FABRICATION AND CHARACTERISATION OF PLASTICS WITH MAGNETIC AND ELECTRICAL PROPERTIES

By

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ABSTRACT

Several studies have reported the use of conductive Poly Lactic Acid (PLA) plastics in various industries; however, there are limited research studies on magnetic PLA. In this research, PLA with electrical and magnetic properties are fabricated. In addition, a neural network is also developed to estimate the parameters required to fabricate a given composite PLA with unique electrical and magnetic properties. Carbon and iron particles are used as electrical and magnetic reinforcements during the fabrication of the composite PLA plastics. The properties of the composites are characterized using an impedance analyzer. The characterization data from the experiment show that the conductivity and magnetic properties of the pure PLA is improved by combining it with carbon and iron particles. Furthermore, it is observed that, at a certain weight percent of carbon, the conductivity threshold is reached. Therefore, any further increase in carbon does not significantly improve the conductivity of the composite PLA. In addition, the developed neural network is able to estimate the parameters required to fabricate a given composite PLA with acceptable accuracy. Between 80 - 90% of the neural network predicted output parameters are within an error of +/- 10% of the actual experimental values.

ACKNOWLEDGMENT

Firstly, I would like to show my appreciation to my advisor, Dr. Olanrewaju A. Ojo for his guidance throughout my postgraduate study. I also extend my sincere gratitude towards my co-advisor Dr. Nan Wu for the continual support he provided through the course of my research. Further appreciation goes to Mike Boskwick, Trevor Smith, Robin Raju and Dr. Gregory Bridges for their Technical support through the course of this research. Finally, I would like to express the highest appreciation to my loving parents, Mr. and Mrs. Oyelaja for their extensive guidance throughout the course of my education.

DEDICATION

I dedicate this thesis to my parents, Mr. and Mrs. Oyelaja.

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ACRONYM AND GLOSSARY

AC – Alternating Current

DC – Direct Current

ANN – Artificial Neural Network

Aspect ratio $= \frac{Length}{diameter}$

CB – Carbon Black

CF - Carbon Fiber

CORR = Correlation Coefficient

High resistivity = Low conductivity and vice versa

RMSE = Root Mean Squared Error

PLA - Poly Lactic Acid

CHAPTER 1

INTRODUCTION

The rise in technological advancements has made conductive and magnetic materials increasingly important. Consequently, there has been a surge in the use of inherently conductive and magnetic materials such as metals. As a result of this, the cost of metals is continually increasing. Therefore, it is imperative to find alternative materials to replace metals in some applications that require conductive and magnetic properties. This research proposes the use of plastic as a substitute. A.R. Blythe [1] studied the nature of various pure polymers and discovered that polymers have high electrical resistivity typically between 10^{12} - 10^{17} ohm-meter. Kausar [2] proposed that the addition of carbon black in polymers improves conductive properties. Other researchers [3][1] have also looked at other forms of carbon such as carbon fiber as it is a conductive particle. In addition, Protoplast [4] developed a magnetic Poly Lactic Acid (PLA) material. Protoplast claims that the thermal conductivity of the base PLA was improved; however the fabricated PLA is nonconductive. Limited work has been performed relating to the development of both conductive and magnetic plastic. As plastic is majorly classed as an inherent insulator and non-magnetic material. It is essential to study the nature of plastic and redesign it to include these required electrical and magnetic properties if they are to be used as substitute for metals in conductive and magnetic applications. This presents the first objective of this research as in section 1.2 (Objective 1)

1.1 Background

In this thesis, PLA is used as the base material for fabricating the conductive and magnetic plastic. PLA's biodegradability, low cost, and relatively high tensile and flexural strength set it apart from other plastics. To improve the conductivity of the pure PLA, carbon particles are used as conductive reinforcements. Carbon particles are typically known for their high conductivity[5]–[8], and although they may not be as conductive as metals, they possess a certain useable level of conductivity. The conductivity of carbon particles varies based on the type of carbon. Dana Pantea et.al [9][10] reported that the structure and specific surface area are considered the two most important properties of carbon. The common types of carbon particles used in conductive applications include carbon black and carbon fiber. Carbon black is made up of aggregated spherical particles. The structure of the carbon black particle influences its level of aggregation [15]. The specific surface area of the particle is inversely proportional to the diameter of the particle [15][11]. In addition, the electrical conductivity of carbon black is influenced by its structure and specific surface area. Due to these properties, conductive carbon particles such as carbon black and carbon fiber have garnered distinctive attention in the conductive polymer composite industry. Furthermore, magnetic particles in a polymer help improve its relative magnetic permeability [12]–[14]. Magnetic permeability is the level of magnetization in a material as a reaction to an external magnetic field [15]. As initially stated, limited research studies have been done relating to conductive plastics with magnetic properties. Eksi's [16] developed a plastic–metal composite which serves as a lightweight support member in vehicles. The study performed by Eksi only focuses on improving the mechanical properties of the base plastic with metal reinforcement. Magnetic composites could be useful in various applications such as fabricating barcode for magnetic encoders, magnetic storage media and electromagnetic shielding. This research also focuses on the use of artificial neural network (ANN) to estimate the quantity of reinforcing particle required to achieve a certain level of electrical and magnetic property. ANN is a system that helps with the prediction and pattern recognitions problems. ANN models usually have a few simple and nonlinear functional links known as 'neurons'. Neurons are connected to each other by parallel weights. Individual weights represent the magnitude of the connections between the units in the neural network. The learning ability of ANN is crucial; this is because the learning process influences the accuracy of the prediction. Chhachhiya [17] successfully implemented MATLAB ANN in the classification of glass. In Chhachhiya work, 108 instances of glass are used. This network proved useful in applications where the type of glass that was not initially exposed to the network is determined using the previously developed network relationship. Al Shamisi [18] also used MATLAB ANN to predict global solar radiation in regions without solar radiation measuring equipment using metrological data. ANN presents a capability to solve problems involving nonlinearities. ANN models are currently exploited mainly in engineering for applications in pattern-recognition. They are also used in dealing with input-output mapping, and adaptive prediction. This presents the second objective of this research as in section 1.2 (Objective 2).

1.2 Research Objectives

1. Fabricate and characterize composite PLA plastics with electrical and magnetic properties.
2. Develop an artificial neural network capable of predicting the fabrication parameters required to produce a composite with unique electrical and magnetic properties.

1.3 Major Findings

To achieve the research objectives, PLA based composite plastics with both electrical and magnetic properties were fabricated using carbon and iron particles. The plastics were characterized at a frequency of 100kHz. The experimental/characterization results show that the conductivity of the pure PLA is improved by smaller sized carbon black particles. A number of research studies claim that 40wt% of conductive reinforcement and above is required to shift a plastic from a nonconductive to conductive domain [17] [105]. In this research, with as low as 5wt% of 1 micrometer carbon black, a reasonably low level of resistivity (high conductivity) of approximately 1 ohm-meter is achieved. There is also a direct link between the relative permeability of the composite PLA to the quantity of iron particle used for fabrication. Furthermore, the dielectric analysis of the fabricated composite PLA indicates that above a certain weight percent of carbon, the composite does not behave like a dielectric due to its high conductivity. Finally, an artificial neural network capable of predicting what parameters are required to fabricate a composite with unique electrical and magnetic properties has been developed in this study. The network is able to estimate the composition of a given composite PLA with acceptable accuracy. Between 80 - 90% of the neural network predicted output parameters are within an error of +/- 10% of the actual experimental values.

1.4 Thesis Structure

Chapter 1: This section provides the background information, the purpose of this research, the objectives of this research and major findings.

Chapter 2: This section presents the literature review of the uses, limitations of PLA as well as current improvements to the properties of PLA.

Chapter 3: This section outlines the materials, fabrication procedures, the experimental methodology and neural network modeling adopted in this research.

Chapter 4: This section outlines the analysis of the results obtained from the experiment. Discussions of the fabricated composite plastic, as well as the electrical and magnetic characterization of the plastic samples, are presented in this chapter.

Chapter 5: This section outlines the design of the artificial neural network capable of estimating the composition of a given composite PLA.

Chapter 6: This section summarizes the research findings and recommendations of further work/study.

CHAPTER 2

LITERATURE REVIEW

2.1 Application and Limitations of PLA

The use of PLA plastic has grown over the years; in general, most plastics are made from either petroleum or natural materials like cellulose as it contains carbon. The long repeating chain of carbon atoms in plastics make them strong, light in weight and flexible [19][20]–[22]. Over the years, the properties of plastics have been modified and tailored to meet specific needs. Plastics have become a pivotal part of the society due to their versatile properties thus making them exceptionally useful in various applications [20]. Overall, plastics provide unmatched versatility to design engineering over an extensive range of applications. This is because they possess advantages such as exceptional strength to weight ratio, cost and resistance to corrosion compared with alternative materials. This makes plastics a very resource efficient material [23][24].

2.1.1 Applications of PLA

Due to the nature of PLA, it is used in a number of applications. Its long shelf life makes it useful in numerous packaging applications. When PLA is disposed properly, it hydrolyzes into harmless natural products. Therefore, PLA provides a means to solve the issue with ridding the environment of harmful plastics. Since the early 90's, about 17.3 billion pounds of disposable plastics have been sold in USA annually [25]. A substantial fraction of this is disposed as waste. This waste becomes a threat to marine life. Death estimate of sea life is about 100,000 sea animals and 1 to 2 million marine birds per year [25]. In addition, compared to other plastics such as ABS and HDPE with 500-1000 years degradation life, PLA has a degradation duration of as short as 6 months [26]. In food packaging applications, PLA is used because very limited migration of chemicals occurs from PLA into the packaged food. The only substance that could migrate is lactic acid. Researchers estimate that the approximate lactic acid intake from PLA is 700 times less that the daily lactic acid intake for infants. For this reason, PLA is said to be safe for manufacturing food articles [GRAS]. In agriculture, PLA is used as mulch to prevent sunlight reaching the soil. This is done to prevent the growth of unwanted plants. In 3D prototyping, PLA is used due to its low melting temperature, this allows it to be easily processed by majority of 3D printers. PLA plastics used in prototyping have been modified over the years to improve properties such as conductivity and strength. Due to the durability of PLA, it is used in numerous long life consumer goods. 'Fujistu' computer housing and keys are made from PLA, 'Toyota' vehicle wheel cover is also made from PLA. Various fabric of clothing apparel is also made form PLA. Unlike some other types of plastics, the manufacture of PLA does not contribute immensely to global warming, as it is not made from petroleum byproducts.

2.1.2 Limitations of PLA

The application and uses of PLA are limited due to a number of reasons, this includes, its weak thermal stability, relatively low toughness and ductility [24]. PLA has a relatively low glass transition temperature of about 55 Celsius making it unsuitable for high temperature purposes. Modifiers are usually added to PLA to improve its stiffness at elevated temperatures. The deformation and softening of the PLA materials could occur in hot weather. As PLA's attractive properties outweigh its limitations in some applications, therefore, a study into the modification of the plastic to improve its property is essential. Protoplast company [4] developed conductive plastics using carbon particles as conductive reinforcement. Suryanegara et al [27] also successfully worked on the enhancement of the thermal and mechanical properties of PLA by reinforcing it with micro fibrillated cellulose.

2.1.2.1 Improvements to PLA Properties

Other developments have also been made to improve the properties of PLA, this includes, improvements to PLA's electrical and thermal conductivity and mechanical properties. Lin et al [28] reportedly studied the enhancement of the thermal conductivity of PLA using fillers. Lin incorporated tannic acid (TA) functionalized graphite nano-platelet (GNP) into PLA matrix. This reinforcement improved the thermal conductivity of the plastic. [28]. Kamthai et al [29] also studied the effect of isosorbide diesters (plasticizer) on the thermal and mechanical properties of PLA and carboxymethyl cellulose composite (PLA/CMCB). Kamthai discovered that an increase in the concentration of the plasticizer resulted in a decrease in melting, glass and decomposition temperatures of the PLA composite. However, the elongation properties of PLA/CMCB composites was improved [29]. Likewise, Yu [30] reported that using acetyl tributyl citrate (ATBC) as a plasticizer in PLA in combination with carbon black improves the electrical conductivity of the PLA. With the increase in the carbon black contents, both the storage modulus, and glass-transition temperature increased. Zhang D et al [31] also researched and characterized the effects of carbon nanotubes in PLA. In Zhang's work, the conductivity of the PLA composite improved with the increase in the carbon nanotube loading. The 'percolation threshold' was observed at 14 weight percent of carbon nanotube loading with a resistivity value of 10 ohm-cm in Zhang's work. Percolation threshold is further explained in as *section 4.2.1.* Numerous conductive polymer composites display percolation traits [32]. The usual curve of conductivity versus quantity of filler is 'S' shaped. This reveals a slim filler content range in which a marginal increase in the quantity of the filler results in an extreme increase in the observed conductivity. This is illustrated in Figure 2.1. There are numerous means to reduce the percolation threshold of conductive filler in a polymeric matrix. These include, the use of additives such as plasticizers, optimizing fabrication parameters, as well as altering the morphology (the size and structure) of the reinforcement. A beneficial property of plastic is that it does not oxidize. Metals corrode in certain environments; this limits the extent of usage and applications. Although, conductive composites plastics could replace metals in some applications, the use of conductive PLA is limited by its conductivity magnitude. According to literature, it is possible to fabricate PLA with high conductivity. Various research studies have experimented with the use of composite plastics in applications such as 'electromagnetic shielding' [33], [34]. Electromagnetic shielding is the process of surrounding electronic devices and cables with conductive or magnetic materials to protect them against inward or outward radiations of electromagnetic frequencies [35][36], [37]. Electromagnetic shielding is used mainly to inhibit electromagnetic interference of sensitive electronic devices [44]. Shields are frequently used to guard a part of a device from affecting another component inside the same device. In mobile phones, electromagnetic shielding protects electronics from its transmitter and receiver. Electromagnetic shielding in smartphones also decreases the amount of radio frequency energy that could be absorbed by the smartphone user. Electromagnetic shielding [37] is generally used in situations where a device with sensitive information needs to be protected. Therefore, to improve the security of air-gapped systems, electromagnetic shielding is recommended. Prior to the development of electromagnetic shielding, physical isolation with lack of external connectivity had been considered suitable for system security. However, proof-of-concept attacks show that exploiting the electromagnetic radiation of a system can facilitate intrusion. Conductive plastics are also used in antistatic applications to prevent the buildup of static electric charge [35] [36]. The most common applications of antistatic include manufacturing of rubber work surfaces, bags and containers and trays. In authentication applications, the electromagnetic signature (electrical and magnetic property) of a material is used to for verification purposes. This includes the verification of the authenticity of coins and currency notes [38]. In detection applications, the electromagnetic signature of surgical devices could be used tracked its location in a patient's body, this makes it useful in medical application [39][40]

2.2 Factors that Affect Conductivity of a Composite Material

The magnitude of conductivity of a composite plastic is influenced by a number of factors. These include the size of the reinforcing conductive particle, type/nature of the conductive particle, and the quantity of the reinforcing particles in the plastic matrix. These factors are usually combined optimally to achieve the required level of conductivity in the composite. Various studies on the effect of the size of conductive particle on the conductivity of composites have been performed. In these studies, it is revealed that smaller reinforcing particles have relatively higher surface area. Therefore, at the same content ratio, smaller sized particles produce more conductive composites compared to large sized carbon particles [2], [6], [41]. Kazuya [41] investigated the effect of graphite particles size on the electrical conductivity of graphite and low-density polyethylene composites. As seen in Figure 2.3, Kazuya observed that the smaller sized graphite particles produced a more conductive composite compared to the larger sized particles at the same volume fraction. The level of conductivity of a composite plastic is also affected by the nature of the conductive particle in its matrix. Due to the chemical composition and shape of the reinforcing conductive particle in the composite, the magnitude of conductivity of the composite is affected. Carbon and metal particles are commonly used to enhance the electrical conductivity of a material. Carbon particles are usually spherical or fiber shaped. Spherical carbon particles are known as 'carbon black' while fiber shaped carbon particles are called 'carbon fibers'. The conductivity of carbon black and carbon fiber has been studied by numerous academia and claims that carbon fiber is inherently more conductive compared to carbon black has been made. Although, the structure of carbon fiber influences its high conductivity, larger carbon fiber particles have poor surface area therefore the resulting conductivity of composites fabricated with carbon fiber could be lower due to the added effects of surface area and the structure of the fibers [5].

