Introduction

Polylactic acid (PLA) is a renewable polyester that can replace typical non-degradable plastics, which has been approved by the US Food and Drug Administration (FDA) for applications in clinical medicine and has been widely used. PLA exhibits several advantages including good biocompatibility as well as excellent mechanical strength and barrier property. On the other hand, it exhibits disadvantages of large volume resistivity (>10^{15} \Omega \cdot cm), facile accumulation of static electricity, slow crystallization speed, and poor thermal stability, limiting its applications in high-value-added fields, such as medical and electronic materials. For example, by using PLA as medical protective clothing and surgical gowns, dust and bacteria are easily survived. By using PLA as electronic industrial products, damage to the integrated circuit is inevitably caused. By using PLA in protective clothing and filter materials in mining and petrochemical industries, spontaneous fire and explosion are inevitable.

Meanwhile, multi-walled carbon nanotube (MWCNT) constitutes one of the hardest and strongest materials known, with advantages of good toughness, high thermal stability, and thermal conductivity.
and excellent electrical and thermal conductivities.\textsuperscript{6-8} MWCNT has been applied in various composite materials, serving as ideal nanofiber filler material for PLA. In this study, PLA and MWCNT were melt-blended for overcoming the disadvantages of PLA, including high resistivity and poor thermal stability, and obtaining PLA/MWCNT composites with good electrical conductivity, heat resistance, and mechanical property.

Demand for protective clothing has increased as new coronavirus outbreaks have erupted around the world. The main raw material of medical protective clothing is undegradable polypropylene non-woven fabric, and a large amount of waste causes great pressure on the environment. If biodegradable antistatic non-woven composites were used to prepare protective clothing, it can effectively solve the above problems and reduce the pressure on the environment.

**Materials and instruments**

**Materials**

MWCNT (FT-7000), with a tube diameter of 8 to 15 nm and a tube length of 5 to 20 $\mu$m, was commercially obtained from Tiannai (Zhenjiang) Material Technology Co., Ltd. PLA (6100D) was purchased from US Nature Works Company.

**Instruments**

XS-60 rubber plastic mixing machine with an LH60 mixer (Shanghai Kechuang Rubber & Plastic Equipment Co., Ltd.), swing-type high-speed grinder (Ruian Yongli Pharmaceutical Machinery Co., Ltd.), STA 449 F5 synchronous thermal analyzer (Germany NETZSCH Company), DZF-6020 vacuum drying box (Shanghai Boxun Industry & Commerce Co., Ltd.), WDW-1 microcomputer control electronic universal testing machine (Jinan Yinuo Century Test Instrument Co., Ltd.), ZC-90G high-insulation resistance measurement instrument (Shanghai Tajiou Electronics Co., Ltd.), and CH-0206 molding machine (Dongguan Chuanghong Instrument Equipment Co., Ltd.).

**Preparation of PLA/MWCNT composites**

First, PLA and MWCNT were dried under vacuum at 45°C for 12 h. Then, the dried PLA and MWCNT were added into the XS-60 rubber-plastic blender for melt-blending. PLA was first added, followed by the MWCNT after the melting PLA claded the hot rolls. The blending temperature was 190°C, with a blending time of 5 min and a blending speed of 50 r/min.

Second, the PLA/MWCNT composites were cooled at room temperature after melt-blending and stored in a desiccator. Then, the PLA/MWCNT composites were pressed by CH-0206 molding machine at 200°C under 10 MPa for 8 min to make plates. Next, the plates were cut in to a small sheet of 10 cm $\times$ 10 cm for conductivity measurement, and the remaining plates were cut into a dumbbell-shaped (4 mm $\times$ 75 mm) for measuring of the tensile strength. The PLA/MWCNT composites and dumbbell-shaped plates were dried before performance test and characterization.

PLA/MWCNT (99/1) refer to a mass ratio of PLA and MWCNT in the composite is 99:1.

