Electrical and Tensile Properties of Carbon Black Reinforced Polyvinyl Chloride Conductive Composites

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Abstract: Conductive polymer composites are becoming more important and useful in many electrical applications. This paper reports on the carbon black (CB) reinforced polyvinyl chloride (PVC) conductive composites. Conductive filler CB was reinforced with thermoplastic PVC by compression molding technique to make conductive composites. The particle size of CB was measured, as it affects the electrical conductivity of the composites. Different types of CB-PVC compression-molded composites were prepared, using CB contents from 5 to 30 wt %. The electrical and tensile properties of these composites were studied and compared. Improved electrical properties were obtained for all CB-PVC conductive polymer composites compared to virgin PVC composite. However, the tensile properties of the CB-PVC composites increased up to 15 wt % CB loading, and then decreased, and elongation at break decreased with increasing CB loading. The structure of the CB, PVC and CB-PVC composites were studied by attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopic analysis. ATR-FTIR spectra provide evidence of the formation of CB-PVC composites. The microstructural analyses showed a good dispersion of CB in PVC matrix.

Keywords: carbon black; polyvinyl chloride; conductive; composites; electrical and tensile properties

1. Introduction

In recent years, the demand for conductive polymer composites has increased dramatically due to their potential application in many areas, including as electromagnetic interference shielding materials, self-regulated heating materials, antistatic coatings, semiconductors, batteries and pressure sensors etc. Different types of conductive fillers, such as carbon black (CB) and metallic powders (Ni, Al, Cu, etc.) have been extensively investigated to effectively improve the electrical conductivity of rubbers and insulating polymers [1]. Electrically conductive chlorosulfonated polyethylene (CSM) composites are widely used in electronic sectors in power distribution, audio, and telephone applications as packaging and semi-conductive polymeric materials due to their good weather resistance, good electrical properties and good aging resistance. The preparation of different types of electrically conductive polymer composites filled with various conductive fillers has already been reported by several researchers [2]. The electrical properties of conductive composites depend mainly on how well the fillers disperse within the matrices, the relevant properties of the filler (e.g., particle size, surface area, aggregate structure and surface activity) and polymer-filler interactions. The incorporation of filler into a polymer enhances the mechanical properties of the final product, and also decreases the cost of the end product. The relation between electrical and mechanical properties of conductive polymer composites has also
been the subject of recent research [1,2]. Polymer/metal composites are extrinsic electrical conductivity materials, and the electrical properties of polymer/metal composites have been the subject of many papers. However, less work has been concerned with the correlations among rheological, mechanical and electrical features [3].

Wiriya et al. [1] reported that a binary composite containing 50 phr carbon black in natural rubber was a superior conductive composite for a pressure sensor, based on its balance of compression set and electrical conductivity. They also found that at the same total filler content, a third metal component filler decreases the electrical conductivity of the binary composites and deteriorates the compression set of the composites. Aleksandra et al. [4] reported on the capacitive properties of polypyrrole/activated carbon composite which can be effectively used in super capacitors and/or batteries. Xiangyang et al. [5] reported on development of a conductive polymer matrix composite with low concentration of the conductive filler by coating process.

Recently, there has been an increase in the use of thermally conductive polymeric composites, where polymers filled with thermally conductive fillers effectively dissipate heat generated by electronic components. The life time of electronic devices is directly dependent on the management of heat. To improve the thermal conductivity of a polymeric material, polymer matrices are usually filled with highly thermally conductive fillers like metal powders, ceramic powders and carbon-based materials. Polymer composites filled with thermally conductive particles have advantages due to their easy processability, low cost, and durability to the corrosion. In particular, carbon-based 1-dimensional nanomaterials such as carbon nanotube (CNT) and carbon nanofiber (CNF) have gained much attention for their excellent thermal conductivity, corrosion resistance and low thermal expansion coefficient in comparison to metals [6]. The composites made by incorporating powdery metal fillers into polymers combine the advantages of metals and plastics. Therefore, conductive polymer composites are also essential to some electrical applications, where their properties are relevant in situations such as the discharge of static electricity, heat conduction, electromagnetic interference shields and switching [7]. A high level of electrical conductivity is obtained when a continuous conducting path is formed at a critical concentration of conductive filler particles. This concentration is called the percolation threshold. A melt-blending or coating process is often used to disperse the conductive particles into the polymer matrix, as conductive filler dispersion (or the coating on the polymer) is the most important stage in the preparation of conducting polymers [5].

