Research Article

Can nanoparticles improve polyaniline electrical conductivity?

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Polyaniline is a conductive polymer that attracts the attention of many researchers around the world. The history of this polymer begins in 1862 when Letheby first reported this material. Since then, a myriad of studies has been conducted on this material, and new works continue to investigate the potential of this material. Polyaniline has been improved with the help of Nanotechnology. The use of nanofillers has been seen as a quick and economical way to modify materials, driving innovations based on new physical and chemical properties from the conductive polymer materials and nanoparticles joining. Several works address the use of different nanoparticles, which leads to the practical impossibility of sifting through all this information. Thus, this work proposes to systematically collect data in the literature and investigate which nanoparticles can increase the electrical conductivity of Polyaniline (PAni). The results obtained demonstrate that among the possible nanofillers, graphene and carbon nanotubes have great prominence. Furthermore, the results of the meta-analysis prove that PAni's conductivity increases when this polymer is modified with the aforementioned nanofillers. This work was developed using VOSViewer, a classification and grouping tool, and Jamovi, a new 3rd generation statistical tool used here for the meta-analysis calculation.

1. INTRODUCTION

Since polyaniline was reported by Letheby in 1862, a profusion of work has been developed on this material (1). Polyaniline (PAni) is a conductive polymer from the flexible polymer family, which has high electrical conductivity (2–13). Among conductive polymers, PAni is unique because of its easiness of synthesis (14–25). Besides, Professor MacDiarmid et al. (26) proved the PAni emeraldine base can be converted from an insulator to a metal-like material by treatment with aqueous HCl, by forming the emeraldine-hydrochloride salt. Such transformation involves non-redox doping, in which a polaron conduction band, with most of the positive charge residing on the nitrogen atoms, is responsible for the electrical conductivity. PAni's doping process produces a nice color range from blue to green, which is useful for developing electrochromic devices, which is further favored by its good environmental stability (27–39). PAni has been one of the most studied polymers for the past 20 years.

Our research group has studied PAni dozens of times. Our first publication on PAni dates back to 2005 when we introduced a methodology for studying the dependence of electrical resistivity with pressure in conducting composites (40). Then, we studied several subjects regarding PAni, such as polymerization in the presence of carbon black (41), DBSA (42), cardanol bio-resin (43), cardanol-furfural bio-resin (44), DMIT (45), polyamide-6,6 (35), SBS

(46, 47), nitrile rubber (48), EVA-NBR (49), poly(lactic acid) (50), curaua fibers (51), coconut fibers (52, 53), coir fibers (54), mango fibers (55), cotton fabrics (56) as well as the influence of plasticizers (57, 58), magnetite (59, 60) and maghemite (61, 62). Next, the characterization of these materials by electrical surface colorimetry (63), resistivity (64), WAXS (65, 66), UV-Vis (67), XPS (68), NIR (69), and SAXS (70–73), were studied. Finally, a paper named "Polyaniline: Trends and perspectives from text-mining analysis" (74) was published in the Brazilian Journal of Experimental Design, Data Analysis, and Inferential Statistics. Thus, as evidenced by this set of studies, our research group has a relevant background in this subject, always looking for new ways to improve this polymer.

PAni is a typical conductive polymer, resulting from the oxidative polymerization of aniline, whose conductivity can be affected by the degree of doping, the type of dopant, the morphology, and the degree of crystallization (4). PAni has attracted attention in recent decades due to its characteristics such as low cost, high conductivity, and good resistance to the environment (18). Besides, PAni has special electronic properties, which can be reversibly controlled by the material's protonation/deprotonation processes (5). In addition, PAni has great potential for high-end applications, such as electrodes (18, 28, 38, 75–83), batteries (15, 84–93), microelectronics (94–102), electrochromic materials used in displays (103–110), sensors (22, 111–118), and electromagnetic shielding (119–127). Despite its excellent properties, several studies have been carried out in an attempt to improve the properties of PAni. Among all of the documents about PAni listed in Scopus database (n = 30,788), almost 75% of them (23,066) involved polyaniline and "nano". Therefore, the use of nanofillers is a quick and economical way to modify materials, driving innovations based on new physical and chemical properties from the conductive polymer materials and nanoparticles joining.