Fabrication process could also affect the level of conductivity of a composite. Composite are generally mixed sufficiently in order to avoid a highly inhomogeneous mixture. Although a perfectly homogeneous mix may be difficult to achieve, it is essential that the conductive particles be evenly distributed. Pinto et al [42] studied composites fabricated with nylon powder and carbon black by using compression molding and melt blend process. Pinto's observed that percolation of the carbon black in the composite was at 9 weight percent. This is a lower threshold compared to 25 weight percent usually needed in a melt blend process. Pinto asserted that fabricating the composite using compression molding improves the conductivity [42]. As illustrated in Figure 2.4 particles are compressed at sufficiently high pressure causing plastic deformation and bonding to occur. The porosity of the composite is also reduced; this results in a higher conductivity.

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R. B. Rosner, “ESD An,” pp. 121–131.

Figure 2.1: Percolation threshold of conductive particle [43]

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R. B. Rosner, “ESD An,” pp. 121–131.

Figure 2.2: Particle network structure [43]

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K. Nagata, H. Iwabuki, and H. Nigo, "Effect of particle size of graphites on electrical conductivity of graphite/polymer composite," Compos. Interfaces, vol. 6, no. 5, pp. 483–495, Jan. 1998.

Figure 2.3: Conductivity of graphite/LDPE vs. volume fraction at different sizes [41]

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Figure 2.4: Compression molding/Tableting process [44]

2.3 Factors that affect the Magnetic Properties of a Composite Material

The magnetic property of a composite is influenced by the nature of the reinforcement in the composite matrix. This is usually a direct result of the magnetic permeability of the reinforcing particle in the composite. Ferromagnetic particles such as iron and nickel based powders are some of the most commonly used magnetic reinforcements. Magnetic composites are appealing to industries that manufacture items that required some form of magnetism to function, for example, the makers of small motors and actuators [45]. Magnetic composite materials have garnered more attention in recent years because they are also detectable through non-contact probing. Although the magnetic composite industry is growing, there are limited studies on PLA with magnetic property. Protoplast [4], a company that manufactures 3D printing filaments, developed a commercially available magnetic, however, non conductive PLA filament. Protoplast claims that the filaments provide the benefits of magnetism to 3D prints.

2.4 Electrical and Magnetic Material Characterization

Resistance, inductance and capacitance are some of the properties analyzed during the material characterization of conductive and magnetic materials. These properties are a function of resistivity, relative permeability, and dielectric constant respectively. Firstly, resistance is an electrical measurement that states how a material inhibits the flow of electric current through it. The unit of resistance is 'ohms'. The resistance of a material is the product of the resistivity and length divided by its area [46]. Secondly, inductance is measured in 'Henry' [47]. When current flows through an inductor or coil of wire, an electromagnetic (EM) field is created. An increase in the current enlarges the electromagnetic field and a reduction in current decreases it. This alternating electromagnetic field results in an induced voltage across the coil in a direction to oppose the changing current. The resulting opposition is known is the inductance.

The measured magnitude of inductance is based on the magnetic permeability of the material at the center of the coil. Finally, capacitance is the electrical quantity of a capacitor. It defines the measure of the ability of a capacitor to store electrical charge. Capacitance is measured in 'Farad' [48]. The magnitude of charge stored by a capacitor is a function of the dielectric constant of the material in the capacitor as seen in Figure 2.5. Alternating (AC) or direct current (DC) can be used for analyzing the properties of materials, however, DC only allows measurement of resistance. The capacitance and inductance of a material can only be measured using AC measurement. Methods such as a four-point probe analysis and impedance analyzer are commonly used for characterizing electrical and magnetic properties of materials. A four-point probe is used to measure the resistance of a material with DC. This technique uses 4 probes to analyze a material's resistivity. A direct current is passed between the outer probes, and a voltmeter is used to measure the potential difference between the inner probes [49]–[51]. From the measured resistance, the resistivity of the material is computed from the physical size of the sample, the current, and voltage measurements [50]. The devices used for this measurement include: a four-point collinear probe, direct current source and a voltmeter as shown in Figure 2.6. As explained, this method only analyzes the resistivity of the material. Other properties such as inductance and capacitance could be measured using more a complex AC measurement method. An impedance analyzer provides more capabilities compared to a four-point probe. It measures the inductance, capacitance and resistance of a material over a range of frequencies[13], [52]–[56]. This device uses alternating current for its measurements. An important feature of the impedance analyzer is that it allows for the study of the electrical and magnetic properties of materials at different AC frequencies.

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https://www.electronics-tutorials.ws/capacitor/cap_1.html.

Figure 2.5: Capacitor [48]

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https://www.evaluationengineering.com/materials-characterization-resistivitymeasurements-using-four-point-collinear-probe.

Figure 2.6: Resistivity measurement using four-point probe [49]

2.5 Data Estimation and Prediction

Data estimation is a branch of statistical analysis that involves approximating values of parameters based on empirical data with arbitrary dependencies. In data estimation, the relationship between data consisting of multiple variables is known as regression analysis. Regression analysis involves identifying the factors that have influence on a certain variable. Some data estimation techniques include, design of experiment and artificial neural network.

2.5.1 Design of experiment

This is an aspect of applied statistics that involves analysing controlled experiments to determine the factors that affect the observed output parameter. By manipulating the input parameters, design of experiment identifies the important factors that may be overlooked. Since most experiments involve fixating certain factors while changing other parameters, some or all of the possible effects of input combinations can be studied using DOE. The major drawback of DOE is the number of experimental runs that need to be performed to achieve a significant statistical relationship. This is because several data points are required for each combination of input. A DOE process can take a substantial amount of time. In addition, the inability to control certain input variables could make it difficult to obtain an optimum statistical relationship.

2.5.2 Neural Network

Neural network is a system that captures and represents intricate input and output relationships. The major benefit of neural networks is their capability to characterize both linear and nonlinear relationships. Traditional linear models like DOE are inadequate when modeling data that contains nonlinear relationships. Minli et al [57] highlighted the advantage of using MATLAB neural network. Minli stated that MATLAB provides an accessible toolbox with neural network functions. The toolbox provides modeling capabilities without the need to write extensive codes. In recent years, MATLAB neural network has been used to approximate the link between input and output data in numerous studies. Anghel et.al [58] also used a MATLAB based neural network tool with 6 input and 3 output parameters to estimate how various working conditions affects the workstations of a manufacturing firm. Therefore, in this research, MATLAB neural network is used to establish a relationship between composite PLA properties and fabrication parameters.

CHAPTER 3

RESEARCH METHODOLOGIES

3.1 Materials

To fabricate the conductive and magnetic composite PLA, the base polymer used is pure Poly Lactic Acid (PLA) in fine powder form. This polymer is relatively easy to process and it possesses relatively good mechanical properties compared to other thermoplastics [22]. The reinforcing particles used to improve the electrical properties of the PLA are: 1, 2, 4 and 100 micrometer carbon black particles, micrometer carbon fiber (diameter in micrometer range, length in micrometer range) and nanometer carbon fiber (diameter in nanometer range, length in micrometer range.) The aspect ratio (length/diameter) of the nanometer carbon fiber is relatively large compared to the micrometer carbon fiber. Iron particle (10 micrometer) is used to improve the magnetic properties of the PLA.

3.2 Preparation

The composite PLA is prepared by combining the reinforcing particles with the pure PLA powder at different weight percent (wt%) ratio accordingly. The resulting compound is mixed using a rotating motor (Dayton AC-DC series motor) for 10 minutes with 5 spherical balls in the compound to aid the shearing action of the mixture. The mixed compound is then compressed as seen in Figure 3.3 at 6 kilopascals for 5 minutes in a precision mold using a compression tool.

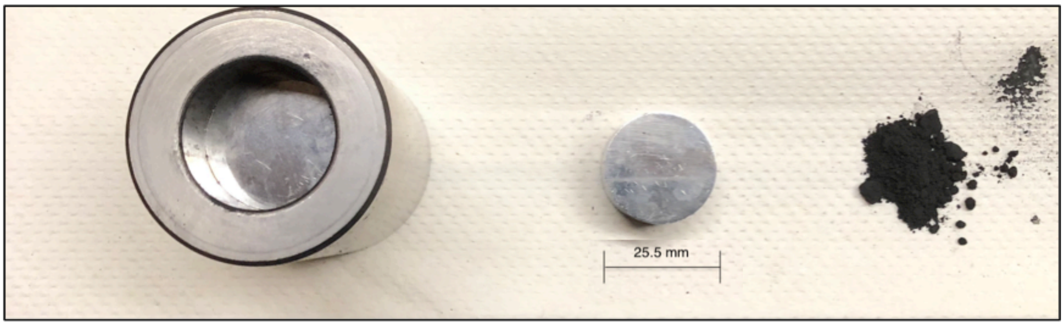

Figure 3.1: Compression mold, punch, & PLA Plastic + Carbon black mixture

Figure 3.2: PLA Plastic + Carbon black mixture in mold

Figure 3.3: Compression molding process

Figure 3.4: Conductive plastic sample (PLA Plastic + Carbon black)

3.3 Electrical and Magnetic Analysis

An "Agilent" brand impedance analyzer is used for the electrical and magnetic analysis of the properties of the fabricated composite PLA. In addition, rather than measuring with a basic direct current instrument such as a multi-meter, the impedance analyzer is used because it studies the behavior of the samples over a broad range of frequencies [59]. This is important because electrical components have distinctive characteristics at different frequencies [55]. The analyzed properties of the composites include resistance, inductance, and capacitance. These properties are a function of the resistivity, relative permeability and dielectric constant of the composites respectively.

3.3.1 Frequency Measurement Range

The samples in this research are analyzed at frequencies of 10 kHz to 10 MHz; however, the frequency used for analysis is 100 kHz. This frequency is commonly used for detecting materials with low conductivity relative to metals [59]. Furthermore, from the experimental results in this research, expected trends in the data are prominent at 100 kHz.

3.3.2 Resistance and Capacitance Measurement

Two circular parallel plates, as shown in Figure 4.2, are used as electrodes to hold the composite PLA sample. The resistance measured is a function of the network created by the carbon in the sample. Furthermore, as a sample is placed between the two parallel plates, the measured capacitance of the plates changes. Figure 3.5 is the circuit used for the resistance and capacitance measurement.

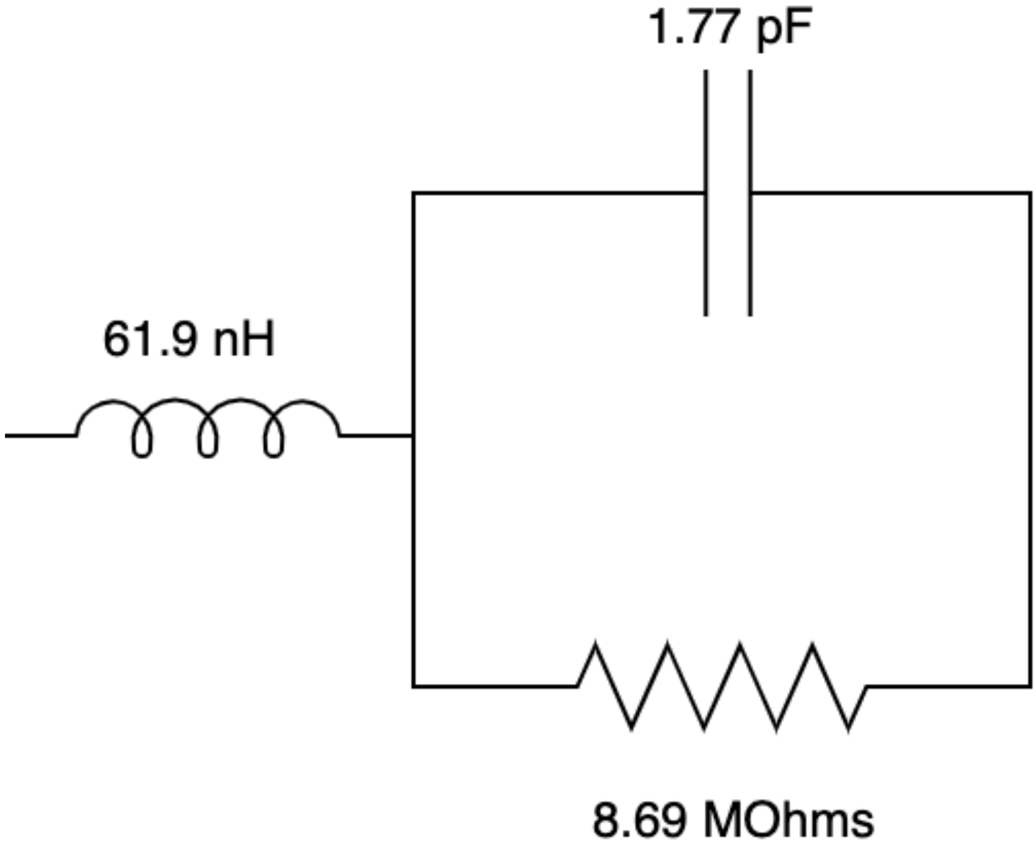

Figure 3.5: Circuit diagram for capacitance and resistance measurement

The resistance and capacitance readings are extracted from the measured impedance. The impedance expression for the circuit i.e., Inductance (L) in series with Resistance (R) and Capacitance (C) in parallel is as follows:

$$Z_{L-R/C} = iwL + (R^{-1} + iwC)^{-1} \qquad \text{Equation 3.1}$$

Separating Equation 3.1 into real and imaginary parts

$$Z_{L-R/C} = iwL + \frac{R}{1+(wRC)^2} - i\frac{wR^2C}{1+(wRC)^2} \qquad \text{Equation 3.2}$$

$$Z_{L-R/C} = \frac{R}{1+(wRC)^2} + i(wL - \frac{wR^2C}{1+(wRC)^2}) \qquad \text{Equation 3.3}$$

$$Z_{Real} = \frac{R}{1+(wRC)^2} \qquad \text{Equation 3.4}$$

$$Z_{Imaginary} = X = (wL - \frac{wR^2C}{1+(wRC)^2}) \qquad \text{Equation 3.5}$$

The resistance and capacitance values of the samples can be computed from the real and imaginary parts of the measured impedance

Computationally by Maple

$$R = \frac{L^2w^2 - 2LwX + X^2 + z_{real}^2}{z_{real}} \qquad \text{Equation 3.6}$$

$$C = \frac{Lw - X}{w(L^2w^2 - 2LwX + X^2 + z_{real}^2)} \qquad \text{Equation 3.7}$$

Resistance is a function of resistivity ρ, which is related to the geometry of a material and is expressed as follows,

$$\rho = R\frac{A}{l} \qquad \text{Equation 3.8}$$

Where

$$\rho = Resistivity\ (ohm.meter)$$

$$A = Area(meter^2)$$

$$l = Thickness\ of\ sample(meter)$$

Capacitance is a function of dielectric constant (k), which is related to the geometry of the material; and is expressed as follows,

$$Capacitance = k\varepsilon_0 \frac{A}{l} \qquad \text{Equation 3.9}$$

$$k = \frac{\varepsilon_r}{\varepsilon_0} \qquad \text{Equation 3.10}$$

$$\varepsilon_r = dielectric\ permitivity\ (Farad/meter)$$

$$\varepsilon_0 = Permitivity\ of\ vacuum\ \ (Farad/meter)$$

$$A = Area(meter^2)$$

$$l = Thickness\ of\ sample(meter)$$

3.3.3 Inductance Measurement

A coil, as shown in Figure 4.1, is used to generate a magnetic field by passing current through it. As the composite PLA sample is dropped in the generated magnetic field, the measured inductance of the coil increases. This is because the coils air core is now comprised of a material with higher relative permeability (Iron particles in the composite PLA sample). Relative permeability is a function of inductance [12], [60]–[64]. To calibrate the coil, the inductance of the wound coil is measured [65]–[68] before the sample is placed in the coil. After each sample is placed in the coil, the increase in the inductance of the coil is recorded. Figure 3.6 is the circuit configuration used for the measurement. The inductance readings are extracted from the measured impedance. The impedance expression for Inductance (L), Resistance (R) and Capacitance (C) in parallel is as follows

$$Z_{L/C/R} = [R^{-1} + (iwL)^{-1} + iwC]^{-1} \qquad \text{Equation 3.11}$$

Where L is inductance in Henry, C is Capacitance in Farad, R is Resistance in Ohms Ω, w is angular frequency in Hertz

Separating Equation 3.11 into real and imaginary parts

$$Z_{L/C/R} = \frac{\frac{1}{R}}{\left(\frac{1}{R}\right)^2+\left(wC-\frac{1}{wL}\right)^2} - \frac{wC-\frac{1}{wL}}{\left(\frac{1}{R}\right)^2+\left(wC-\frac{1}{wL}\right)^2} i \qquad \text{Equation 3.12}$$

$$Z_{Real} = \frac{\frac{1}{R}}{\left(\frac{1}{R}\right)^2+\left(wC-\frac{1}{wL}\right)^2} \qquad \text{Equation 3.13}$$

$$Z_{Imaginary} = X = -\frac{wC-\frac{1}{wL}}{\left(\frac{1}{R}\right)^2+\left(wC-\frac{1}{wL}\right)^2} \qquad \text{Equation 3.14}$$

The inductance change is computed.

