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Effect of CNT additives on the electrical properties of derived nanocomposites (experimentally and numerical investigation)

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ABSTRACT

In this work, two simulations models have been developed to study the electrical percolation and the electrical conductivity of epoxy-based nanocomposite containing Multi-walled Carbon Nanotubes. The models are based on resistor-model and finite element analysis. The former was evaluated using MATLAB code and the finite element analysis using DIGMAT software. The maximum tunnelling distance and its influence on the percolation probability and final electrical conductivity were studied. Electrical measurements on the samples were conducted for numerical validation. The experimental data showed a percolation achievement around 2 wt%, which was confirmed in the numerical simulations. This study provides evidence of the effectiveness of the resistor model and finite element method approach to predict the electrical conductivity of nanocomposites.

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1. Introduction

In the last decades, the demand for enhanced materials has faced a rapid increment. For instance, in many industries such as in the military, aerospace and construction, better physical properties (i.e. mechanical, electrical and thermal.) are essentially demanded to meet the rapid advancement of new technologies [1,2]. The utilization of neat materials is inevitably confined because of their basic low inherent properties. On the other hand, composite materials have demonstrated the capability to fulfil those requirements [3]. Particularly, polymeric nanocomposites have attracted considerable research interest as they have high strength to weight ratio, high resistance of corrosion, inexpensive, and easy to be fabricated in difficult shapes [4]–[6]. Different nanometric fillers have been used to improve the mechanical performance, thermal and electrical conductivity of the polymeric matrix. Normally, the material composition consists of fillers in form of fibres or different types of particles [5]

Carbon additives have been employed to enhance the properties of pristine polymeric matrices. Among the most popular are graphene, graphite, carbon blacks, carbon nanotubes, and carbon

nanofibers, [8][4,5,7]–[9]. Carbon nanotubes are the most attractive as their present unique physical properties i.e. high strength to weight ratio and

aspect ratio [10]. Moreover, due to their superior mechanical and electrical properties, CNTs are widely employed for tailoring multifunctional properties of polymer nanocomposites for nano-electromechanical sensors [11,12]. Additionally, it is well known that the addition of a small number of CNTs into dielectric polymeric matrices will result in a drastic improvement in the electrical conductance of the composite [13]. The critical volume fraction of CNT that triggers a severe escalation in the conductivity of the composite is called the electrical percolation threshold (EPT). At the percolation threshold, the electrical conductivity is exponentially increased due to the formation of connected networks [14].

Many numerical studies have been performed to study the influence of carbon nanotubes in polymeric matrices. There are many model methods to study the electrical conductivity and the percolation behaviour of polymer-based composites such as Monte Carlo, resistor-model, finite element and mean-field homogenization [15,16]. The majority of these methods are based on 3D Representative volume element (RVE), Bao et al. [13] employed Monte Carlo simulations with a network recognition approach to determine current continuity. For instance, Hu et al. [17] used a 3D resistor-model network to estimate the electrical properties

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of CNT nanocomposites. Also, Manta and Tserpes [18], simulated the electrical behaviour of carbon nanotube-filled polymers through a finite element (FE) model based on the tunnelling effect. Extensive numerical simulations can be found in the literature regarding the prediction of electrical conductivity and percolation probability. However, the correct applications are still underdeveloped as the percolation threshold is a very complex phenomenon to understand and not simple to model [19]. Moreover, when the percolation is achieved the conduction takes place due to three different sources: the intrinsic conductance of the nanotubes, direct contact conductance, and conductance resulting from electron tunnelling. Thus, many models take into account the contact conductance, but few the tunnelling effect.

Thus, it is important to perform numerical simulations because is a potent and inexpensive tool and it has become a normal procedure for design and characterization [5]. Therefore, MATLAB software and DIGMAT-FE were selected to perform the numerical simulations for each weight percentage. Using MATLAB software we have studied the influence of CNT randomly distributed and the influence of tunnelling distance through a resistor model. The latter was used to performed Finite element analysis.