From a specific point of view, such as when studying a well-defined nanofiller-matrix system, synthetic issues must be considered for improvements in electrical properties. Besides that, it is crucial to understand which superficial modifications are necessary for achieving the desired improvement (127). However, the premise of this study was to proceed with a broad search on which nanoparticles could increase the conductivity of systems based on polyaniline.

Thus, the effect of the quantity in different nanofillers on the conductivity of the polyaniline matrix was studied. As the conductivity studies were conducted differently by their respective authors, the primary efforts were to analyze the slope and correlation between conductivity and the amount of different nanofillers. This procedure allowed for inferring that, among several possibilities, specific nanofillers, such as carbon nanotubes and graphene, are more exploited, aiming to increase the matrix conductivity. Furthermore, the meta-analysis results prove that PAni conductivity increases when this polymer is modified using the nanofillers mentioned earlier.

Of course, other nanofillers with the potential to increase conductivity may have escaped this study, but until new evidence emerges, carbon nanotubes and graphene should be the two preferred nanofillers for studies that seek to increase the conductivity of polyaniline.

2. METHODS

All the steps of this study were derived from a question: "***Could nanoparticles improve polyaniline electrical conductivity or electrical resistivity?***"

2.1. Article search

Research articles containing the term “polyaniline” in the title, keyword and abstract were collected using the Scopus database. The used key was TITLE-ABS-KEY (polyaniline). The number of papers published per year was plotted using QtiPlot Software. Articles with publication year of 2021 and 2022 were excluded, as the final number of articles is not consolidated yet. The date of the retrieval was 2nd July, 2021. Then, a second search key, more complex than the first, was used to refine the number of documents retrieved. In its complete form, the second search key was written as: TITLE-ABS-KEY (nano* AND polyaniline AND improv* AND "electrical conductiv*" OR "electrical resistivit*") AND (LIMIT-TO (DOCTYPE , "ar")). Here, there was no limitation of the researched years. The RIS file containing these data is available at https://github.com/ftir-mc/Nanoparticles-improving-Pani-conductivity.git.

2.2. Scientific scenarios evaluation with VOSviewer

The bibliographical information for the documents containing abstract, author keyword and index keywords were exported as a RIS document and imported by VOSviewer (version 1.6.10) software (128). Different trending topics and themes were identified from the Titles and Abstracts. Then, the search was refined again. In its complete form, the third search key was written as: TITLE-ABS-KEY (nano* AND polyaniline AND improv* AND "electrical conductiv*" OR "electrical resistivit*" AND graphene OR "carbon nanotub*") AND (LIMIT-TO (DOCTYPE , "ar ")). Once more, there was no limitation of the researched years.

2.3. Data Extraction

The articles were analyzed according the following criteria: nanofillers concentration in PAni, number of replicates, and standard deviation. Based on the inclusion criteria, information from all eligible publications were extracted. The following information were included in each study: name of first author, year of publication, filler (CNT or graphene), number of replicates, and the correlation between the electrical conductivity or electrical resistivity versus the percentual amount of nanofiller. When the document did not present the data tables, data was extracted from plots curves. Engauge Digitizer 3.0 (by Mark Mitchell) software was used to extract data from these figures. From data extracted, the 95% confidence intervals (CI95%) were calculated.

2.4. Statistical Analysis and Meta-analysis

As several electrical measurement techniques were performed, the effect of nanofillers on the electrical conductivity of materials was evaluated by correlating the conductivity data and the mass quantity of the nanofiller in the material. The Jamovi (version 1.6.23.0) module MAJOR (129) was used to obtain Meta-analysis and Forest plot. The analysis was carried out using the Fisher r-to-z transformed correlation coefficient as the outcome measure. A random-effects model was fitted to the data. The amount of heterogeneity (i.e., tau^2), was estimated using the restricted maximum-likelihood estimator (130). In addition to the estimate of tau^2, the Q-test for heterogeneity (131) and the I^2 statistic are reported. In case any amount of heterogeneity is detected (i.e., $tau^2 > 0$, regardless of the results of the Q-test), a prediction interval for the true outcomes is also provided. Studentized residuals and Cook's distances are used to examine whether studies may be outliers and/or influential in the context of the model. Studies with a studentized residual larger than the 100 x (1 - 0.05/(2 X k))th percentile of a standard normal distribution are considered potential outliers (i.e., using a Bonferroni correction with two-sided alpha = 0.05 for k studies included in the meta-analysis).