**Performance test and characterization**

**Conductivity test.** According to the resistivity test method (National Standard GB/T1410-2006), at room temperature with a controlled relative humidity of 60% to 70%, the 10 cm $\times$ 10 cm PLA/MWCNT composite sheet was placed into an electrode box, in which the metal electrode block was added. The circuit was connected according to the connection mode shown in Figure 1. The displayed data were recorded after 1 min, representing the measured
volume resistance $R_s$ or the surface resistance $R_s$ for the PLA/MWCNT composites. After discharging the sample sheet, the above-mentioned measurement was repeated 5 times, and the average value was taken. Then, the volume resistivity $\rho_v$ and the surface resistivity $\rho_s$ of the sample were calculated according to Formulas (1) and (2), respectively.

$$\rho_v = R_s \frac{A}{h} \quad (1)$$

where $\rho_v$ = Volume resistivity, $\Omega$·cm.

$A$ = Effective area of the protected electrode (21.24 cm$^2$), cm$^2$.

$h$ = Sample average thickness, cm,

$$\rho_s = R_s \frac{P}{g} \quad (2)$$

where $\rho_s$ = Surface resistivity, $\Omega$.

$P$ = Effective circumference of the protected electrode (16.34 cm), cm.

g = Distance between the two electrodes (0.2 cm), cm.

**Mechanical properties.** According to the National Standard GB/T1040-92 plastic tensile performance test, the tensile strength $\sigma$ and elongation at break $\varepsilon$ of the PLA/MWCNT composites were measured at room temperature by using WDW-1 microcomputer controlled electronic universal testing machine. The stretching speed was 15 mm/min.

**Thermogravimetric analysis.** Approximately 5 mg of PLA/MWCNT composite was loaded into an alumina crucible and placed in the STA449 F5 synchronous thermal analyzer sample cell under high-purity N$_2$ gas sweeping with a flow rate of 50 mL/min. The temperature was increased from 30°C to 500°C at a rate of 10°C/min to obtain thermogravimetric analysis (TGA) curves and the first-order differential thermogravimetric (DTG) curve of the composite.

**Differential scanning calorimetry.** Approximately 5 mg of PLA/MWCNT composite was loaded into an alumina crucible and placed in the STA449 F5 synchronous thermal analyzer sample cell under high-purity N$_2$ gas sweeping at a flow rate of 50 mL/min. The temperature was increased from 30°C to 200°C at a rate of 10°C/min to obtain a differential scanning calorimetry (DSC) curve of the composite.

The crystallinity $X_c$ of the composite was calculated according to Formula (3):

$$X_c = \frac{\Delta H_m - \Delta H_{c1} - \Delta H_{c2}}{\omega_{PLA} \cdot \Delta H_m^{PLA}} \quad (3)$$

where $\Delta H_m$ is the enthalpy absorbed by the PLA/MWCNT composite during the melting, J/g.

$\Delta H_{c1}$ is the enthalpy released during the cold crystallization of the PLA/MWCNT composite, J/g.

$\Delta H_{c2}$ is the enthalpy released by the $\alpha'$ crystal form of the PLA rearranged into the $\alpha$ crystal form of the PLA, when the composite approaching the melting temperature, J/g.$^9$

$\omega_{PLA}$ is the mass fraction of PLA in the sample.

$\Delta H_m^{PLA}$ is the ideal melting enthalpy for 100% crystalline PLA (93.1 J/g).$^{10}$

**Scanning electron microscopy.** The internal microstructure of the PLA/MWCNT composite was observed using an S-3500 N-type scanning electron microscope (Hitachi Company). The sample is prepared by freezing PLA/MWCNT composite in liquid nitrogen followed by application of a high-speed impact to create freshly fractured surfaces. The fractured surface was covered with gold before observation.

**Results and discussion**

**Conductivity measurements**

First, MWCNT and PLA were melt-blended according to the ratio of the added MWCNT, which was 0.0 wt%, 0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%, and 3.0 wt%, of the total mass of the composite, respectively. The results of volume resistivity $\rho_v$ and surface resistivity $\rho_s$ are shown in Table 1. $\rho_v$ represents the quotient of the direct-current electric field strength and the steady-state current density in the material, that is, the volume resistance per unit volume. $\rho_s$ represents the quotient of the direct-current electric field strength in the surface layer and the linear current density of the material, that is, the surface resistance per unit area.