In this paper, we aimed to investigate the electrical behaviour of polymeric nanocomposites through a combination of experimental and two computational methods. The influence of the tunnelling distance in the final electrical conductivity and percolation probability was studied and compared with experimental data for different percentages of nanofillers. This unique approach had explored a new method to characterize the final electrical conductivity of nanocomposites with different percentage of aggregation. Finally, the combination of this two numerical methods is highly effective when compared to the experimental data.

2. Materials and experimental methods

The CNT/Polymer composites were fabricated by mixing CNTs into an epoxy Matrix. Multi-walled carbon nanotubes (MWCNT) purchased from Applied sciences Inc were dispersed into Epoxy 862 with different percentage i.e., 0.5, 1, 2, 4, 5 wt%. The MWCNT dimensions range from 100 nm to 200 nm for the diameter and 30 μm to 100 μm for the length. The samples electrical conductivity was measured using four-probe measurements (Loresta equipment).

The calculation of the model's electrical conductivity was carried out through two different numerical simulation approaches. To predict the electrical conductivity of the nanocomposite, MATLAB and DIGMAT FE were chosen to estimate the accuracy of the models. The physical properties of the constituents utilized in the numerical simulations are displayed in Table 1 [13,20,21].

The MATLAB simulation involved two steps. First, the generation of the 3D geometry Representative Volume Element (RVE) with the nanofillers randomly distributed within the volume. For each sample, the number of nanofillers was aggregated according to the volume calculation based on the weight fraction (Fig. 1). The second step was the characterization of the networks and the calculation of their electrical properties. The simulation emphasized only the quantum tunnelling effect. This resistance predominantly affects the final conductivity [22,23]. Thus, the first

assumption was that the CNTs were not able to cross each other or expand outside the RVE volume. And the second assumption was that the matrix's electrical resistivity does not affect the outcome. As its value is significantly lower than the CNT's intrinsic resistivity [24].

In the initial part, the RVE dimensions were set to 20 μm (length) \times 20 μm (width) \times 20 μm (thickness). The shapes of the CNTs were considered cylinders. The diameter of each CNT was set to vary between 100 nm and 200 nm, and the length was set to change to any value between 30 μm and 100 μm following the manufacturing data. The potential formation of CNT networks is dependent on the minimum distances between two CNTs i.e., tunnelling distance. It was calculated using eq. (1).

$$a, b, c, d, S, T \in R^N$$

$$s, t \in R$$

$$S = \{ s \times b + (1 - s) \times a \mid 0 \leq s \leq 1 \}$$

$$T = \{ t \times b + (1 - t) \times a \mid 0 \leq t \leq 1 \}$$

$$(S, T) = \sqrt{\sum_{i=1}^n (s \times b_i + (1 - s) \times a_i - w \times c_i - (1 - w) \times d_i)^2} \quad (1)$$

When a minimal distance between two points, depicted in Fig. 2 with S and T, located in the axis of the CNTs, is lower than the pre-set tunnelling distance then the two nanofillers are considered in junction.

The minimal distance calculation is carried out for every nanofiller, thereby the potential formed networks are saved. If a network was built within the RVE volume, the final electrical conductivity can be calculated. The network should connect two opposite faces of the RVE throughout the percolation pathway to obtain the equivalent resistivity. When this is achieved the circuits formed by the CNTs are arranged into a matrix equation. The circuit is then solved by Kirchhoff's circuit laws and node theory [Fig. 3 [25]–[27]].