Studies with a Cook's distance larger than the median plus six times the interquartile range of the Cook's distances are considered to be influential. The rank correlation test and the regression test, using the standard error of the observed outcomes as predictor, are used to check for funnel plot asymmetry.

3. RESULTS

From the Scopus database, 29584 documents were collected between 1969 and 2022. All the conference papers (3,028), reviews (533), book chapters (252), and conference reviews (188) were excluded from the search. Thus, the documents were restricted to articles, remaining 25,396 titles. The number of documents published per year is shown in Figure 1.

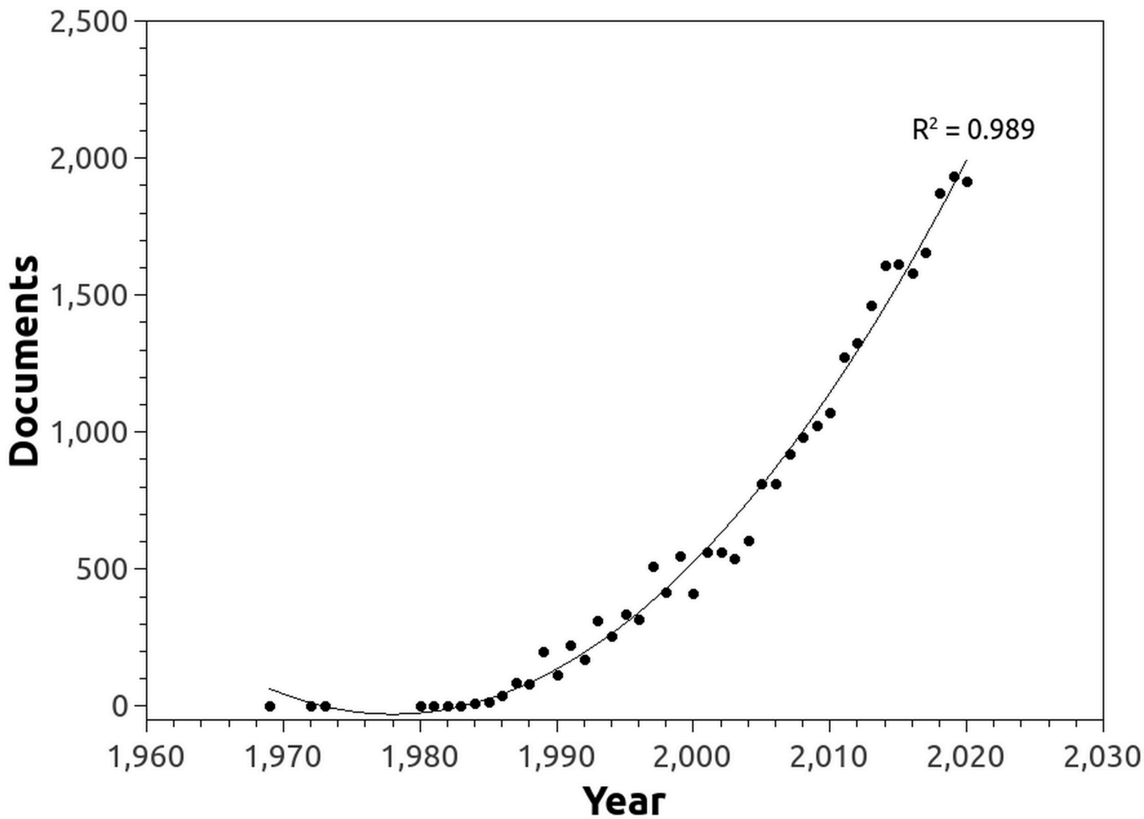

Figure 1 - Number of documents published per year using the key TITLE-ABS-KEY (polyaniline).

The data in Figure 1 follow a polynomial of order 2. The coefficient of determination (R^2) found was equal to 0.989. The model and the associated R^2 numerically demonstrate that interest in the PAni topic is accelerating since the 1980s. With the advent of nanotechnology, it is evident that these numbers will continue to grow over the next years.

Then, focusing on the key question of this research, 392 documents were found using the search key TITLE-ABS-KEY (nano* AND polyaniline AND improv* AND "electrical conductiv*" OR "electrical resistivit*") AND (LIMIT-TO (DOCTYPE , "ar")). The titles and abstracts of these documents were saved in RIS format and analyzed using

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VOSviewer software. The obtained results are shown in Figure 2. Besides, the *MAP.txt and *NET.txt files generated by VOSviewer are available at https://github.com/ftir-mc/Nanoparticles-improving-PAni-conductivity.git.