Computationally by Maple

$$L = \frac{X^2+z_{real}{}^2}{w(CX^2w+Cz_{real}{}^2w+X)} \qquad \text{Equation 3.15}$$

$$R = \frac{X^2+z_{real}{}^2}{z_{real}} \qquad \text{Equation 3.16}$$

With the above formula, the changes in the measured inductance as the samples go in and out of the coil is extracted. Relative permeability μ_r is then extracted from the measured inductance as follows [67]

$$L_{coil} = N^2\, \mu_0\mu_r\left(\frac{D}{2}\right)\left[\ln\left(\frac{8D}{d}\right) - 2\right] \qquad \text{Equation 3.17}$$

Where

$$L_{coil} = Inductance\ of\ coil\ in\ Henries$$

$$N = Number\ of\ turns$$

$$\mu_0 = Permeability\ of\ free\ space = 4\pi\times10^{-7}$$

$$\mu_r = Relative\ permeability$$

$$D = Loop\ diameter$$

$$d = Wire\ diameter$$

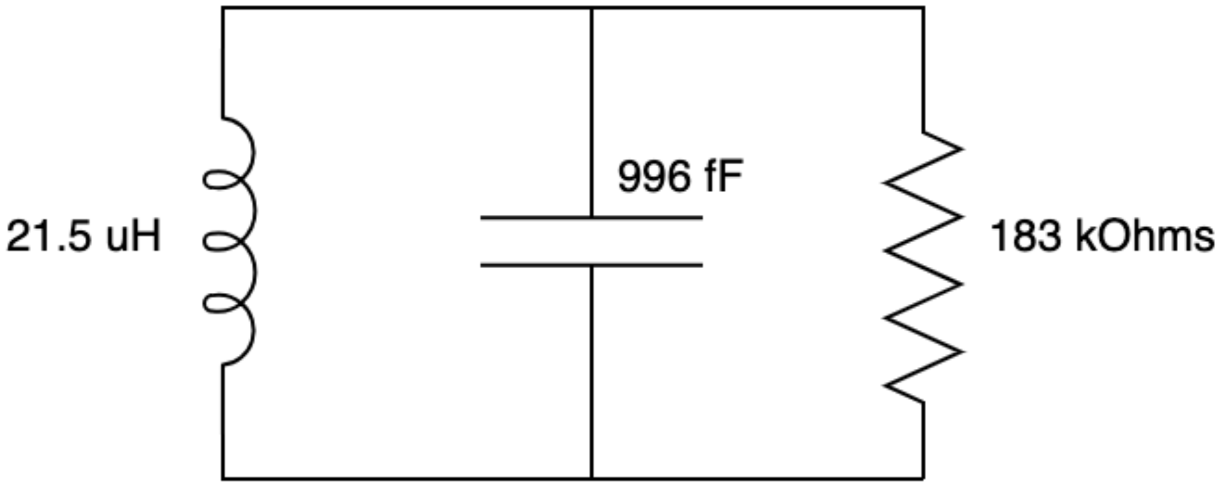

Figure 3.6: Circuit diagram for inductance measurement

3.4 Neural Network Modeling

There are numerous types of algorithms that could be used for function approximation problems of neural networks. However, Levenberg-Marquardt (LM) and Resilient Back-Propagation (RP) algorithms are recommended in many cases, these algorithms have the ability to achieve low root mean square errors compared to the other algorithms in the MATLAB program [69]. Both algorithms are trialed during the design of the model to determine the best algorithm for the model. Nevertheless, the efficiency of the applied algorithm depends on using the optimal number of neurons when designing the model. A number of neurons are trialed and tested during the design of the model to determine the optimal number of neurons required. In addition, a feed forward back propagation approach is used for designing the network. This approach comprises of a number of layers with the first layer serving as the input to the network. The neurons and functions used to establish the input-output relationship are present in a hidden layer. The output is at the last layer of the network. When building the network, the optimal number of neurons required is determined by retraining the network with different numbers of neurons each time. The structure of the neural network is presented in Figure 3.7.

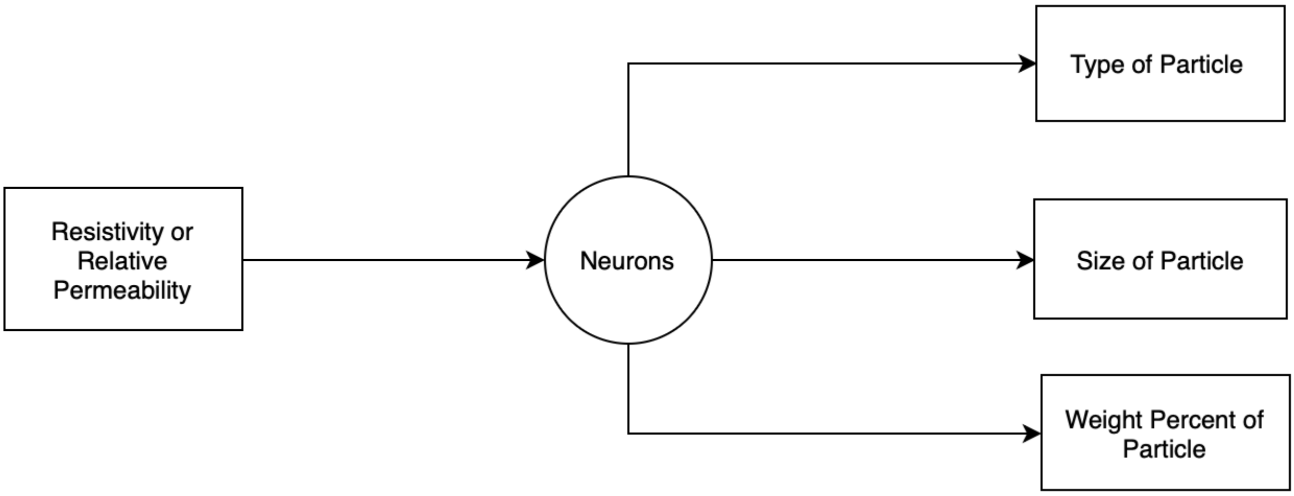

Figure 3.7: Artificial Neural Network Structure

CHAPTER 4

EXPERIMENTAL RESULTS AND DISCUSSION

In this research, composite PLA samples are fabricated by mixing reinforcing particles (Carbon black, Carbon fiber and Iron powder) with pure PLA. This mixture is compressed to produce composite PLA plastics with unique electrical and magnetic properties. The electrical and magnetic characterization of the fabricated conductive and magnetic PLA plastic is performed using the equipment as shown in Figures 4.1 - 4.3. The data from the experiment indicate that the following factors have an effect on the electrical and magnetic properties of the composite PLA:

a) The type and nature of reinforcing particle

b) The size of the reinforcing particle

c) The weight percent of the reinforcing particle

4.1 Magnetic Property of all the Fabricated Composites

As iron is ferromagnetic and carbon particles are not, Figure 4.4 shows that neither the carbon fiber nor carbon black has any effect on the observed relative permeability of the composite PLA. At the same weight percent of iron, composites fabricated with carbon black have the same relative permeability as those fabricated with carbon fiber. Hence, with this knowledge, the content of iron in all the fabricated composite PLA is fixed at 10wt%. It is also observed that regardless of the quantity of iron in the composite, the measured electrical properties do not change. This observed behavior is perhaps attributed to the minuscule quantity of iron in the composite. Also, due to the fact that the iron particles have been exposed to the atmosphere, they may have been oxidized to a certain extent making them highly resistive.

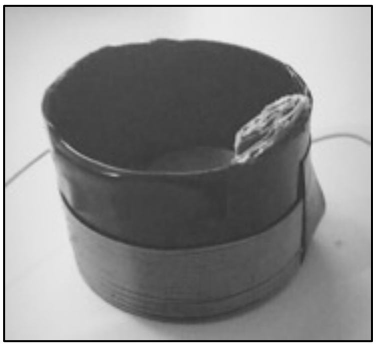

Figure 4.1: Coil for composite inductance measurement

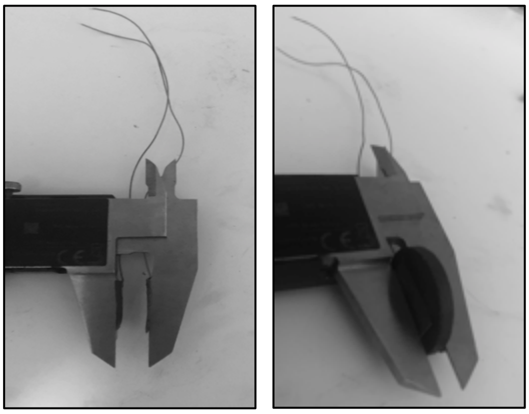

Figure 4.2: Probe for composite resistance and capacitance measurement

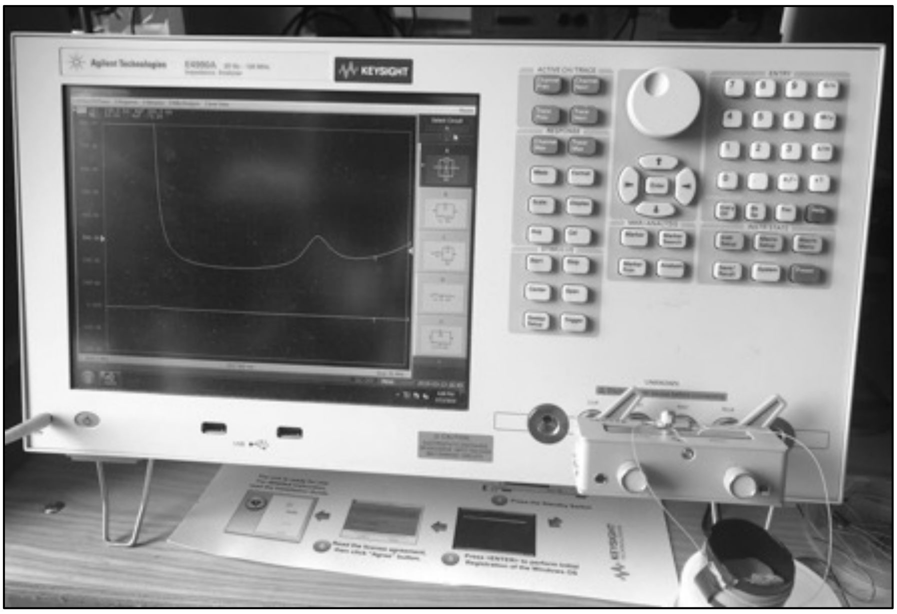

Figure 4.3: Impedance analyzer

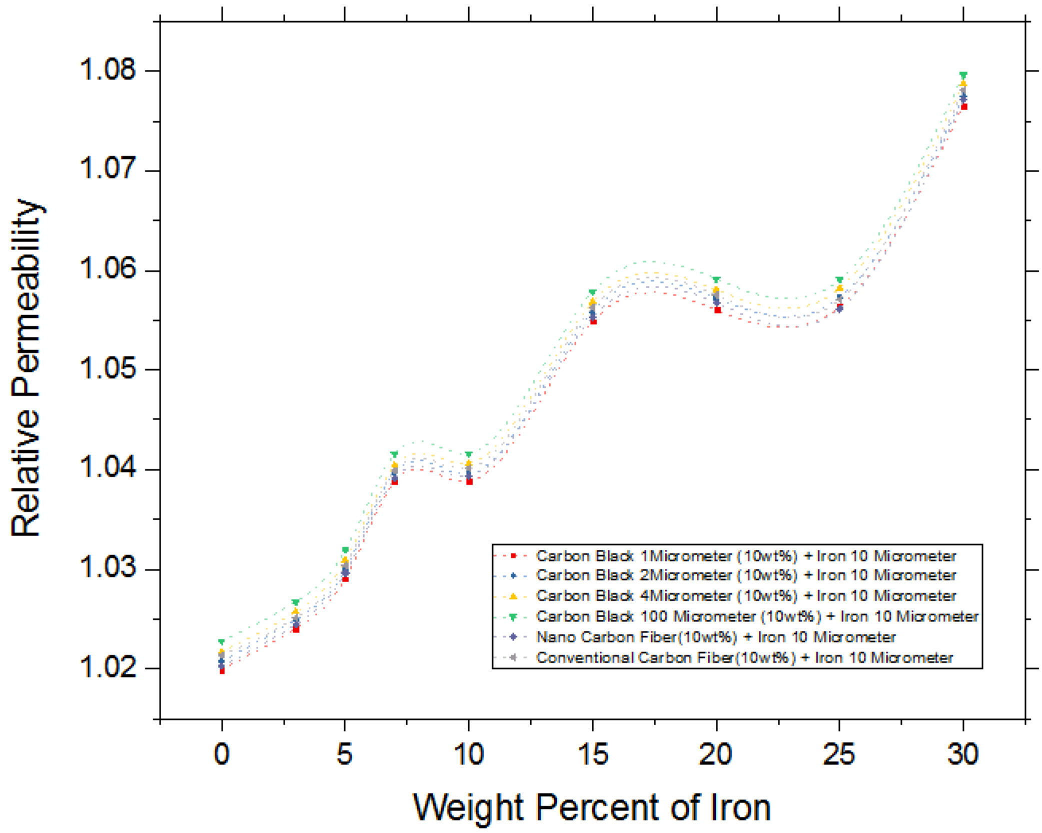

Figure 4.4: Variation of relative permeability with weight percent of iron [10 micrometer]

4.2 Conductive Property of Composites Fabricated with Carbon Black

Composite PLA samples are fabricated by combining pure PLA with different carbon black sizes at various weight percent ratio. The sizes of carbon black used for fabrication are 1, 2, 4 and 100 micrometer. The weight percent of carbon black in the composite is varied between 1 - 20%. A fixed 10wt% iron particle is also added to every composite. The effects of size and weight percent of carbon black on the conductive property of the fabricated samples are discussed as follows.

4.2.1 Effect of carbon black size on conductivity of the composite PLA

The experimental data in Figure 4.5 shows the effect of carbon black sizes on the conductivity of the composite PLA. It is observed that at a given weight percent of carbon black, for example at 10wt%, the resistivity of the composites fabricated with small carbon black particles is relatively low compared to larger carbon black particles. This observation also correlates with the experimental results of Nagata et al. [41]. The high conductivity observed in the composites fabricated with small carbon particles is likely a result of the high specific surface area and inherent chainlike aggregate network of the particle. Carbon black is mainly made up of round particles that bond together to form aggregates. The extent of bonding/aggregation between the individual particles of could affect the inherent level of conductivity of the carbon black. In addition, due to the high surface area of small particles, the individual particles have high surface energy [70]. Typically, to reduce the high surface energy, individual particles may bond with adjacent particles, this could result in the formation of aggregates [70]. Hence, compared to large carbon black particles, smaller particles may tend to bond together to form a more continuous chainlike aggregate network, which could facilitate electrical conductivity [70].