Eq. (2) represents the matrix form of a system with N nanotubes and L junctions between them and the conductive faces as shown in Fig. 4.

$$I = RxV \quad (2)$$

The matrix I is null except for the first and last element which are pre-set to the applied electrical potentials of the two faces. V represents the electrical potential of the CNTs, in which the first element and the last are pre-set to the electrical potentials of the two conductive faces (Fig. 4). The matrix R, which is dependent on the minimal junction distances, is used to calculate the resistivity of each CNT that are only participating in the network with eq. (3). Where l_{jk} is the length of the CNT between the nodes j and k, σ_{cnt} is the intrinsic electrical conductivity and D the diameter. Lastly, the final resistivity and the conductivity are then calculated using eqs. (4) and (5). Where G_{rve} is the effective electrical conductance, V_{f1}, V_{f2} are the applied voltages on the conductive RVE faces. The parameter t the RVE's thickness and σ_{rve} is the effective electrical conductivity of the nanocomposite [9,12,17,26].

$$R_{jk} = \frac{4l_{jk}}{\pi\sigma_{cnt}D^2} \quad (3)$$

$$G_{rve} = \frac{I_{total}}{(V_{f1} - V_{f2})} \quad (4)$$

$$\sigma_{rve} = \frac{G_{rve}}{t} \quad (5)$$

Table 1
Properties of the constituents.

	Epoxy EPON 862	Carbon nanotubes (MWCNT)
Density	1.21 g/cm ³	2.1 g/cm ³
Electrical conductivity	2.1×10^{-7} S/cm	5×10^1 S/cm – 5×10^5 S/cm

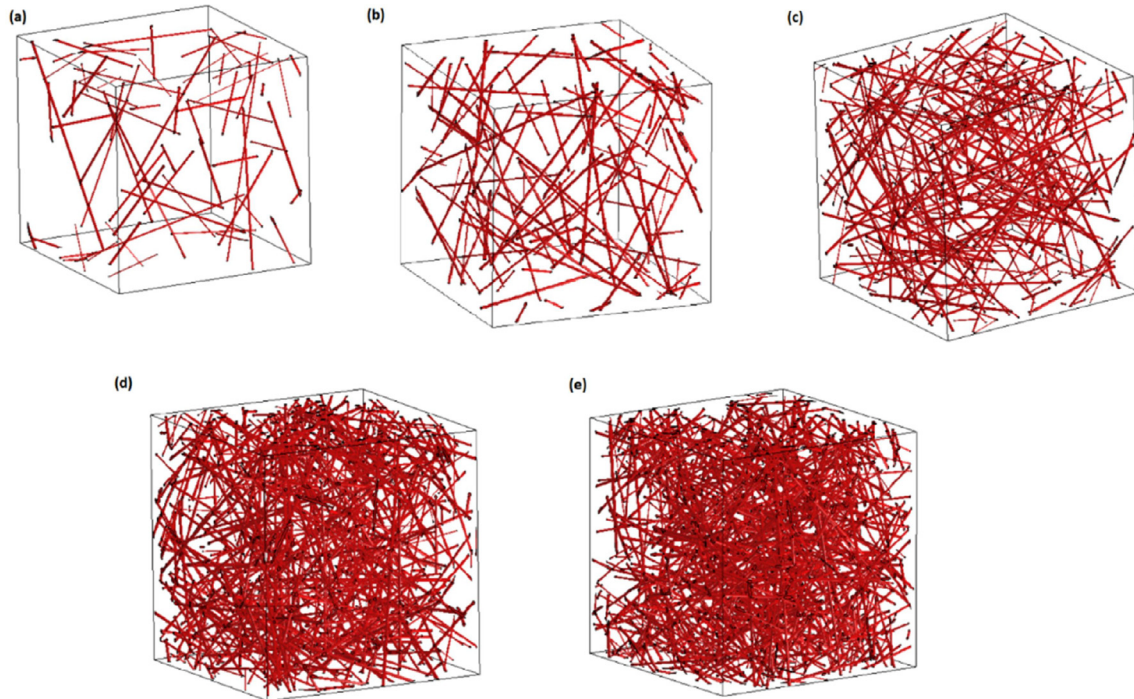


Fig. 1. Generation of RVEs as a function of the percentage of nanofillers (a) 0.5 wt%; (b) 1 wt%; (c) 2 wt%; (d) 4 wt%; (e) 5 wt%

