REPORT**  
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# CONTENTS

ACKNOWLEDGEMENTS .....	2
CONTENTS.....	3
ABSTRACT.....	4
SYMBOLS.....	6
ABREVIATIONS .....	7
1) INTRODUCTION .....	8
1.1 Design and Synthesis of Flame-Retardant Pcl Electrospun Nanofibers .....	8
1.2 Electrospinning .....	9
1.3 Nanoparticles .....	10
1.4 Tensile Test.....	15
2) MATERIAL AND METHODS .....	18
3) CALCULATIONS AND RESULTS.....	28
4) CONCLUSION AND DISCUSSION.....	56
5) APPENDICES .....	57
REFERENCES .....	62

## ABSTRACT

The design and synthesis of flame-retardant polycaprolactone (PCL) electrospun nanofibers have gotten a lot of attention because of its potential applications in a variety of industries that require increased fire safety. This review paper examines the tactics used to increase the fire resistance capabilities of flame-retardant PCL nanofibers and offers an overview of the research development in this area.

In order to improve flame retardancy, flame retardant additives like as halogen-based or phosphorous-based chemicals are commonly incorporated into the PCL matrix. During combustion, these additives can encourage the production of a solid phase char, which acts as a physical barrier against heat and oxygen transport. Furthermore, they can interfere with combustion reactions and disturb the combustion process by lowering flammable gas evolution.

Electrospinning, a versatile nanofiber fabrication technology, provides advantages in the creation of flame-retardant PCL nanofibers. The electrospinning technique produces continuous nanofibers with high surface-to-volume ratios, which can improve flame retardant properties. To achieve the required fiber morphology and qualities, several parameters such as polymer concentration, applied voltage, and collector design can be optimized.

The flame retardant chemicals used, as well as their concentration and dispersion within the PCL matrix, are important elements impacting the flame retardant performance of the resultant nanofibers. PCL and flame retardant additive compatibility is critical for uniform dispersion and efficient flame retardant activity. To further improve the flame retardancy of PCL nanofibers, synergistic effects between different additives might be investigated.

Flame-resistant PCL electrospun nanofibers are used in a variety of industries, including automotive, aerospace, construction, textiles, and electronics. These materials have the potential to be utilized in the creation of fire-resistant components, insulating materials, protective apparel, and filtration systems. Understanding and controlling the design and synthesis of flame-retardant PCL nanofibers allows for the creation of new materials with improved fire safety qualities.

This project gives insights into current research efforts and future objectives in the field of flame-retardant PCL electrospun nanofibers, emphasizing the necessity of customizing the composition, shape, and processing parameters to obtain desired flame retardant qualities.

## **SYMBOLS**

<b>mm</b>	: Millimeter
<b>g</b>	: Gram
<b>kV</b>	: Kilovolts
<b>F<sub>t</sub></b>	: Tensile Force
<b>t</b>	: Thickness
<b>A</b>	: Cross-sectional Area
<b>σ</b>	: Stress
<b>ε</b>	: Strain
<b>E</b>	: Elastic Modulus
<b>L</b>	: Final Length
<b>L<sub>0</sub></b>	: Initial Length
<b>MPa</b>	: Megapascal
<b>N</b>	: Newton

## **ABREVIATIONS**

**PCL** : Polycaprolactone

**TiO<sub>2</sub>** : Titanium Dioxide

**C<sub>2</sub>H<sub>3</sub>N** : Acetonitrile

**CHCl<sub>3</sub>** : Chloroform

# **1) INTRODUCTION**

## **1.1 Design and Synthesis of Flame-Retardant PCL Electrospun Nanofibers**

Flame-retardant materials play a crucial role in various industries, including textiles, construction, transportation, and electronics, where fire safety is of paramount importance. Among the diverse range of flame-retardant materials, polymeric nanofibers have gained significant attention due to their unique properties and wide-ranging applications. Electrospinning, a versatile and scalable technique, has emerged as a prominent method for fabricating nanofibers with precise control over morphology, diameter, and composition. In this context, the design and synthesis of flame-retardant poly( $\epsilon$ -caprolactone) (PCL) electrospun nanofibers have garnered considerable interest as an effective approach to enhance the flame retardancy of polymeric materials.

Poly( $\epsilon$ -caprolactone) (PCL), a biodegradable and biocompatible polyester, possesses favorable characteristics such as good processability, mechanical strength, and flexibility. These attributes make PCL an ideal candidate for electrospinning, enabling the fabrication of nanofibers with high aspect ratios and large surface areas. However, the intrinsic flammability of PCL limits its applicability in fire-prone environments. To address this limitation, incorporating flame-retardant additives into PCL nanofibers has emerged as a promising strategy to enhance their flame-retardant properties.

Flame-retardant additives can be broadly categorized into three classes: halogen-containing compounds (e.g., bromine, chlorine), phosphorus-based compounds, and nitrogen-based compounds. These additives function through various mechanisms, including the release of flame-inhibiting gases, formation of a protective char layer, and interruption of the combustion process. By incorporating these additives into PCL electrospun nanofibers, it is possible to impart flame-retardant properties to the resulting materials, thereby significantly improving their fire safety performance.

The design and synthesis of flame-retardant PCL electrospun nanofibers require careful consideration of several factors, including the selection of suitable flame-retardant additives, incorporation methods, and optimization of electrospinning parameters. Additionally, thorough characterization techniques must be employed to assess themorphology, thermal stability, and flame-retardant effectiveness of the resulting nanofibers.

This research aims to explore the design and synthesis of flame-retardant PCL electrospun nanofibers as a means to enhance fire safety in various applications. By

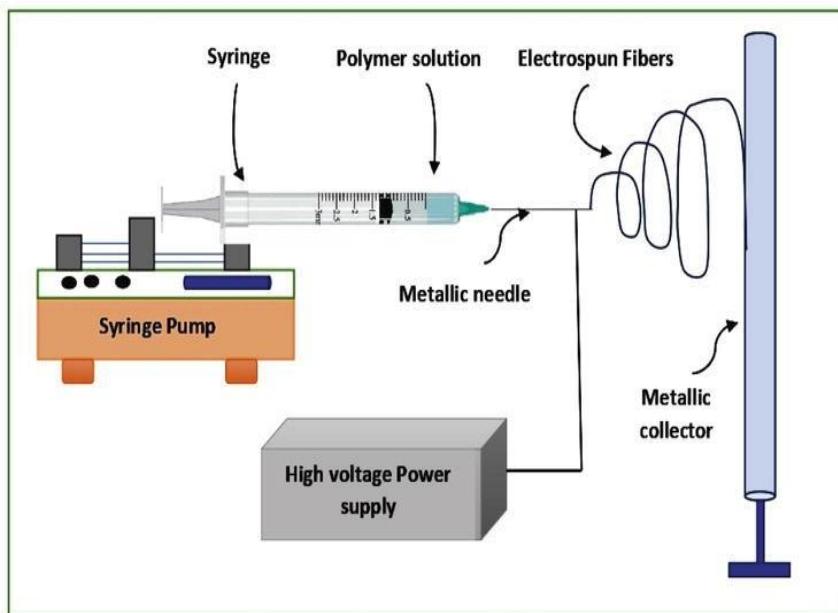
investigating different flame-retardant additives, incorporating them into PCL nanofibers, and optimizing the electrospinning process, it is anticipated that this work will contribute to the development of advanced flame-retardant materials with improved performance and functionality.

In summary, the design and synthesis of flame-retardant PCL electrospun nanofibers hold great promise for enhancing fire safety in diverse industries. This research endeavors to explore the incorporation of flame-retardant additives into PCL nanofibers through electrospinning, aiming to develop materials with superior flame-retardant properties and expand their applications in fire-prone environments.

## **1.2 Electrospinning**

Electrospinning is the most widely used method for nanofiber production [1]. Electrospinning is a method that uses electric force to draw charged threads of polymer solutions or melted polymer to produce nanofibers [2]. An electro spinnable polymer melt or solution must be able to carry an electric charge and must have enough viscosity to be stretched without breaking up into droplets [3]. Three components are required for the electrospinning process to be completed: a voltage supplier, a capillary tube with a small- diameter needle (often a syringe), and a grounded collecting screen/drum. When the voltage (range of 1-30 kV) is applied, the hanging drop of polymer solution becomes highly electrified with an electric charge induced on the liquid surface. It causes deformation of liquid drops into a conical object which named Taylor Cone [4].

Electrospun nanofiber membranes are a revolutionary type of material with a wide range of applications, including wound dressings. These membranes have a high surface-to-volume ratio, are versatile, and have a lot of microporosity. Electrospun nanofiber wound dressings, which are created using electrospinning technology, have various advantages. For starters, their structure and biological functions are very similar to that of the natural extracellular matrix (ECM), resulting in an ideal milieu for cell adhesion, proliferation, migration, and differentiation. Second, the polymer matrix utilized in electrospinning enables the combination of natural polymer biocompatibility and synthetic polymer mechanical strength [5].



**Figure 1:** Schematic view of electrospinning process.

### 1.3 Nanoparticles

#### **Polycaprolactone (PCL):**

- PCL is a semi-crystalline, biodegradable polyester derived from the ring-opening polymerization of caprolactone monomers.
- It has a low glass transition temperature ( $T_g$ ) of approximately -60°C and a relatively low melting point ( $T_m$ ) of around 60-65°C, which makes it suitable for various processing techniques, including electrospinning.
- PCL exhibits good mechanical properties, such as flexibility, toughness, and elongation, making it suitable for applications requiring robust materials.
- It is biocompatible and has been widely used in biomedical applications, such as tissue engineering scaffolds and drug delivery systems.
- PCL has hydrophobic characteristics, which can be advantageous for applications where water resistance or moisture barrier properties are desired.
- To improve fire resistance, flame-retardant chemicals can be mixed with PCL nanofibers. Depending on the required flame-retardant mechanism, various types of additives can be considered.

- Phosphorus-containing flame retardants, such as ammonium polyphosphate (APP) or phosphorous-containing polyols, can be used to impart flame retardancy. They emit phosphorus-containing gases that act as flame retardants.

### **Electrospinning of PCL Nanofibers:**

- Electrospinning is a versatile technology for creating polymer nanofibers with diameters ranging from a few nanometers to several micrometers.
- The process involves the use of an electric field to draw a charged polymer solution or melt into a fine fiber.
- Dissolving PCL and the desired flame-retardant additives in a suitable solvent, such as chloroform or dichloromethane, yields a polymer solution containing the desired flame-retardant additives.
- A fine needle or nozzle is used to load the polymer solution into a syringe.
- An electric field is created by applying a high voltage between the syringe needle and a grounded collector.
- As it travels towards the collector, the electric field forces the polymer solution to form an elongated charged jet that solidifies into nanofibers.
- Post-treatment processes, such as solvent evaporation or thermal annealing, can be used to eliminate remaining solvent and improve fiber shape and stability. [7].

### **Acetonitrile ( $C_2H_3N$ ):**

Acetonitrile is frequently employed as a solvent, reaction medium, or additive in the manufacture of flame retardant compounds.

- Acetonitrile is a common solvent used to dissolve flame retardant chemicals, especially in liquid formulations. Many flame retardant substances, such as those based on phosphorous or halogens, are only partially soluble in water or other typical solvents. Acetonitrile is a well-liked option for creating flame retardant formulations due to its excellent solubility and compatibility with a variety of flame retardant additives.
- In some instances, the synthesis of flame-retardant polymers involves using acetonitrile as a reaction medium. For instance, it might act as a solvent when monomers are polymerized or when flame-retardant moieties are added to polymers. Acetonitrile is useful for assisting chemical processes involved in the synthesis of flame retardant materials due to its capacity

to dissolve a wide range of reactants and its stability at high temperatures.

- Acetonitrile has some flame retardant characteristics and can be used as an addition to improve the flame retardancy of materials. Because of its high autoignition temperature (525°C) and low flammability, it is useful for enhancing the fire resistance of specific formulations. Acetonitrile can be used to improve the resistance of coatings, adhesives, and other materials to ignition or flame propagation.

While acetonitrile offers advantages in flame retardant material production, it is important to consider safety aspects associated with its use:

- Acetonitrile is a volatile organic compound (VOC) with a low flash point (-17°C) and is flammable. Proper handling, storage, and ventilation are crucial to ensure safety
- If inhaled, swallowed, or absorbed via the skin, acetonitrile can be hazardous. When working with acetonitrile, adequate personal protective equipment and safety procedures should be used.
- To avoid environmental pollution, acetonitrile should be disposed of in accordance with applicable legislation and recommendations.

It is vital to note that the usage of acetonitrile in the production of flame retardant materials varies based on the specific application, formulation, and regulatory constraints. When working with acetonitrile or any other chemical, proper handling and adherence to safety requirements are critical.

### **Chloroform(CHCl<sub>3</sub>):**

Chloroform, or trichloromethane, is an organic chemical compound with the formula CHCl<sub>3</sub>. It is a colorless, easily evaporating, fragrant, non-flammable, and heavy liquid at room temperature. It is frequently used as a solvent and is produced in large quantities for use in the production of PTFE and some refrigerants.

While chloroform has traditionally been utilized in a variety of industries, including the creation of certain flame retardant compounds, it is crucial to note that its use has dropped dramatically in recent years due to safety and environmental concerns. Chloroform is a dangerous compound that is both a probable carcinogen and a reproductive toxicant. It is now widely recognized as a dangerous chemical, and its usage is controlled or prohibited in many countries.

However, because of its ability to dissolve a wide range of flame retardant chemicals, chloroform was previously utilized as a solvent in the manufacturing of flame retardant polymers. It was extensively utilized to make polymer solutions or formulations containing flame retardant chemicals. To improve the flame retardant qualities, the dissolved chemicals would be absorbed into the polymer matrix.

It is critical to underline that the use of chloroform or any other chemical should be done with utmost caution, in accordance with all safety rules and regulations. In the case of flame retardant compounds, less harmful and more ecologically friendly solvents, such as safer organic solvents or water-based systems, are increasingly being used. These alternatives provide a safer working environment while lowering the environmental effect of the manufacture and use of flame retardant materials.

**Talc:**

Talc is a mineral that occurs naturally and is made up of magnesium, silicon, and oxygen. It is well-known for its chemical inertness, excellent thermal stability, and capacity to increase material mechanical properties. Talc, when used as an addition in flame retardant formulations, can increase the material's thermal deterioration and water resistance. It does not directly extinguish the flame, but rather works in tandem with other flame retardant substances to improve overall fire resistance.

Talc can be used in a variety of flame retardant materials, including plastics, coatings, and textiles. In plastics, talc can improve the mechanical properties and flame retardancy of the material when incorporated into the polymer. In coatings, it can improve adhesion and mechanical properties. In textiles, talc can be added to clothing fabrics to enhance their fire retardancy. When exposed to heat or flames, talc promotes the creation of a solid, insulating char layer. This char layer works as a physical barrier, delaying the combustion process by decreasing heat and oxygen transfer to the underlying substance. The production of a stable char layer can aid in the prevention or delay of ignition, the reduction of flame propagation, and the limitation of the release of flammable gases.

Talc can help flame retardant chemicals disperse and distribute more evenly throughout the polymer matrix. Its lamellar structure and tiny particle size help to the development of a homogenous mixture, ensuring that flame retardant chemicals are distributed uniformly throughout the material. This dispersion improves fire resistance and

the efficiency of the flame retardant system.

Talc has strong thermal stability and can resist high temperatures without degrading much. This makes it suited for applications involving the exposure of flame retardant materials to high temperatures. Talc's heat resistance contributes to the flame retardant system's integrity and stability during fire exposure.

Talc can give mechanical support to the polymer matrix in addition to its flame retardant qualities. It increases the stiffness, dimensional stability, and impact resistance of the material, making it useful for structural applications that require both flame retardancy and mechanical performance.

Talc is found in a variety of flame retardant materials and compositions, such as plastics, thermosets, coatings, and fabrics. It is used in industries such as automotive, construction, electronics, and textiles where fire safety and flame resistance are essential.

It is important to note that the precise formulation and composition of flame retardant materials can differ based on the desired level of flame retardancy, the intended use, and regulatory restrictions. [8].

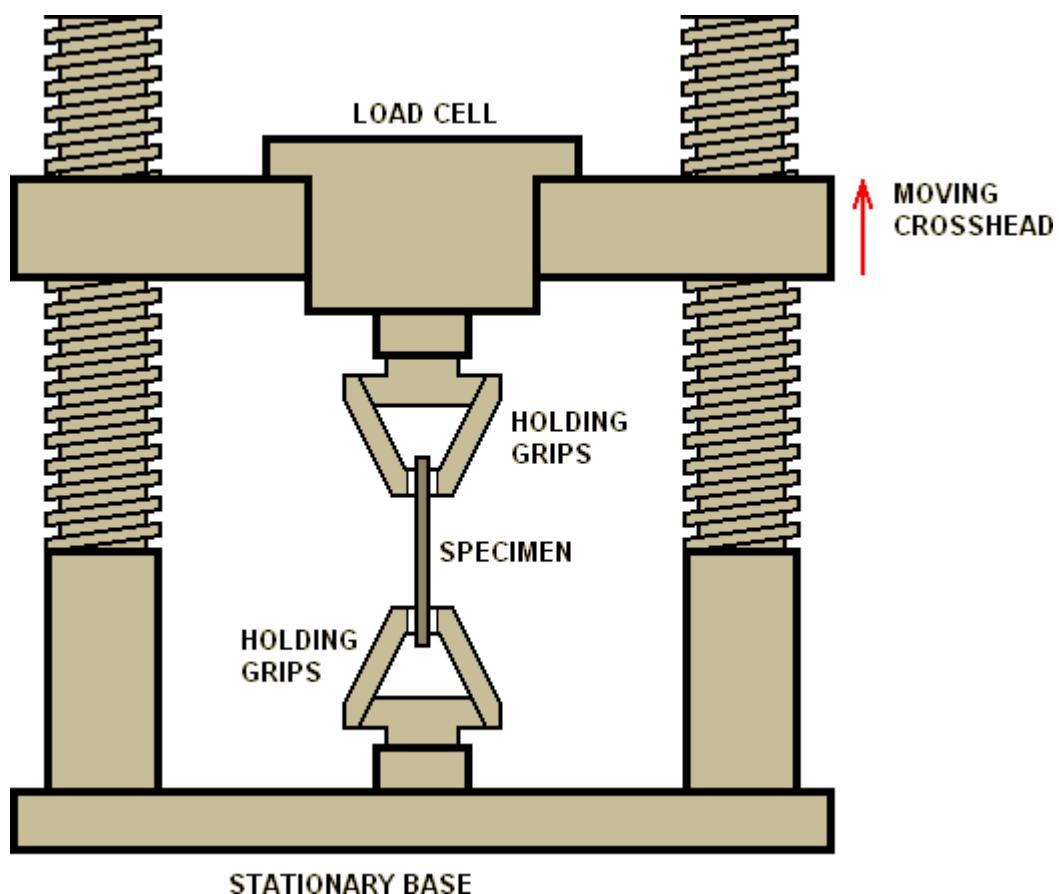
### **Titanium Dioxide( $\text{TiO}_2$ ):**

- Titanium dioxide possesses photocatalytic qualities that may help PCL nanofibers be flame resistant.  $\text{TiO}_2$  nanoparticles produce reactive oxygen species when exposed to UV light, which can oxidize and degrade combustible gases and volatile organic compounds produced during combustion. This oxidative process aids in stopping the chain reaction of combustion and containing flame spread.
- To create a homogeneous mixture,  $\text{TiO}_2$  nanoparticles can be scattered throughout the PCL polymer matrix. Effective flame retardancy and improved overall performance of the nanofibers require proper  $\text{TiO}_2$  dispersion. The compatibility between  $\text{TiO}_2$  nanoparticles and PCL can be enhanced through methods including surface modification or the application of surfactants, which will facilitate their dispersion.
- $\text{TiO}_2$  nanoparticles may be introduced into the PCL polymer melt or solution during the electrospinning procedure.  $\text{TiO}_2$  serves to disperse the flame retardant activity throughout the PCL nanofibers, enhancing the material's fire resistance. By generating continuous nanofibers with a high surface-to-volume ratio using electrospinning, it is possible to further improve the flame retardant qualities of the resulting nanofiber mats.