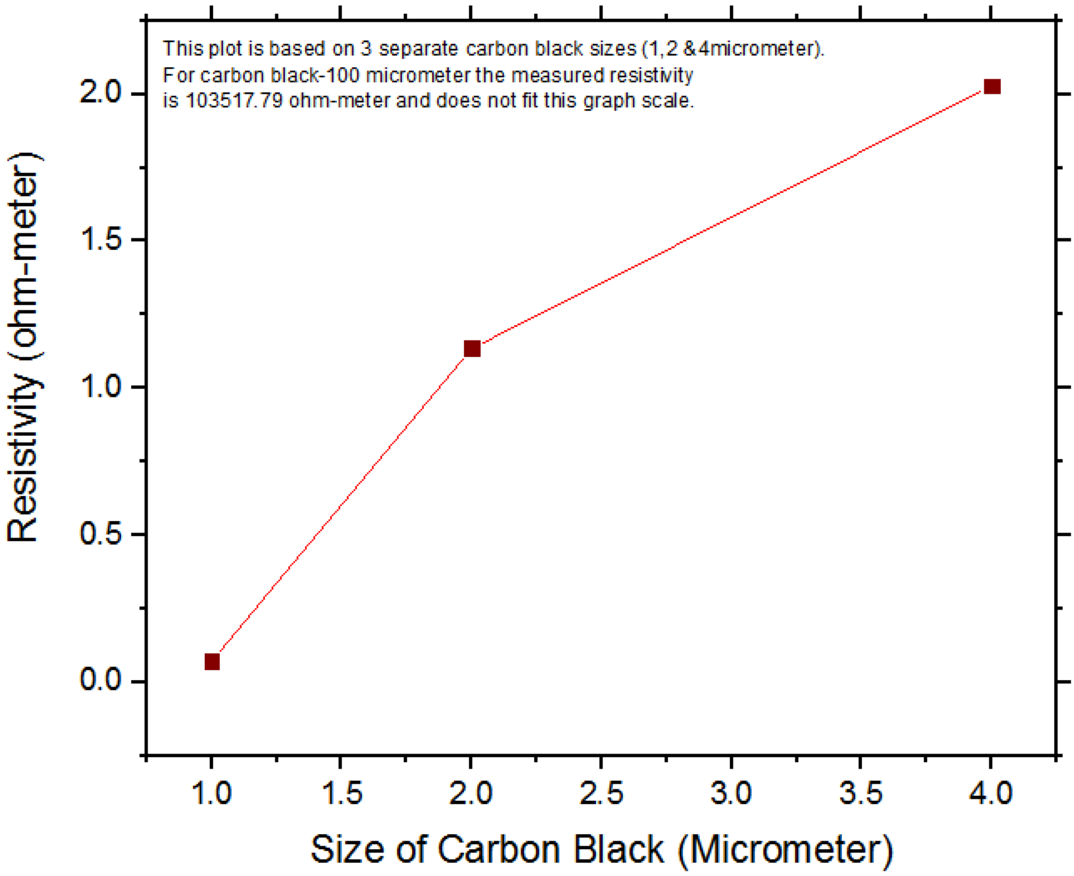

Figure 4.5: Variation of resistivity with carbon black size (Carbon black sizes are compared at 10wt %)

4.2.2 Effect of carbon black weight percent on conductivity of the composite PLA

Presented in Figures 4.6 - 4.7 are the experimental data that show the variation of resistivity of the composite with weight percent of carbon black. It is observed that the conductivity of the composite is directly proportional to the quantity of reinforcement in the composite. This implies that there is a reduction in the measured resistivity as the quantity of carbon black in the composite is increased. As discussed in section 4.2.1, the conductivity achieved in the composite PLA could be attributed to the carbon black network in the composite. This facilitates the transfer of electrons between the carbon black particles in the PLA matrix. At low weight percent of carbon black, a continuous conductive network has most likely not been formed; therefore, the conductivity of the composite is perhaps dependent mainly on the extremely low conductivity of the base PLA. However, at a higher weight percent, a conductive network has most likely been formed and the conductivity in this case depends mainly on the conductivity of the carbon black. Composites with low weight percent (<3wt%) of carbon black are almost non conductive. A usable level of conductivity is observed for most of the composites above 3wt% of carbon black. In summary, electrical conduction may occur in the composites either through the physical contact of the carbon black particles or through the high probability of *'electron tunneling'*.

4.2.2.1 Electron Tunneling

Electron tunneling is a channeling effect that occurs when quantum particles (in this research the electron) move through a barrier that according to the theories of classical physics should be impossible [71]. The barrier may be a physically impassable medium, such as an insulator, in the case of this study, the pure PLA in the composite matrix. In classical mechanics, a particle with inadequate energy would not be able to overcome a potential barrier. However, as seen in Figure 4.8, in quantum mechanics, particles habitually display wave-like behaviors.

Therefore, on encountering an impasse, a quantum wave does not abruptly end; rather, there is an exponential decrease in the amplitude of the wave. In a case where the barrier is thin enough to accommodate a drop in the amplitude of the wave through the barrier without reaching zero, there is a finite probability that some of the electrons will channel through the barrier [71]. Thus, the possibility of electron tunneling could be enhanced by the increase in the weight percent of the carbon black in the composite. This could be as a result of the consequent decrease in the gaps between disconnected carbon black particles when the quantity of carbon black in the composite is increased. It should also be noted that as the weight percent of carbon black is increased, '*percolation*' phenomenon might be experienced. In addition, above a certain weight percent of carbon black, known as the *'percolation threshold'*, only a marginal increase in conductivity is observed. The concepts of percolation and percolation threshold are discussed further in section 4.2.2.2

4.2.2.2 Percolation

Percolation is an aspect of statistical theory that describes the mechanics of interconnectivity between isolated components of a given system. When percolation occurs, there is a change in the overall behavior of the system. In this research, percolation most likely occurs due to the interconnectivity between individual carbon particles in composite as illustrated in Figure 4.9. **Percolation threshold** is the lowest quantity of reinforcement in the composite after which there is only a marginal change in the measured level of conductivity [43]. In summary, the observed magnitude of conductivity of the composites is most likely influenced by the formation of a conductive channel within the PLA [72]. On another note, the threshold at which percolation occurs is also dependent on the size of the carbon black in the sample. Low percolation threshold is observed for smaller carbon black sizes. This could be a result of the surface area and high degree of aggregation in small carbon black particles. However, for larger particles, percolation occurs at a higher weight percent. This could be due to the low degree of aggregation and highly discontinuous nature of the carbon black structure. Danae [5] also observed a similar trend when studying the electrical conductivity of different carbon black particles.

4.3 Dielectric Constant of Composites Fabricated with Carbon Black

Dielectric constant is a measure of a material's capacity to store electrical energy in an electric field. Polarization and dielectric leakage are some of the factors that determine the dielectric constant of a material. When a material is immersed in an electric field, the positive and negative charges of the material's atoms displace according to the direction of the field resulting in polarization. The extent of polarization determines the material's dielectric constant. Dielectric leakage occurs when a material, which is intended to serve as a dielectric, behaves instead like a short circuit/conductor. This could occur when the material is too conductive to accommodate the electric field formation needed for charge storage to occur. The concepts of polarization and dielectric leakage are further discussed as follows.

4.3.1 Polarization

Matter comprises of atoms arranged in a spatial domain adjacent to one another. The nucleus of an atom has a positive charge and is usually surrounded by one or more electrons. Some materials may have atoms arranged in such a way that electrons are free to move within the material. This is particularly common in metals and doped semiconductors. Insulators have their electrons bound in a molecular orbit; therefore, they are not free to move. When an insulator is placed in an electric field, the field pulls the electron and repels the positively charged nucleus. This results in a material with a net atomic dipole or polarization. The sum of all the microscopic polarization of the atoms represents the macroscopic polarization of the material. Therefore, as illustrated in Figure 4.10, there is a net electrical field E_0 in the material. **Dielectric constant** k is the ratio of the applied field E to the net field generated in the material due to polarization ($\frac{E}{E_0}$). The capacitance of a capacitor increases by a factor of the dielectric constant.

$$C_{New\ capacitance} = k \times C_{initial\ capacitance} \qquad \text{Equation 4.1}$$

4.3.2 Dielectric leakage

Dielectric leakage is the loss of charge in a capacitor due to the inability of the capacitor's dielectric to prevent flow of electric current. Insulators are excellent dielectric materials because they allow low dielectric leakage. They allow a net electric field formation within them. Hence, the more conductive a material is, the more leakage it is prone to have [73]. The dielectric constant of a composite is studied ideally at an extremely low conductivity. An infinite dielectric constant represents a highly conductive material such as metals since they are highly polarizable. This also implies that a net electric field formation is not possible in highly conductive materials. A literature study of the dielectric constant of epoxy resin shows that adding carbon to the epoxy resin matrix increases the dielectric constant magnitude of the resin [74]. However, if charges are free to move throughout the resin, the material becomes conductive making it lose its dielectric properties as a result of high dielectric leakage [73]. Dielectric leakage is extremely high after the **dielectric threshold**. The dielectric threshold of a material is the point at which the material has an unacceptably high dielectric leakage as a result of its high conductivity.

4.3.3 Effect of carbon black size on the dielectric constant of the composite PLA

As shown in the experimental data in Figure 4.11, at the same weight percent, the smaller the size of the carbon black particles, the higher the observed dielectric constant.

As established in section 4.3.2, highly conductive materials have infinite dielectric constant. Thus, due to the inherent high conductivity of the small carbon black particles, the dielectric constant is high in comparison to larger particles.

4.3.4 Effect of carbon black weight percent on dielectric constant of the composite PLA

Figure 4.12 shows the effect of carbon black weight percent on the measured dielectric constant of the composites. The increase in the weight percent of carbon black results in an increase in the magnitude of the dielectric constant. Also, as described in section 4.3.2, it is observed that above the dielectric threshold weight percent, the material is highly conductive and cannot act as a dielectric. As previously stated, conductive materials like metals have infinite dielectric constant. Therefore, as the weight percent of the carbon black increases, the magnitude of the dielectric constant increases. After the dielectric threshold is reached, the composites can no longer be used as a dielectric in a capacitor. This perhaps is due to extremely high dielectric leakage resulting from the conductivity of the composite. According to previous research from literature review [75], dielectric properties of composites are usually studied at very low weight percent, less than 0.5wt% of conductive reinforcement in most cases. In this research, the dielectric nature of the composite is studied between 1 - 20wt% of carbon. Therefore, a more detailed explanation of the effect of carbon black weight percent cannot be established. This is because majority of the fabricated samples do not behave like a dielectric after 3wt%. On another note, the size of the carbon black used for fabrication also has a relationship with the observed dielectric threshold since the size of the carbon black affects the conductivity of the composite. In fact, only composites fabricated with carbon black (100 micrometer) behave like dielectrics after 3wt%. This could be attributed to the inherent low conductivity of the large carbon black particle.

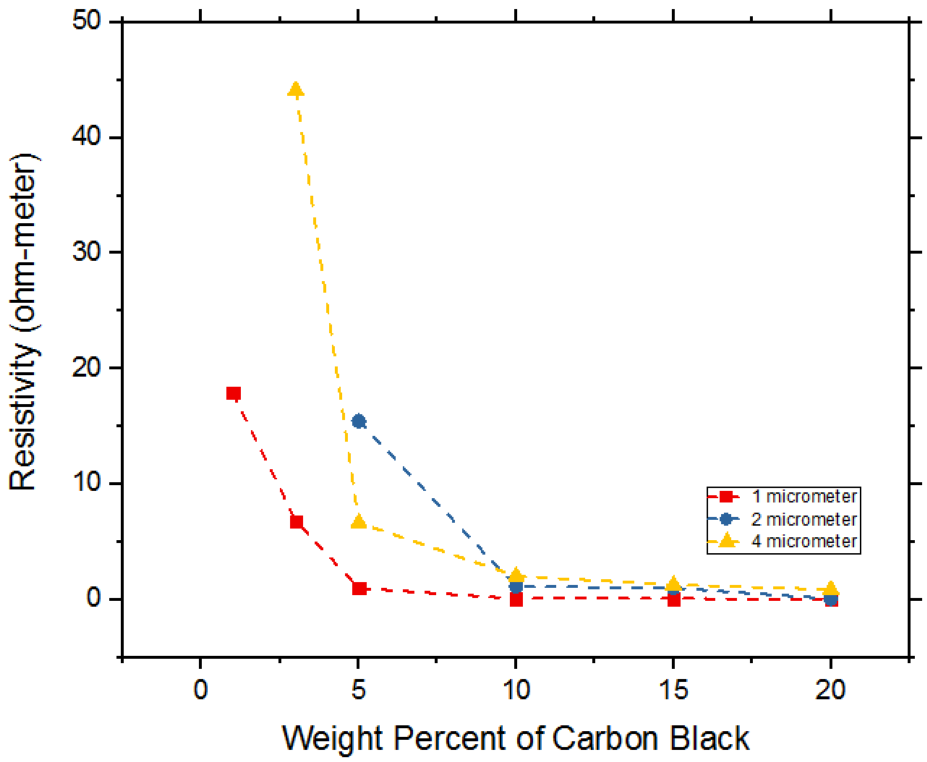

Figure 4.6: Variation of resistivity with weight percent of carbon black [1 – 4 micrometer]

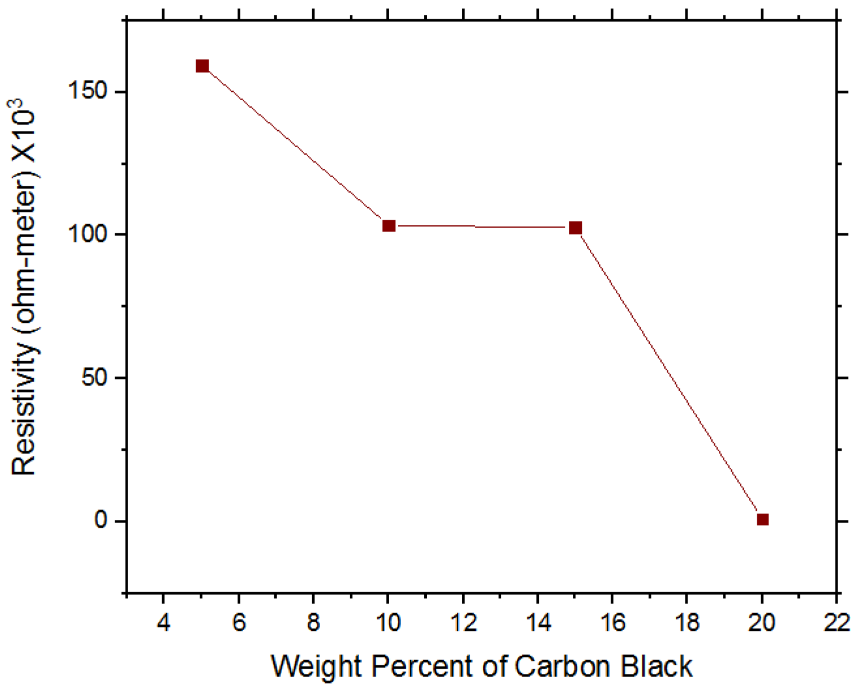

Figure 4.7: Variation of resistivity with weight percent of carbon black (100 micrometer)

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Figure 4.8: Barrier Penetration (U is the potential energy of the impasse) [71]

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Chapter 2 Electrical Conductivity, Percolation Theory, Electromagnetic Interference (EMI) and Its Mechanisms

Figure 4.9 Percolation Pathway (Blue boxes represent conducting reinforcement and white boxes represent insulator matrix) [76]

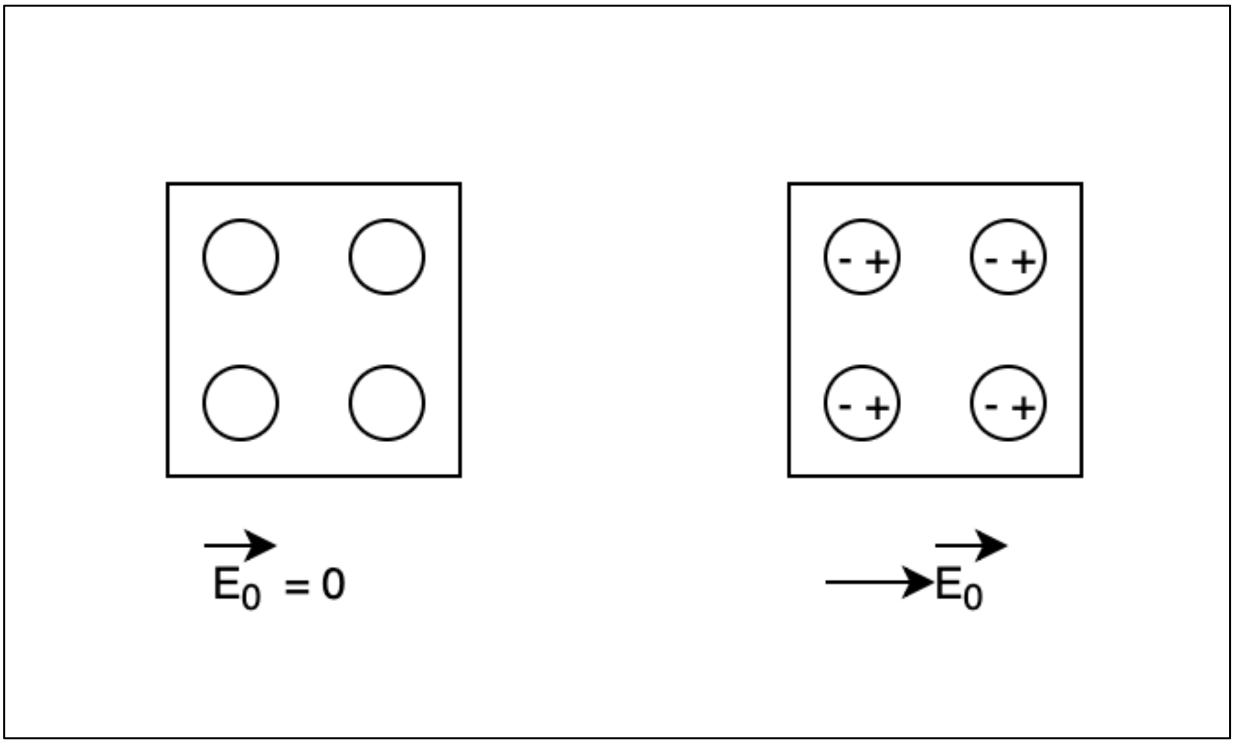

Figure 4.10: Polarization of a material (E: Electric field) [77]