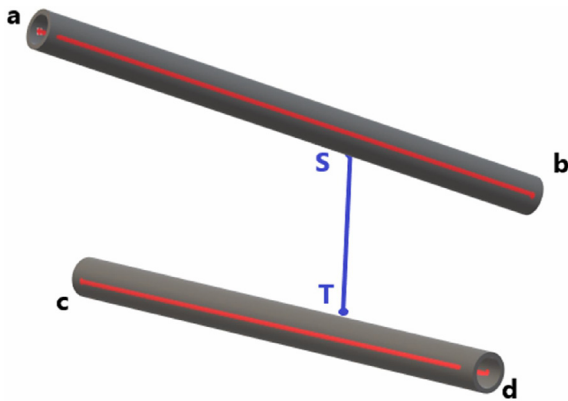


Fig. 2. Minimal distance calculation between two carbon nanotubes inside resistor model.

The DIGMAT simulations included the well-known Finite Element Method (FEM). As a result, a two-phase RVE was generated, enclosing the matrix and the nanofillers. The dimensions of the RVE, the diameter and the length of the CNTs were set the same as the parameters in the MATLAB simulation. Additionally, random distribution and interpenetration between CNTs were assumed. The RVE generation was according to the samples shown in Fig. 1. Additionally, a mesh convergence study was performed, finding that 420 000 number of elements is the perfect parameter where the final electrical conductivity convergence (Fig. 5).

3. Results and discussion

The low electrical conductivity of neat epoxy is affected by the addition of a small percentage of MWCNT. For every percentage, the electrical conductivity is increased. Also, it is observed that a sharp increase in the conductivity was achieved at 2 wt%. This indicates that the percolation threshold occurs around this filler per-

centage. The experimental results were confirmed with simulation models finding a good correlation. The small addition of nanofillers exponentially increased the conductivity, Fig. 6. Moreover, the percolation achievement is also affected by the addition of MWCNT. The formation of networks depends on the nanofillers aspect ratio, dispersion and distribution within the matrix. The increase of the filler aspect ratio makes the formation of networks easier. Therefore, decreasing the percolation threshold percentage at which is triggered [15,28]. Additionally, the maximum tunnelling distance affects mainly after the percolation threshold.

Thus, in our simulations, the influence of the contact resistance and the maximum tunnelling distance on the percolation threshold and the final electrical conductivity was studied. Firstly, the percolation probability was investigated with three maximum tunnelling distance for each sample i.e., 0.3 μm , 0.4 μm , and 0.5 μm . A total of 750 simulations were performed inside MATLAB simulations, 50 simulations for each weight percentage. The results of the percolation probability are depicted in Table 1.

As the number of nanofillers augments the percolation probability is increased. However, it is noticeable that the tunnelling effect plays a significant role, Fig. 7 [29]. The maximum tunnelling distance alters the volume at where the percolation threshold is achieved [30]. According to the aspect ratio, the first distance studied was 0.3 μm . At this distance, the percolation threshold takes place at 4 wt%. With the addition of more nanofillers, the percentage of probability is altered. Every time the percentage is augmented the probability is significantly increased. However, at 2 wt% close to 50% of percolation probability was observed. Clearly the distance shifted the percolation threshold to 4 wt% where 97% is obtained. At the higher number of nanofillers, the probability reached 100%. Subsequently, the distance 0.4 μm was studied. This distance showed an initial probability of 10%. The 1 wt% showed a 18% of probability higher than showed with 0.3 μm . A huge sharp increment occurs around 2 wt%, where a high probability of 98% was achieved. Above 2 wt% the percolation probability reached 100% and stabilized. Finally, a 0.5 μm of maximum tunnelling distance was analysed. After 500 simulations the distances 0.4 μm ,