## 1.4 Tensile Test

Tensile testing is a material science and engineering test that measures the behavior of a material against tensile force. In this test, a sample is subjected to a controlled tension until it fails. This test allows to measure the properties of the material such as ultimate tensile strength, breaking strength, maximum elongation and reduction in cross-sectional area. From these measurements, other properties of the material such as Young Modulus, Poisson ratio, yield strength and strain hardening characteristics can also be determined.

Tensile testing is the most common method to obtain the mechanical characteristics of isotropic materials. Some materials use biaxial tensile testing. The main difference between these testing machines is how force is applied to the material. [9]



**Figure 2:** Schematic diagram of tensile testing machine [10].

Tensile testing may have various purposes, such as:

- Select a material or item for an application.
- Predict how a material will perform in use: normal and extreme forces.
- Determine if, or verify that, the requirements of a specification, regulation, or contract are met.
- Decide if a new product development program is on track.
- Demonstrate proof of concept.
- Demonstrate the utility of a proposed patent.
- Provide standard data for other scientific, engineering, and quality assurance functions.
- Provide a basis for technical communication.
- Provide a technical means of comparison of several options.
- Provide evidence in legal proceedings.

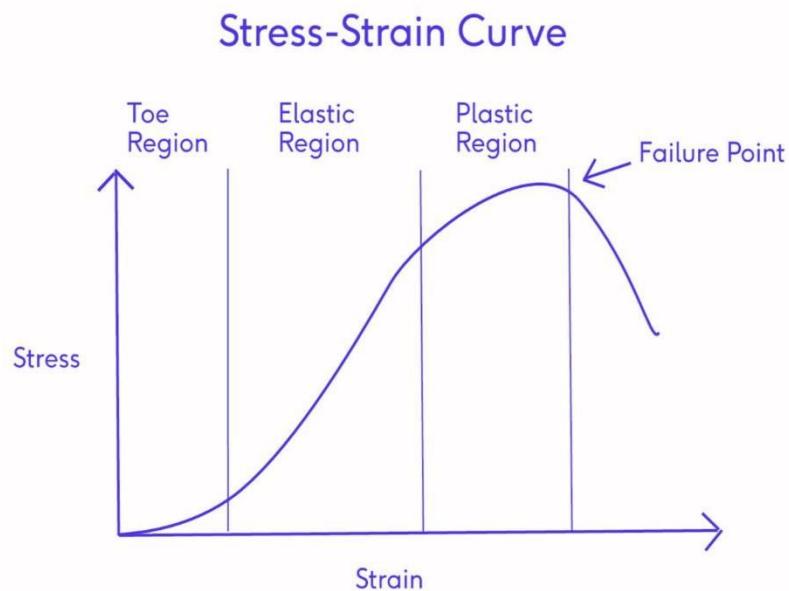
The preparation of test specimens depends on the purpose of testing and on the governing test method or specification. A tensile specimen usually has a standardized cross-sectional area. It has two shoulders and a gauge (section) in between. The shoulders and grip section are generally larger than the gauge section by 33% so they can be easily gripped. The smaller diameter of the gauge section also allows the deformation and failure to occur in this area. [11]

Stress and strain calculation depends on the applied force and the dimensions of the material. Stress is the ratio of the applied force to the cross-sectional area of the material. Strain is the ratio of elongation of the material.[4] According to these definitions, the following formulas are used:

$$\varepsilon = \frac{L - L_0}{L_0} \quad (1)$$

Yield point is the point where the elastic behavior of the material ends, and the plastic behavior begins. At this point, the material is permanently deformed and will not return to its original size and shape, even when the applied force is completely removed. The yield point corresponds to the yield strength on the stress-strain graph. Yield strength is the minimum stress required for the material to enter plastic deformation.

Ultimate stress is the maximum stress that the material can withstand before breaking. This value corresponds to the ultimate tensile strength on the stress-strain graph. Ultimate tensile strength is the point where the cross-sectional area of the material decreases rapidly, and a phenomenon called necking occurs.



**Figure 3:** Stress-Strain Curve [12]

## 2) MATERIAL AND METHODS

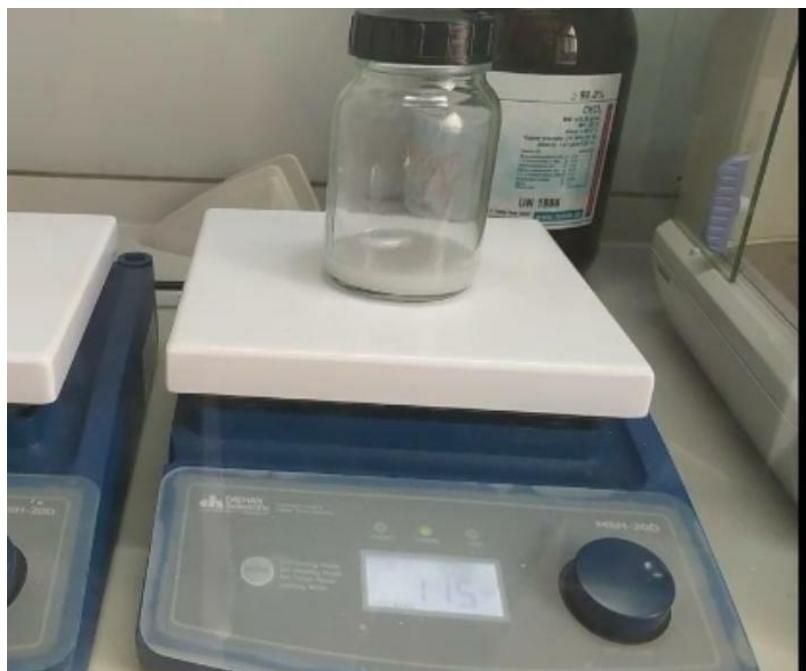
### 2.1 NANOFIBROUS MATS

#### 2.1.1 Materials

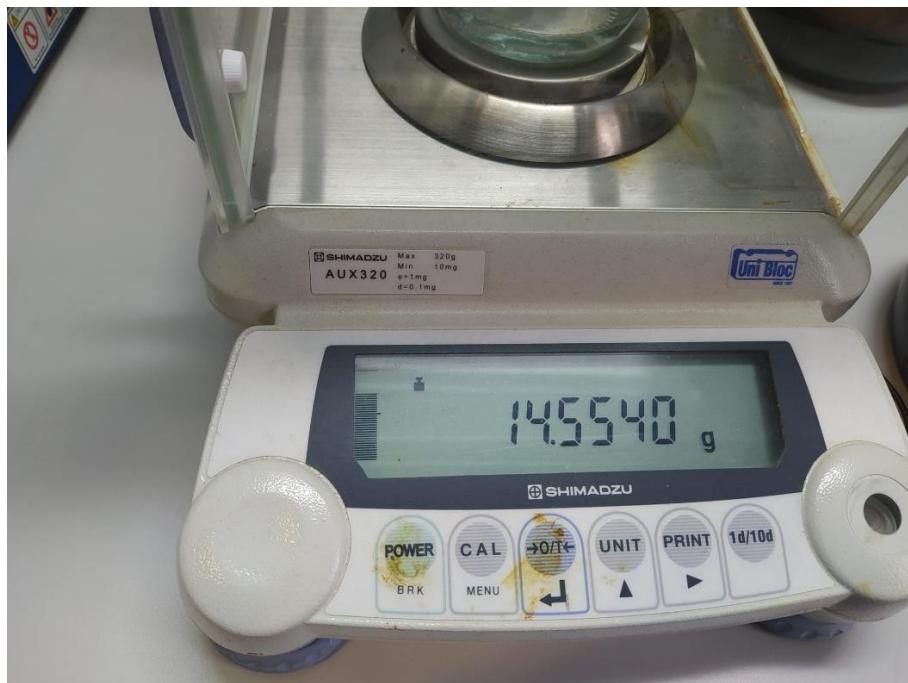
- **PCL**
- **Chloroform**
- **Acetonitrile**
- **Talc**
- **TiO<sub>2</sub>**

#### 2.1.2 Preparation of Solutions for Electrospinning

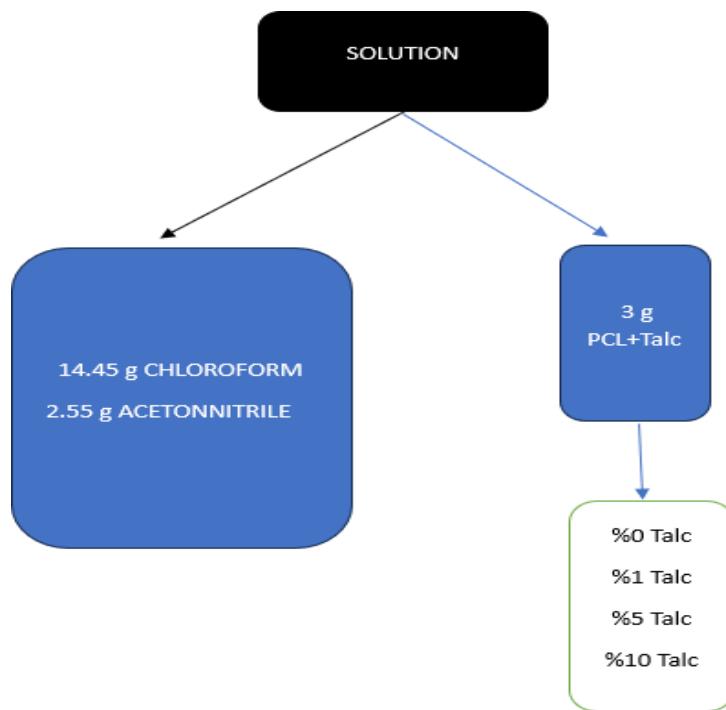
The following procedure was performed to prepare the solutions. A mixture of chloroform (14.45 g) and acetonitrile (2.55 g) was prepared first. Then PCL (3 g) was added to this mixture. For other solutions, talc was added at different concentrations (1%, 5%, 10% and 15%) of the total mass of 3 g, and the rest was PCL. The same method was applied for TiO<sub>2</sub> solutions. The resulting mixtures were magnetically stirred at 50°C for 12 hours at a speed of 115-150 rpm.



**Figure 4:** Solution left to spin on hot plate



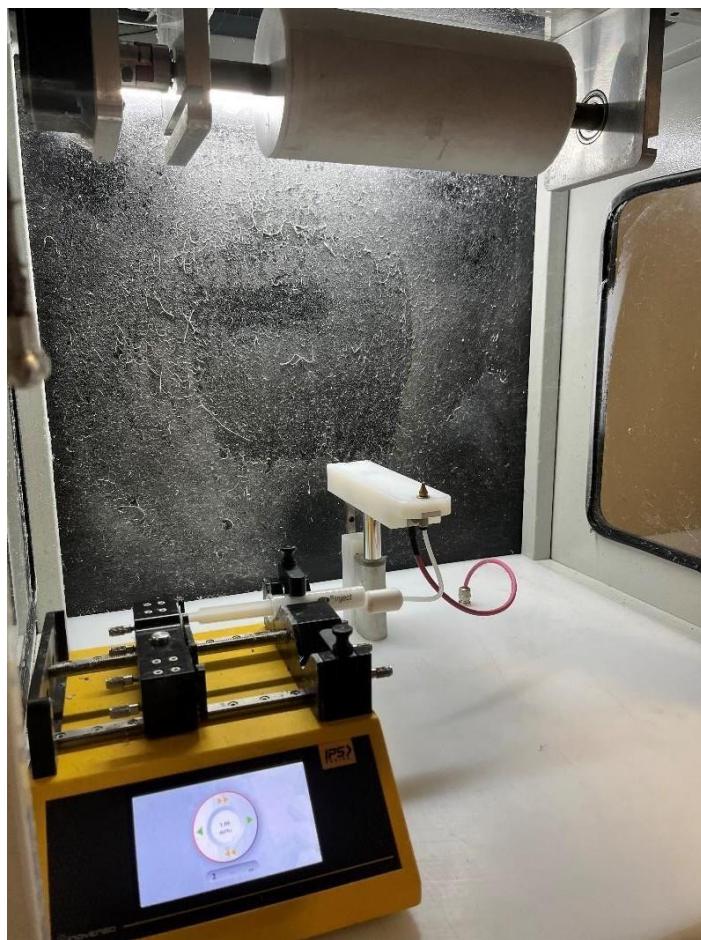
**Figure 5:** Preparation of the solution



**Figure 6:** Quantity of Nanoparticles in Solution

### 2.1.3 Nanofiber fabrication

The solution was 10 mL of it was drawn with a syringe. An “Inovenso NE 200 Nanospinner” electrospinning machine was used. The syringe was connected to the pump and the solution flow rate was set to 1 mL/h. The nozzle and the rotating drum collector were 25 cm apart. A voltage of 20 kV was applied. The temperature was maintained at 25°C throughout the fabrication process. Also, greaseproof paper was used to cover the collector drum for easy removal of the fibers from the drum. The nanofiber solution could dry and harden at the nozzle tip, which could cause nozzle clogging. Therefore, the nozzle was regularly cleaned. All nanofiber mats were fabricated with the same parameters and procedures.



**Figure 7:** Electrospinning setup.



**Figure 8:** Electrospun nanofiber

#### 2.1.4 Tensile Test

Electrospun mats were cut into specimens of 60x10mm size for tensile testing. Also, paper frames of 60x45mm size and 10mm thickness were prepared. The fiber mat was fixed to the frame with water-based adhesive and double-sided tape. Moreover, cardboard was attached to both sides of the grip to increase the thickness of the grip. A “Shimadzu AGS-X 50 kN” tensile testing machine was used. The test specimen (the fiber membrane with the test window frame) was placed on the tensile tester such that the tabs holding the fiber membrane were on the grip. The gauge length was 40 mm.



**Figure 9:** Tensile Test Machine



**Figure 10:** Tensile Test Machine



**Figure 11:** Test Specimen

## 2.1.5 Thermal Test

The main goal of our study was the creation of flame-resistant nanofiber materials. Thermal efficiency testing was very important for assessing how heat was responded to by small samples. An experimental setup was created by us by adopting an engineer's mindset and taking into account the available resources and parameters. Among other things, these characteristics included ambient density, temperature, humidity, and pressure. In order to reduce the impact of different physical elements, the thermal heat test was carefully carried out in a closed room, under controlled circumstances, at a certain time of day. Our main objective was to maintain a constant atmosphere by minimizing the effects of variables like pressure, humidity, and temperature variations brought on by the sun's position and angle. All efforts were taken to stabilize these ambient conditions as much as feasible, despite the fact that a completely sealed system was not practicable. By preventing misleading data from ambient conditions, this project attempted to make it possible to compare the samples accurately. Such stringent monitoring was necessary to guarantee accurate results throughout the experiment. A material's ignition and combustion properties are not always determined by direct contact with a heat source. Instead, their heat resistance without coming into touch with the ignition source should also be considered when their flame retardant qualities are evaluated. Moreover, a more effective configuration was created by us by including a bigger number of factors in the experimental setting. Compared to the ignition test, the results from the thermal heat test were clearer because the material did not completely burn up but instead shrank over time. The purpose of the heat test is to examine how the material responds to heat under conditions. The thermal heat test involves evaluating the sample's numerous physical characteristics as a function of temperature and time, including its melting point, dehydration point, isomer transition point, weight, and conductivity. The changes in these physical attributes were intended to be observed and quantified by us by exposing the material to controlled heat within predetermined boundaries. Notably, the purposeful shrinkage of the material upon exposure to the heat source was caused by the design of our experimental apparatus. Given our small number of sample pieces, this design decision was taken by us to investigate the material's sensitivity to heat and collect as much information as feasible. In the sections that follow, the parameters used in our painstakingly created experimental setup will be explained by us and an explanation for the thinking behind their selection will be given.