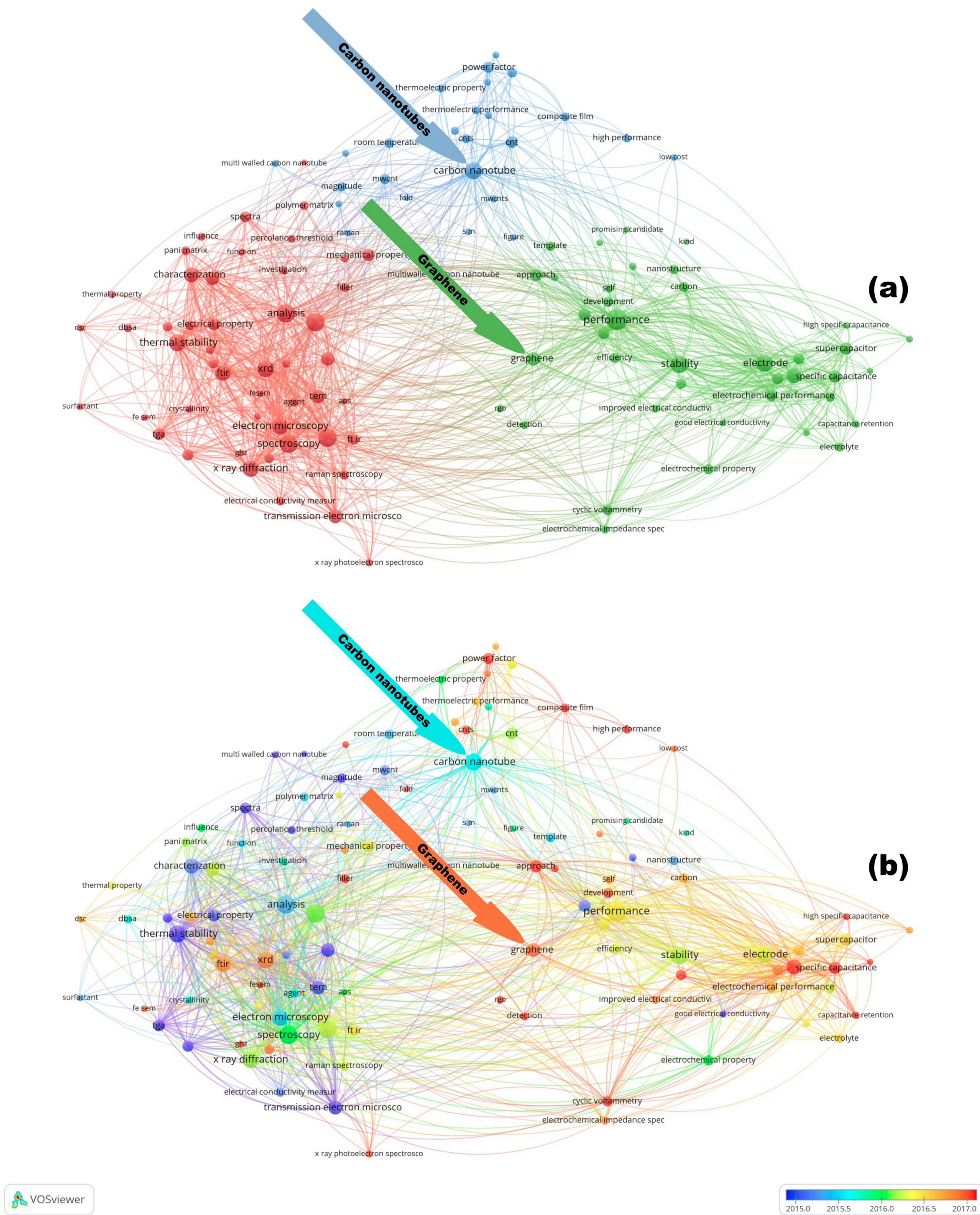

Figure 2 – VOSviewer network (a) and overlay visualizations (b) from the key TITLE-ABS-KEY (nano* AND polyaniline AND improv* AND "electrical conductiv*" OR "electrical resistivit*") AND (LIMIT-TO (DOCTYPE , "ar")).