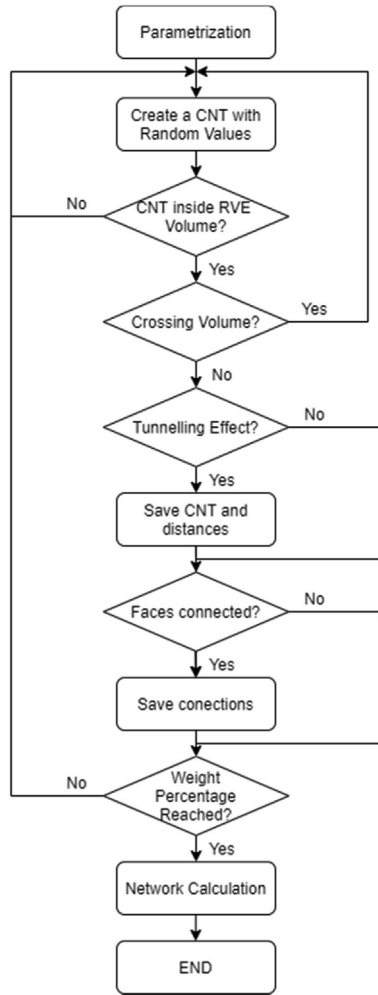


Fig. 3. Flow chart of the numerical process.

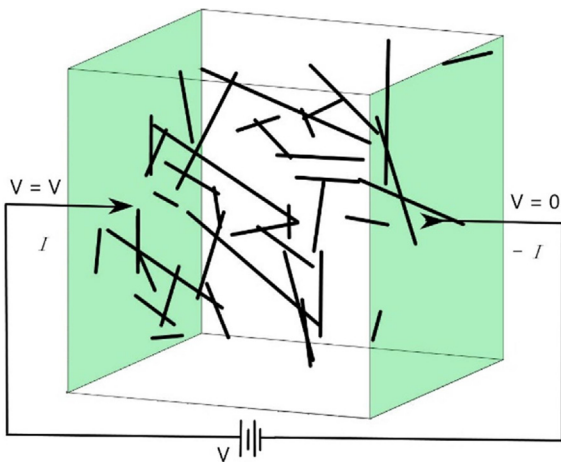


Fig. 4. 3D resistor network model of CNT randomly distributed.

and 0.5 μm showed similar results in the percolation probability, the only disagreement occurs at 1 wt%, where there is a difference of 8% of probability. Also, the 0.5 μm tunnelling distance showed the percolation threshold at around 2 wt%. This percolation threshold is represented with a red line in Fig. 7. Although, similar values are obtained in the percolation threshold, the final electrical con-

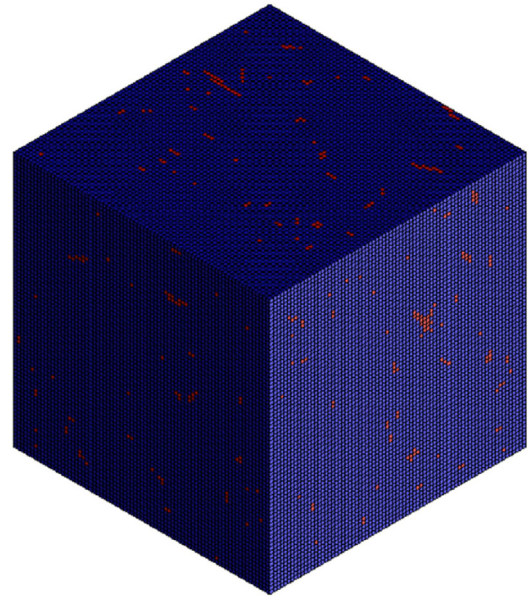


Fig. 5. Representative volume element of the matrix and MWCNT in DIGMAT FE.