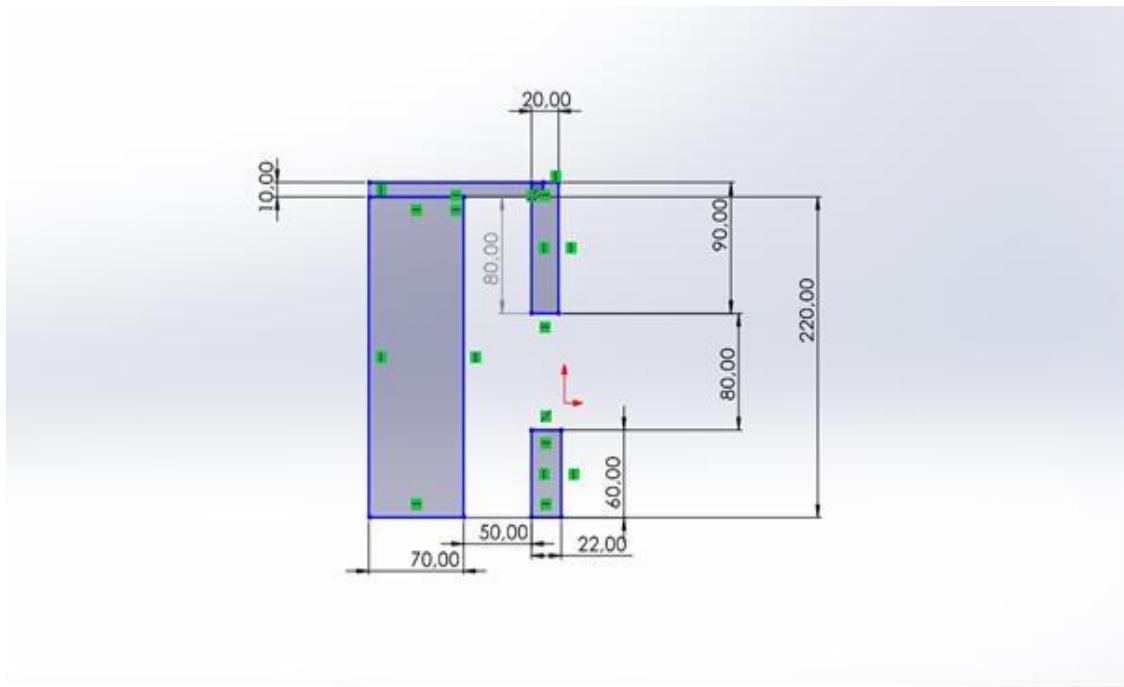
As can be seen in the figure, we divided the samples into 4 equal parts so that we can observe how the heat affects the sections on the sample. At the same time, this allows us to obtain more data and make a detailed examination. Each sample has a length of 2 cm by 9 cm and a length of 2 cm by 4 cm. It is divided into equal parts. It is a share so that it can hold a 1 cm caliper. As it is seen in the figure, when the heat is given, the samples shrunk completely by shrinking in turn. The heat progresses in sequence on the steps. In this way, it is possible to examine in each region the effect of the samples in which region with heat from start to finish. Where the starting point is the side close to the heat source and the end is the side close to the caliper. the heat travels from the starting point to the end point by shrinking each chamber. Thanks to the pen that we separated each part of, a momentary pause was observed during the shrinkage transition between the partitions, thus enabling us to observe the transition between the partitions and to get the data in the right number of times. As a result, dividing it into four compartments has enabled us to examine the effect of heat from the beginning to the end of the sample in detail, to obtain more data, and to make our observation much clearer. We schematized our experimental setup in solid, here we will explain the parameters in detail.



**Figure 12:** After and Before Heat Process

The object with a length of 7 cm and a length of 22 cm, visible on the below, was chosen to keep the heat source at a constant distance from the sample. In addition, as seen in the diagram, it allows a fixed 8 cm distance between the heat source and the samples. In addition, the fixed distance of the heat source to the box is 5 cm. The height of the heatsource from the ground is 6 cm. The object that raises the caliper from the ground, thanks to

the width of the caliper, the samples rose 14 cm from the ground. The reason why all the distances are applied consistently is to ensure that all samples are exposed to heat equally. The heat source was turned on at the same degree each time. Since the distances were predetermined, all samples were exposed to the heat at the same location. All samples were observed under equal parameters.

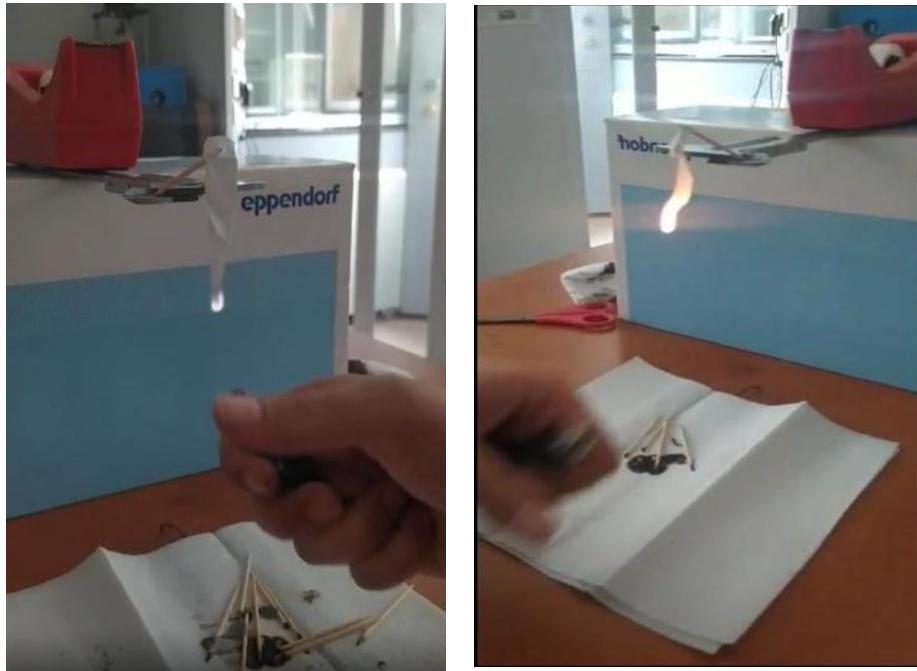


**Figure 13:** Test Setup

### 2.1.6 Ignition Test

The setup we designed for the ignition test is shown in Figure 14. While designing the experimental setup, we aimed to test the samples with the same parameters. Temperature, humidity, pressure, etc., which depend on the environment, are among these parameters. We carried out our experiment in a short time without changing the ambient conditions in a closed room to keep the values constant (at least close). The aim is to complete the experiment in a short time, in which factors such as the angle of the sun, the position of the earth, etc. affect variables such as temperature, humidity, etc. at the least. Although we tried to do it in a short time, we placed the samples one by one carefully and according to the standard dimensions that we determined beforehand. The experimental setup mechanism was kept constant, the samples were placed in the experimental setup in order, but as it is known, the thermal test and the ignition test are affected by different parameters. In the thermal test,

while variables such as the distance of the temperature source to the sample, the sample location, the intensity of the temperature are very important, in the ignition test, since the temperature source is in direct contact with the sample, the distance and the setup It is not as important as in the thermal test. Since the temperature source directly contacts the sample and ignites it, the most important parameter in the experimental setup is the temperature source. That's why we kept the temperature source setting we used here constant. Other effective parameters are humidity, temperature, pressure, etc. are environmental factors. For this reason, we performed it in a completely closed room without a heat exchanger, humidity exchanger, etc.



**Figure 14:** After and Before Ignition Process

Here, as seen in Figure 14, we used a box to raise it from the ground. We placed a caliper on the box to hold the material. Afterwards, we decided to attach the caliper to the caliper with a disposable material so that the caliper would not be damaged by ignition. This material was toothpick. Also, its length was 8 cm and width 2 We marked this region by adding 0.5 cm to our material which is cm. The purpose here was to leave a margin for the part where it joins with the toothpick. The toothpick is the only variable in the experimental setup. But as I mentioned before, since the sample would burn, it could damage the materialto which it was fixed. This was also a problem to be solved. Because the setup had to be fixedto compare the samples. In this way, both the mechanism we fixed was not damaged and the experimental setup contained the same parameters for all samples. Since we have explained the mechanism, we can talk about ignition. As seen in Figure 14, the heat source was brought.

close to the sample to ignite. Then, the heat source was turned off when ignition started in each sample. As seen in Figure 14, ignition progressed from beginning to end on the sample and finished the sample. We recorded these times. Of course, we need to mention about the samples. To give accurate data and to make a correct comparison, the sizes of the samples are cut exactly equal. These dimensions are 2 cm in width and 8 cm in length. Also, the samples were taken from the middle part of the produced nanofibers to have the same thickness. This can be explained by briefly explaining the electrospin process. Electrospin is made by throwing electrons transversely into a rotating cylinder. In this case, the cross-sections on the nanofiber contain much lower errors. Thus, the transverse samples have almost the same thickness. Let's take a look at the importance of the size of the samples in the ignition test. Our ignition test can be explained in its simplest form as follows: propagation of ignition from the starting point of the sample to the ending point. Our aim in all these tests and experiments is flame retardation, it should not be forgotten, it is not to obtain a completely fireproof material. As can be seen in ignition Figure 14, starting from the beginning, the material is finished, and the material is in Figure 14. We can compare this to a vehicle moving on a straight road, like going from point a to point b. The time passing from point A to point B can be thought of as the data in our table. Here point A can be compared to the starting point of the sample, point B to the end point of ignition in the vehicle. The reason why we make our explanation here is both to fully understand the parameters of the test and to see how well the comparison is made. Ignition can be compared to the vehicle here. The sample is road. As we mentioned before, we can say that the vehicle is the same as the ignition comes from a constant heat source. The lengths of the samples must also be the same so that the path taken by the ignition is the same in this test where we use constant ignition. In this way, it can be observed how the material contained in the sample changes the burning time. As a result, samples with the same width and thickness were used. The heat source is constant. Ambient conditions were tried to be kept constant. In short, the only variable that is variable is the density of the material contained in the sample. In this way, we can examine the flame retardancy effects of the samples by considering the burning time.

### 3) CALCULATIONS AND RESULTS

#### 3.1 NANOFIBROUS MATS

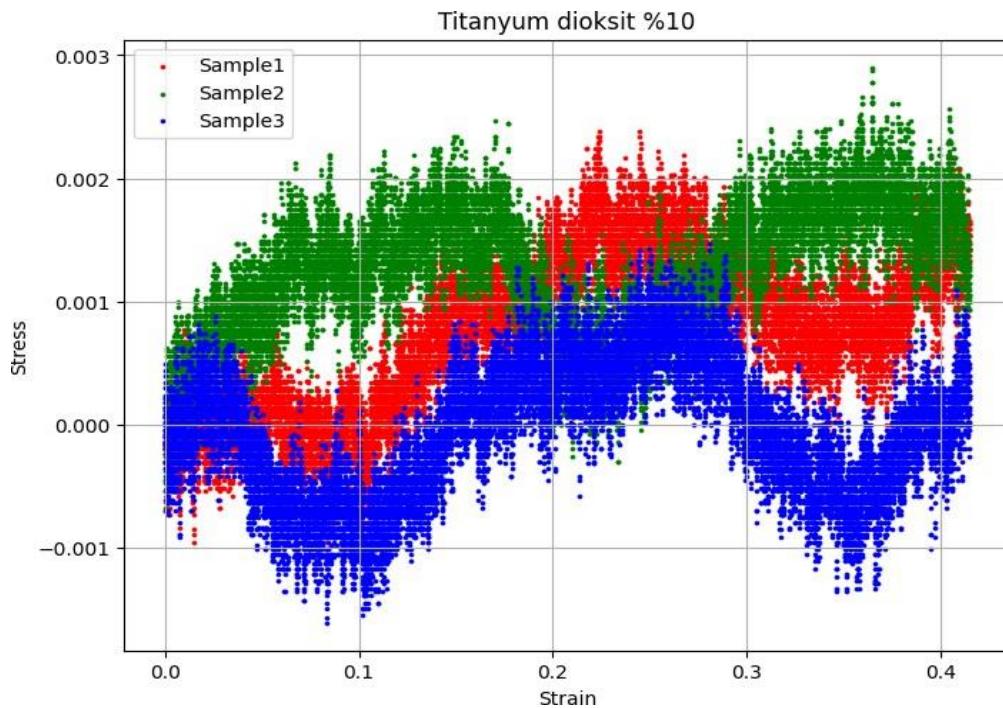
##### 3.1.1 Tensile Test

Tensile stress and strain can be calculated with the formulas below:

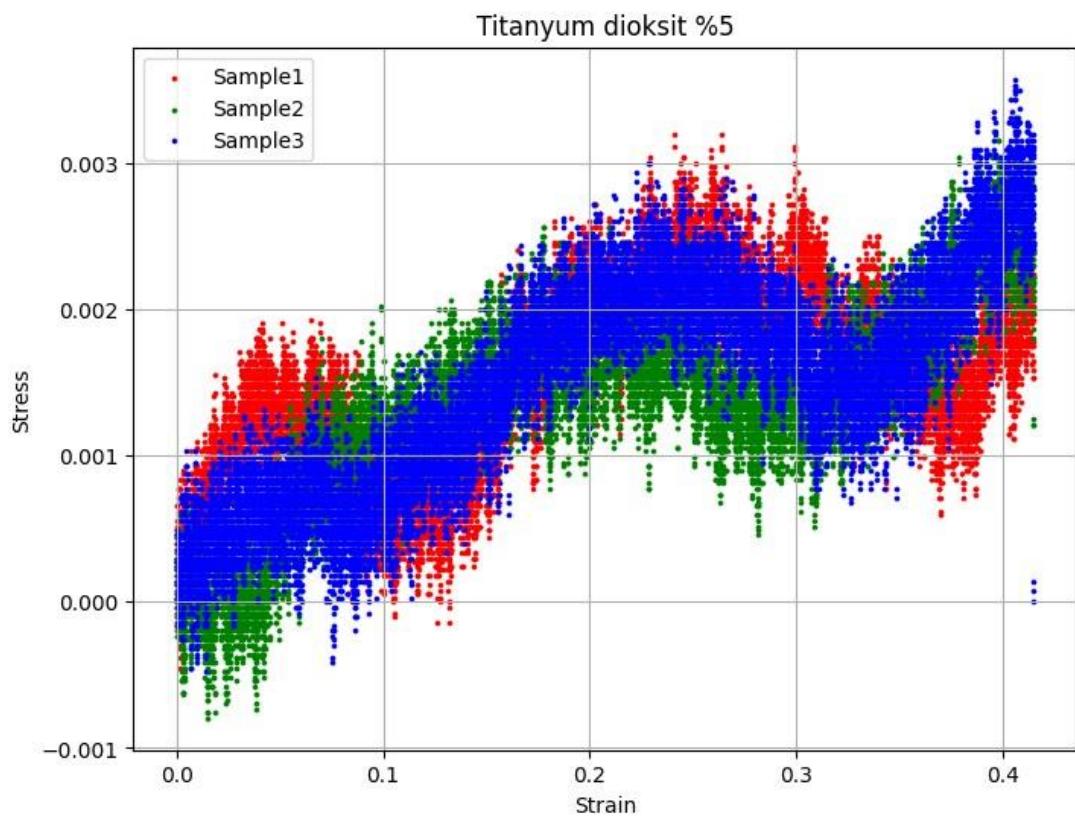
$$\sigma = \frac{F}{A} \quad (3)$$

$$\varepsilon = \frac{\Delta L}{L_0} \quad (4)$$

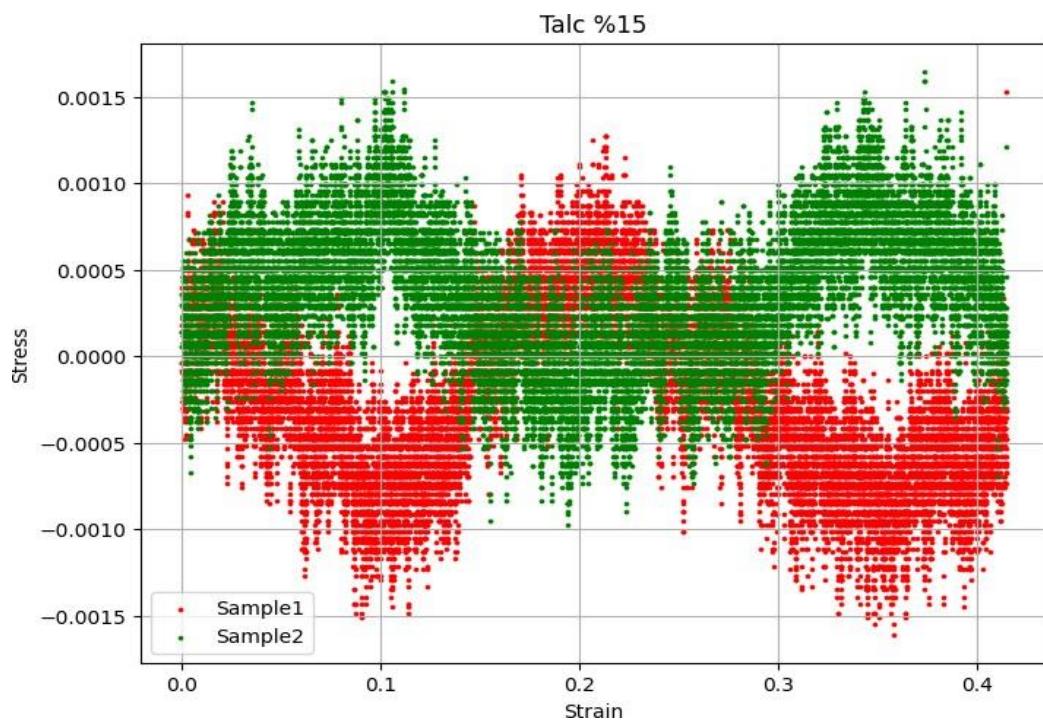
Stress and strain calculations were made with the data obtained from the tensile test results. The graphs obtained as a result of these calculations are given below:



**Figure 15:** 10% TiO<sub>2</sub> Solution

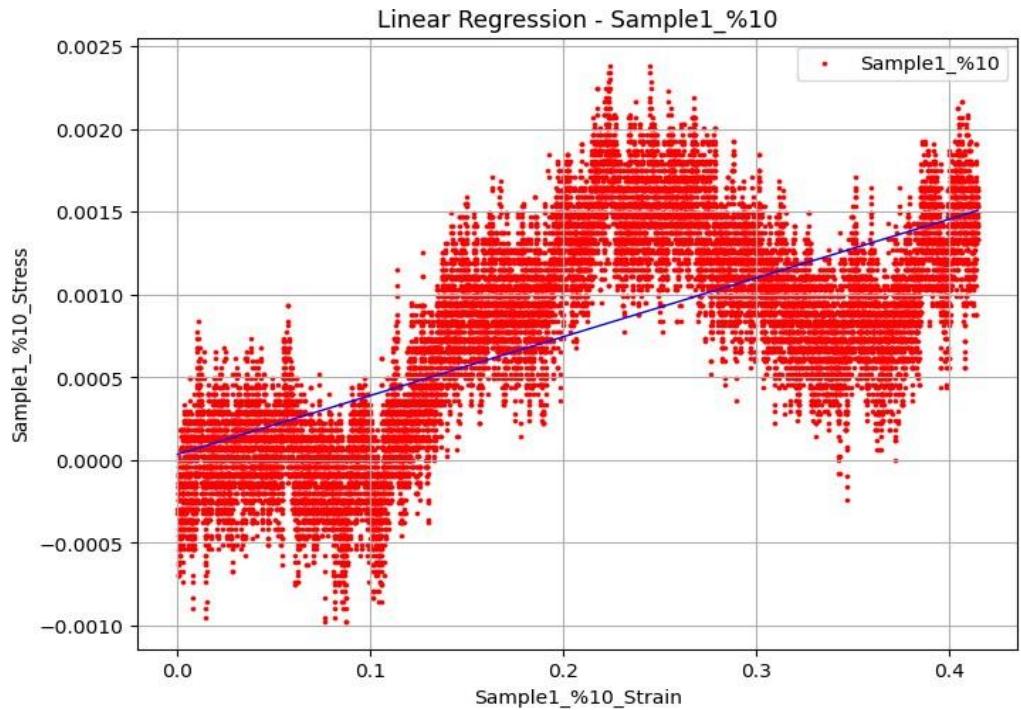


**Figure 16:** 5%  $\text{TiO}_2$  Solution

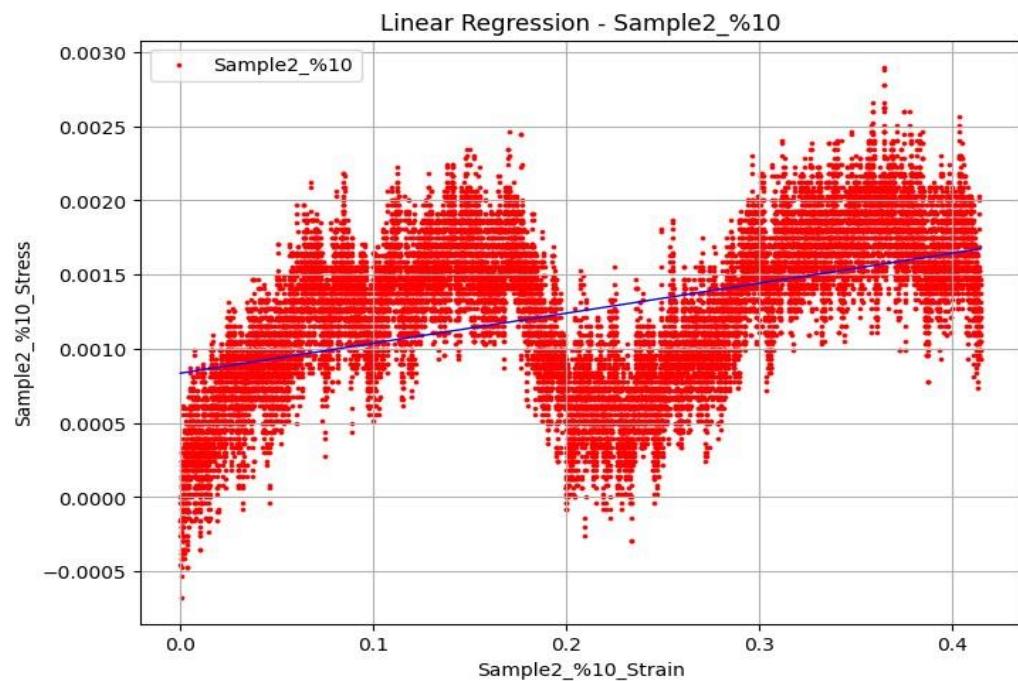


**Figure 17:** 15% Talc Solution

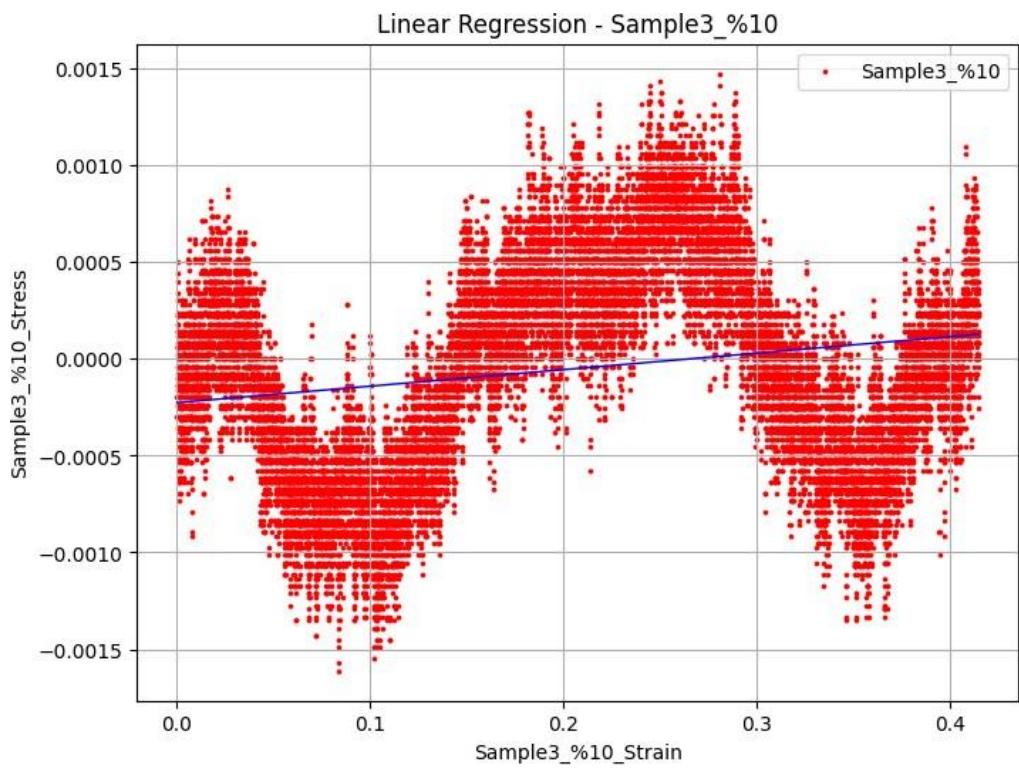
Polynomial and linear regression methods were employed to obtain a better fit for the irregular shapes of the graphs.