As shown in Table 1, with the increase in the loading amount of MWCNT, the $\rho_v$ and $\rho_s$ of the PLA/MWCNT composites decreased considerably. With a MWCNT loading of 3.0 wt%, $\rho_v$ decreased from $1.95 \times 10^{15}$ $\Omega$ cm for pure PLA to $2.48 \times 10^{6}$ $\Omega$ cm. With a MWCNT loading of 3.0 wt%, $\rho_s$ also decreased by 9 orders of magnitude.

| Sample | Surface resistivity $\rho_v$ ($\Omega$) | Volume resistivity $\rho_s$ ($\Omega$·cm) |
|--------|--------------------------------------|----------------------------------------|
| PLA    | $1.95 \times 10^{15}$                | $5.20 \times 10^{15}$                  |
| PLA/MWCNT (99.5/0.5) | $3.99 \times 10^{14}$ | $7.31 \times 10^{14}$                  |
| PLA/MWCNT (99.0/1.0) | $1.42 \times 10^{12}$ | $6.95 \times 10^{11}$                  |
| PLA/MWCNT (98.5/1.5) | $2.08 \times 10^{8}$   | $3.00 \times 10^{8}$                   |
| PLA/MWCNT (98.0/2.0) | $1.63 \times 10^{7}$   | $5.42 \times 10^{7}$                   |
| PLA/MWCNT (97.0/3.0) | $2.48 \times 10^{6}$   | $9.98 \times 10^{6}$                   |
Currently, three conductive mechanisms exist for the composite materials prepared using conductive fillers: 

(a) Conductive path mechanism: The conductive filler in the composite material contact each other, yielding a chain-shape conductive path, endowing the material with conductive performance. (b) Tunneling effect mechanism: with the sufficiently near distance of the conductive filler, the electrons can jump over the potential barrier of the matrix to the adjacent conductive filler, forming a large tunnel current under thermal vibrations. (c) Field-emission mechanism: under the action of a strong internal electric field, even with a large distance of the conductive filler, the electrons can jump over the base potential and completely transition into the adjacent conductive filler, affording a field-emission current.

PLA/MWCNT composites are known as composites comprising the conductive nanofiller MWCNT filled with PLA. Combined with the analysis shown in Table 1, the conductivity of the composite material clearly improved considerably; that is, the surface resistivity $\rho_s$ decreased with an MWCNT loading of 0.5 wt% to 1.5 wt%, and the volume resistivity $\rho_v$ in the content interval from 0.5 wt% to 2.0 wt%. Resistivity significantly decreased in these intervals, corresponding to the transformation of the conductive mechanism in PLA/MWCNT composites.

With the increase in the loading amount of MWCNT, the formation threshold of the lowest conducting path (i.e. the percolation threshold) was exceeded. As shown in in Figure 2, the actual conductive mechanism of the network was transformed from tunneling effect mechanism dominated to conductive path mechanism dominated. After reaching the percolation threshold, a conductive network was constructed, and a clear conductive effect was achieved with the further increase in the loading amount of MWCNT.

The surface resistivity $\rho_s$ reached the percolation threshold at a lower concentration than the volume resistivity $\rho_v$ because during the melting and blending of composite materials, the MWCNT tended to accumulate on the surface of PLA/MWCNT composites due to the interfacial effect, leading to a relatively higher content of MWCNT on the surface of PLA/MWCNT composites. This high content of MWCNT makes it more easier to form a conductive network. Therefore, with the same loading amount of MWCNT, the surface resistivity was relatively smaller, and the threshold was also relatively easier to achieve. Thus, with a loading amount of 1.5 wt% to 3.0 wt% of MWCNT, $\rho_s$ decreased more significantly compared to $\rho_v$, which has reached the percolation threshold.