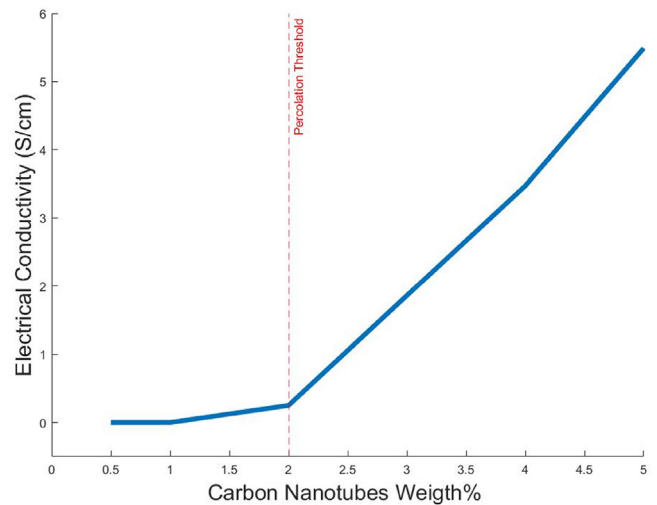


Fig. 6. Electrical Conductivity for Epoxy/MWCNT.

ductivity for each samples seems affected, which is discussed in the upcoming section [31]. This is due that the higher the distance the more CNT are considered in the formation of the network [32]. This is in good agreement with experimental measurements.

The electrical conductivity was studied using two approaches. First, the electrical conductivity was calculated for each sample in MATLAB. The electrical conductivity for each (Table 2). These values were calculated through the average electrical resistivity obtained after every time the percolation was achieved. The values for the electrical conductivity with the 0.3 μm distance, are in good agreement even though the percolation threshold occurs at a different nanofiller percentage than the experimental results. Above the 2 wt% the model could predict the conductivity for each sample. Nevertheless, for each tunnelling distance, it was not possible to predict the final electrical conductivity below percolation. The disagreement before the percolation threshold is due to that the resistor network does not take into account the electrical resistivity of the matrix. Which before the formation of the percolation networks the composite's conductivity is close to the matrix [33].

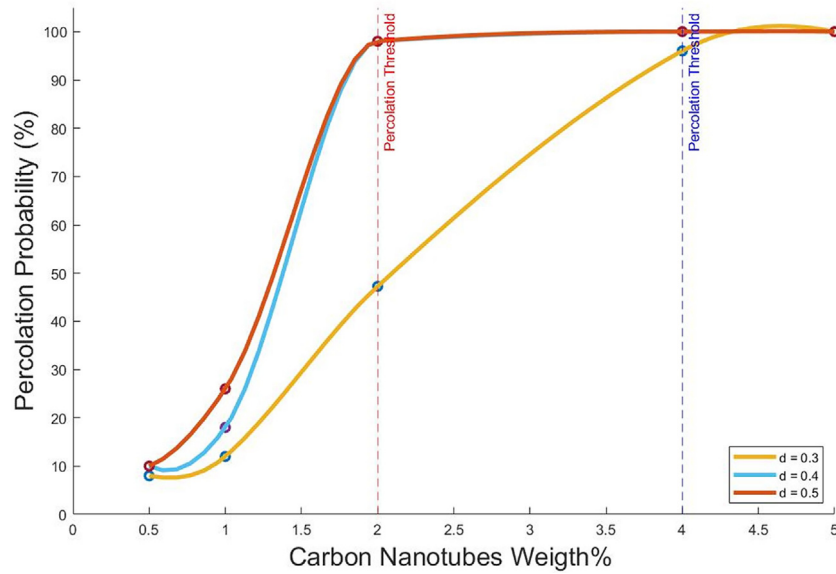


Fig. 7. Percolation probability with different tunnelling distance.

Table 2
Percolation probability for different tunneling distances.