**Figure 18:** Linear Regression 10%  
TiO<sub>2</sub> Solution Sample 1

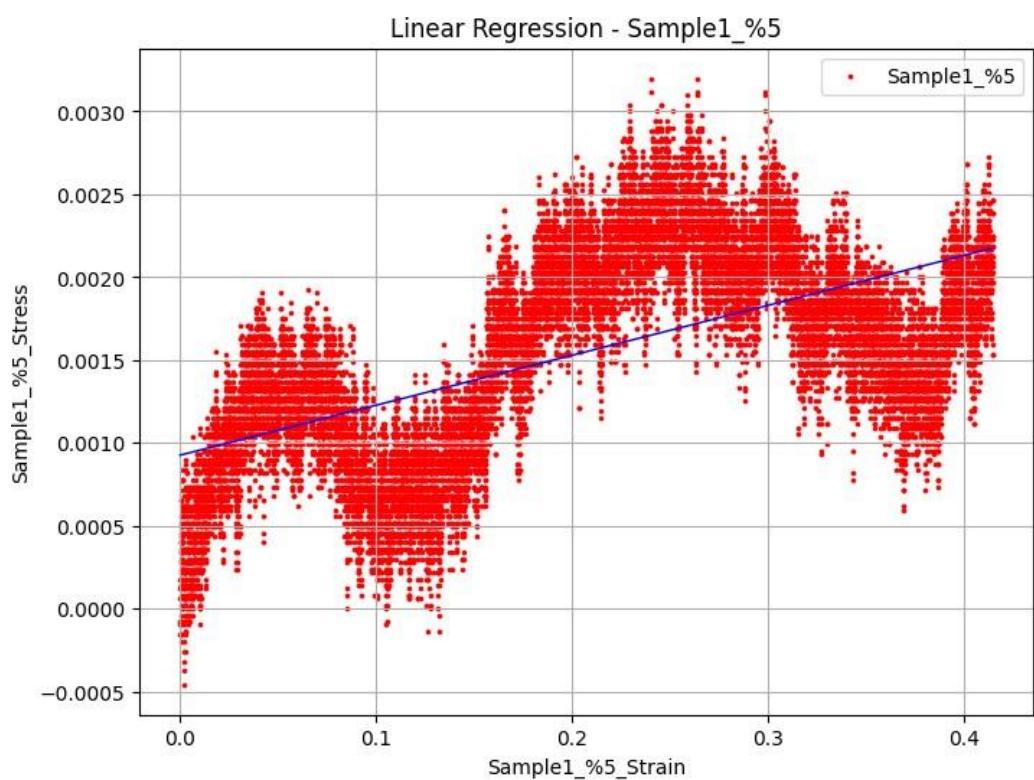


**Figure 19:** Linear Regression 10%  
TiO<sub>2</sub> Solution Sample 2

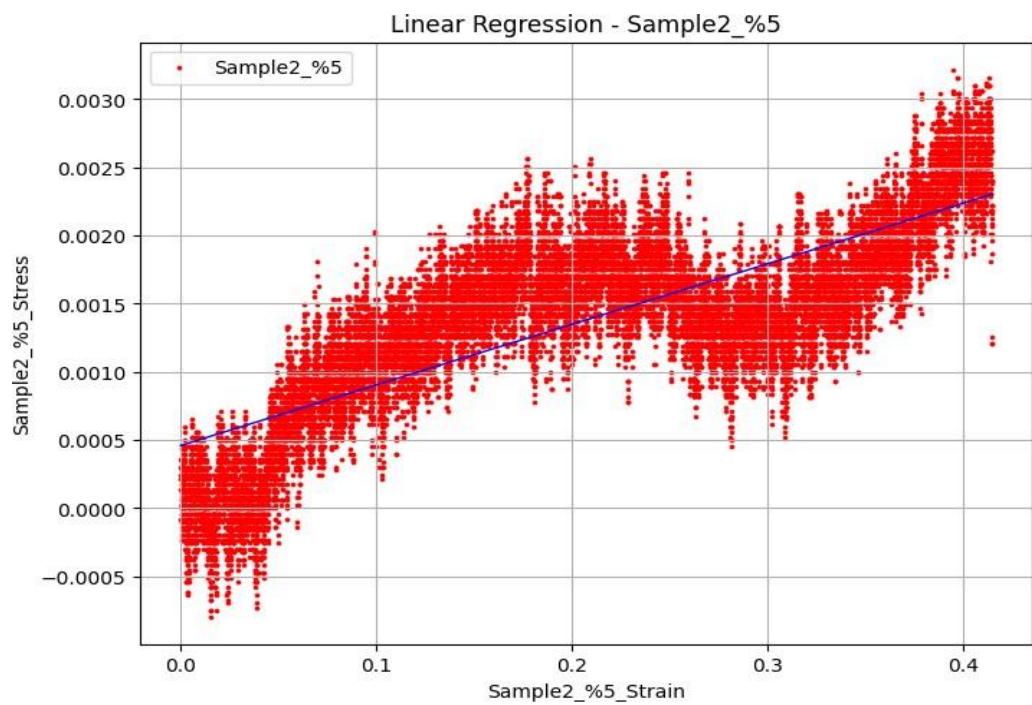


**Figure 20:** Linear Regression 10% TiO<sub>2</sub> Solution Sample

3

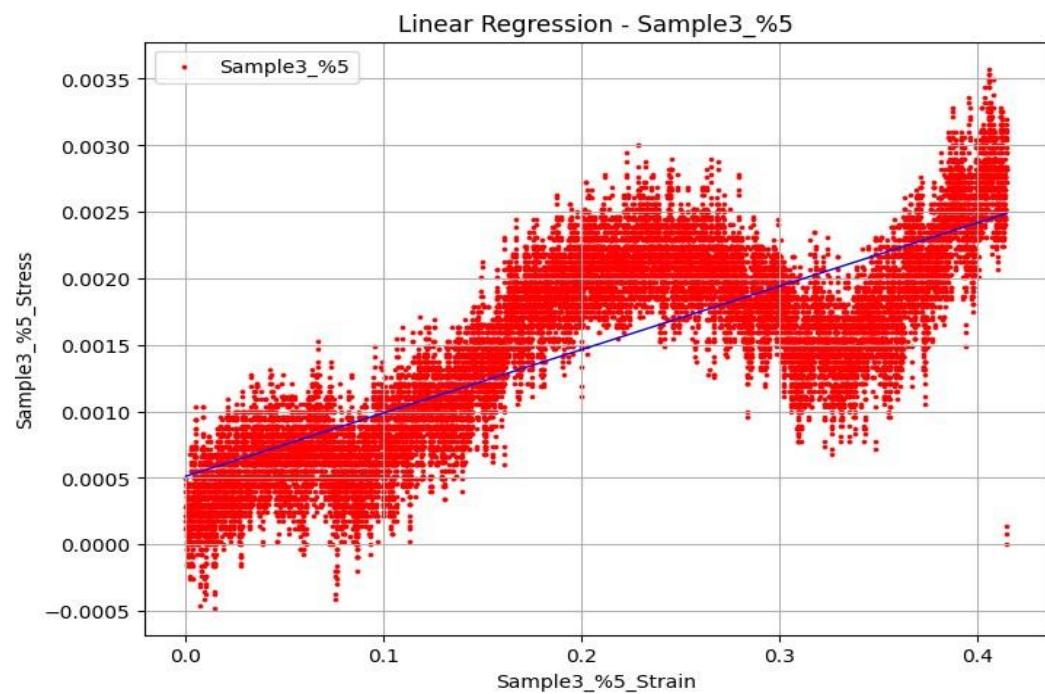


**Figure 21:** Linear Regression 5% TiO<sub>2</sub> Solution Sample 1

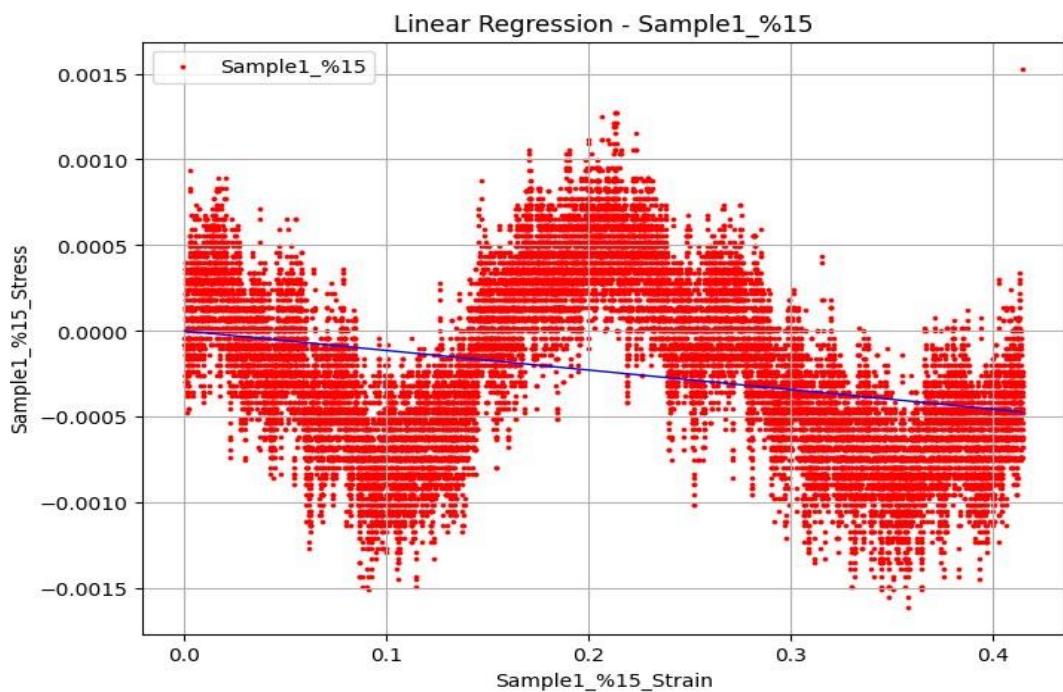


**Figure 22:** Linear Regression 5% TiO<sub>2</sub> Solution Sample

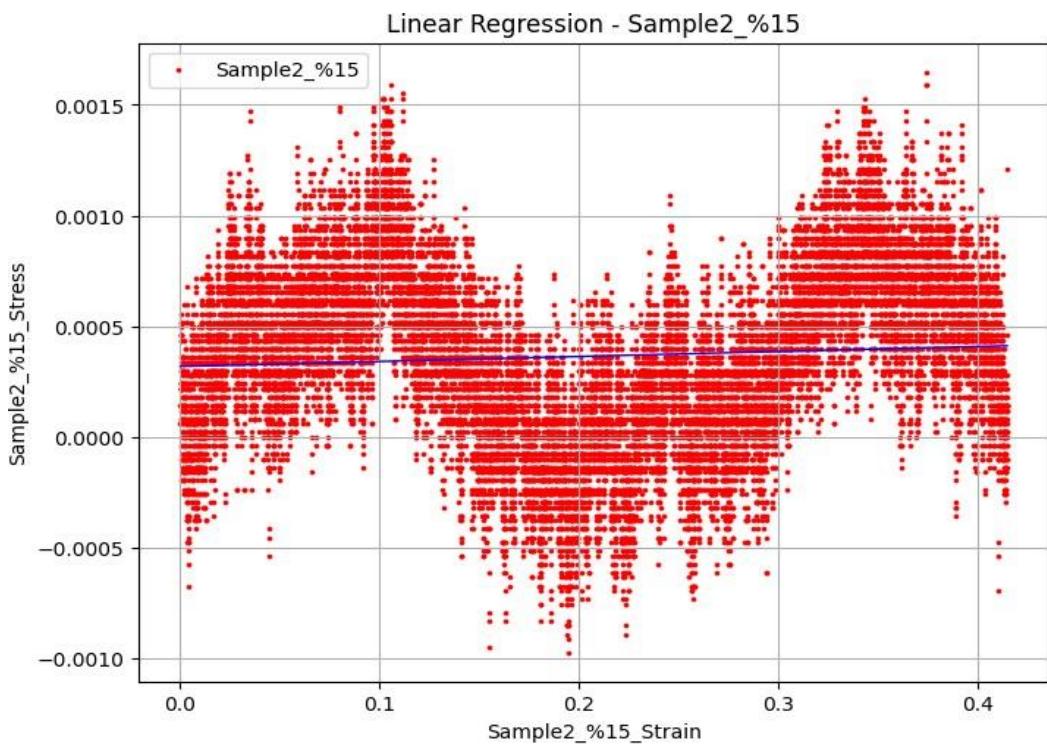
2



**Figure 23:** Linear Regression 5% TiO<sub>2</sub> Solution Sample 3

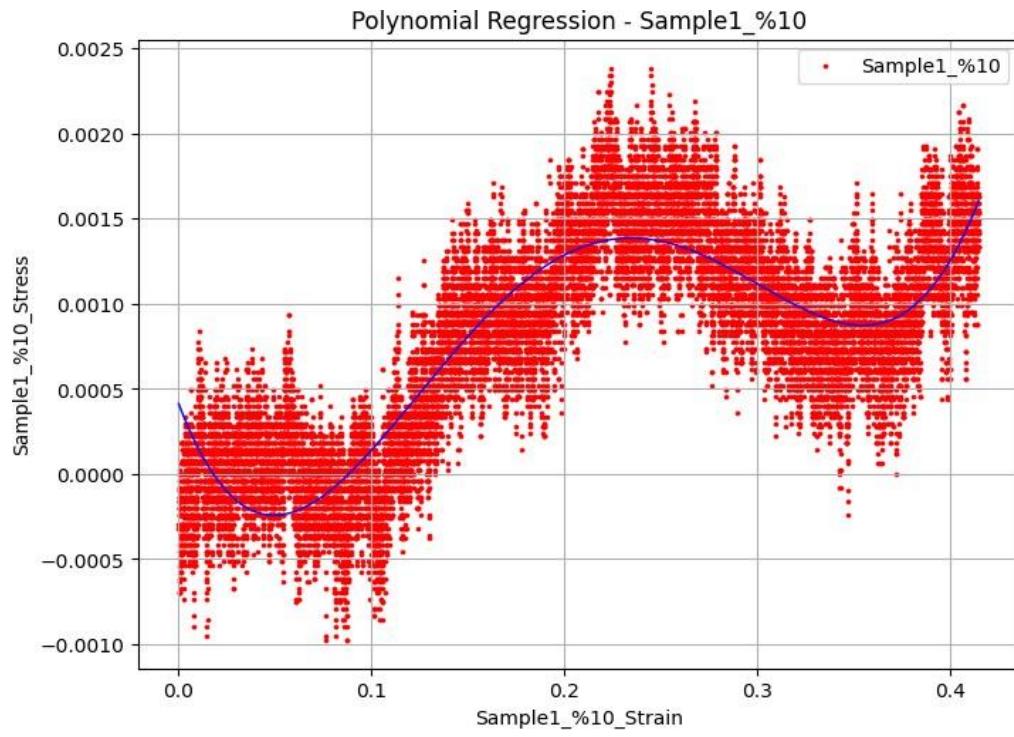


**Figure 24:** Linear Regression 15% Talc Solution Sample 1

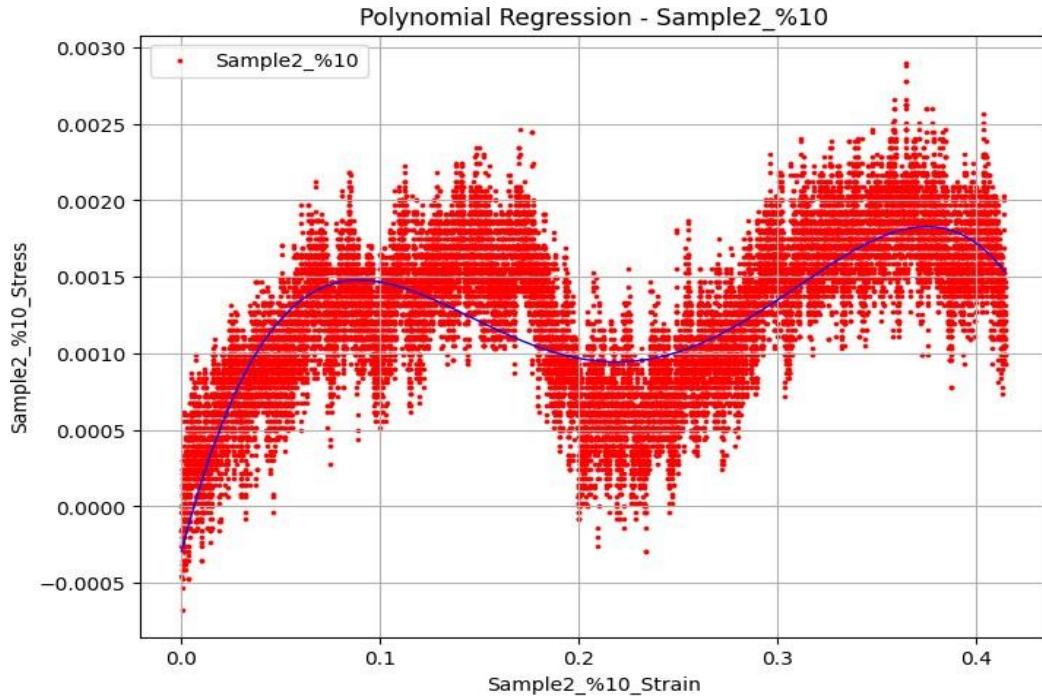


**Figure 25:** Linear Regression 15% Talc Solution Sample 2

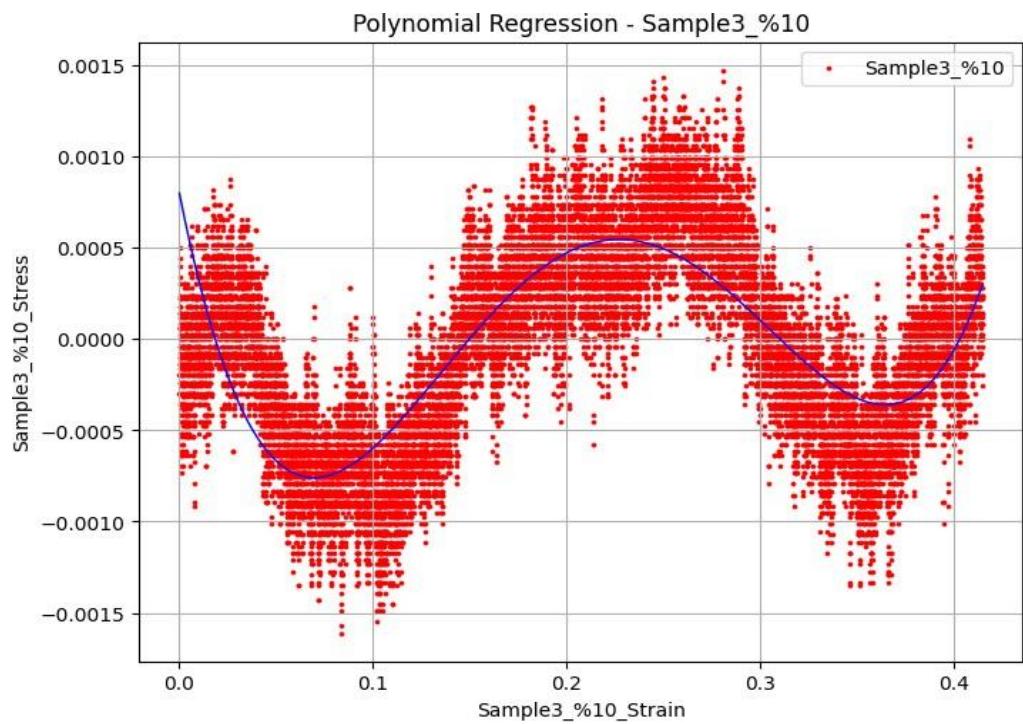
26



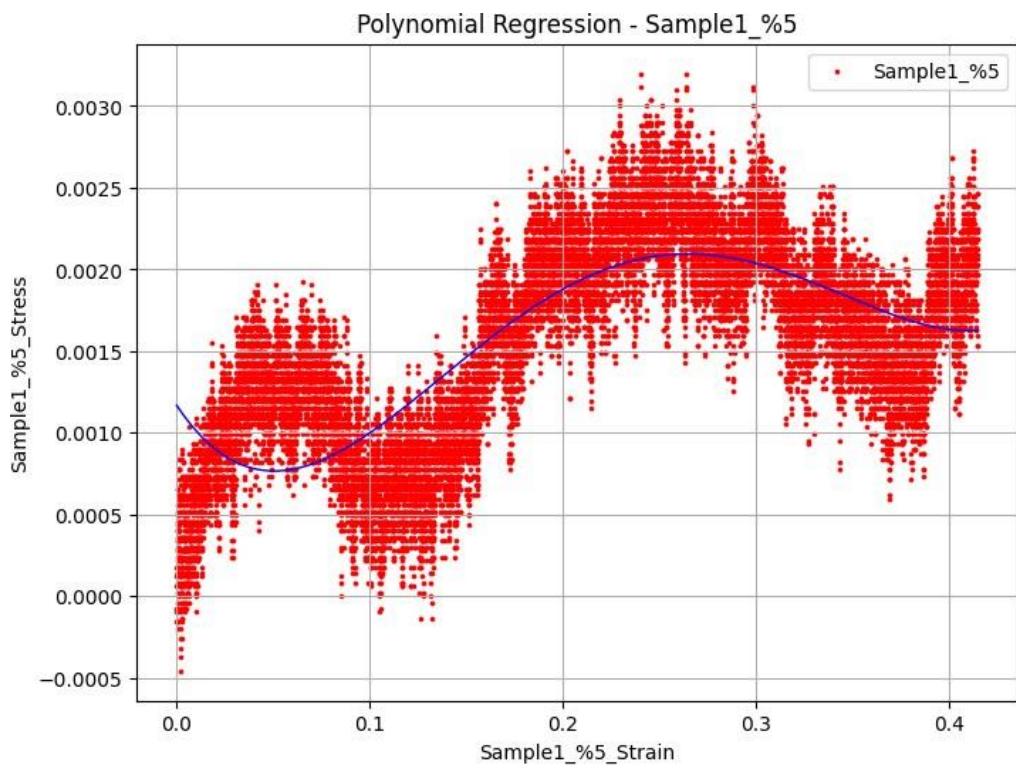
**Figure 26 :** Polynomial Regression 10% TiO<sub>2</sub> Solution Sample 1



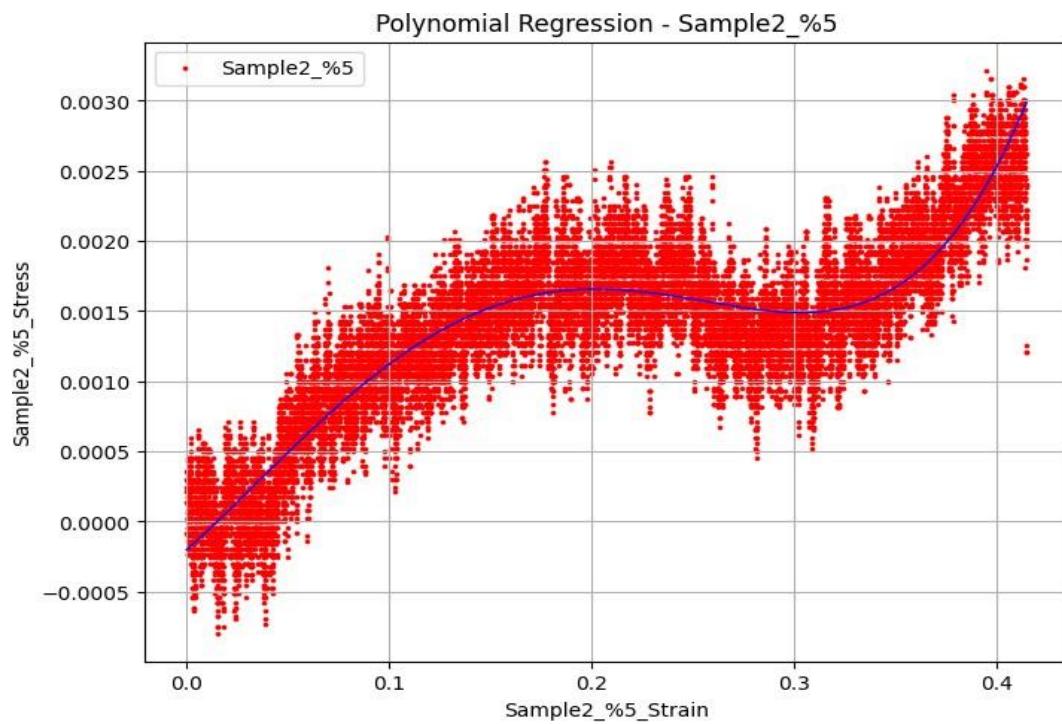
**Figure 27:** Polynomial Regression 10% TiO<sub>2</sub> Solution Sample 2



**Figure 28:** Polynomial Regression  
10% TiO<sub>2</sub> Solution Sample 3

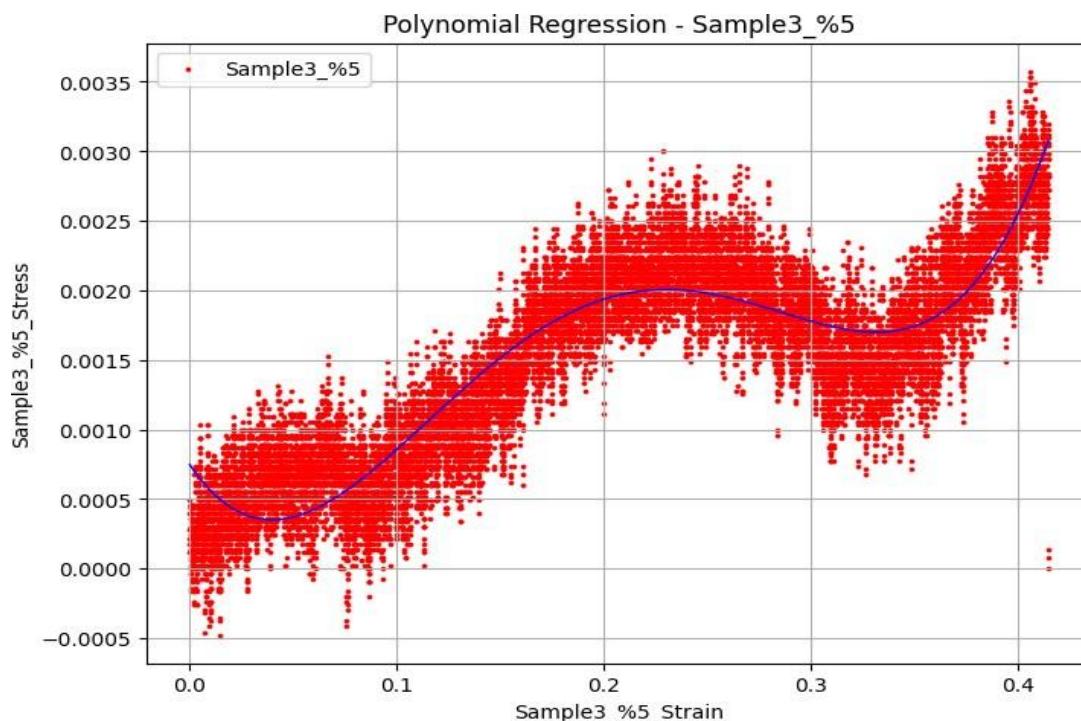


**Figure 29:** Polynomial Regression  
5% TiO<sub>2</sub> Solution Sample 1

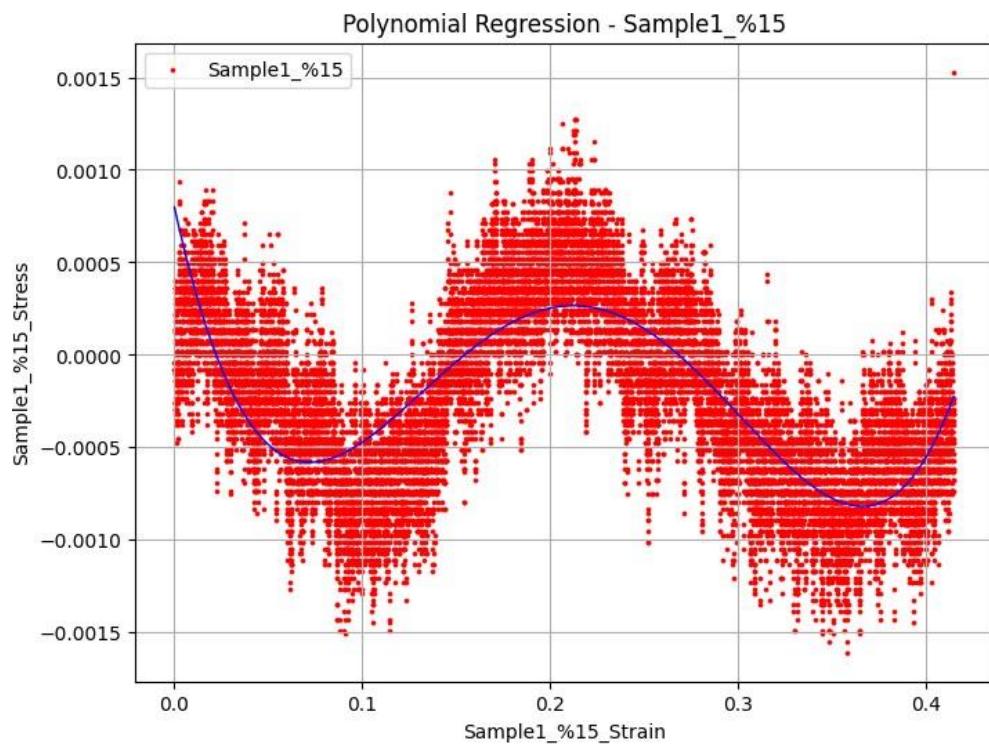


**Figure 30:** Polynomial Regression 5%  $\text{TiO}_2$  Solution Sample

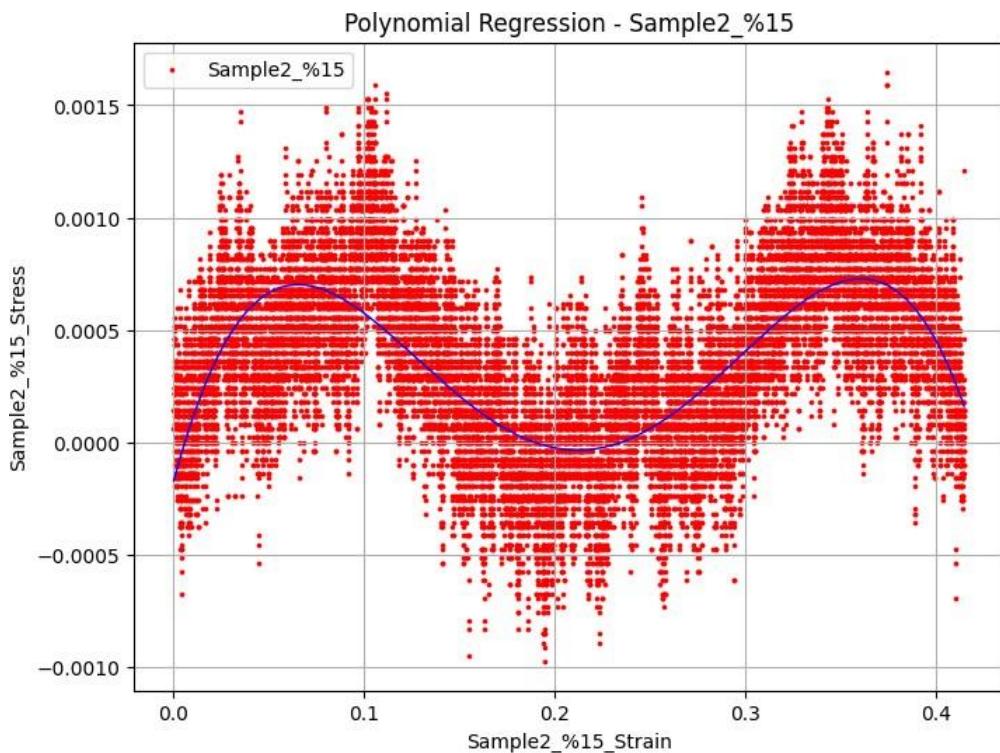
2



**Figure 31:** Polynomial Regression 5%  $\text{TiO}_2$  Solution Sample 3



**Figure 32:** Polynomial Regression 15% Talc Solution Sample 1



**Figure 33:** Polynomial Regression 15% Talc Solution Sample 2

The reason for using polynomial and linear regression for stress strain curves is to better describe and predict the mechanical properties of the material. Polynomial regression is used to capture the nonlinear behavior of the material. For example, events such as plastic flow or necking create a curved or broken shape on the stress strain graph. To obtain a curve that will fit this shape better, a polynomial equation must be used. Polynomial regression can also be used to determine important parameters of the material such as elastic modulus, yield strength, ultimate tensile strength.