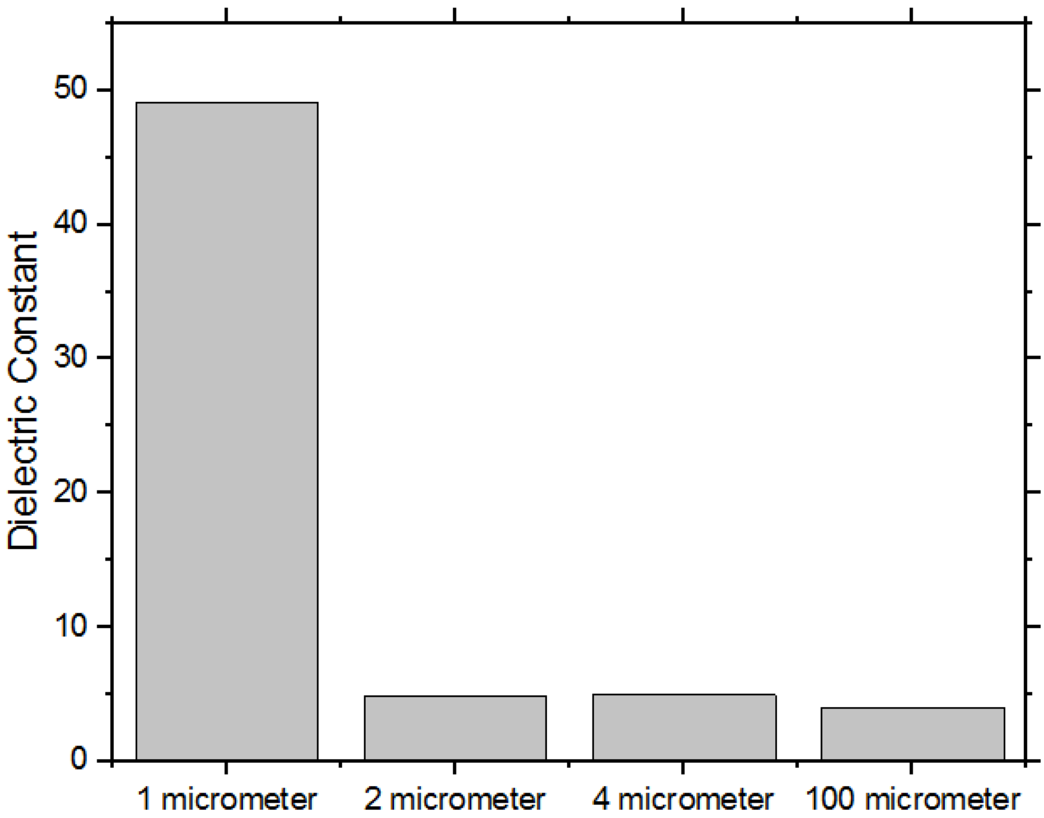

Figure 4.11: Variation of dielectric constant with carbon black size (Carbon black sizes are compared at 1wt %)

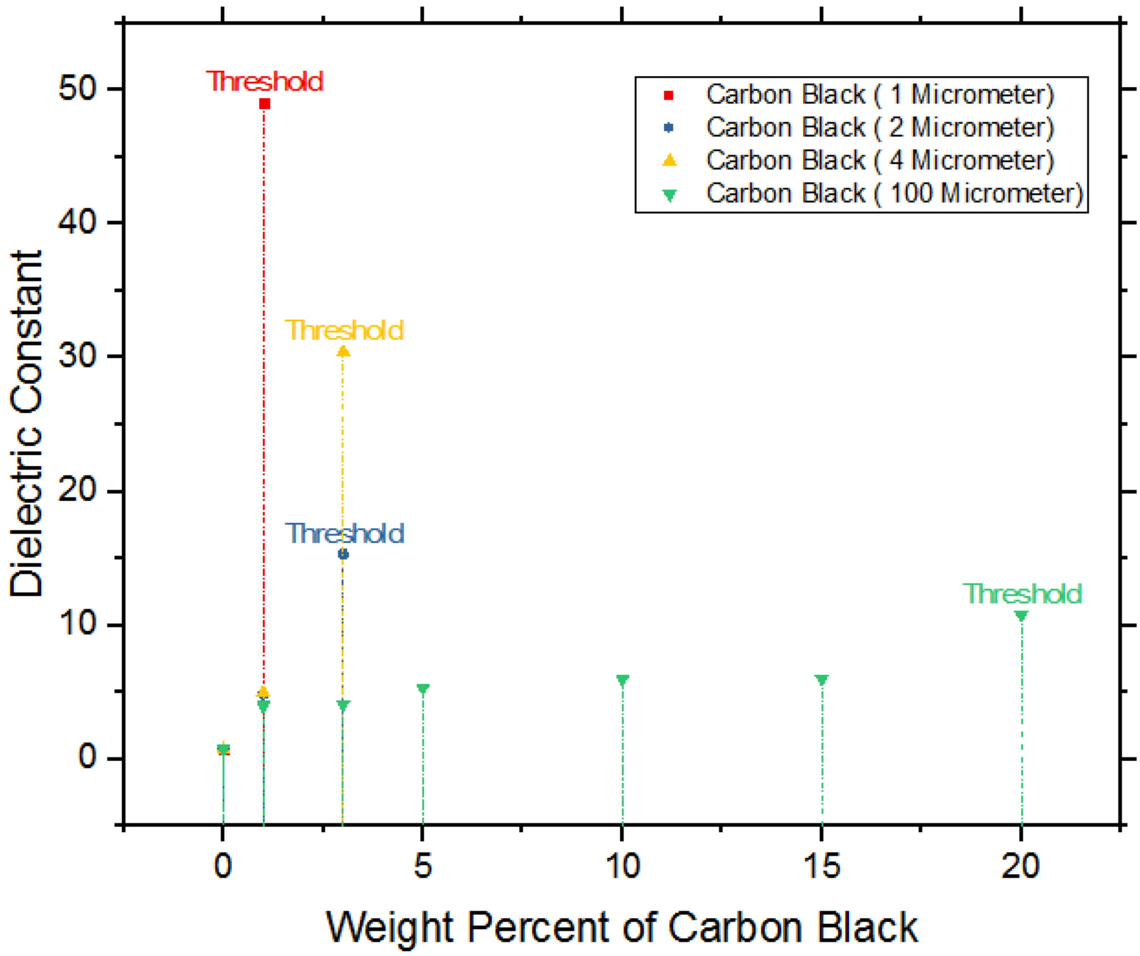

Figure 4.12: Variation of dielectric constant with weight percent of carbon black

4.4 Conductive Property of Composites Fabricated with Carbon Fiber

Another set of composite PLA samples are fabricated by combining pure PLA with two different sizes of carbon fiber at various weight percent ratio. The carbon fiber sizes used are each within the micrometer and nanometer range in diameter. The weight percent of carbon fiber in the composite is varied from 1 - 20%. A fixed 10wt% iron particle is also added to every composite. The effects of size and weight percent of carbon fiber on the conductive property of the fabricated sample are discussed as follows.

4.4.1 Effect of carbon fiber size on conductivity of the composite PLA

Although both fibers have similar lengths (micrometer range), the nanometer carbon fiber is smaller in diameter compared to the micrometer carbon fiber; therefore, it has a higher aspect ratio (length/diameter). At any given weight percent, composite PLA fabricated with nanometer carbon fiber are more conductive compared to those fabricated with micrometer carbon fiber. Figure 4.13 shows the huge difference in the observed level of resistivity between both fibers.

The specific surface area of carbon fiber is a direct function of its aspect ratio. Carbon fibers with high surface area have high inherent conductivity [78]. Due to its small diameter, the nanometer carbon fiber most likely possesses a chainlike network that facilitates electrical conductivity [79]. On the other hand, low conductivity is observed in composites fabricated with micrometer carbon fiber, this could be as a result of its large diameter. It is relatively difficult for the large carbon fiber to form interconnected aggregates. As reported by Shen [79], smaller sized carbon fibers easily form chainlike aggregates. This phenomenon is also highlighted in Section 4.2.1, which describes why small carbon black particles tend to have a continuous chainlike network.

4.4.2 Effect of carbon fiber weight percent on conductivity of the composite PLA

The conductivity of the composite is proportional to the quantity of carbon fiber used for fabrication. Percolation threshold, as previously described in Section 4.2.2.2 is observed between 15 - 20wt% for micrometer carbon fiber as shown in Figure 4.14. However, as shown in Figure 4.15, percolation occurs at a lower weight percent (10wt%) for nanometer carbon fiber composites.

The conduction channel and electron tunneling theories of quantum mechanics as described in section 4.2.2 may further be used to explain the effect of weight percent of the carbon fiber on the conductivity of the composite. When the weight percent of the carbon fiber is high, there is an improvement in conductivity. This could be due to the corresponding increase in the physical contact between the individual carbon fibers as the average gap between the individual particles is most likely reduced. When the weight percent of the carbon fiber is low, the gaps between the carbon fibers are most likely large. Therefore, the probability of contact between individual fibers is small; consequently, an effective conductive channel may not be formed. Also, the probability of electron tunneling is small due to the large barrier of pure PLA between the carbon particles. This could be due to the insufficient carbon fiber in the composite matrix; therefore, a low conductivity (high resistivity) is observed at low weight percent of carbon fiber. The same phenomenon is also observed in a study of the effect of carbon fiber on High Density Polyethylene composites [79].

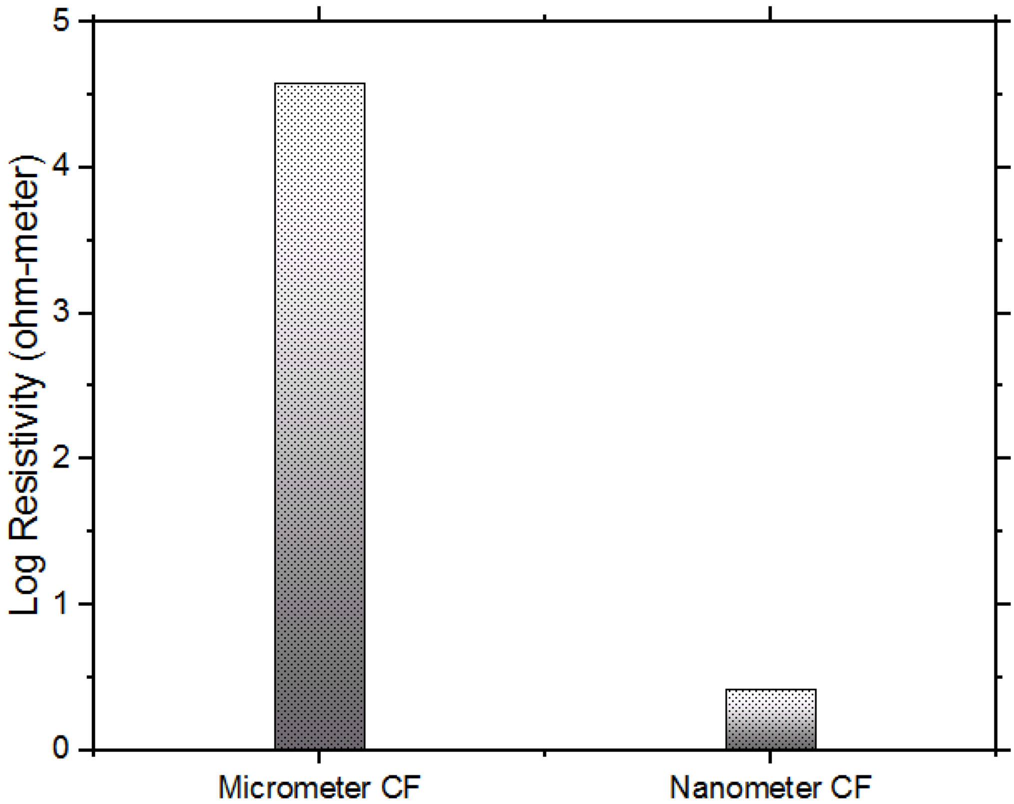

Figure 4.13: Variation of resistivity with carbon fiber size (Carbon fiber sizes are compared at 10wt %)

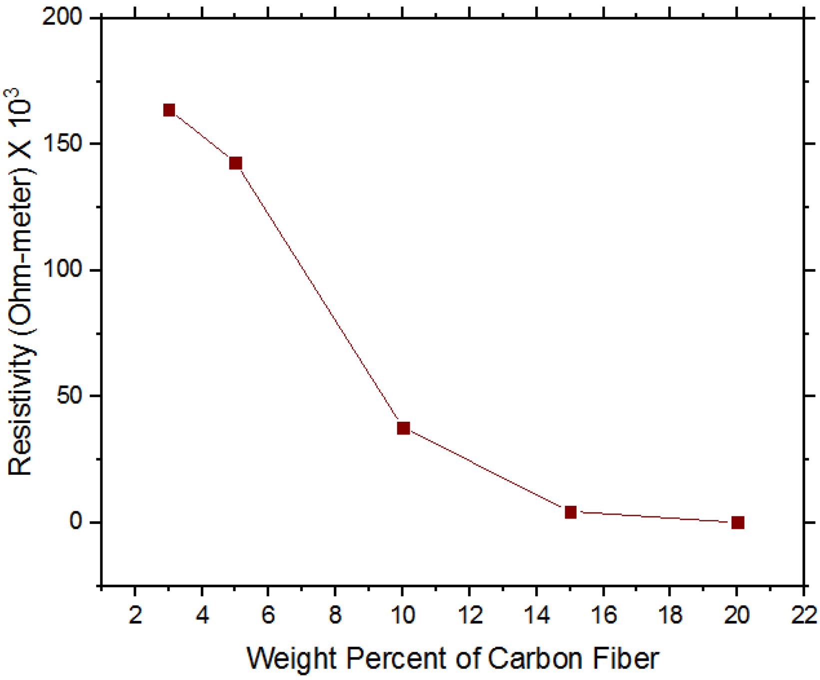

Figure 4.14: Variation of resistivity with weight percent of micrometer carbon fiber

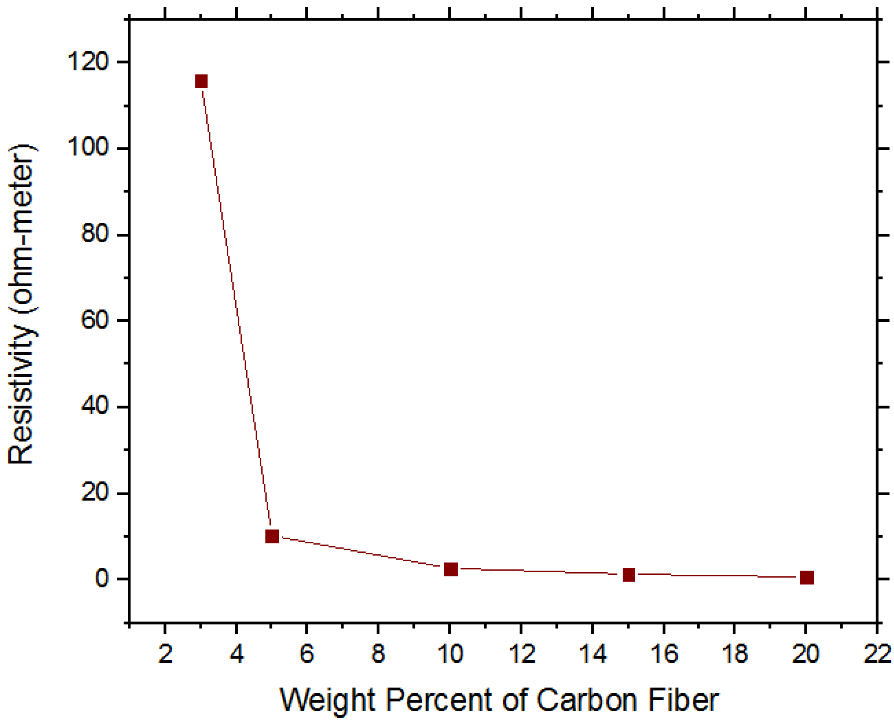

Figure 4.15: Variation of resistivity with weight percent of nanometer carbon fiber

4.5 Dielectric Constant of Composites Fabricated with Carbon Fiber

The effects of size and weight percent of carbon fiber on the dielectric constant of the fabricated composites are discussed as follows.