Rungsima et al. [8] worked on carbon filler-reinforced polypropylene composites by injection-molding technique for a bipolar plate application. They reported that the orientation of carbon fibers in composites is affected by the injection flow geometry. Ritu et al. [9] worked on conducting polymer composites of polyvinyl-pyrrollidione (PVP) with potassium hydroxide (KOH). They used solution-cast techniques to prepare composites, and investigated the structural and mechanical properties of the polymer composite specimen. The results revealed that the conductivity and hardness of the polymer composite increased with the concentration of KOH. Kristin et al. [10] investigated PVC composites incorporating multi-walled carbon nanotubes (MWCNT) by three roll mill techniques for use in antistatic coatings. They reported that CNT contents ≤0.5% are sufficient for use in antistatic coatings. Remo et al. [11] worked and reported on conducting composites of multi-walled carbon nanotubes (MWCNTs) and ionic liquid (IL)-functialized nano fillers (IL-f-MWCNTs) with ethylene octane copolymers (EOC). They observed that MWCNTs and IL-f-MWCNTs were both effective in increasing the storage modulus, tensile modulus, stress-at-break, and electrical conductivity of EOC-based nanocomposites with increasing nano filler content. Bahaa et al. [12] studied the effect of filler content on DC electrical properties of zinc powders filled polyvinyl chloride composites. They reported that the DC electrical conductivity of the polyvinyl chloride increases with increasing zinc concentrations and temperature. Cheng-Ho et al. [13] reported on rigid and conductive PVC/CB composites prepared by Haake torque rheometer. They observed that the rigidity of the PVC/CB composite increased with an increase in CB content, thus also increasing the electrical and mechanical properties. Saad et al. [14] worked on the electrical and mechanical properties of polyvinyl chloride (PVC) mixed with electrically
Conductive additives, and reported that a carbon black (CB)-filled PVC–CB system containing a high CB content possesses a much higher electrical conductivity, but a higher CB loading causes deterioration of the mechanical properties.

Jinhong et al. [15] provided a review of the electrical, thermal and mechanical properties of graphene/polymer composites fabricated by solution-casting method. They also provided guidance for stimulating the development of wider applications for conductive polymer composites and nano composites in structural, functional and electrical areas. Conductive filler-filled thermoplastic composites and nanocomposites could also be used for structural and functional applications in spite of the present predominance of electrical applications [15,16]. Carbon fiber-reinforced polymer composites have been demonstrated to have many applications in diverse fields, including the aerospace and automotive industries, and related to engineering components such as bearings, gears, fan blades, and chassis structures, due to their ability to obtain a synergetic effect when combining the toughness of the thermoplastic polymer with the stiffness and strength of carbon fibers [17]. Recently, extensive studies have focused on the effect of carbon fibers for developing conductive thermoplastic composites [18]. Tailorable properties provide applications for conductive carbon-based composite materials in many industries as electronic and constructional components: in microelectronics, antistatic, electrostatic dissipation, in electromagnetic shield fabrication, for aircraft structures, aircraft brakes, concrete structures, etc. [18–20]. Very few research works were found on carbon black-reinforced thermoplastic composites through compression-molding processing techniques for electrically conductive applications. Carbon black-based conductive composites produced by compression-molding processing techniques can be used in a wide range of applications in the structural and electrical areas. Selection of composite materials for the proper end-use application also depends on the desired mechanical and electrical properties. In this research, carbon black (CB) was used to reinforce thermoplastic polymer polyvinyl chloride (PVC) in order to prepare CB-PVC composites using different wt fractions of CB by compression-molding technique. This research work focuses on the manufacturing process and the properties of the CB-PVC composites in order to determine their potentialities in both electrical and structural applications. Thus, the aim and objective of this work is to assess the effect of CB loading on the electrical and tensile properties of compression-molded CB-PVC composites. The influence of CB content on the structure of CB-PVC composites was also studied by ATR-FTIR spectroscopic analyses and microstructural analyses.