**Mechanical properties**

MWCNT and PLA were melt-blended, hot-pressed into a plate, and cut into dumbbell-shaped (4 mm × 75 mm) with MWCNT loading amounts of 0.0 wt%, 0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%, and 3.0 wt% of the total mass of composite. Then, the tensile strength $\sigma$ and elongation at break $\varepsilon$ of the composites were measured by WDW-1 microcomputer control electronic universal testing machine, the results are shown in Figures 3 and 4.
As shown in Figure 3, within the scope of the investigation, with the increase in the MWCNT loading amount, the $\sigma$ of the PLA/MWCNT composite first increased and then decreased. With a loading amount of MWCNT of 1.0 wt%, the maximum tensile strength was $62.04 \pm 1.43$ MPa, which was increased by $9.66$ MPa compared to $52.38 \pm 1.27$ MPa for pure PLA. Subsequently, with a MWCNT loading of 1.5 wt%, $\sigma$ significantly decreased, while with the further increase in the MWCNT loading amount to 2.0 wt% and 3.0 wt%, a significant change was not observed possibly because as one of the enhanced nanofillers, MWCNT added in the PLA matrix rendered an enhanced effect at a lower content. Moreover, MWCNT played a role in the heterogeneous nucleation of the PLA matrix, which decreased the initial nucleation (induction) of PLA; accelerated the crystallization rate of PLA; and increased the crystallinity of PLA; hence, the tensile strength increases. The PLA matrix exhibited a limited bearing capacity for MWCNT, for example, with an MWCNT loading of 1.5 wt%, MWCNT formed a conductive path in the PLA matrix, and more stress concentration points were observed, leading to the facile expansion of the internal cracks during tensile tests; hence, the tensile strength decreases accordingly.

Similarly, as shown in Figure 4, under the combined action of various aspects of MWCNT, with the increase in the loading amount of MWCNT, the $\varepsilon$ of the PLA/MWCNT composites first increased and then decreased. With an MWCNT loading of 1.0 wt%, the highest $\varepsilon$ was observed ($4.10 \pm 0.33\%$). This value increased by 0.46% compared with that of pure PLA ($3.64 \pm 0.23\%$) because of the high toughness of MWCNT. The MWCNT dispersed in the PLA matrix exhibited a toughening effect at low concentrations. With the increase in the loading amount, MWCNT gradually contacted each other to form a conductive network. Then, the weak phase interface between MWCNT and PLA led to the facile expansion of the internal cracks of the composites, and the elongation at break decreased accordingly.

**Figure 4.** Elongation at break $\varepsilon$ of PLA/MWCNT composites.

**Figure 5.** TGA curves (a) and DTG curves (b) of PLA/MWCNT composites.

**TGA**

MWCNT and PLA were melt-blended with the MWCNT loading amount of 0.0 wt%, 0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%, and 3.0 wt% of total mass of the composite. Then, TGA curves and DTG curves of the PLA/MWCNT composites were recorded on the STA449 F5 synchronous thermal analyzer as shown in Figure 5(a) and (b), respectively. Table 2 summarizes the 5% decomposition temperature ($T_{5\%}$), 50% decomposition temperature ($T_{50\%}$), 95% decomposition temperature ($T_{95\%}$), and maximum decomposition temperature ($T_p$) data of the PLA/MWCNT composites, where $T_{5\%}$, also known as the initial thermal degradation temperature, is an important parameter for evaluating the heat resistance of the PLA/MWCNT composites.