Samples	$d1 = 0.3$	$d2 = 0.4$	$d3 = 0.5$
0.5 wt%	8	10	10
1 wt%	12	18	26
2 wt%	47.27	98	98
4 wt%	96	100	100
5 wt%	100	100	100

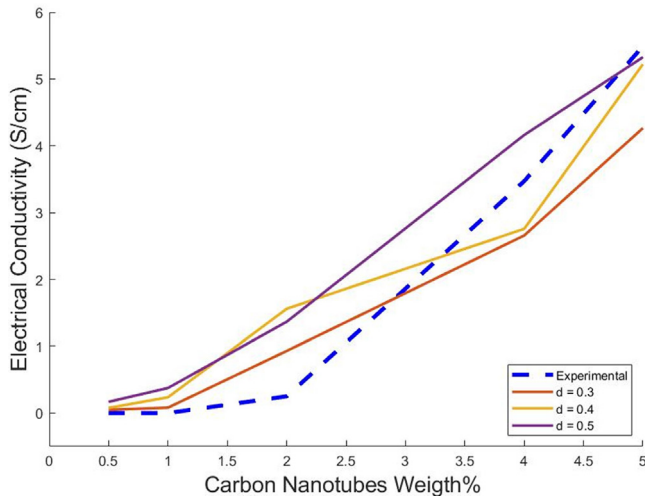


Fig. 8. Comparison of electrical conductivity of MATLAB simulations with experimental data.

Table 3
Electrical conductivity (S/cm) results.

Samples	Experimental	Finite Element	$d1 = 0.3$	$d2 = 0.4$	$d3 = 0.5$
0.5 wt%	2.123×10^{-5}	2.15×10^{-7}	4.9×10^{-2}	7.774×10^{-2}	1.68×10^{-1}
1 wt%	5.195×10^{-4}	1.167×10^{-6}	8.13×10^{-2}	2.361×10^{-1}	3.7754×10^{-1}
2 wt%	0.2512	0.222	0.9300	1.5609	1.366
4 wt%	3.472	3.160	2.658	2.7572	4.161
5 wt%	5.549	5.2190	4.2644	5.2208	5.32

The best fit possible is the 0.4 μm distance. The model could predict the final electrical conductivity above the percolation threshold i.e., 2 wt%, 4 wt%, 5 wt%. At this distance, the electrical conductivity showed the same exponentially growth, Fig. 8. At the 2 wt%, the electrical conductivity is overestimated by the model, being close to 1 S/cm. Whereas for 4 wt% and 5 wt% the conductivity is underestimated. However, the results are in good agreement with the experimental validation. For the final tunnelling distance of 5 μm , the same phenomena happened. Above the percolation threshold, there is a good agreement in the conductivity. Nevertheless, the model tends to overestimate the final conductivity. For instance, at 2 wt% the difference is higher than 1 S/cm. Also, at 4 wt% the disagreement is above 0.5 S/cm. Finally, a good prediction was seen at 5 wt%, where an underestimation of just 0.2 S/cm was calculated Table 3.

Furthermore, the predictions on the final electrical conductivity of the nanocomposites were carried out in DIGMAT software. For each sample, the nanofillers were calculated based on the weight percentage, Fig. 1. The values for the final electrical conductivity are shown in Table 2. The model showed anisotropic behaviour. Thus, the values are the average of the three electrical conductivities resulting from each axis in the finite element method. In the finite element method, an exclusive percolation threshold studied was not performed. Instead only the final conductivity was calculated. Similarly, below the percolation threshold, the model is not capable to match the experimental results. As the finite element method overestimates the matrix electrical resistivity. On the other hand, the model could vastly predict the final conductivity. There is a huge agreement with the experimental validation, Fig. 9. From 2 wt% the path of the electrical conductivity follows the same increment as in the measured conductivity. For instance,

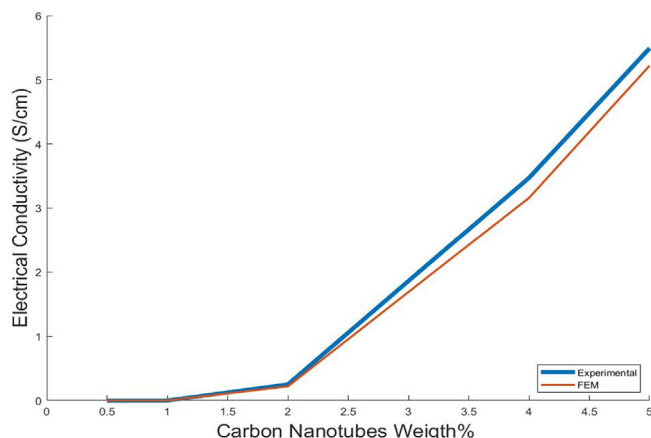


Fig. 9. Comparison of electrical conductivity of Finite Element with experimental data.