2. Materials and Methods

2.1. Materials

A reagent-grade carbon black (activate charcoal) was used in this study. It was made by Uni-Chem, Wuxi, China. A commercial-grade polyvinyl chloride (PVC) was used in this study, and it was supplied by Thai Plastic and Chemicals Public Company Limited.

2.2. Analyses of PVC

Measurement of moisture content of PVC:

\[ \text{% of Moisture} = \frac{(\text{Initial wt} - \text{Final dried wt})}{(\text{Initial wt})} \times 100 \]

Measurement of ash content of PVC:

\[ \text{% of Ash} = \frac{(\text{Weight of ash})}{(\text{Weight of sample})} \times 100 \]

The melting point of PVC was determined using a Gallenkamp melting point apparatus, made in London, UK.
Moisture content, ash content and melting point of PVC were measured, and had values of 1.25%, 0.03% and 185 °C, respectively.

2.2.1. Particle Size Measurement of PVC

The particle size of granular PVC was measured using an analytical sieve machine (Fritsch, Idar-Oberstein, Germany). In this experiment, the distribution of particle sizes of PVC were measured by sieve analyses using 60, 70, 100 and 120 mesh. There was a mixture of different particle sizes of PVC ranging from 125 to 177 µm.

2.2.2. Particle Size Measurement of Carbon Black

The experiment was carried out using 4 wt % CB with de-ionized water, which was diluted in water at a ratio of 1:100 (v/v) at a temperature of 25 °C. The sample was then ultrasonicated for 30 min in an ultrasonic bath (Unique, model: VGT-1860, Berlin, Germany) (40 kHz ultrasound frequency, 150 W ultrasonic power). The particle size of the CB was measured using a Malvern Zetasizer Nano ZS (Model: ZEN 3600, Worcestershire, UK). A disposable cuvette DTS0012 was used to measure size. The average size value of the CB was measured based on the values of three measurements. The results are presented in the results and discussion section.

2.3. Preparation of Carbon Black (CB)-Polyvinyl Chloride (PVC) Conductive Composites by Compression-Molding Method

Dried granulated PVC and CB were mixed in a blender for one minute at a speed of 1000 rpm and molded by a compression-molding machine (Paul–Otto Weber Press Machine) at a molding temperature of 185 °C. The pressure, temperature, heating time and cooling time of the samples were controlled at the same rate in the preparation of all composites. The applied pressure was 200–250 kN, and the heating time was 10 min. After the completion of the heating, an additional pressure of 50 kN was applied in order to obtain void-free compression-molded specimen. The specimen was allowed to cool and cure by tap water through the outer area of the heating plates of the hydraulic press machine. The completely cured composite was taken out of the mold. The compression-molded composite was circular in shape and had a diameter of 14 cm and a thickness of 4 mm. The sample was then cut to make specimens of suitable dimensions for different tests. The composites with a high CB loading of over 30 wt % were too brittle and unstable to measure any properties. Different types of composites were prepared, and their compositions are presented in Table 1.

<table>
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<tr>
<th>Reinforcing Material CB (%)</th>
<th>Polymer Matrix PVC (%)</th>
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2.4. Mechanical Properties of the Conductive Polymer Composites

In order to investigate the mechanical properties of the CB-PVC composites, tensile tests of all composites were carried out. For these tests the appropriate American Society for Testing and Materials (ASTM) methods were followed.
A Universal Testing Machine (Model-Titan5, Brand-James Heal, Halifax, UK) was used to perform tensile properties for all specimen groups. Tests were performed according to ASTM D882-02, (Standard Test Method for Tensile Properties of Thin Plastic Sheeting). The speed of the tensile test was 20 mm/min, and the dimensions of the specimens were 128 mm length, 10 mm width and 4 mm thickness. The average values and standard deviations of tensile strength and elongation-at-break were calculated using the five maximum values selected from the test results of seven specimens in each group. The load vs. elongation curves were obtained from the instrument. The highest load in the tensile test gives the ultimate tensile strength ($\sigma_{UT}$), and was calculated from the following equation:

$$\text{Tensile strength, } \sigma_{UT} = \frac{W}{A_T}$$

where $W$ is breaking load and $A_T$ is the cross-sectional area.

### 2.5. Electrical Properties of the CB-PVC Conductive Polymer Composites

In order to investigate the electrical properties of the prepared CB-PVC composites, the four-probe method was used to measure the DC electrical specific conductivity ($\sigma_{dc}$) at room temperature.