Linear regression is used to capture the linear behavior of the material. For example, according to Hooke's law, there is a linear relationship between stress and strain. To obtain a line that will fit this relationship better, a linear equation must be used. Linear regression can also be used to determine an important parameter of the material such as Young Modulus. [13]

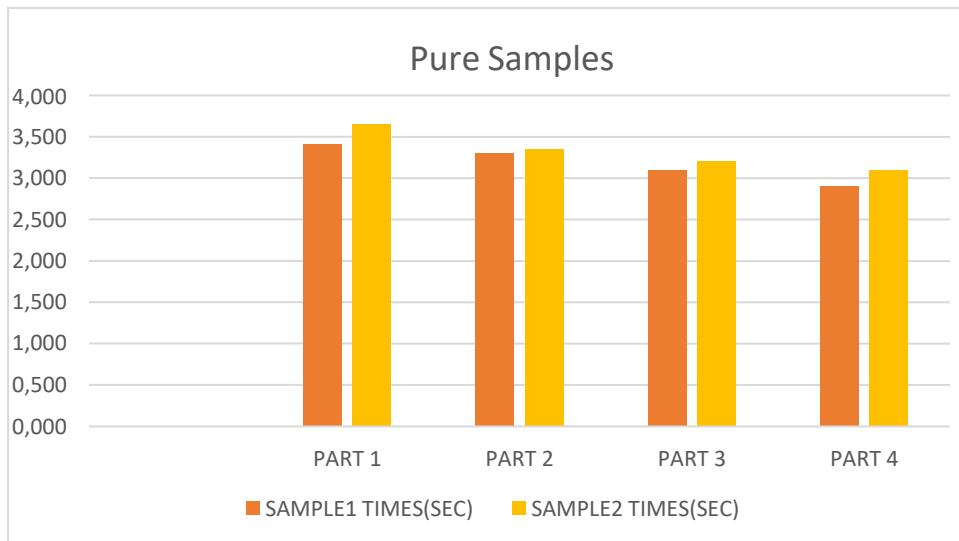
When the tables are examined, it is seen that the solution with the highest Ultimate Stress value is 5% TiO<sub>2</sub> solution. A result of approximately 0.003 MPa was observed. If the material has a high ultimate stress, the following types of movements are expected from that material:

- The material can withstand a high stress before breaking. This means that the material has a high strength.
- The material undergoes less deformation before breaking. This means that the material has less elasticity.
- The material absorbs less energy when it breaks. This means that the material has less toughness.

### 3.1.2 THERMAL EFFICIENCY TEST

PURE	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	1,23	1,30
PART 2	1,10	1,15
PART 3	1,04	1,14
PART 4	0,98	1,02

**Table 1:** Pure Thermal Efficiency

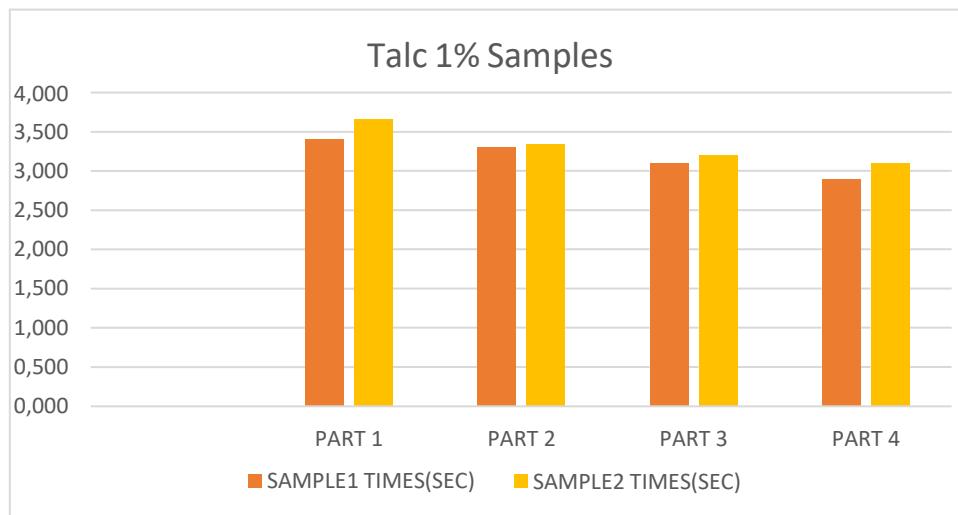


**Figure 34:** Pure Thermal Efficiency

When sample 1 and sample 2 are examined, the biggest difference is seen in part 4 as 0.17 seconds. Since these small differences between the parts are too small to be considered, we can say that samples gave similar results. The reason why we use it with two samples is to obtain reliable results pure samples and when the table is examined, the margin of error is seen as 18.88% at maximum part 4 sample 1 0.9 second part sample 2 1.07. Also, when the parts are examined sequentially, the burning time of part 1 is greater than part 2, which is greater than part 3, which is also part 4. This is true for both samples, which means that once the material starts to heat up, the shrinkage reaches the end of the material with an increasing acceleration.

TALC 1%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	1,30	1,28
PART 2	1,15	1,19
PART 3	1,00	1,10
PART 4	0,90	1,07

**Table 2:** 1% Talc Thermal Efficiency



**Figure 35:** 1% Talc Thermal Efficiency

According to the table, in part 1, if we compare pure sample 1 with talc 1% sample 1; we see that pure sample 1 shrank in 1.23 seconds, talc 1% sample 1 shrank in 1.30 seconds. According to the shrinkage times, we say that talc 1% sample 1 shrinks 0.07 seconds later than pure sample 1. Talc 1% sample 1 contracted 5.69% slower than pure sample 1. The reason for this is the difference in thermal expansion coefficients they have. As engineers, it is important for the thermal stability and durability of materials to consider the coefficient of thermal expansion in our designs. We proceeded by showing all samples in no order and the percentages of change between them.

If we compare part 1 pure sample and part 1 talc 1% on sample 2; We see that talc%1 sample 2 shrunk in 1.28 seconds and pure sample 2 in 1.30 seconds. Pure sample 2 contracted 0.02 seconds slower than 1% of talc sample 2. Based on this, it is possible to say that it shrinks 1.56% later. The fact that the difference between the two is not high provides us with the cost of the material to be selected.

Part 2 pure sample 1 shrank in 1.10 seconds as seen in the table. In the table, we see

that part 2 talc 1% sample 1 also shrunk in 1.15 seconds. According to the data, it is possible to say that part 2 talc 1% sample 1 shrinks 0.05 seconds later than part 2 pure sample 1. Proportionally, we get the result that talc 1% sample 1 shrinks 4.54% later than pure sample 1. We can say that Talc 1% sample 1 is more heat resistant than pure sample 1 in terms of part 2.

From the table, we see that pure sample 2 shrank in 1.15 seconds according to part 2, and talc 1% sample 2 shrank in 1.19 seconds according to part 2. According to the data talc 1% sample 2 shrank 0.04 seconds slower than pure sample 2. Talc 1% sample 2 shrank 3.47% slower than pure sample 2. We conclude that talc 1% sample 2 is 3.47% more durable than pure sample 2.

In part 3, the shrink time of pure sample 1 is 1.04 seconds, and in part 3, the shrink time of talc 1% sample 1 is 1.00 seconds. In part 3, we concluded that pure sample 1 contracted 0.04 seconds slower than talc 1% sample 1. It is also possible to say that in part 3, pure sample 1 shrinks 4% later than talc 1% sample 1. This result can show us that late shrinkage can be achieved without using talc, we need to evaluate and interpret all the data to be able to say this statement.

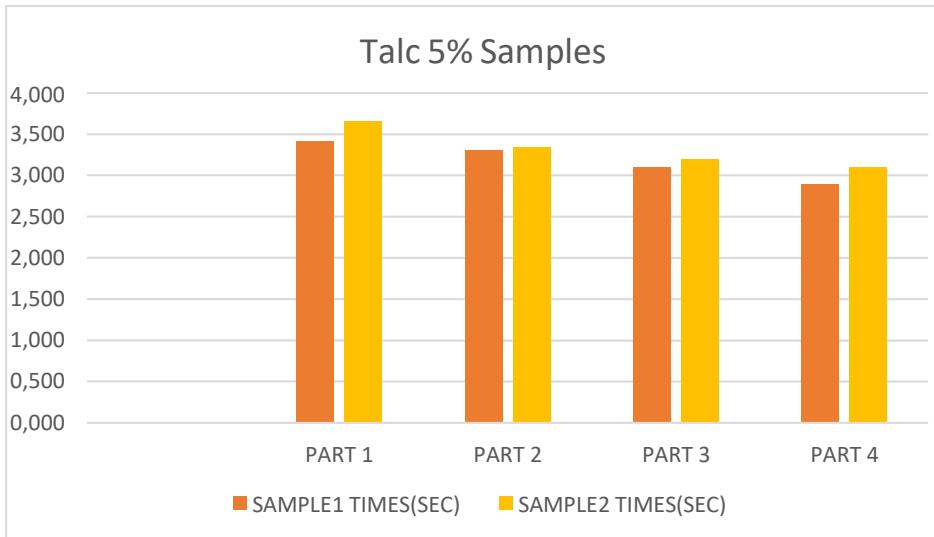
In part 3, the shrink time of pure sample 2 is 1.14 seconds and the shrink time of talc 1% sample 2 is 1.10 seconds. The shrinkage time difference between them is 0.04 seconds. In Part 3, it is possible to say that pure sample 2 shrinks 3.63% later than talc 1% sample 2. In Part 3, we see that example 1 and example 2 give close results. For Part 3, we can say that both samples of pure substances shrink later than talc 1% samples.

In part 4 we see that pure sample 1 shrank in 0.98 seconds, in part 4 we see that talc 1% sample 1 shrank in 0.90 seconds. For part 4, we can say that pure sample 1 shrinks 0.08 seconds later than talc 1% sample 1. This gives us the result that pure sample 1 shrinks 8.88% slower than talc 1% sample 1. Here, we got the result that the pure sample shrinks later than the talc 1% sample, and we continued to interpret the data to obtain healthier data.

It is possible to say that in part 4, pure sample 2 shrank in 1.02 seconds, in part 4, talc 1% sample 2 shrank in 1.07 seconds. In part 4; talc 1% sample 2 shrank 0.05 seconds slower than pure sample 2. So, in part 4, we can say that talc 1% sample 2 shrinks 4.90% slower than pure sample 2. The differences we caught between example 1 and example 2 in Part 4 can give us a better idea of the material selection to be used.

TALC 5%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	1,57	1,60
PART 2	1,48	1,54
PART 3	1,37	1,40
PART 4	1,28	1,22

**Table 3:** 5% Talc Thermal Efficiency

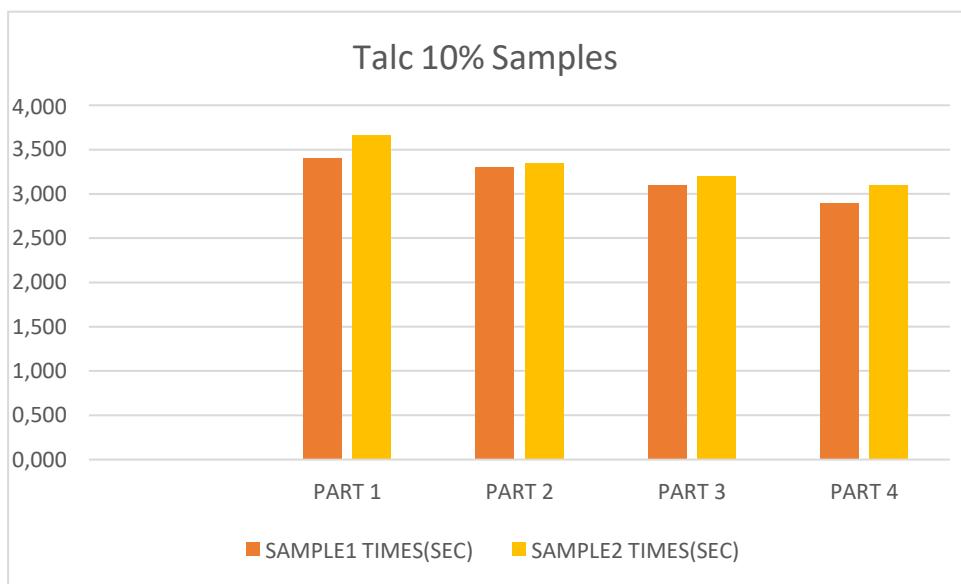


**Figure 36:** 5% Talc Thermal Efficiency

Compared to talc 5% sample 1 part 1 and talc 1% sample 1, it shrunk 20.76% later with a 5% deviation, which means it was 20.76% more resistant to heat. When the part two are compared, it is seen in the table that the 28.69% talc 5% sample burns later. With a late burning rate of 37.00%, the part lasted much longer. Based on this, we can say that the state of being affected by more heat was slowed down by accelerating towards the end of the sample for part 3. With the 33.33% slower burning rate of the talc 5% sample in part 4, the situation we mentioned for part 3 is proven to be true. Let's check the sample 2 data by examining it. Talc 5% sample 2 part 1 is 1.60 seconds, talc 1% sample 2 part 1 is shrunk in 1.28 seconds, which means talc 2 part 1 with a deviation of 5% is 25.00% more successful. Let's compare other parts. When we come to part 2, talc with a late burning rate of 29.41% proves that it is still successful with a deviation of 5%. Talc 5% sample 2 part 3 is 27.27% with rate more successful than talc 1% part 3. Towards the end we said for sample 1, we continue to obtain data that proves that the state of being affected by the heat is accelerated. Although part 4 is still more successful by 5%, the rate has decreased to 14.01%.

TALC 10%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	2,37	2,10
PART 2	2,18	1,97
PART 3	1,93	1,80
PART 4	1,68	1,59

**Table 4:** 10% Talc Thermal Efficiency

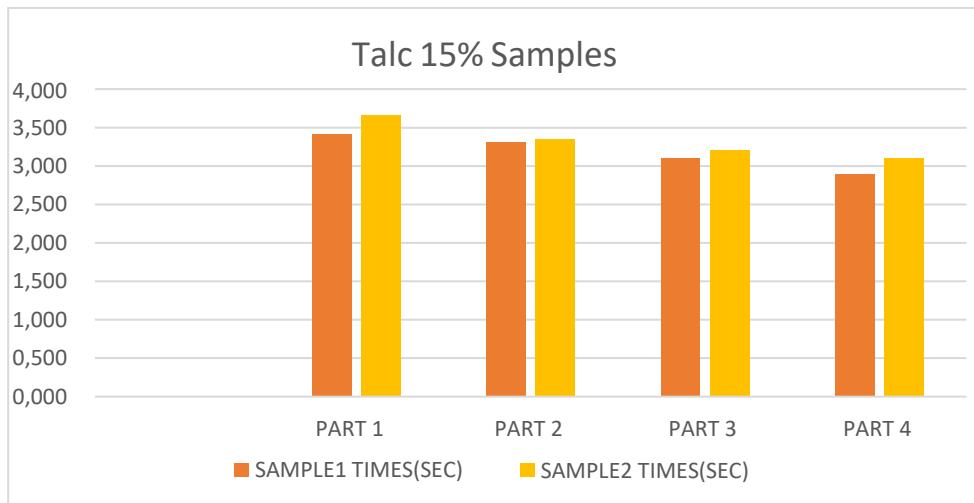


**Figure 37:** 10% Talc Thermal Efficiency

When talc 10% sample 1 talc 5% sample 1 part 1 is compared, with a 50.95% increase, 10% sample Ignition test produced the most successful part, let's see what the other part comparisons will show. Talc 10% part 2 talc 5% part 2 sample 1 47.29% more successful, repeating the success we just said. It is successful in part 3 by shrinking 40.87% later, but the compensation of the strength decreased towards the end of the sample is seen to be less successful than talc 5% in this part. It provided almost the same delay in part 4 with a rate of 40.87%. Let's make more sense how accurate our ratios are by comparing samples 2 talc 5% sample 2 part 1 1.60 seconds talc 5% sample 2 part 1 2.10 seconds shrunk. Talc 10% part 1 sample 2 seems 31.25% more successful. When compared to part 2, talc 10% is again successful with a success rate of 27.92%, but the strength that decreases towards the end of the sample is also unsuccessful here than talc 5%. In part 3, we can say that this momentum was somewhat preserved with a rate of 28.57%.