As can be observed in Figure 5(a) and (b) and Table 2, with the increase in the MWCNT loading amount, the $T_{5\%}$ of the composites increased by 6.45°C, 8.54°C, 9.91°C, 8.16°C, and 4.01°C compared with that of pure PLA. $T_{50\%}$ increased by 3.10°C, 3.12°C, 3.64°C, 3.56°C, and 2.46°C compared with that of pure PLA. $T_p$ increased by 1.11°C, 2.09°C, 1.99°C, 1.91°C, and 1.09°C compared with that of pure PLA. Based on the comprehensive data analysis, the
decomposition temperature $T_{-5\%}$, $T_{-50\%}$, and $T_P$ of the composites increased first and then decreased with the increase in the MWCNT loading amount. With a MWCNT loading amount of 1.5 wt%, the highest decomposition temperature and best thermal stability were achieved, related to the good thermal conductivity of the MWCNT, and the thermal conductivity of the composites increased with the MWCNT loading amount, leading to the increase in the decomposition temperature. With a MWCNT loading amount of 1.5 wt%, the MWCNT constituted a conductive network and formed a good thermal conductivity network for composite materials. The subsequent decomposition temperature decreased with the increase in the loading amount of MWCNT, accompanied by the additional agglomeration of MWCNT. Owing to the large difference in thermal conductivity between MWCNT and PLA, the agglomeration point of each MWCNT was a heat concentration point, promoting the thermal decomposition of the composite material, therefore, the decomposition temperature decreases. By compared comprehensively, the loading of MWCNT enhanced the thermal stability of the composite.

### Differential scanning calorimetry

MWCNT and PLA were melt-blended with the MWCNT loading amounts of 0.0 wt%, 0.5 wt%, 1.0 wt%, 1.5 wt%, 2.0 wt%, and 3.0 wt% of the total mass of composite. Then, the DSC curves of the PLA/MWCNT composites were recorded on the STA449 F5 synchronous thermal analyzer as shown in Figure 6. Tables 3 and 4 summarize the thermodynamic performance data of PLA/MWCNT composites. Six DSC curves exhibited glass transition temperature $T_g$ ranging from 58°C to 60°C. With the further increase in the temperature, a strong cold crystallization exothermic peak was observed near 96°C. The corresponding cold crystallization temperature to $\Delta H_{cc1}$ was $T_{cc1}$. As the temperature approached 160°C, a small exothermic peak was observed in the DSC curves, corresponding to a process in which a previously formed imperfect $\alpha'$ crystal form further developed to a more perfect $\alpha$ crystal structure at high temperature. The exothermic enthalpy was $\Delta H_{cc2}$. With the increase in the temperature to $-175^\circ C$, a strong endothermic peak was observed in the DSC curve, corresponding to the melting enthalpy of the crystalline part of the composite. It was denoted as $\Delta H_m$, and the corresponding melting temperature was $T_m$. The crystallinity $X_c$ of the PLA/MWCNT composite was calculated by Formula (3).

From the data obtained from Figure 6 as well as Tables 3 and 4, with the increase in the MWCNT, the amplitude of variation in the glass transition temperature $T_g$, cold crystallization temperature $T_{cc1}$, melting point temperature $T_m$, and crystallinity $X_c$ of PLA/MWCNT composites was not large. These values increased first and then decreased. From analysis, at a low concentration of the loaded MWCNT, MWCNT mainly served as a heterogeneous nucleating agent in the composite material, increased the number of crystal nuclei for cold crystallization, promoted the crystallization of PLA, and improved the crystallinity. However, with the increase in the loading of MWCNT, MWCNT

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**Table 2. Thermodynamic performance data of PLA/MWCNT composites.**

| Sample                  | $T_{-5\%}$ (°C) | $T_{-50\%}$ (°C) | $T_{-95\%}$ (°C) | $T_P$ (°C) |
|-------------------------|-----------------|-----------------|-----------------|-----------|
| PLA                     | 309.75          | 354.92          | 373.2           | 361.83    |
| PLA/MWCNT (99.5/0.5)    | 316.20          | 358.02          | 375.39          | 362.94    |
| PLA/MWCNT (99.0/1.0)    | 318.29          | 358.04          | 373.73          | 363.92    |
| PLA/MWCNT (98.5/1.5)    | 319.66          | 358.56          | 377.52          | 363.82    |
| PLA/MWCNT (98.0/2.0)    | 317.91          | 358.48          | 378.63          | 363.74    |
| PLA/MWCNT (97.0/3.0)    | 313.76          | 357.38          | 380.81          | 362.92    |