#### 2.5.1. Four-probe Method for Resistivity

In the four-probe method, a battery serves as the DC power source, a volt meter measures the potential difference across the sample, and an ammeter measures the current in the circuit. Using this method, a small current in the microampere range ($\mu A$) was passed through the sample and the voltage, i.e., emf, across the sample was monitored as a function of temperature at constant supply voltage. The resistances (R) of the composites were measured using Ohm’s law. The resistivity ($\rho$) and the conductivity ($\sigma_{dc}$) of the composites were calculated using the dimensions of the composites.

#### 2.5.2. Calculation of Specific DC Conductivity

The specific conductivities ($\sigma_{dc}$) of all composite specimens were measured by the application of Ohm’s law. The specific conductivities ($\sigma_{dc}$) are defined as the inverse of resistivity ($\rho$). The amount of electrical current which flows through the sample is restricted by the amount of resistance (R) present.

The electric resistances of all samples have been measured using the Equation (1) which is the ratio of potential difference across the sample and the amount of current flowing through the sample:

$$\text{Resistance (R)} = \frac{V}{I}$$  \hspace{1cm} (1)

where, $V =$ voltage and $I =$ current pass.

$$\text{Resistivity (} \rho \text{)} = RA/l$$  \hspace{1cm} (2)

where, Area of the sample $A = \pi r^2$; $r =$ radius of sample, $l =$ thickness of sample and $R =$ Resistance.

$$\text{Specific conductivity (} \sigma_{dc} \text{)} = 1/\rho$$  \hspace{1cm} (3)

where, $\rho =$ Resistivity.

### 2.6. Structural Characterization of PVC, CB and CB-PVC Composites

#### 2.6.1. ATR-FTIR Spectroscopic Characterization of PVC, CB and CB-PVC Composites

Attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR) spectra were taken to study the structure of PVC, CB and CB-PVC composites. The spectra were recorded on a FT-IR/NIR Spectrometer (Forn-tier, PerkinElmer, Waltham, MA, USA) using ATR sampling technique
in the wave number range of 650 to 4000 cm\(^{-1}\). To take ATR-FTIR, the samples were used directly in the solid form, and the spectra were obtained in printed form.

2.6.2. Microstructural Characterization of CB, PVC and 30 wt % CB-PVC Composites

The microstructure of CB, PVC and 30 wt % CB-PVC composites and the dispersion of CB in the PVC matrix were analyzed using optical microscopy (Model: Humanscope, Cat.No-16100, Wiesbaden, Germany) at 100 times magnification. The microscopic images of samples were captured by camera. CB and PVC were used directly to take images. The tiny pieces of 100% PVC and 30 wt % CB-PVC composites were used to take images.

3. Results and Discussion

In this work, conductive polymer composites were prepared, and their electrical and mechanical properties were studied. PVC polymers in the 100–177 \(\mu\)m size range were used as matrix materials. Nirvana et al. [18] reported on three types of PVC-based conductive composites, with carbon black, carbon fiber and synthetic graphite, respectively. They reported that carbon black was more effective than carbon fiber and synthetic graphite at low weight fractions. In this research, carbon black (CB) was used as a conductive ingredient to prepare conductive composites. The particle size of CB used in this research was measured, and was found to be 2705 nm or 2.70 \(\mu\)m (Figure 1). The conductivity of the composites depends on the shape, particle size and properties of the conductive fillers applied [18].

The DC conductivity of the prepared CB-PVC compression-molded composites was measured by applying four probe methods at room temperature. The results of DC conductivity obtained in this study are presented in Figure 2. It is observed from Figure 2 that the DC conductivity of the composites increases with increasing CB loading. The electrical conductivity of the composites increases with increasing the conductive filler content [9,12,21]. Bahaa [22] stated that the increase in conductivity with increased concentration of conductive filler was due to the increase in the charge carriers, which increase with increasing filler contact. These filler contact networks generate electrical contacts and enhance the conductivity. In this research, the DC conductivity (\(\sigma_{dc}\)) of 100% PVC was found to be 0 S·cm\(^{-1}\), or could be so small that it was undetectable with the setup used.

![Graph of size distribution by intensity of carbon black](image-url)

Figure 1. Size distribution by intensity of carbon black.