Figure 2 (a) shows the existence of three main clusters. The first one, in red, is dominated by several nodes related to the characterization techniques used by the authors in their studies. The second cluster, in blue, is dominated by the "carbon nanotube" node, a key constituent for the production of nano-modified materials based on PAni. Finally, the third cluster, in green, is dominated by the performance, stability, and electrode nodes. Afterward, the most important material node is the "graphene", showing that this material is another relevant nanofiller in the context of improving the electrical properties of PAni. The comparison between the strengths of the terms CNT and graphene was calculated based on the "*Total link strength*" values recorded in the *MAP.txt file. The calculation allows inferring that graphene corresponds to 68.3% of the relative strength of the CNTs in the references analyzed. Furthermore, Figure 2 (b) allows determining that the year of maximum interest for the terms carbon nanotube (2015.65) and graphene (2016.89) nanofillers differs by about 1.24 years. So, they are contemporary themes.

Thus, two new keywords, "carbon nanotubes" and "graphene", gained relevance. So these two new words were added to the search key (TITLE-ABS-KEY (nano* AND polyaniline AND improv* AND "electrical conductiv*" OR "electrical resistivit*" AND graphene OR "carbon nanotub*") AND (LIMIT-TO (DOCTYPE , "ar "))), in a new refinement, which returned 177 documents (132–308).

All of these 177 documents were downloaded and their data were scratched looking for statistically relevant information for constructing the analysis here proposed. Table 1 shows the 1st author of the study and the year of publication, the used nanofiller as well as the number of experimental conditions tested, the total number of replicates, and the Adjusted Correlation (adjR).

From the 177 selected documents, 15 presented useful data for the proposed meta-analysis. However, among the documents evaluated, some had more than one useful case. So, a total of k=20 studies were included in the analysis. The observed Fisher r-to-z transformed correlation coefficients ranged from 0.3713 to 3.1320, with the majority of estimates being positive (100%). The estimated average Fisher r-to-z transformed correlation coefficient based on the random-effects model was $\hat{u} = 1.6441$ (95% CI: 1.3285 to 1.9596). Therefore, the average outcome differed significantly from zero ($z = 10.2119$, $p < 0.0001$). According to the Q-test, the true outcomes appear to be heterogeneous ($Q(19) = 165.5133$, $p < 0.0001$, $tau^2 = 0.4428$, $I^2 = 86.1258\%$). A 95% prediction interval for the true outcomes is given by 0.3022 to 2.9859. Hence, even though there may be some heterogeneity, the true outcomes of the studies are generally in the same direction as the estimated average outcome. An examination of the studentized residuals revealed that none of the studies had a value larger than ± 3.0233 and hence there was no indication of outliers in the context of this model. According to the Cook's distances, none of the studies could be considered to be overly influential. Neither the rank correlation nor the regression test indicated any funnel plot asymmetry ($p = 0.7584$ and $p = 0.3707$, respectively).

Table 1 – Statistically relevant data scratched from selected documents

Authors	Year	Filler	Experiments	Total replicates	adjR
Tung et al. (258)	2011	Graphene	5	15	0.9880
Abadad et al. (218)	2013	Graphene	11	33	0.9962
Liu & Yu. (303)	2014	CNT	5	15	0.9403
Wang et al. (274)	2015	DWCNT	4	12	0.9592
Wang et al. (274)	2015	DWCNT	6	18	0.9302
Wang et al. (178)	2016	CNT	8	24	0.9732
Yazdi & Motlagh. (295)	2017	Graphene	5	15	0.5220
Mao et al. (146)	2018	CNT	5	15	0.7332
Li et al. (228)	2018	CNT	8	24	0.5138
Kumar et al. (270)	2018	MWCNT	5	15	0.8398
Pathak et al. (220)	2019	Graphene	6	18	0.7614
Li et al. (236)	2019	CNT	5	15	0.3551
Zhang et al. (260)	2020	Graphene	5	15	0.9503
Amirabad et al. (264)	2020	Graphene	5	15	0.9643
Feng et al. (197)	2021	CNT	5	15	0.9338
Feng et al. (197)	2021	CNT	5	15	0.9812
Cho et al. (245)	2021	SWCNT	5	15	0.9233
Cho et al. (245)	2021	DWCNT	5	15	0.9785
Cho et al. (245)	2021	MWCNT	5	15	0.9236
Cho et al. (245)	2021	Graphene	6	18	0.9241

Funnel plot is shown in Figure 3.