4.5.1 Effect of carbon fiber size on dielectric constant of the composite PLA

Similar to carbon black composites, the size of carbon fiber influences the observed dielectric constant of the composites. As shown in Figure 4.16, composites fabricated with nanometer carbon fiber have higher dielectric constant compared to those fabricated with micrometer carbon fiber. As established in section 4.3.2, highly conductive materials have infinite dielectric constant. Thus, due to the high conductivity of the nanometer carbon fiber particles as explained in section 4.4.1, the dielectric constant is high in comparison to micrometer carbon fiber particles.

4.5.2 Effect of carbon fiber weight percent on dielectric constant of the composite PLA

The experimental result in Figure 4.17 shows the effect of weight percent of carbon fiber on the dielectric constant of the fabricated composites. The magnitude of the observed dielectric constant increases with the increase in the weight percent of carbon fiber; this occurs up until the dielectric threshold. The observed threshold is at 20wt% for micrometer carbon fiber composites, this is high compared to the 3wt% observed in nanometer carbon fiber composites. The nanometer carbon fiber most likely induces high dielectric leakage in the composites due to its inherent high conductivity, hence the observed low dielectric threshold. The higher dielectric threshold observed in samples fabricated with micrometer carbon fiber is most likely attributed to the low conductivity of the fiber.

As previously explained in section 4.3.2, conductors are not good dielectrics due to their extremely high dielectric leakage. Therefore, the dielectric leakage of the composite could be minimized when small quantity of carbon fiber is used for fabrication. However, it should be noted that composites fabricated with nanometer carbon fiber are still highly conductive even at low weight percent.

4.6 Combining both carbon black and carbon fiber composite PLA

A final set of composite PLA samples are fabricated by combining pure PLA with both carbon black and carbon fiber. In this case, a combination of carbon black (5wt%) and carbon fiber (5wt%) is used for comparison purposes. Similar to the previous samples, a fixed 10wt% iron particle is maintained in the composites. Only the effect of the size of carbon fiber and carbon black on the conductive properties of the composite is discussed in this section. It should be noted that the effect of weight percent of carbon on conductivity is not studied as it has been established in sections 4.4.2 and 4.5.2. Also, the dielectric constant of the composite is not discussed; this is because the composites are highly conductive at 10wt% of carbon and do not behave like dielectrics as explained in section 4.3.2.

4.6.1 Conductive Property of Composites Fabricated with both carbon black and carbon fiber

As shown in Figures 4.18 - 4.21, samples fabricated with only carbon black (1 micrometer) are highly conductive. However, at the same weight percent, an equal combination ratio of either nanometer or micrometer carbon fiber with carbon black (1 micrometer) worsens the conductivity. The same behavior is observed for composites fabricated by combining 2 or 4 micrometer carbon black with either of the two carbon fiber sizes. In addition, composites fabricated with only carbon black (100 micrometer) have poor conductivity, however, the combination of nanometer carbon fiber with carbon black (100 micrometer) immensely improves the level of conductivity.

The data shown in Figure 4.21 is supported by Shen [80]. Shen observed that, at the same weight percent, composites fabricated by combining both carbon fiber and carbon black are more conductive compared to composites fabricated with only carbon black. However, as shown in Figures 4.18 - 4.20, sometimes, using solely carbon black produces higher conductivity. This observation is supported by Drubetski [81]. Overall, the relative surface area of the carbon particle is likely the main factor that affects the achieved level of conductivity. As emphasized in section 4.2.1, smaller sized carbon black has a high surface area; therefore, combining it with carbon fiber of lesser surface area worsens the conductivity of the composite. A general rule of thumb is that, unless the carbon black to be used has a lower surface area property compared to the carbon fiber, it could be counterintuitive to combine both carbon fiber and carbon black in an attempt to improve the conductivity of the composite.

4.7 Applications and uses of the achieved resistivity (conductivity), relative permeability and dielectric constant

In this research, various composites with different levels of conductivity have been fabricated. These composites could be used in various applications. The highest level of conductivity achieved in this research is 0.06 ohm-meter. This level of conductivity could be used for the fabrication of electromagnetic interference shields. Electromagnetic interference (EMI) shielding is the process of reflecting and/or adsorbing electromagnetic radiation. EMI shielding material are used to protect/shield components from EMI radiations. The main mechanism of shielding/protection is reflection, and for this to occur, the shield must have mobile charge carriers (electrons). Therefore, shields tend to be highly conductive and can function with a resistivity of 0.06 ohm-meter. The secondary mechanism of EMI shielding is adsorption. To achieve this, the shield has to be electrically conducting and/or magnetic. This is to enable the shield to interact with the interrogating/unwanted electromagnetic radiation. The electric and magnetic dipoles needed for the interaction could be provided by carbon - iron composite PLA developed in this research. Adsorption or reflection mechanism is determined by the electrical conductivity and the relative magnetic permeability of the material (shield). Chung stated that the adsorption loss is a function of the product å and μ_r, while the reflection loss is a function of the ratio $å/\mu_r$; where å is the conductivity of the material in relation to copper and μ_r its relative permeability [82]. Also, the low conductivity achieved in some of the samples could be used for fabricating static dissipative materials. Static dissipative materials are used to control the flow of charges from the source to the ground more slowly in order to prevent damage to electronic components. In static dissipative applications, a low conductive material is needed. Usually, a resistivity between 1 x 10^2 – 10^8 ohm-meter is required. Furthermore, the relative permeability of the fabricated composites could be used for producing magnetic storage devices. It could also be used to induce a magnetic signature into objects for traceability purposes, for example, fabricating traceable medical devices or for authentication purposes, as in currency coins. Also, the highest dielectric constant achieved in this research is 50. This could be used to fabricate capacitors with high charge storage capacity.

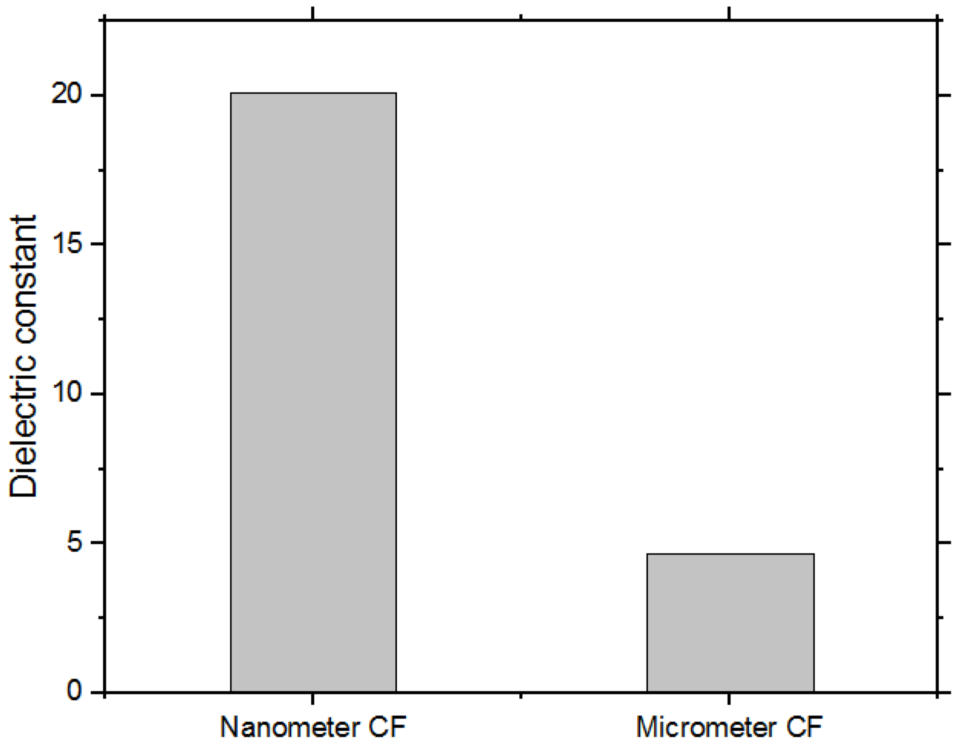

Figure 4.16: Variation of dielectric constant with carbon fiber size (Carbon fiber sizes are compared at 3wt %)

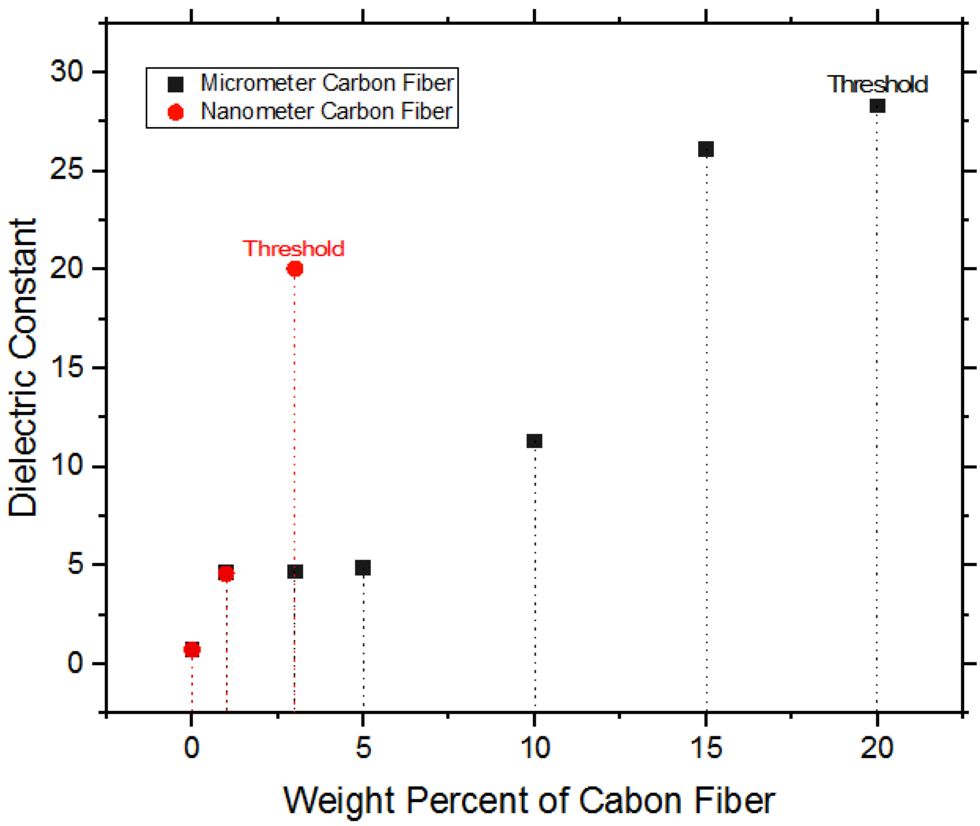

Figure 4.17: Variation of dielectric constant with weight percent of carbon fiber

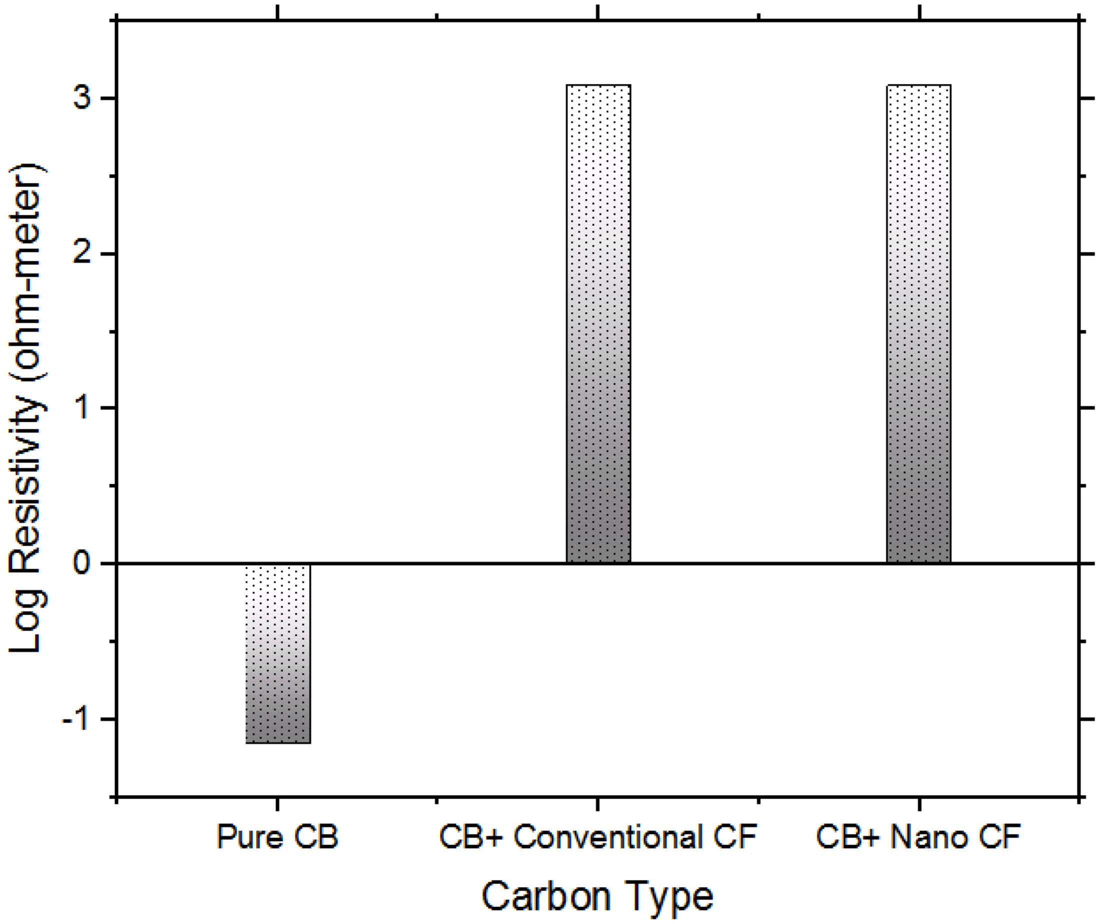

Figure 4.18: Variation of resistivity with carbon type: (10wt% Pure CB 1 micrometer), (5wt% CB 1 micrometer + 5wt% micrometer CF) & (5wt% CB 1 micrometer + 5wt% nanometer CF)

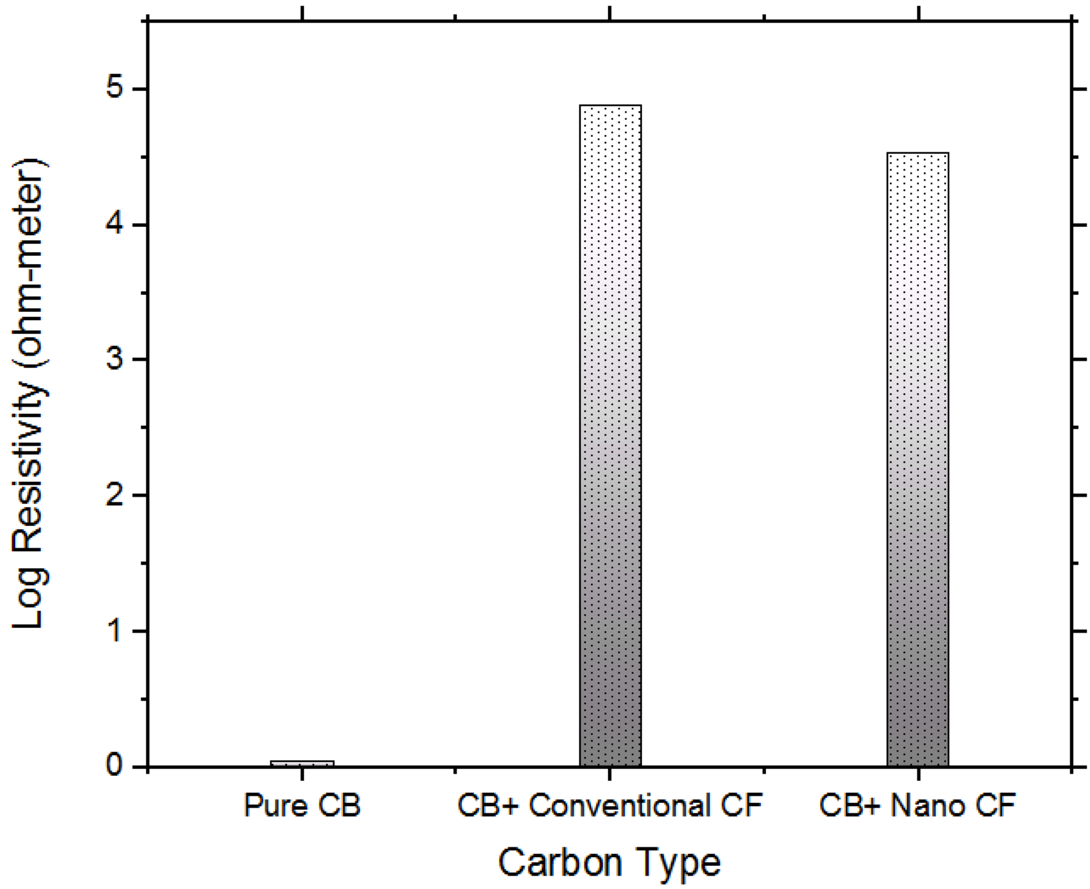

Figure 4.19: Variation of resistivity with carbon type: (10wt% Pure CB 2 micrometer), (5wt% CB 2 micrometer + 5wt% micrometer CF) & (5wt% CB 2 micrometer + 5wt% nanometer CF)