TALC 15%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	2,70	2,48
PART 2	2,50	2,36
PART 3	2,08	2,10
PART 4	1,78	1,93

**Table 5:** 15% Talc Thermal Efficiency



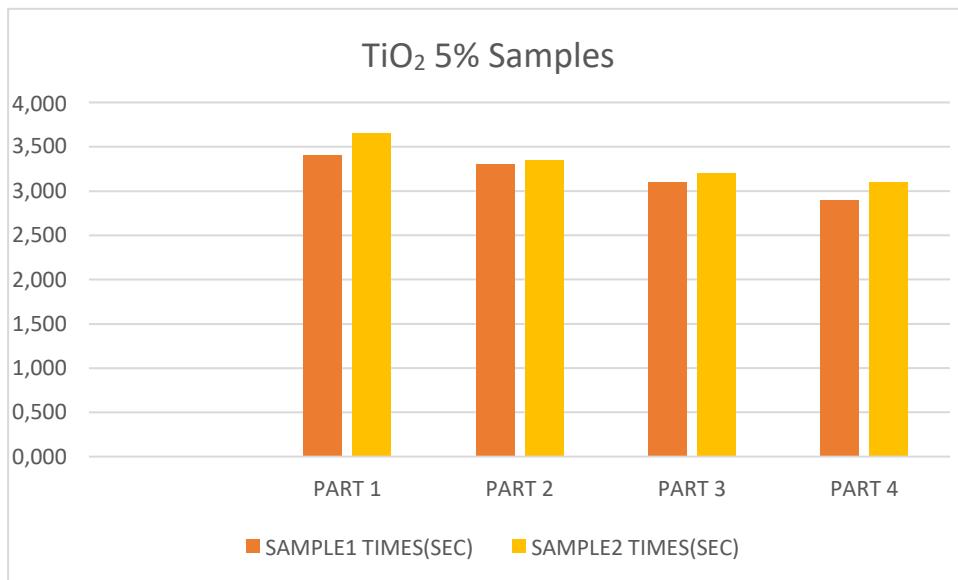
**Figure 38:** 15% Talc Thermal Efficiency

15% samples Ignition test was more unsuccessful than 10% samples. Let's examine what will happen in the thermal test by comparing talc 15% and talc 10%. In part 1, pure sample 1 shrunk in 1.23 seconds, in part 1 talc 10% sample 1 shrunk in 2.37 seconds. The shrinkage time difference between samples is 1.14 seconds. For Part 1; talc10% sample 1 shrank 92.68% slower than pure sample 1. In part 1, pure sample 2 shrunk in 1.30 seconds. In part 1, talc was 10%, and sample 2 shrunk in 2.10 seconds. The shrinkage time difference between Talc 10% sample 2 and pure sample 2 is 0.8 seconds. Talc 10% sample 2 shrank 61.53% slower than pure sample 2. In Part 2, pure sample 1 shrunk in 1.10 seconds, talc 10% sample 1 shrunk in 2.18 seconds. The shrinkage time difference between them is 1.08 seconds. Talc 10% sample 1 contracted 1.08 seconds later in part 2 than pure sample 1. Talc 10% sample 1 shrank 98.18% slower in part 3 than pure sample 1. In part 2, pure sample 2 contracted in 1.15 seconds. In part 2, talc 10% of example 2 shrunk in 1.97 seconds. In part 2, 10% of talc sample 2 shrank 0.82 seconds and 71.30% later than pure sample 2. In part 3, pure sample 1 contracted in 1.04 seconds. In part 3, talc 10% shrunk in sample 1.93 seconds. In Part 3, 10% of talc sample 1 contracted 0.89 seconds later than pure sample 1. Talc 10% sample 1 shrank 85.57% slower in part 3 than

pure sample 1. In part 3, pure sample 2 contracted in 1.14 seconds. In part 3, talc 10% of example 2 shrunk in 1.80 seconds. Here, talc 10% sample 2 shrank 0.66 seconds and 57.89% slower than pure sample 2. In part 4, pure sample 1 contracted in 0.98 seconds. In part 4, talc 10% of sample 1 shrunk in 1.68 seconds. In part 4, talc 10% of sample 1 shrank 0.7 seconds. 71.42% later than pure sample 1. In part 4, 10% talc sample 2 shrank 0.57 seconds and 55.88% later than pure sample 2.

TiO <sub>2</sub> 5%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	1,57	1,70
PART 2	1,37	1,40
PART 3	1,26	1,37
PART 4	1,10	1,20

**Table 6:** 5% TiO<sub>2</sub> Thermal Efficiency



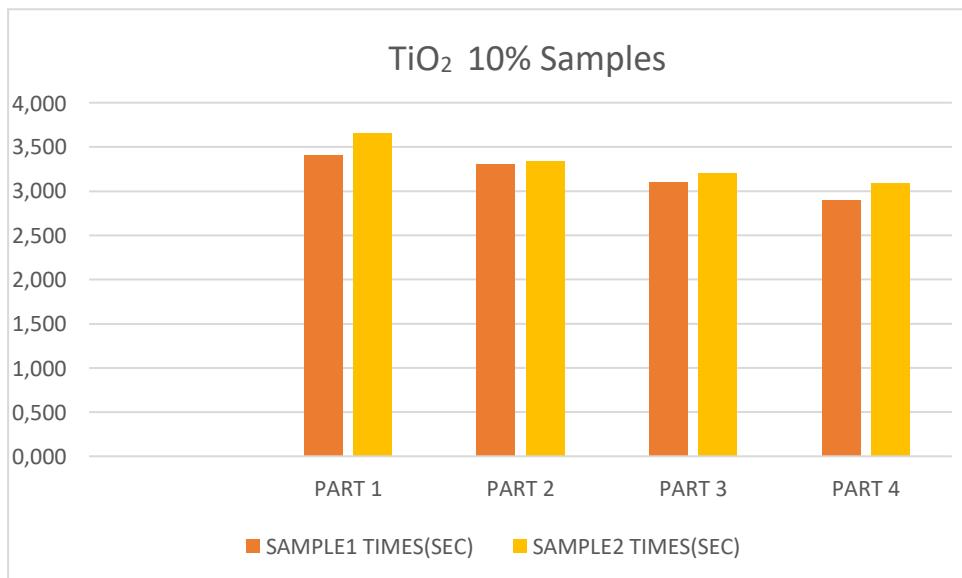
**Figure 39:** 5% TiO<sub>2</sub> Thermal Efficiency

Titanium dioxide has always been more successful than talc samples in Ignition and thermal tests. For the first time in thesis, the trace time is equal, namely titaniumdioxide. Between sample 1 titanium dioxide 5% and talc 5%, in part 2 talc 5% 8.02% shrank later. The dominance of titanium dioxide in the Ignition tests was broken here. In part 3, the talc sample proved to be successful with a rate of 8.73%. In part 4, it was seen that talc was more successful by 16.36% and 5%.Contrary to the Ignition test and our visible observations, talc was more successful than 5% sample 1 titanium dioxide 5% sample 1 out of part 1. Since

the Ignition tests are based on the point where the sample directly touches the fire, it can be said just by looking here that talc is more successful when it is not in direct contact with the heat source or is not nearby. titanium dioxide is more successful in direct exposure to heat. When samples 2 are compared, the rates are approximately the same and the situation is the same as seen in the table.

TiO <sub>2</sub> 10%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	2,54	2,63
PART 2	2,43	2,54
PART 3	2,10	2,29
PART 4	1,70	1,99

**Table 7:** 10% TiO<sub>2</sub> Thermal Efficiency

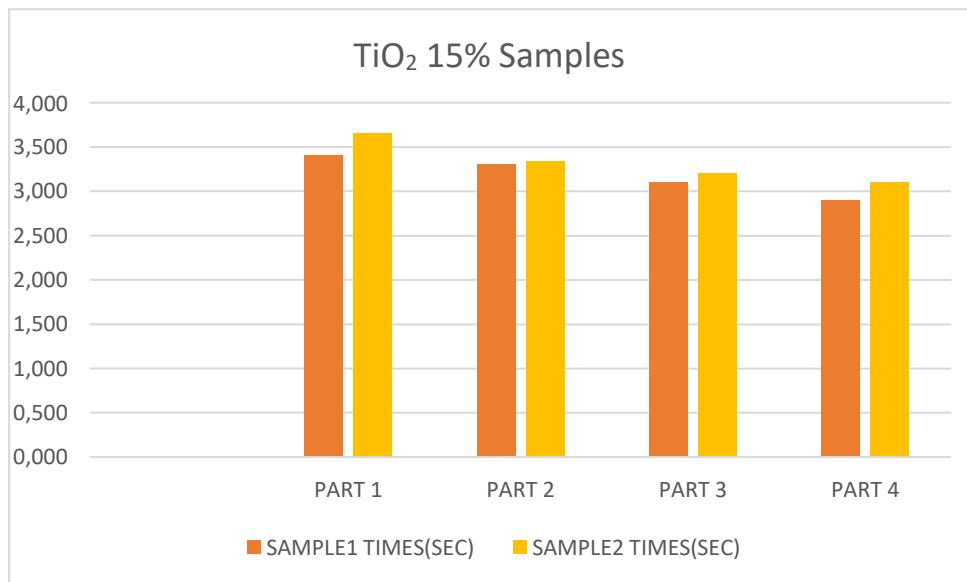


**Figure 40:** 10% TiO<sub>2</sub> Thermal Efficiency

When titanium dioxide 10% and talc 10% are compared, sample 1 talc was more successful, sample 2 titanium dioxide was more successful, that is, it shrank later. The data was not consistent and did not serve a purpose. It is seen in the table.

TiO <sub>2</sub> 15%	SAMPLE1 TIMES(SEC)	SAMPLE2 TIMES(SEC)
PART 1	3,40	3,65
PART 2	3,30	3,34
PART 3	3,10	3,20
PART 4	2,89	3,09

**Table 8:** 15% TiO<sub>2</sub> Thermal Efficiency



**Figure 41:** 15% TiO<sub>2</sub> Thermal Efficiency

### 3.1.3 IGNITION TEST

MATERIAL	SAMPLE 1	SAMPLE 2
PURE PCL	2,10	2,40
TALC 1%	2,05	2,30
TALC 5%	3,10	3,30
TALC 10%	3,40	3,49
TALC 15%	2,98	2,83
TITANIUMDIOXIDE 5%	3,98	3,89
TITANIUMDIOXIDE 10%	4,10	3,65
TITANIUMDIOXIDE 15%	3,30	3,28

**Table 9:** Ignition Test Results (in seconds)

- **PURE SAMPLES**

When we look at the sample 1 value of the pure material from the Table, we see that it was 2,10 seconds, and sample 2 burned in 2,40 seconds. There is a 14.28% margin of error between the burning time of these two samples. When the other samples are examined, it will be understood that there is a reasonable margin of error. Pure substance samples will always be the first of our reference points because this is the material we were trying to develop before adding talc and titanium dioxide. Flame retardancy of this material is the main purpose of our experiment. The main purpose is to observe the change that occurs when we add talc and titanium dioxide. Since sample 1 burned in 2,1 seconds, sample 2 in 2,4 seconds, we can say that our average value is 2,25 seconds. Our pure sample burned in an average of 2,25

seconds, these will be our reference values. Also, it should not be forgotten that the margin of error is 14.28%.

- **TALC (1%) SAMPLES**

Now our first sample, 1% talc, can be examined. By looking at the Table, 1 of talc 1% in the sample 1 burned in 2.05 seconds and sample 2 in 2.3 seconds, the change is 12.19%. The average of the two values is 2.175 seconds. The average burning time of pure samples was 2.25 seconds. The average burning time of 1% talc samples was 2.175 seconds. The average burning time of pure samples burned 3.44% later than the talc 1% samples. If we have to compare individual samples, the burning times are pure sample 1 2.1 seconds, talc 1% sample 2.05 seconds. This means that pure sample 1 burned 2.43% later than talc 1% sample 1. Pure sample 2 burn time is 2.4 seconds and talc 1% sample burn time is 2.3 seconds. This means that pure sample 2 burned 4.34% later than talc 1% sample 2. pure sample 1 burn time is 2.10 seconds, talc 1% sample 2 burn time is 2.3 seconds. This means that pure sample 1 burned 17.073% later than talc 1% sample 2. Finally, if we need to compare pure sample 2 and talc 1% sample 1, the difference is 14.58%. In addition, pure samples burned 0.075 seconds later on average than talc 1% samples. The biggest percentage change in between is between pure sample 2 and talc 1% as explained. The duration is 0.35 seconds. In the light of this information, it can be easily said that when pure samples 1% talc is added, on the contrary to late burning, earlier burning was observed, with a maximum of 17.03%. the only late burning data here is seen between pure sample 1 and talc 1% sample 2 data. Finally, when the data presented in detail is examined, it will be seen that the 1% talc mixture did not exhibit flame retardant properties, on the contrary, it lagged the pure samples, albeit a very small amount. Finally, when the data presented in detail is examined, it will be seen that the 1% talc mixture did not exhibit flame retardant properties, on the contrary, it lagged the pure samples, albeit a very small amount. We can say that talc 1% sample is ineffective.

- **TALC (5%) SAMPLES**

The next sample we will see in Table 17 is talc 5%. Since we can't get meaningful data from talc 1%, we won't make a comparison. At the same time, since pure samples exhibits better properties than talc 1% samples, comparison with talc 1% will be meaningless. So, it would make sense to compare talc 1% samples with pure samples. We will already compare all samples with pure samples because first it is our reference point. As can be seen, talc 5% sample 1 burned in 3.10 seconds. Sample 2 burned completely in 3.30 seconds. two samples average burning time is 3.20 seconds. The burning time of sample 2 is more than sample 1 with

an increase of 6.45%. Also, it burned 0.20 seconds later. The margin of error 6.45% is an acceptable margin of error. It is worth remembering that pure samples burn in an average of 2.25 seconds. When pure samples average burning time and talc 5% samples average burning time are compared, talc 5% samples burned later with an increase rate of 42.22%. At the same time, it burned 0.95 seconds later average. When these values are examined, the results are promising. The purpose of the samples, which we said before, is notto be completely fireproof, but to ensure later combustion. It can be clearly said that talc 1% samples failed, but talc 5% samples burned 42% later on average and served the purpose of the experiment. The reason why we specifically refer to percentiles comes from the pathtaken logic that we exemplified earlier. If the samples had increased in much more quantity, the time difference would have increased in quantity, although the percentage would be similar. Therefore, the percentage change is more reliable at this stage, but we see a benefit interms of comparison when looking at the time. Now let's go back to the comparison between talc 5% and pure samples. Pure samples 1 talc 5% sample 1 is 2.10 and 3.10. When compared talc 5% sample 1 burned 47.61% later. We can also observe that it burns 1 secondlater in duration. There is a significant delay. Although talc 1% was unsuccessful, we can say in the light of previous information that talc 5% showed a significant delay. Now let's look at the relationship between talc 5% sample 1 and pure sample 2. We can say that it burned 29.16% later, that is, talc burned 38.38% later on average than 5% pure samples. Now let's compare talc 5% sample 2 with pure samples. It was observed that talc 5% sample 2 burned 57.14% more in pure sample 1. It burned 37.50% later than talc 5% pure sample 2. On average, 5% sample 2 burned 47.32% later than pure samples. it is also 1.20 seconds ahead of pure sample 1 by 1.50 seconds from pure sample 2 burning was observed. In the light of these data, samples of 5% talc gave successful data.

- **TALC (10%) SAMPLES**

If we examine the sample containing 10% talc, we observe that sample 1 burned in 3.40 seconds and sample 2 burned in 3.49 seconds. The difference between these two samples is 2.64%. The average of the two values is 3.445 seconds. The average burning time of pure samples was 2.25 seconds. The average burning time of 10% talc samples is 3.445 seconds. The burning of 10% talc samples occurs 8.66% later than pure samples. When we compare pure sample 2 with 10% talc sample 2, we see that 10% talc sample 2 burns 45.41% later. When pure sample 2 is compared with 10% talc sample 1, it is concluded that 10% talc sample 1 burns 41.66% later than pure sample 2. When pure sample 1 is compared with 10% talc sample 2, the difference is found to be 57.91%. On average, 10% talc samples burned 1.195 seconds later than pure samples. The largest percentage change is observed between pure sample 1 and 10%

talc sample 2. This change lasted 1.39 seconds. In conclusion, the addition of 10% talc sample to the pure sample resulted in delayed burning up to 61.90%. The minimum burning data obtained is between 10% talc sample 1 and pure sample 2. Now, we will compare talc 10% samples with talc 5%. The aim here is how the increase in the talc5% rate that we get late burning causes a proportional increase in talc 10%. We remind you that we did not make this comparison for talc 1% because the data were not recorded. Since we obtained the late burning with talc 5%, we can make our study more detailed. Talc 10% sample 1 burned 9.67% later than talc 5% sample. We can also say that it burned 0.3 seconds later. Talc 10% sample1 burned 3.03% later than talc 5% sample 2. This is 0.1 second. It means that it burns later. On average, we can say that it burns 6.35% later. The rate has not changed much. According to the intended use, we can say that talc 5% can be used instead of talc 10% for cost. On the other hand, talc 10% sample 2 burned 12.58% later than pure sample 1 and 5.75% later than pure sample 2. This means a delay of 0.39 and 0.19 seconds, respectively. On average, we can say that it delayed by 9.16%. Talc 5% can be used instead of talc 10% with a reasonable margin of error.

- **TALC (15%) SAMPLES**

The combustion behavior of samples containing 15% talc was investigated. Sample 1, with a 15% talc content, exhibited a combustion time of 2.98 seconds, while Sample 2, also with a 15% talc content, showed a combustion time of 2.83 seconds. The small difference of 0.084 seconds between these two values indicates a subtle variation in the burning characteristics. To gain further insights, the average combustion time of the two samples was calculated to be 2.905 seconds. In comparison, the pure samples without any talc content exhibited a combustion time of 2.25 seconds. Therefore, it is evident that the presence of 15% talc in the samples resulted in a significant delay in the combustion process, amounting to a 29.11% increase in burning time compared to the pure samples. Analyzing the individual samples in detail, Sample 1 without talc ignited in 2.1 seconds, while Sample 1 with 15% talc ignited in 2.98 seconds, indicating a substantial delay of 41.90% in the burning time.

Similarly, Sample 2 without talc exhibited a combustion time of 2.40 seconds, whereas Sample 2 with 15% talc had a combustion time of 2.83 seconds, showing a delay of 17.91% compared to the pure sample. Furthermore, comparing Sample 1 without talc with Sample 2 containing 15% talc, it becomes apparent that the latter exhibited a significant delay of 34.76% in ignition time. On the other hand, when comparing Sample 2 without talc with Sample 1 containing 15% talc, the 15% talc sample ignited 20% later. The most substantial variation was observed between Sample 1 without talc and Sample 1 with 15% talc, highlighting the impact of talc content on the combustion behavior. The combustion time of the 15% talc samples exhibited an average delay of 0.655 seconds compared to the pure samples.

These findings elucidate the influence of talc content on the burning characteristics, indicating a clear trend of increased combustion time with higher talc concentrations. The detailed analysis of individual samples provides valuable insights into the effects of talc on the combustion behavior, contributing to our understanding of the fire-retardant properties of the material under investigation.

When we compare the talc 15% values with the talc 10% values, we see how important it is in the project to compare the materials themselves. Talc 15% sample 1 burned in 2.98 seconds, talc 15% sample 2 burned in 2.83 seconds. On the other hand, it burned 0.51 seconds earlier than talc 10% sample 2 and burned 17.11% earlier. It was observed that it burned 15.6% earlier on average. Talc 15% sample 2 burned 0.57 seconds earlier than talc 10% sample 1 and burned 20.14% earlier. On the other hand, it burned 0.66 seconds earlier than sample 2 and burned 23.32% earlier. This means that 10% talc samples are the most ideal among the 3 talc samples that give meaningful results according to the pure substance.