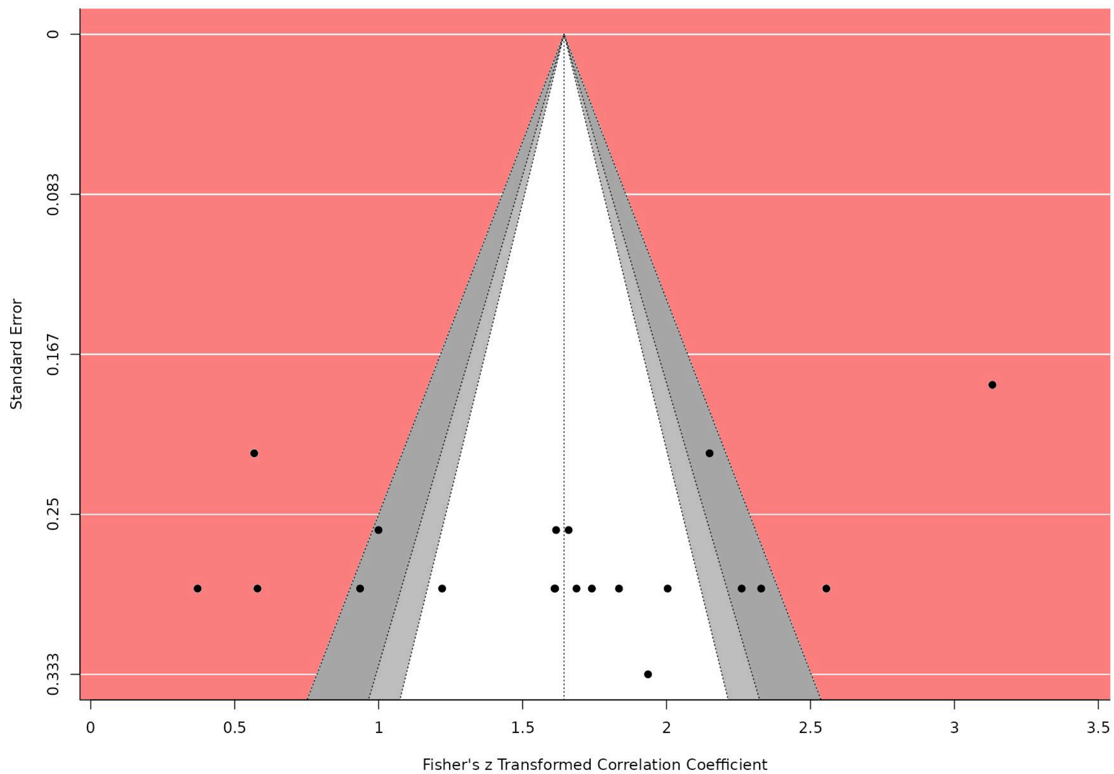

Figure 3 – Funnel plot for the selected studies.

Fisher's transformation is commonly used to eliminate a possible bias in the untransformed correlation coefficient (309). As the population value of correlation becomes further from zero, the sampling distribution of correlation coefficients becomes skewed, and Fisher's transformation normalizes this sampling distribution. Empirical evidence suggests that transforming the correlation coefficient can be beneficial (310). Mainly because many meta-analytic methods assume that the sampling distribution of observed results is normal. When the correlation in a particular study is far from zero, and the sample size is small, then the gross sample distribution of the correlation becomes skewed, no longer being closely approximated by a normal distribution. In this context, Fisher's r-to-z transformation is an effective normalization transformation, which makes the statistical analysis of correlations independent of unknown quantities (311).

On Fisher's transformation of the correlation coefficient, the actual effect is 1.64. Most studies are in the Standard Error region between 0.25 and 0.33, that is, at the base of the Funnel Plot. As described in line 287 of Jamovi MAJOR Module source code (312), the data are distributed among regions comprising 90% (white), 95% (gray), 99% (dark gray), and the rest are beyond 99% (red) probability.

Forest plots are a key-graphical method used in meta-analysis. The forest plot is the graphical representation resulting from quantitative systematic reviews. This representation is designed to compare the effects of treatments in quantitative studies. The term "forest" comes from the idea that the graph resembles a forest of lines. Originally

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the forest plot was designed to compare randomized clinical trials that addressed a common theme. Currently, however, this representation is quite prevalent in observational studies, to visually present the mathematical significance of the joint conclusions of several works analyzed as a block (313–315).