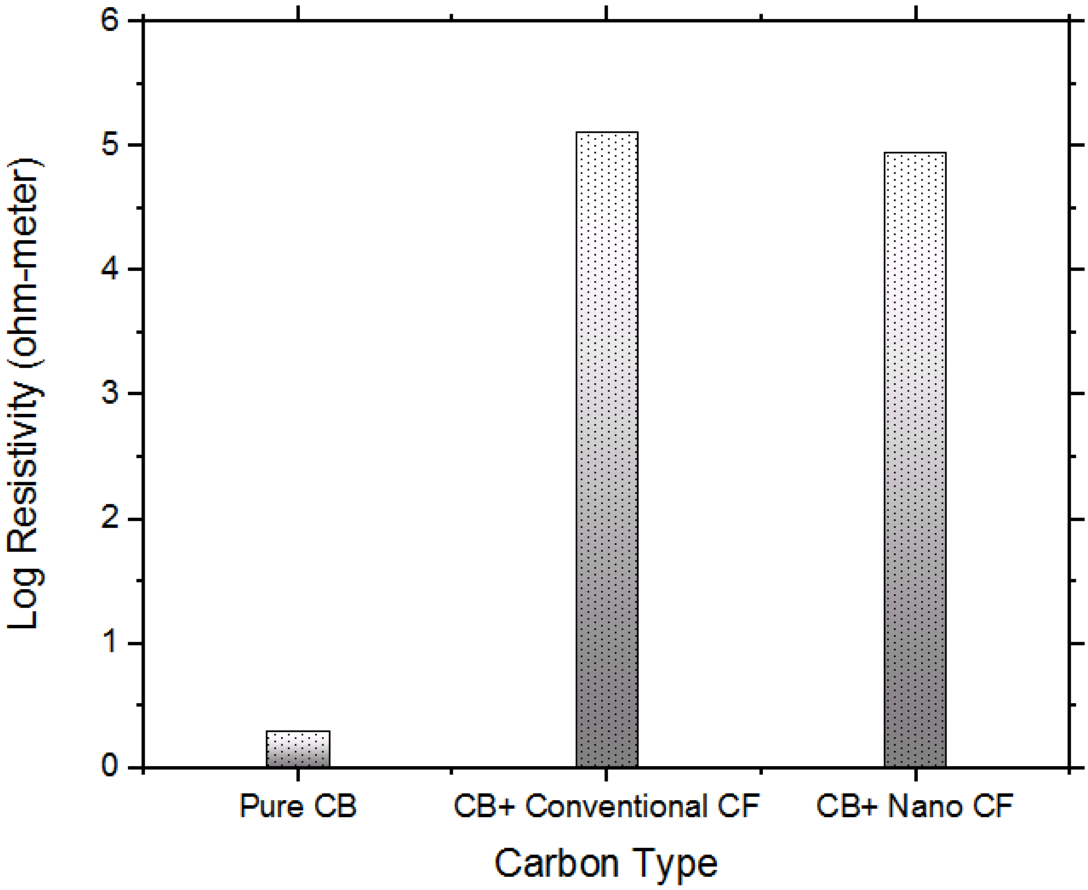

Figure 4.20: Variation of resistivity with carbon type: (10wt% Pure CB 4 micrometer), (5wt% CB 4 micrometer + 5wt% conventional CF) & (5wt% CB 4 micrometer + 5wt% nanometer CF)

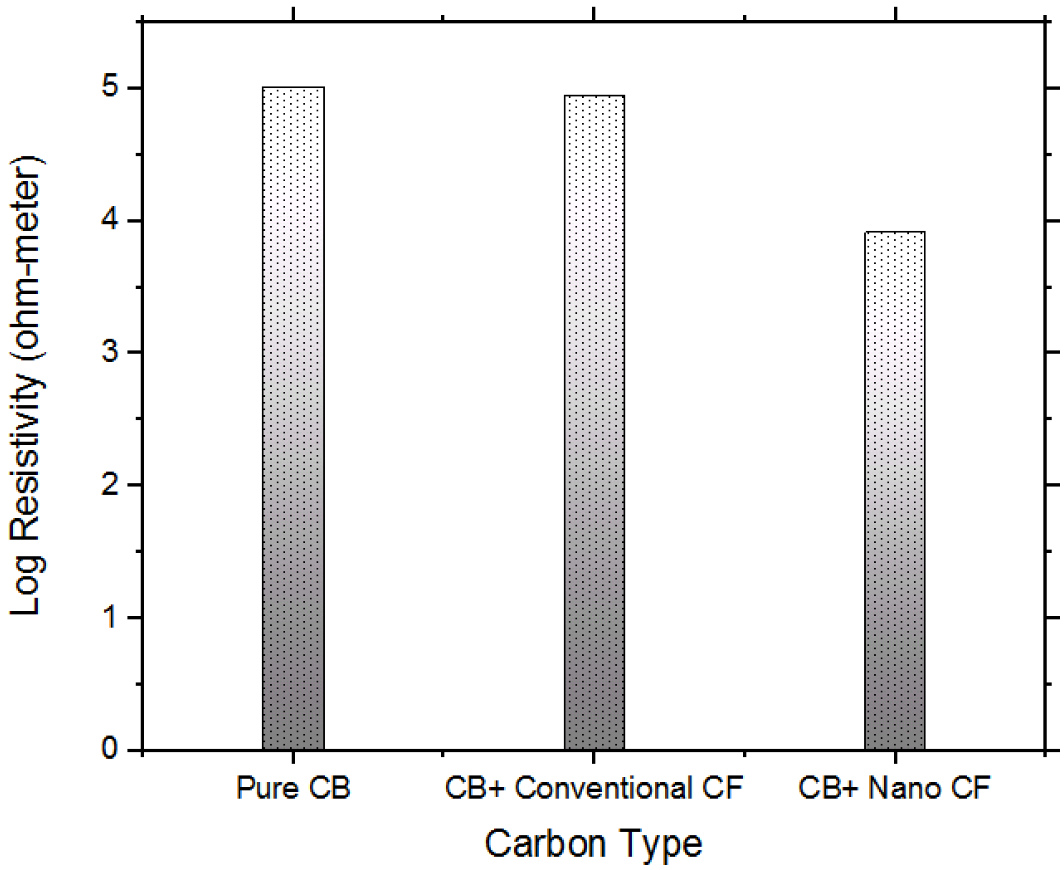

Figure 4.21: Variation of resistivity with carbon type: (10wt% Pure CB 100 micrometer), (5wt% CB 100 micrometer + 5wt% conventional CF) & (5wt% CB 100 micrometer + 5wt% nanometer CF)

CHAPTER 5

MODEL DESIGN AND IMPLEMENTATION

5.1 Scope and purpose of the neural network model

As previously established in Chapter 4, the parameters that affect the properties of the composite include: the size, type and weight percent of the reinforcing particle. Using these data from the experiment, a model is developed to predict what combinations of these parameters are required to fabricate a composite with unique resistivity and/or relative permeability. It should be noted that since only 10 - 15% of the fabricated samples behaved like a dielectric material, the dielectric constant data is not used for the model development. This is due to the very limited dielectric constant data available.

5.2 Model Design

The design of an artificial neural network (ANN) model usually involves a number of steps that follow a systemic process. The basic modeling steps for an ANN are shown in Figure 5.1. MATLAB neural network toolbox developed by Mathworks Incorporation is used for the development of the ANN. Shown in Figure 5.2 is the neural network toolbox, it provides a user interface that replaces the need to write extensive codes and scripts. The ability to choose and modify a number of algorithms to fit specific needs makes the tool both versatile and reliable.

5.2.1 Preprocessing

After the resistivity and relative permeability data of all the composites have been gathered, preprocessing is performed in order to train the ANN more efficiently. Preprocessing procedure includes:

1) Sorting and solving the problem of erratic data & 2) Normalization of the measured data.

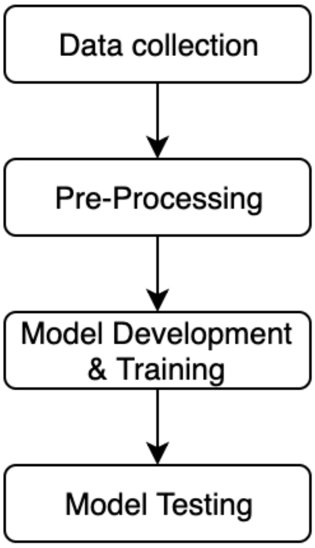

Figure 5.1: Basic process of designing an artificial neural network

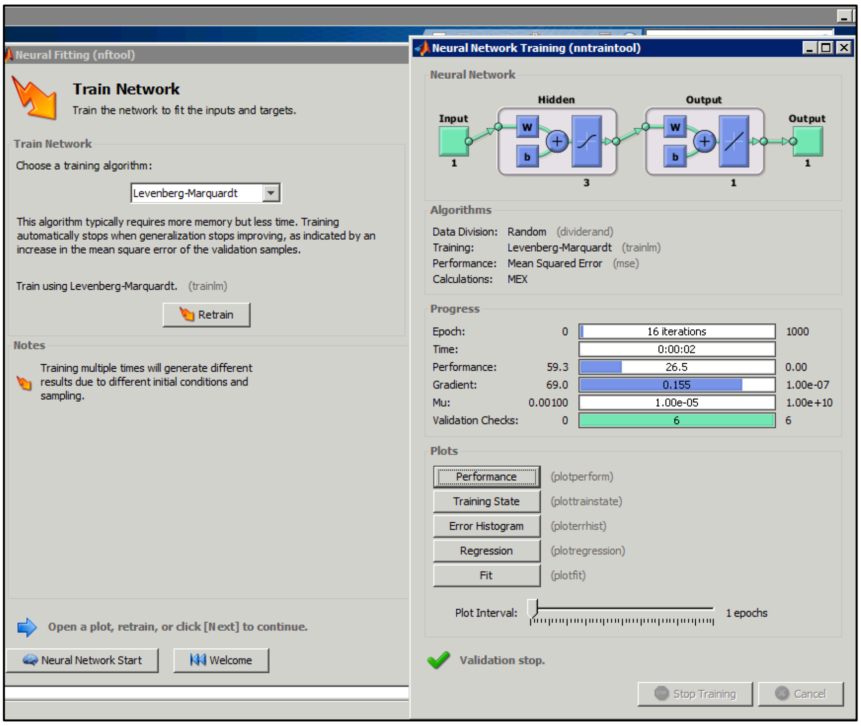

Figure 5.2: Artificial neural network user interface [MATLAB]

5.2.1.1 Imputing erratic data

About 3% of all the fabricated composites display properties that are not consistent with the other samples. The properties of these composites are replaced by taking the mean value of neighboring data. This is known as data imputation. The practice of data imputation has also been used in other ANN development studies Mavani [83].

5.2.1.2 Data Normalization

Normalization of data is generally a good practice that should be performed before the training data is presented to the network. As the neural network cannot distinguish the importance of each data, exposing the network to a mixture of very large and very small values may cause confusion for the network. Therefore, to prevent confusion, all the input values are normalized using the *mapminmax* script in MATLAB.

5.2.2 Model Development

Over 100 composite PLA samples have been fabricated in this research. These samples have unique properties. The data from the experimental characterization process are used to train and test the neural network.

5.2.2.1 Data partition

Several studies in machine learning use a 70-30% rule. This implies that 70% of the data is used for training and 30% is used for validation and testing. However, if the training data set contains minimal noise/erratic data, the training data percent ratio could be reduced. In this research, only 3% of the data are identified as noise/erratic. Hence, 65% of the data from the experimental characterization are used for training, 15% for validation and 20% for testing.

5.2.2.2 Transfer functions

A neural network consists of three layers, the input, hidden and output layer. These layers are interconnected by transfer/activation functions generated through the training process. Transfer functions are the processing units of a neural network and they are either linear or nonlinear. They usually have a range between 0 to 1 or -1 to 1. Some useful functions include pure linear, log sigmoid and hyperbolic tan as shown in

Figure 5.3. Log sigmoid and hyperbolic tangent are two of the most used functions since they somewhat represent nonlinear relationships modeled in a neural network. Hyperbolic tangent function connects the input and hidden layers together. A linear transfer function connects the hidden layer to the output layer.

5.2.2.3 Training algorithm

Of the various types of neural network training algorithms, the most commonly used are Resilient Back Propagation (trainrp) and Levenberg-Marquardt (trainlm) as they produce low errors [69]. The resilient back propagation algorithm disregards the magnitude effect of the partial derivative of the network performance function. Instead, it uses the sign of the derivative to update the weights and biases of the network. It is based on the steepest descent method [83]. On the other hand, the levenberg-marquardt algorithm reduces the performance function of the network by updating the weights and biases with each iteration. This algorithm ensures the performance function is continually reduced after each iteration [69]. Although this algorithm is relatively quick, a major drawback is that it requires large computing memory.

5.2.3 Model Validation and Testing Procedures

The simulation of the network is a very crucial step that ensures the trained network generalizes and produces desired outputs when presented with new data. In this research, 20%

of the recorded composite PLA properties are used for testing the neural network model. Afterwards, both the qualitative and quantitative assessments of the network are performed.

5.2.3.1 Quantitative Assessment

It is advisable to use more than one quantitative measure to analyze the performance of the model [84]. The quantitative assessment of the model performance could be evaluated by computing performance indicators such as correlation coefficient and root mean square error. Correlation coefficient (CORR) measures how strong the relationship is between the actual/experimental (x) and ANN model predicted (y) values.

$$CORR = \frac{n(\sum xy) - (\sum x)(\sum y)}{\sqrt{[n\sum x^2 - (\sum x^2)][n\sum y^2 - (\sum y)^2]}} \qquad \text{Equation 5.1}$$

CORR generally measures how linearly related two variables are. It provides an appropriate method to analyze the relationship between the predicted and actual data. A major drawback to CORR is that it does not indicate the magnitude of the error values. Therefore, it is advisable to use CORR with another performance metric. In conjunction with CORR, the root mean square error (RMSE) metric is used to measure the network performance. The RMSE between the observed and predicted data indicates the deviation of the predicted values from the experimentally observed values. A lower RMSE indicates a closer agreement between both values. RMSE is used when large errors are undesirable. To compute the RMSE, error values are squared before calculating the mean. This is a benefit of RMSE metric because it helps identify large errors in a neural network. The RMSE is calculated as follows:

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(Predicted_i - Actual_i)^2}{N}} \qquad \text{Equation 5.2}$$

5.2.3.2 Qualitative Assessment

The qualitative analysis of the scatter plots is performed visually. Visual assessment helps answer questions such as:

1) Is the model performance higher within a certain range of input?

2) Does the model under or overestimate output values?

The aim of this study is to qualitatively assess whether the model accurately predicts the fabrication parameters required to achieve a certain level of resistivity and/or magnetic permeability.

5.3 Model Performance

This section describes the performance/results generated by the trialed model designs. The label **'CN'** identifies the model for predicting the parameters required to fabricate a composite with unique resistivity. The label **'FE'** identifies the model for predicting the parameters required to fabricate a composite with unique relative permeability. Presented in Tables 5.1 - 5.2 are the performance statistics and the overall assessment of all the trialed CN and FE model designs. The best CN and FE design is selected based on the CORR and RMSE of the training and validation data.

5.3.1 Effects of model design parameters

The parameters that affect the accuracy of the neural network include normalization, training algorithm and number of neurons in the hidden layer. The effects of these parameters are discussed as follows

5.3.1.1 Input data normalization

To examine the importance of normalizing the input values, a separate CN and FE design is developed and assessed without the *(mapminmax)* normalization script. As shown in Tables 5.1 - 5.2, it is observed that the designs with no normalized inputs have relatively low CORR and large RMSE in comparison to the subsequent designs with normalized input. This highlights the importance of normalization as previously stated in section 5.2.1.2. This approach is also used in numerous ANN research papers, for example, Heydari [85] used normalization technique to develop a neural network model to predict water quality.

5.3.1.2 Training algorithm

As shown in Tables 5.1 - 5.2, compared to trainrp, the trainlm algorithm generates the best predictions according to the CORR and RMSE values. Theo et al. [86] also observed in his study that trainlm algorithm provides the best estimation for predicting the quality of marine beach water.