**Table 3. Thermodynamic properties of PLA/MWCNT composites.**

| Sample                  | $T_g$ (°C) | $T_{cc}$ (°C) | $T_m$ (°C) |
|-------------------------|-----------|---------------|-----------|
| PLA                     | 59.32     | 95.49         | 175.35    |
| PLA/MWCNT (99.5/0.5)    | 59.48     | 97.67         | 174.99    |
| PLA/MWCNT (99.0/1.0)    | 59.50     | 97.51         | 175.16    |
| PLA/MWCNT (98.5/1.5)    | 58.88     | 96.61         | 175.25    |
| PLA/MWCNT (98.0/2.0)    | 59.26     | 95.51         | 175.68    |
| PLA/MWCNT (97.0/3.0)    | 58.20     | 93.54         | 175.34    |
exhibited increased agglomeration and decreased nucleation. Simultaneously, excess MWCNT can cause steric hindrance in the PLA melt, hinder PLA molecular chain segment movement, increase the system viscosity and the activation energy of crystal growth, thereby decreasing the crystallization rate and crystallinity. Based on comprehensive data analysis, in the simultaneous presence of the two contrasting effects, the nucleation effect of MWCNT played a major role in the composite, leading to a slightly higher crystallinity of the composite compared to pure PLA. With a MWCNT loading amount of 0.5 wt%, the composite exhibited the highest crystallinity, and $X_c$ was 20.43%.

**Conclusion**

1. With the increase in the MWCNT loading, the electrical conductivity of the PLA/MWCNT composites improved considerably. The surface resistivity $\rho_s$ decreased from $1.95 \times 10^{15}$ Ω for pure PLA to $2.48 \times 10^6$ Ω with a MWCNT loading of 3.0 wt%, and the volume resistivity $\rho_v$ decreased from $5.20 \times 10^{15}$ Ω cm for pure PLA to $9.98 \times 10^6$ Ω cm with a MWCNT loading of 3.0 wt%, which decreased by $10^9$ times. In addition, the surface resistivity $\rho_s$ of the tested material was relatively lower with the loading of the same MWCNT amount, and the penetration threshold $\rho_s$ was also relatively easier to achieve.

2. With the increase in the MWCNT loading, the tensile strength $\sigma$ and elongation at break $\varepsilon$ of the PLA/MWCNT composites first increased and then decreased. The best data were obtained with a loading amount of 1.0 wt%, $\sigma$ was 62.04 ± 1.43 MPa, which was 9.66 MPa greater than that of pure PLA. $\varepsilon$ was 4.10 ± 0.33%, which was 0.46% greater than that of pure PLA.

3. With the increase in the MWCNT content, the decomposition temperatures $T_{\text{d}}$, melting point temperature $T_{\text{m}}$, and crystallinity $X_c$ of the PLA/MWCNT composites did not change significantly, and it basically demonstrated an increasing trend first and then decrease trend. Typically, the loading of MWCNT increased the crystallinity of the composites and enhanced heat resistance.

**Declaration of conflicting interests**

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**Table 4. Crystallinity of PLA/MWCNT composites.**

| Sample       | Melting enthalpy $\Delta H_m$(J/g) | Cold crystallization enthalpy $\Delta H_{cc1}$(J/g) | Cold crystallization enthalpy $\Delta H_{cc2}$(J/g) | Crystallinity $X_c$ (%) |
|--------------|-----------------------------------|---------------------------------------------------|---------------------------------------------------|-------------------------|
| PLA          | 40.61                             | 22.58                                             | 1.57                                              | 17.70                   |
| PLA/MWCNT (99.5/0.5) | 43.02                             | 22.82                                             | 1.30                                              | 20.43                   |
| PLA/MWCNT (99.0/1.0) | 41.09                             | 21.08                                             | 1.33                                              | 20.29                   |
| PLA/MWCNT (98.5/1.5) | 37.58                             | 19.00                                             | 1.30                                              | 18.86                   |
| PLA/MWCNT (98.0/2.0) | 36.89                             | 18.85                                             | 0.88                                              | 18.83                   |
| PLA/MWCNT (97.0/3.0) | 35.61                             | 18.52                                             | 0.89                                              | 17.95                   |

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