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Tensile strength and elongation-at-break of the different types of CB-PVC composites were determined following the ASTM method described in the experimental section. The tensile strengths of the CB-PVC compression-molded composites are presented in Figure 3. It is found from Figure 3 that the tensile strength of 100% PVC composite is higher than that of all CB-PVC composites. The lower tensile strength of the CB-PVC composite system is due to the inability of the filler particles to transfer the applied stress for weak interfacial adhesion between the filler and matrix molecules [7,23,24]. It is also found from Figure 3 that the tensile strengths of the CB-PVC composites increase with increasing CB content up to a CB loading of 15 wt %, and then decrease. This may be due to vander Waals force between CB molecules in the CB-PVC composites. The distance between the CB molecules decreases at higher loading of CB, so they tend to stack together due to the vander Waals force in the composites. This tendency of CB reduces their uniform dispersion and load transfer in the CB-PVC composites. As a result, tensile strength of the CB-PVC composites decreases at CB loadings greater than 15 wt %. A similar observation was reported by Jinhong et al. [15]. Enoki et al. [25] reported that when a component with low molecular weight is increased in a matrix, then the tensile strength decreases. Sahai et al. [26] worked on and reported the effect of particle size and concentration of fly ash on the mechanical properties of polyphenylene oxide composites. They reported on the effect of particle size on tensile behavior, and stated that filler agglomerate with lower particle size at higher concentration reduced the tensile strength of composites with a filler loading above 10 wt %. Bikiaris [27] reported on multi-walled carbon nanotube (MWCNT)/polypropylene (PP) nanocomposites, and this review also stated that at low content, MWCNTs act as reinforcement agents, increasing the tensile strength of nanocomposites, but that at higher MWCNT contents, the size of aggregates increases and the formed aggregates act as mechanical failure concentrators. Thus, MWCNTs/PP nanocomposites exhibiting their mechanical performance at concentrations of 2–2.5 wt %, while with a further increase of MWCNT content, the tensile strength of PP/MWCNTs nanocomposites decreases. In this research, the lower particle size CB fillers may have a tendency to agglomerate at high filler contents of more than 15 wt %, resulting in improper bonding between the CB and the PVC, such that the tensile strength of CB-PVC composites decreases with the addition of CB above 15 wt %. At low CB content (5–15 wt %), partial tensile strain can be transferred to CB embedded in the PVC matrix under tensile stress, which leads to an increase in tensile strength [27]. However, elongation-at-break of the CB-PVC composites decreases with increasing PVC content (Figure 4). Elongation is the percentage increase in original length (strain) of a specimen as a result of tensile force (stress) being applied to the specimen [28]. It also indicates that

![Figure 2. Conductivity vs. Carbon Black loading (wt %) chart for Carbon Black-Polyvinyl Chloride composite.](image-url)
the toughness or ductility of the CB-PVC composites decreases with increasing CB loading. The error bars of Figures 3 and 4 show the standard deviation of tensile strength and elongation-at-break for all of the composites. The low standard deviations found in each group of composites indicate good uniformity between the samples. These small standard deviations of composites are due to variations in the sample manufacturing processes [29].

![Figure 3](image3.png)

**Figure 3.** Tensile strength (MPa) vs. CB loading (wt %) chart for CB-PVC composites.

![Figure 4](image4.png)