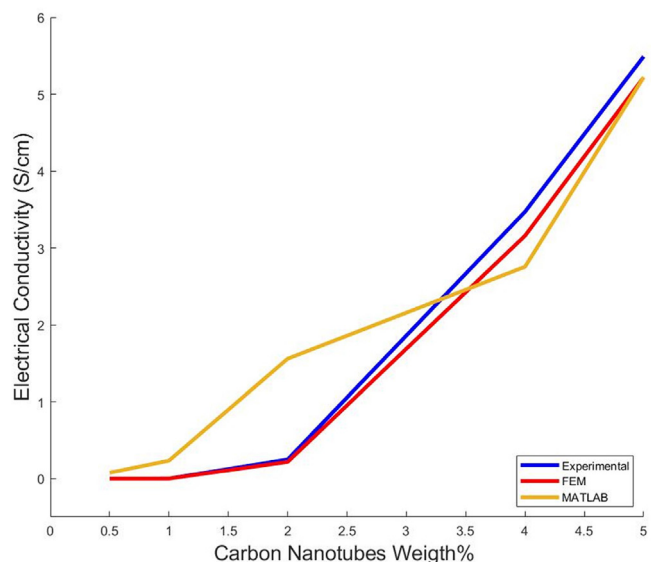


Fig. 10. Comparison of electrical conductivity of MATLAB simulations and Finite Element with experimental data.

the difference at 2 wt% is only 0.029 S/cm. Also at 4 wt% and 5 wt%, the difference is only 0.3 S/cm.

Lastly, both simulation approaches showed an incredible capability to predict and match the electrical conductivity with the experimental data. In Fig. 10, the best fit from MATLAB simulations (i.e., 0.4 μm) is compared with the finite element method along with the experimental measurements. The data suggest that the percolation threshold is achieved just between 1 wt% and 2 wt%. Suggesting that at this very low nanofiller concentration the electrical conductivity is highly augmented [34]–[36]. This result is due to that the higher the aspect ratio of carbon nanotubes the lower the percentage at where the percolation threshold is achieved [36]–[38]. The impact of high aspect ratios is more evident at lower percentages of MWCNT [15,39].

4. Conclusions

The electrical behaviour of polymer nanocomposites with CNT as nanofillers was numerically investigated. Two simulations methods were developed to predict the electrical percolation and the electrical conductivity. The simulations results were compared

with experimental measurements for each MWCNT/EPOXY nanocomposite sample. Three tunnelling distances were studied, finding that the maximum tunnelling distance highly affects the volume at where the percolation probability is achieved. Also, the tunnelling distance plays an important role in the final conductivity of the model. The experimental results along with the numerical showed that the percolation threshold is achieved just before 2 wt%. The 3D resistor network model was successfully capable to estimate the final electrical conductivity for randomly nanofillers distributions. In the same manner, it was found that the Finite Element Method could match the experimental results. Although both methods cannot predict the electrical behaviour of the samples before the percolation threshold. Both methods have shown a high accuracy above the percolation threshold.

CRedit authorship contribution statement

S. Tamayo-Vegas: Writing – original draft, Investigation, Formal analysis, Software. **A. Muhsan:** Investigation. **M. Tarfaoui:** Supervision, Formal analysis, Validation. **K. Lafdi:** Supervision, Project administration, Validation, Conceptualization, Methodology. **L. Chang:** Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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