5.3.1.3 Numbers of neurons

The number of neurons used in the hidden layer are 3, 5,10 and 20. As shown in Tables 5.1 - 5.2, an increase in the number of neurons reduces the RMSE. The best results are achieved when 10 neurons are used in the hidden layer. Above 10 hidden neurons, it is observed that the model generates relatively poor results. A higher RMSE is observed in the predicted output data when the number of hidden neurons is 20 and above, this could be as a result of over fitting. **Over fitting** could be detected when the training RMSE decreases while the validation RMSE increases after a certain number of iterations. To prevent over fitting, methods such as reducing the training capacity of the network or increasing the training data set could be employed. However, increasing the training data set is not always feasible due to time and technical constraints. Furthermore, reducing the training capacity allows the network to focus on learning the relevant patterns rather than memorizing the training data.

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J. Mavani, "Artificial Neural Network," 2014.

Figure 5.3 MATLAB Transfer Function [83]

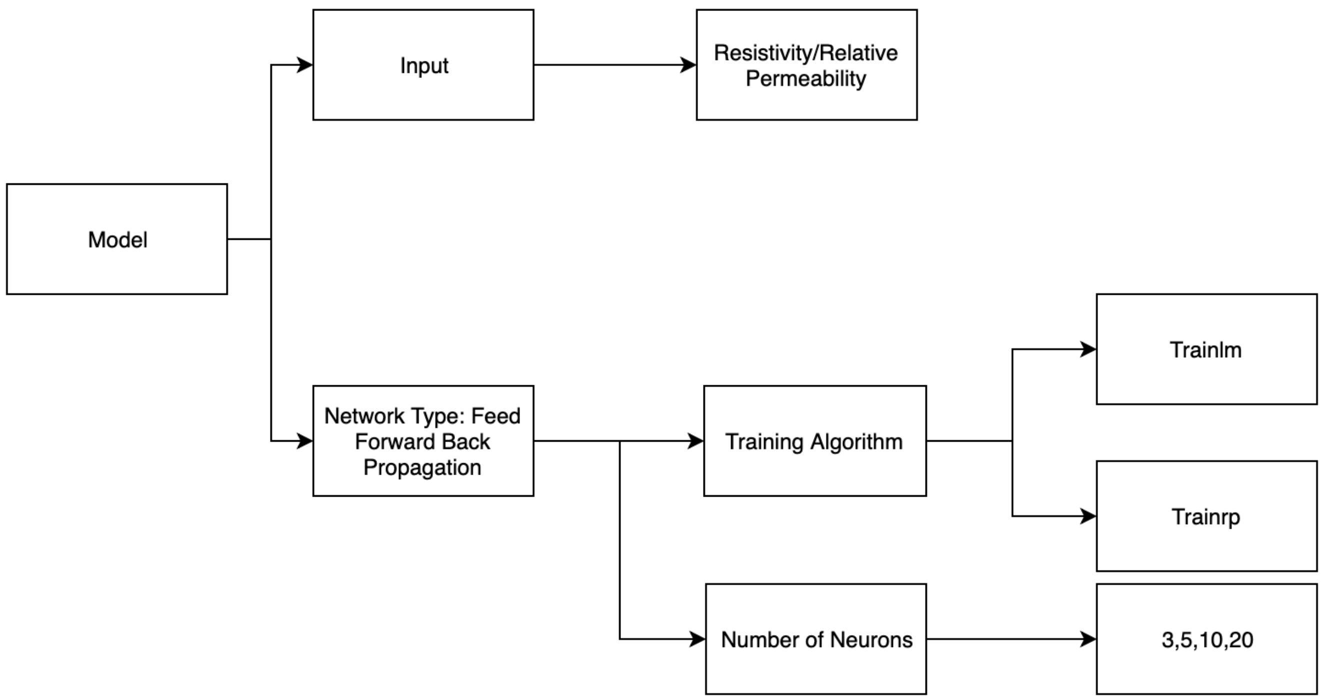

Figure 5.4: Design of Artificial Neural Network

Table 5.1: Performance comparison of trialed designs for predicting carbon particle effect on resistivity

Model	**Training algorithm**	**# of neurons**	**Input norm.**	**Training data**		**Validation data**	
				RMSE	**CORR**	**RMSE**	**CORR**
CN	Trainlm	10	No	6.22	0.81	7.11	0.87
	Trainlm	3	Yes	5.36	0.85	5.15	0.79
	Trainlm	5	Yes	3.44	0.90	3.32	0.83
	Trainlm	**10**	**Yes**	**0.55**	**0.91**	**0.46**	**0.98**
	Trainlm	20	Yes	0.41	0.94	2.27	0.79
	Trainrp	3	Yes	7.73	0.82	6.33	0.75
	Trainrp	5	Yes	5.41	0.78	4.43	0.77
	Trainrp	10	Yes	2.55	0.93	0.46	0.91
	Trainrp	20	Yes	1.34	0.88	5.27	0.81

Table 5.2: Performance comparison of trialed designs for predicting iron particle effect on relative permeability

Model	**Training algorithm**	**# of neurons**	**Input norm.**	**Training data**		**Validation data**	
				RMSE	**CORR**	**RMSE**	**CORR**
FE	Trainlm	10	No	5.55	0.82	5.92	0.86
	Trainlm	3	Yes	9.12	0.93	15.11	0.77
	Trainlm	5	Yes	6.78	0.65	11.23	0.79
	Trainlm	**10**	**Yes**	**2.74**	**0.90**	**2.34**	**0.98**
	Trainlm	20	Yes	1.22	0.88	12.53	0.84
	Trainrp	3	Yes	12.84	0.55	19.74	0.77
	Trainrp	5	Yes	9.74	0.47	15.68	0.79
	Trainrp	10	Yes	6.36	0.59	15.34	0.87
	Trainrp	20	Yes	5.28	0.56	21.18	0.45

5.3.2 Graphical and Visual Assessment

The performance statistics of the neural network provides an indication of its prediction capability. Figures 5.5 - 5.6 show a point-by-point plot comparison of the experimental data (x-axis) and neural network model predicted data (y-axis).

5.3.2.1 Best performing model design for predicting resistivity

Figure 5.5 is a plot of the observed and predicted weight percent of carbon required to achieve a certain level of resistivity. This plot is based on the best CN model design as highlighted in Table 5.1. By specifying the preferred level of resistivity, the model identifies the carbon particle type and size that could be used to achieve the required resistivity. It then provides an estimation of the corresponding weight percent for each of the identified carbon particle. The data used to simulate the accuracy of the model shows a CORR value of 0.98. This indicates that the predicted weight percent of carbon has a strong correlation with the actual weight percent used for fabrication during the experiment. In addition, an acceptable RMSE of 0.46 is observed.

5.3.2.2 Best performing model design for predicting relative permeability

Figure 5.6 is a plot of the observed and predicted weight percent of iron particle required to achieve a certain level of relative permeability. This plot is based on the best FE model design as highlighted in Table 5.2. The data used to simulate the accuracy of the model shows a CORR value of 0.98. Therefore, the predicted iron quantity has a strong correlation with the quantity used for the fabrication during the experiment. In addition, comparing the predicted data to the experimental data, it is observed that the RMSE is 2.34.

In summary, it is essential to combine visual assessment with statistical assessment. This is done in order to provide an overall assessment of the ANN performance. To conclude, qualitative and quantitative assessment helps to understand the ANN model behavior as well as assessing the suitability of the model for prediction applications.

5.4 Applications and uses of model

The developed model can be used to predict fabrication parameters for applications in electromagnetic interference shielding, static dissipative and authentication. The aforementioned list of applications is by no means exhaustive. It could be used for estimating fabrication parameters for a wide range of conductive and magnetic applications if the required levels of conductivity and relative permeability are given. More specifically, if a resistivity of 0.06 ohm-meter and above or a relative permeability of 1.07 and below is needed, the model could be used to identify the nature and corresponding weight percent of reinforcing particles required for fabrication.

5.4.1 Electromagnetic interference shielding

The concept of electromagnetic interference (EMI) shielding has been explained in section 4.7. A functional EMI shield could have a resistivity level of 1 ohm-meter, and relative permeability level of 1.07. Therefore, to use the developed neural network model to predict the parameters required to fabricate the EMI shield, the abovementioned levels of resistivity and relative permeability are inputted into the network.

Experimentally, 27wt% of iron (10 micrometer) is required to achieve 1.07 relative permeability, the model predicts 25.5wt% of iron (10 micrometer). Through extrapolation from experimental data, 25.5wt% should produce a relative permeability of 1.06. In addition, experimentally, 5wt% of carbon black (1 micrometer) is required to achieve 1 ohm-meter; the model predicts 4.6wt% carbon black (1 micrometer). Through extrapolation from experimental data, 4.6wt% should produce a resistivity of 2.1 ohm-meter. The errors between the extrapolated experimental values and the model predicted values of the resistivity and relative permeability are negligible.

5.4.2 Static dissipative materials

The concept of static dissipation has also been established in section 4.7. A functional static dissipative material could have a resistivity level of 37 kohm-meter. Therefore, to use the developed neural network model to predict the parameters required to fabricate the static dissipative material, the aforementioned level of resistivity is inputted into the network.

Experimentally, 10wt% of carbon fiber (micrometer) is required to achieve 37 kohm-meter, the model predicts 10.2wt%. Through extrapolation from experimental data, 10.2wt% of carbon fiber (micrometer) should produce a resistivity of 39 kohm-meter. The error between the extrapolated experimental value and the model predicted value of the resistivity is relatively small.

5.4.3 Authentication

In authentication applications, the conductive and magnetic properties of a material are sometimes used for identification purposes. For example, if a material with a resistivity level of 6 ohm-meter, and a relative permeability of 1.04 is required, the model could be used to predict the parameters to fabricate the specified material. The aforementioned levels of resistivity and relative permeability are inputted into the network.

Experimentally, 10wt% of iron (10 micrometer) is required to achieve 1.04 relative permeability, the model predicts 9.9wt% of iron (10 micrometer). Through extrapolation from experimental data, 9.9wt% of iron should produce a relative permeability of 1.039. Furthermore, experimentally 6wt% of carbon black (4 micrometer) is required to achieve 6 ohm-meter; the model predicts 4.5wt% carbon black (4 micrometer). Through extrapolation from experimental data, 4.5wt% should produce a resistivity of 7.8 ohm-meter. The error between the extrapolated experimental value and the model predicted values of the resistivity and relative permeability are negligible.

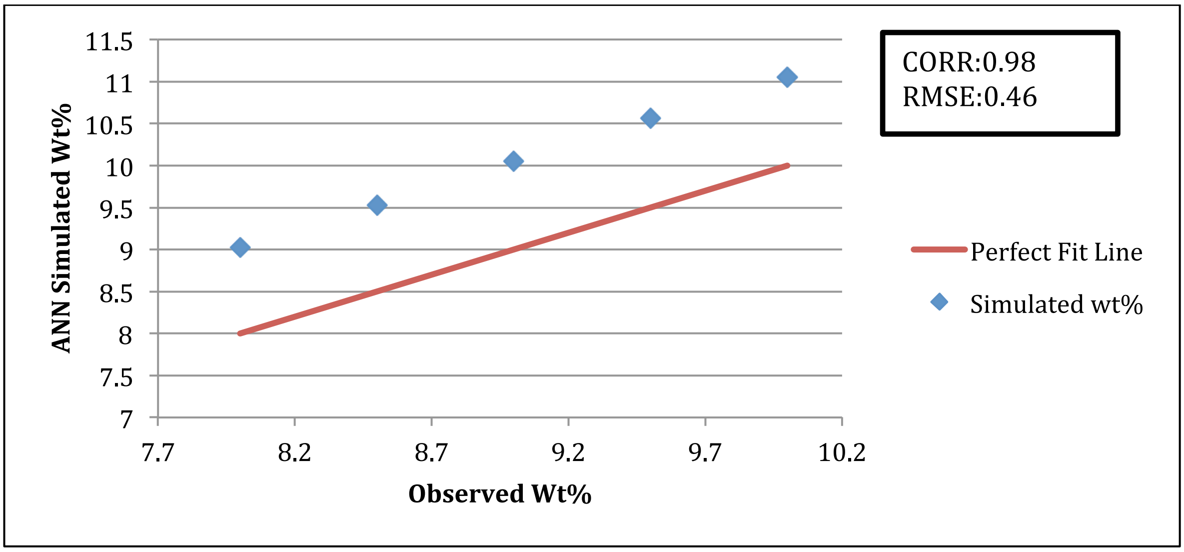

Figure 5.5: Observed vs. predicted wt% of carbon [Input: resistivity and Size of carbon particle]

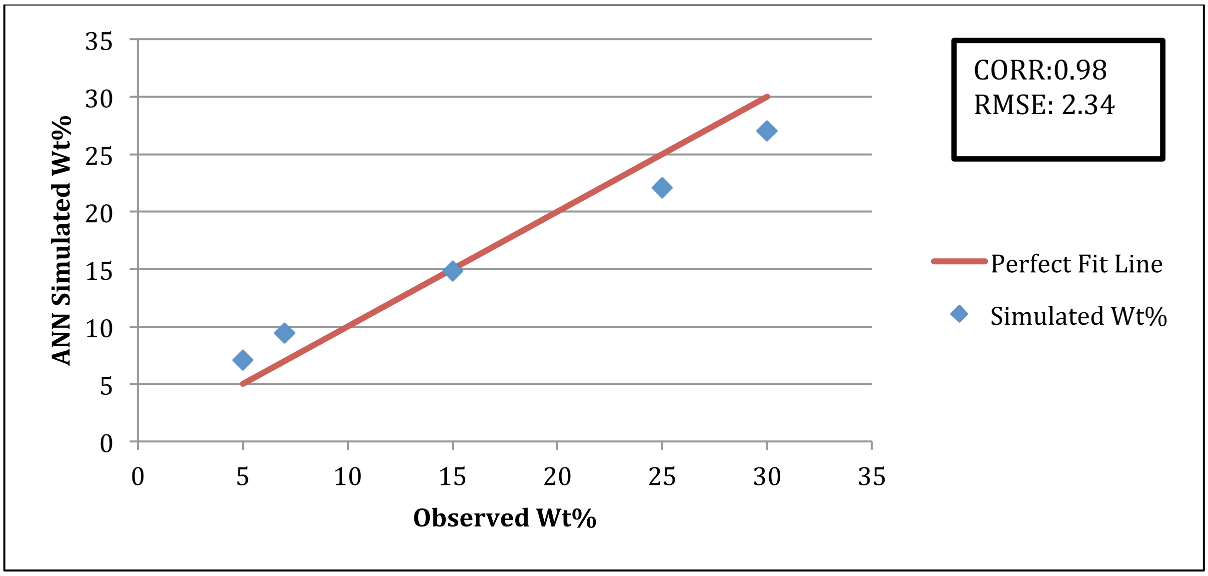

Figure 5.6: Observed vs. predicted wt% of Iron [Input: relative permeability and 10 micrometer Iron]

CHAPTER 6

SUMMARY & CONCLUSION

The fabrication and characterization of composite PLA plastics with electrical and magnetic properties have been performed in this research. Based on the experimental results, an artificial neural network model capable of predicting the parameters required to produce a composite PLA with unique properties has also been developed.

1. Composite PLA plastics with both electrical and magnetic properties have been successfully fabricated by combining carbon and iron particles with pure PLA.
2. The conductivity of the composite improves with the increase in the weight percent of carbon particles.
3. The conductivity of the pure PLA is improved by using smaller sized carbon particles as conductive reinforcement.
4. The iron particles do not influence the electrical properties of the composite PLA. This could be due to the oxidized nature of the iron particles.
5. There is a direct relationship between the observed relative permeability of the composite PLA to the quantity of iron particles in the composite.
6. The dielectric constant of the composite increases with the increase in weight percent of carbon particles up until a certain weight percent, thereafter, the composite is highly conductive and does not behave like a dielectric.
7. The applied training algorithm and number of neurons affects the accuracy of the developed neural network model.
8. The model is most accurate when the trainlm algorithm and 10 neurons are used for its design. Above 10 neurons, the error in the prediction is high; this could be attributed to data overfitting during the training process.

9. Between 80 - 90% of the neural network predicted output parameters are within an error of +/- 10% of the actual experimental values.

6.1 Suggestions for Further Work

1. The mechanical properties of the composite PLA could be improved. In this research, the fabricated samples are brittle due to the method of fabrication employed (compression molding, no heat applied). Further work can be done to improve the mechanical properties of the composite PLA by using different fabrication techniques.
2. The dielectric properties of the composites could be improved. A material with high dielectric properties can be fabricated by reinforcing PLA with carbon particles; however, the carbon particle weight percent in the composite should be very miniscule, less than 0.5wt% is advised. In this case, the material will have an extremely low conductivity.

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