The Forest Plot, shown in Figure 4, lists all selected studies. The relevance of the studies is presented in percentage form. All studies had similar percentage relevance. The effects and their 95% probability confidence limits are shown to the right side of the percent relevance.

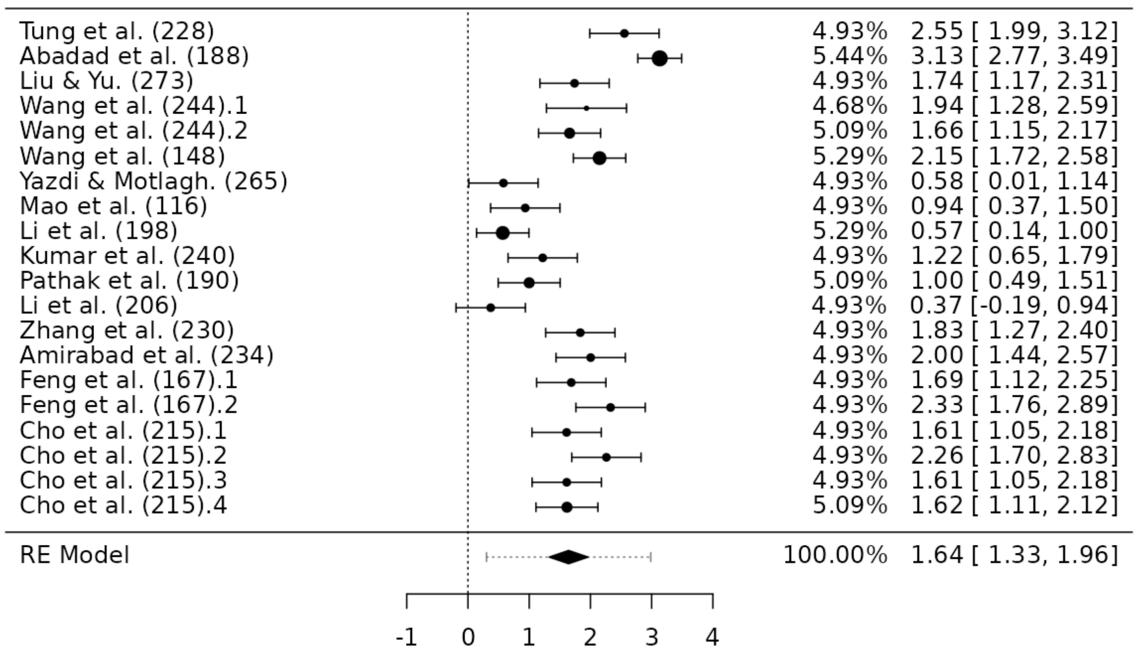

Figure 4 – Forest plot for the selected studies.

The modeled effect is equal to 1.64, with the lower bound equal to 1.33 and the upper bound equal to 1.96. Thus, the modeled effect is positive and nonzero. So, the meta-analysis proved that the nanofillers in this study increase the conductivity of PAni. Therefore, graphene and CNT should be prioritized in future works involving PAni until new evidence suggests the use of different nanofillers.

4. CONCLUSIONS

This study conducted a systematic search for several scientific pieces of research that would allow us to determine whether nanofillers can improve the electrical conductivity of PAni. Thousands of works were analyzed using VOSviewer, which concluded that graphene and carbon nanotubes are the two most studied nanofillers. Thus, hundreds of works involving PAni's modification via these nanofillers were identified from these papers. From them, fifteen presented extractable and helpful information to the meta-analysis. Meta-analysis proved that these nanofillers could effectively increase the conductivity of PAni. Therefore, graphene and CNT should be prioritized in future works involving PAni until new evidence suggests using different nanofillers.

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Sample CRediT author statement

Fernando G. de Souza Junior: Conceptualization, Methodology, Data analysis, and Writing-Original draft preparation and Supervision. **Thaís Nogueira Barradas:** Conceptualization, Methodology, Data analysis and Revision. **Vinicius Freitas Caetano:** Methodology, Data analysis and Revision. **Angela Becerra:** Methodology, Data analysis and Revision.

Conflict of interest: None

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