**Figure 4.** Elongation at break (%) vs. CB loading (wt %) chart for CB-PVC composites.

The structures of the CB-PVC composites were studied by ATR-FTIR spectroscopic analysis. ATR-FTIR spectra of the PVC, CB and three types CB-PVC composites are presented in Figure 5.
Anjalin [30] also reported on the structure of a multi-walled carbon nanotube (MWCNT)-filled poly (vinylidene fluoride) (PVDF) nanocomposite by FTIR spectroscopic analysis. In Figure 5a, the characteristic bands of PVC found at 2916 and 1426 cm\(^{-1}\) are due to the CH\(_2\) stretch. The bands at 1330, 1253 and at 695 cm\(^{-1}\) are related to the CH bend out of phase, the CH bend into phase, and the C–Cl bonds respectively. From Figure 5b, it is observed that the characteristic bands of CB found at 1739, 1578 and 1365 cm\(^{-1}\) are due to the C=O, C–O and C–C bond vibrations, respectively. An almost identical FT-IR spectrum of carbon was reported by Meytij et al. [19]. All of these characteristic bands of CB and PVC are found in the CB-PVC composites in Figure 5c–e, respectively. The broad band near 3400 cm\(^{-1}\) in Figure 5e indicates the presence of hydroxyl groups on the carbon surface. The hydroxyl groups came from water that had been absorbed by the carbon black [19]. The peak intensity of the hydroxyl groups increased from 10 wt % CB-PVC composite to 30 wt % CB-PVC composite with increasing CB content. It is also found from the ATR-FTIR spectra of the CB-PVC composites that the intensity of CB increases with increasing of CB loading in the composites, except for the 30 wt % CB-PVC composite. In 30 wt % CB-PVC composite, the peak intensity of both CB and PVC decreases, but the peak intensity of the hydroxyl group increases due to the presence of more CB.

![ATR-IR spectra](image)

**Figure 5.** ATR-IR spectra of (a) PVC; (b) CB; (c) 10 wt % CB-PVC composite; (d) 20 wt % CB-PVC composite and (e) 30 wt % CB-PVC composite.

Microstructural characterization of CB, PVC particles, 100 wt %PVC and 30 wt % CB-PVC composites were performed by optical microscopy. Dispersion of the filler particles in the composites was evaluated by optical microscopic images analysis [12,22,31,32]. In this work, the microscopic structures of all ingredients and the filler dispersion in the composites were observed by microscopic image analysis. The 100 times magnified images of PVC (125–177 μm) and CB (2.70 μm) particles are shown in Figure 6a,b, respectively. It is clear from the figures that the CB and PVC are mixtures of different sizes of particles. The tiny pieces of 100% PVC and 30 wt % CB-PVC composites were examined using the microscope to observe the microstructure and filler dispersion. The microstructural images of 100 wt % PVC and 30 wt % CB-PVC composites at 100 times magnification are shown in Figure 7a,b, respectively. The microstructural analysis of the composites indicates a good dispersion of CB throughout the PVC.
The DC conductivity of the composites increases with increasing CB loading in the CB-PVC composites. These tensile and electrical characteristics of the compression-molded CB-PVC composites showed that the tensile strength of the composites increased with an increase in CB loading up to 15 wt %, and then decreased, but elongation at break decreased with increasing CB loading. Therefore, the ductility and toughness of the composites decrease with increasing CB loading. The structure of the CB-PVC composites was confirmed by ATR-FTIR spectroscopic analysis. Microstructural analysis also exhibited a uniform dispersion of CB particles in the PVC matrix. The electrical and tensile properties of the compression-molded CB-PVC composites were stable up to 30 wt % loading of CB. In this work, higher conductivity was found for 30 wt % CB-loaded CB-PVC composites. The tensile properties of CB-PVC composites showed that the tensile strength of the composites increased with increasing CB loading, but the tensile strength of the composites increased with the addition of CB up to 15 wt %. These tensile and electrical properties of the composites may give them potential in both electrical and structural applications in the future.

4. Conclusions

CB-PVC composites can be prepared by the compression molding method. CB-PVC compression-molded conductive composites can be applied in many fields of electronic and structural materials. The DC conductivity of the composites increases with increasing CB loading in the CB-PVC composites. Compression-molded CB-PVC composites are stable up to 30 wt % loading of CB. In this work, higher conductivity was found for 30 wt % CB-loaded CB-PVC composites. The tensile properties of CB-PVC composites showed that the tensile strength of the composites increased with an increase in CB loading up to 15 wt %, and then decreased, but elongation at break decreased with increasing CB loading. Therefore, the ductility and toughness of the composites decrease with increasing CB loading. The structure of the CB-PVC composites was confirmed by ATR-FTIR spectroscopic analysis. Microstructural analysis also exhibited a uniform dispersion of CB particles in the PVC matrix. The electrical and tensile properties of the compression-molded CB-PVC composites revealed that the electrical properties of the composites increased with increasing CB loading, but the tensile strength of the composites increased with the addition of CB up to 15 wt %. These tensile and electrical properties of the composites may give them potential in both electrical and structural applications in the future.
characteristics of the compression-molded CB-PVC composites may give them potential in both electrical and structural applications in the future.

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

References


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