- **TITANIUMDIOXIDE(5%)**

For 5% titanium dioxide, sample 1 burned in 3.98 seconds, sample 2 containing 5% titanium dioxide burned in 3.89 seconds. The average burn time between them is 3.935 seconds. The variation between these two samples is 2.31%. It was stated that pure sample 1 burned in 2.10 seconds, while pure sample 2 burned in 2.40 seconds. The average burning time of pure samples is 2.25 seconds. The variation between them is 14.28%. It was calculated that 5% titanium dioxide sample 2 burned 85.23% slower than pure sample 1. The 5% titanium dioxide sample 2 burned 62.08% later than the pure sample 2. Titanium dioxide 5% sample 1

burned 89.52% later than pure sample 1. 5% titanium dioxide sample 1 burned 65.83% later than pure sample 2. With the 5% titanium dioxide sample, a large rate of late burning has been demonstrated. The 5% titanium dioxide samples burned on average 1.685 seconds later than the pure samples. On average, 5% titanium dioxide samples burned 1.685 seconds later than pure samples. In the case of adding 5% titanium dioxide sample to the pure sample, up to 89.52% delayed combustion was observed.

Since titanium dioxide 5% and our other material talc 5% are proportionally different as the same material, a comparison should be made. Titanium dioxide 5% sample 2 burned 0.88 seconds later than talc 5% sample 1 and burned 28.38% later. It has been observed that titanium dioxide burns 0.59 seconds, 20.60% later than 5% sample 1 talc 5% sample 2. As a result, titanium dioxide was much more successful, although it was added at the same rate. We need to check whether we get meaningful data by comparing sample 1 we use with talc 5% samples. Titanium dioxide burned 0.79 seconds later than 5% sample 1 talc 5% sample 1 and was 25.48% more successful in flame retardant. It can be said that it is 17.87% more successful by burning 0.69 seconds later than talc 5% sample 2. The success of titanium dioxide is not accidental. The average titanium dioxide 5% and talc 5% values will also prove this. Titanium dioxide 5% samples burned in an average of 3.935 seconds, and talc 5% samples burned in an average of 3.20 seconds. As a result, titanium dioxide 5% samples burned 22.96% later than talc 5% samples. As seen known titanium dioxide 5% can prefer to talc 5% because material quantity the same but success 22.96% more than the talc 5% samples.

- **TITANIUMDIOXIDE(10%) SAMPLES**

It is seen that sample 1 containing 10% titanium dioxide burned in 4.10 seconds, while sample 2 burned in 3.65 seconds. The average of the two values is 3.875 seconds. The average burning time of the pure samples was 2.25 seconds. The average burning time of 10% titanium dioxide samples is 3.875 seconds. The combustion of 10% titanium dioxide samples was 72.22% later than the pure samples. Pure sample 1 in 2.1 seconds; 10% titanium dioxide sample 1 burned in 4.10 seconds. 10% titanium dioxide sample 1 burned 95.23% slower than pure sample 1. Pure sample 2 in 2.40 seconds; 10% titanium dioxide sample 2 burned in 3.65 seconds. 10% titanium dioxide sample 2 burned 52.08% slower than pure sample 2. If we look at pure sample 1 and 10% titanium dioxide sample 2; 10% titanium dioxide sample 2 burned 73.80% slower than pure sample 1. If we look at pure sample 2 and 10% titanium dioxide sample 1; 10% titanium dioxide sample 1 burned 70.83% later than pure sample 2.

Titanium dioxide 10% samples burned on average 1.625 seconds later than pure samples.

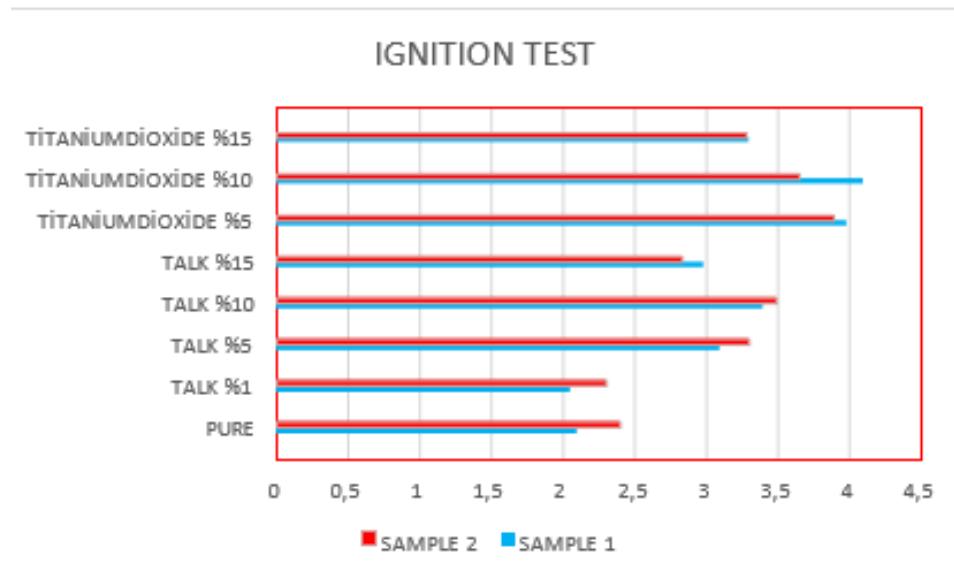
It should be noted that the samples with 10% concentration gave the best results among all samples, so the comparison to be made is important. Titanium dioxide was 20.58% more successful by burning 0.7 seconds later than 10% sample 1 talc 10% sample 1. On the other hand, sample 2 burns 0.16 seconds later than talc 5% sample 2 and is 4.58% more successful in flame retardant. Also, if we go over the average values, we can say that 5.04% titanium dioxide is more successful.

- **TITANIUMDIOXIDE(15%)**

When pure samples with 15% titanium dioxide samples are examined; 15% titanium dioxide sample 1 burned in 3.30 seconds, 15% titanium dioxide sample 2 burned in 3.28 seconds. Pure sample 1 in 2.10 seconds; pure sample 2 burned in 2.40 seconds. The average burning time of 15% titanium dioxide samples is 3.29 seconds. The burn time of pure samples was 2.25 seconds. The 15% titanium dioxide samples burned 46.22% later than the pure samples. The 15% titanium dioxide sample 1 burned 57.14% later than the pure substance sample 1. The 15% titanium dioxide sample 2 burned 36.66% later than the pure substance sample 2. 15% titanium dioxide sample 2 burned 56.19% later than the pure substance sample 1. The 15% titanium dioxide sample 1 burned 37.5% later than the pure substance sample 2. The 15% titanium dioxide samples burned on average 1.04 seconds later than the pure samples.

In addition, the decrease in talc 15% samples is also seen in titanium dioxide 15% samples. However, we see that it is beneficial to compare talc 15% and titanium 15%. As in all tests, it is seen that titanium dioxide is more successful. The data can be said to be significant since it is the third example that confirms this.

If we examine the 15% talc samples and the 15% titanium dioxide samples; The average burning time difference between them is 0.77 seconds. 15% titanium dioxide sample 1 burned 10.73% later than 15% talc sample 1. 15% titanium dioxide sample 2 burned 15.90% later than 15% total sample 2. 15% titanium dioxide sample 1 burned 16.60% later than 15% total sample 2. 15% titanium dioxide sample 2 burned 10.06% later than 15% total sample 1.



**Figure 41:** Ignition Test Results

In all comparisons, we see that sample 1 and sample 2 are close to each other. It has been shown that there is no significant difference between pure samples for 1% talc samples. A significant delay was obtained between the burning times of 5% talc samples and pure samples, and no significant difference was observed between 5% talc sample 1 and 5% talc sample 2. When 10% talc samples were compared with pure samples, a significant delay was observed in terms of burning times. It was observed that 10% talc sample 1 and 10% talc sample 2 values were very close to each other. It was observed that 15% talc samples burned later than the pure sample, but it was observed that it burned earlier compared to 10% talc samples. We see that the combustion delay difference of 5% titanium dioxide samples compared to the pure substance is very high. It is shown in the table that the burning times of 5% titanium dioxide sample 1 and 5% titanium dioxide sample 2 are very close to each.

## **4) CONCLUSION AND DISCUSSION**

In conclusion, flame retardant PCL electrospun nanofibers have a lot of potential to address fire safety issues in a range of applications. Poly(-caprolactone) (PCL) as a basic polymer has several benefits, including biocompatibility, biodegradability, and simplicity of electrospinning processes. By incorporating flame retardant chemicals into the PCL nanofibers, we were able to boost the fire resistance and decrease the flammability of the final materials in this study.

Due to its adaptability, scalability, and capacity to create materials with high surface-to-volume ratios, electrospinning is a frequently utilized method for producing nanofibers. High voltage is applied to a polymer solution or melt throughout the procedure, which creates ultrafine fibers. Numerous benefits, including high porosity, a sizable specific surface area, and an interconnected network, are provided by the PCL electrospun nanofibers' resultant nanofibrous structure, which can further improve their flame-retardant qualities.

The combined effects of the polymer matrix and the flame-retardant additives give the flame-retardant PCL electrospun nanofibers better fire resistance. The development of a protective char coating, the production of gases that suppress flames, and a decrease in heat transfer are some examples of these impacts. The char layer's presence serves as a physical barrier that stops the spread of the flames and the emission of combustible gases. The combustion process can be suppressed, and the amount of heat produced during a fire decreased by the emission of flame-inhibiting gases. Furthermore, the nanofibrous structure's decreased heat transport capabilities can restrict flame ignition and spread.

However, more research is needed to optimize the design and synthesis of flame-retardant PCL electrospun nanofibers. This includes investigating new flame-retardant additives, comprehending the mechanics of flame retardancy in nanofibrous structures, and assessing the long-term stability and endurance of these materials under various environmental circumstances. Furthermore, the scalability and cost-effectiveness of large- scale production methods should be studied to promote the actual application of flame- retardant PCL electrospun nanofibers in diverse industries.

To summarize, the development of flame retardant PCL electrospun nanofibers has a lot of promise for addressing fire safety concerns and furthering the field of fire resistant materials. This research could generate materials with increased fire resistance and offer up new options for safer and more efficient uses in a variety of sectors by combining the specific features of PCL and flame retardant chemicals with the adaptable electrospinning process.

## 5) APPENDICES

```
# Grafik 1: Sample1_%10_Stress - Sample1_%10_Strain
sample1_10_stress = df['Sample1_%10_Stress']
sample1_10_strain = df['Sample1_%10_Strain']
plt.scatter(sample1_10_strain, sample1_10_stress, s=5, linewidth=0.4)
plt.xlabel('Sample1_%10_Strain')
plt.ylabel('Sample1_%10_Stress')
plt.title('Grafik 1: Sample1_%10_Stress - Sample1_%10_Strain')
plt.grid(True)

plt.show()

# Grafik 2: Sample2_%10_Stress - Sample2_%10_Strain
sample2_10_stress = df['Sample2_%10_Stress']
sample2_10_strain = df['Sample2_%10_Strain']
plt.scatter(sample2_10_strain, sample2_10_stress, s=5, linewidth=0.4)
plt.xlabel('Sample2_%10_Strain')
plt.ylabel('Sample2_%10_Stress')
plt.title('Grafik 2: Sample2_%10_Stress - Sample2_%10_Strain')
plt.grid(True)

plt.show()

# Grafik 3: Sample3_%10_Stress - Sample3_%10_Strain
sample3_10_stress = df['Sample3_%10_Stress']
sample3_10_strain = df['Sample3_%10_Strain']
plt.scatter(sample3_10_strain, sample3_10_stress, s=5, linewidth=0.4)
plt.xlabel('Sample3_%10_Strain')
plt.ylabel('Sample3_%10_Stress')
plt.title('Grafik 3: Sample3_%10_Stress - Sample3_%10_Strain')
plt.grid(True)

plt.show()

# Grafik 4: Sample1_%5_Stress - Sample1_%5_Strain
sample1_5_stress = df['Sample1_%5_Stress']
sample1_5_strain = df['Sample1_%5_Strain']
plt.scatter(sample1_5_strain, sample1_5_stress, s=5, linewidth=0.4)
plt.xlabel('Sample1_%5_Strain')
plt.ylabel('Sample1_%5_Stress')
plt.title('Grafik 4: Sample1_%5_Stress - Sample1_%5_Strain')
plt.grid(True)

plt.show()

# Grafik 5: Sample2_%5_Stress - Sample2_%5_Strain
sample2_5_stress = df['Sample2_%5_Stress']
sample2_5_strain = df['Sample2_%5_Strain']
plt.scatter(sample2_5_strain, sample2_5_stress, s=5, linewidth=0.4)
plt.xlabel('Sample2_%5_Strain')
plt.ylabel('Sample2_%5_Stress')
plt.title('Grafik 5: Sample2_%5_Stress - Sample2_%5_Strain')
```

```

plt.grid(True)
plt.show()

# Grafik 6: Sample3_%5_Stress - Sample3_%5_Strain
sample3_5_stress = df['Sample3_%5_Stress']
sample3_5_strain = df['Sample3_%5_Strain']
plt.scatter(sample3_5_strain, sample3_5_stress, s=5, linewidth=0.4)
plt.xlabel('Sample3_%5_Strain')
plt.ylabel('Sample3_%5_Stress')
plt.title('Grafik 6: Sample3_%5_Stress - Sample3_%5_Strain')
plt.grid(True)
plt.show()

# Grafik 7: Sample1_%15_Stress - Sample1_%15_Strain
sample1_15_stress = df['Sample1_%15_Stress']
sample1_15_strain = df['Sample1_%15_Strain']
plt.scatter(sample1_15_strain, sample1_15_stress, s=5, linewidth=0.4)
plt.xlabel('Sample1_%15_Strain')
plt.ylabel('Sample1_%15_Stress')
plt.title('Grafik 7: Sample1_%15_Stress - Sample1_%15_Strain')
plt.grid(True)
plt.show()

# Grafik 8: Sample2_%15_Stress - Sample2_%15_Strain
sample2_15_stress = df['Sample2_%15_Stress']
sample2_15_strain = df['Sample2_%15_Strain']
plt.scatter(sample2_15_strain, sample2_15_stress, s=5, linewidth=0.4)
plt.xlabel('Sample2_%15_Strain')
plt.ylabel('Sample2_%15_Stress')
plt.title('Grafik 8: Sample2_%15_Stress - Sample2_%15_Strain')
plt.grid(True)
plt.show()

# Grafik 1: İlk üç sample
plt.figure(figsize=(8, 6))
plt.scatter(df['Sample1_%10_Strain'], df['Sample1_%10_Stress'], s=5,
            linewidth=0.4, color='red', label='Sample1')
plt.scatter(df['Sample2_%10_Strain'], df['Sample2_%10_Stress'], s=5,
            linewidth=0.4, color='green', label='Sample2')
plt.scatter(df['Sample3_%10_Strain'], df['Sample3_%10_Stress'], s=5,
            linewidth=0.4, color='blue', label='Sample3')
plt.xlabel('Strain')
plt.ylabel('Stress')
plt.title('Titanyum dioksit %10')
plt.grid(True)
plt.legend()

```

```

# Grafik 2: Sonraki üç sample
plt.figure(figsize=(8, 6))
plt.scatter(df['Sample1_%5_Strain'], df['Sample1_%5_Stress'], s=5,
            linewidth=0.4, color='red', label='Sample1')
plt.scatter(df['Sample2_%5_Strain'], df['Sample2_%5_Stress'], s=5,
            linewidth=0.4, color='green', label='Sample2')
plt.scatter(df['Sample3_%5_Strain'], df['Sample3_%5_Stress'], s=5,
            linewidth=0.4, color='blue', label='Sample3')
plt.xlabel('Strain')
plt.ylabel('Stress')
plt.title('Titanyum dioksit %5')
plt.grid(True)
plt.legend()

# Grafik 3: Son iki sample
plt.figure(figsize=(8, 6))
plt.scatter(df['Sample1_%15_Strain'], df['Sample1_%15_Stress'], s=5,
            linewidth=0.4, color='red', label='Sample1')
plt.scatter(df['Sample2_%15_Strain'], df['Sample2_%15_Stress'], s=5,
            linewidth=0.4, color='green', label='Sample2')
plt.xlabel('Strain')
plt.ylabel('Stress')
plt.title('Talc %15')
plt.grid(True)
plt.legend()

# Grafikleri göster
plt.show()

```

```

import pandas as pd
import matplotlib.pyplot as plt
from sklearn.linear_model import LinearRegression

# Sample sütunlarını groplara ayır
samples = [
    {'name': 'Sample1_%10', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample2_%10', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample3_%10', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample1_%5', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample2_%5', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample3_%5', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample1_%15', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample2_%15', 'x_column': 'Strain', 'y_column': 'Stress'}
]

```

```

# Her bir sample için lineer regresyon ve grafik çizimi
for sample in samples:
    # Verileri al
    X = df[sample['name']+ '_' +sample['x_column']].values.reshape(-1, 1)
    y = df[sample['name']+ '_' +sample['y_column']].values.reshape(-1, 1)

    # Lineer regresyon modelini oluştur ve eğit
    regressor = LinearRegression()
    regressor.fit(X, y)

    # Grafik çizimi
    plt.figure(figsize=(8, 6))
    plt.scatter(X, y, s=5, linewidth=0.4, color='red', label=sample['name'])
    plt.plot(X, regressor.predict(X), color='blue', linewidth=1)
    plt.xlabel(sample['name']+ '_' +sample['x_column'])
    plt.ylabel(sample['name']+ '_' +sample['y_column'])
    plt.title(f'Linear Regression - {sample["name"]}')
    plt.grid(True)
    plt.legend()
    plt.show()

```

```

import pandas as pd
import matplotlib.pyplot as plt
from sklearn.preprocessing import PolynomialFeatures

# Sample sütunlarını gruptara ayır
samples = [
    {'name': 'Sample1_%10', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample2_%10', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample3_%10', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample1_%5', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample2_%5', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample3_%5', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample1_%15', 'x_column': 'Strain', 'y_column': 'Stress'},
    {'name': 'Sample2_%15', 'x_column': 'Strain', 'y_column': 'Stress'}
]

# Her bir sample için polinom regresyon ve grafik çizimi
for sample in samples:
    # Verileri al
    X = df[sample['name']+ '_' +sample['x_column']].values.reshape(-1, 1)
    y = df[sample['name']+ '_' +sample['y_column']].values.reshape(-1, 1)

    # Polinom özelliklerini oluştur
    polynomial_features = PolynomialFeatures(degree=4) # İstediğiniz dereceyi belirleyebilirsiniz
    X_poly = polynomial_features.fit_transform(X)

```

```
# Polinom regresyon modelini oluştur ve eğit
regressor = LinearRegression()
regressor.fit(X_poly, y)

# Grafik çizimi
plt.figure(figsize=(8, 6))
plt.scatter(X, y, s=5, linewidth=0.4, color='red', label=sample['name'])
plt.plot(X, regressor.predict(X_poly), color='blue', linewidth=1)
plt.xlabel(sample['name']+'_'+sample['x_column'])
plt.ylabel(sample['name']+'_'+sample['y_column'])
plt.title(f'Polynomial Regression - {sample["name"]}')
plt.grid(True)
plt.legend()
plt.show()
```

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