POLYANILINE/GRAPHENE NANOPLATELETS-SILVER (PANI/GNPs-Ag) NANOCOMPOSITES TREATED VIA NON-COVALENT SURFACE FUNCTIONALIZATION METHOD

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Abstract. PANI/GNPs is one of the most promising conductive polymer nanocomposite materials due to its unique properties, easy synthesis, and low cost. The main aim of this study was to prepare and characterize the morphological and non-covalent functionalization of GNPs nanofiller for improving the matrix-filler interaction between the PANI matrix and GNPs nanofiller. PANI/GNP nanocomposites were prepared via oxidation polymerization of aniline with various GNP nanofiller loadings at 0.25 wt.% into 1.00 wt.% of non-covalently treated GNPs. Subsequently, the PANI/GNPs nanocomposite with excellent conductor of electricity has been selected for producing PANI/GNPs-Ag hybrid-filled nanocomposites with variable Ag nanofiller loadings. The resulting PANI nanocomposite filled with 1.00 wt.% weight percent Ag nanofiller and non-covalently treated GNPs has been characterized by SEM, TEM, and FTIR analysis. SEM micrographs show that GNPs nanofillers show good interaction and dispersion within the PANI matrix. Transmission electron microscopy (TEM) demonstrated the non-covalent surface treatment influencing polymer chains in suspensions and preventing the re-agglomeration of GNPs. FTIR peaks confirmed the formation of the C-N stretching band of aromatic amine, the C-C stretching vibration in the quinoid and benzenoid rings and the C-C stretching vibration of the pyrene ring and carboxylic acid. Overall, it has been proven that the combination of conductive polymer and metal-GNP hybrid composite could change the functional group and physical characteristics of PANI-based nanocomposites.

Keywords: Polyaniline, graphene nanoplatelets, nanocomposite, morphological, non-covalent

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Introduction

In developed economies, conducting polymers play a key role as novel energy storage systems. Among all engineering materials, polyaniline (PANI) is extremely attractive as it has been applied in the field of electronics energy storage due to its high conductivity polymer, interesting redox properties, and environmental stability [1]. However, pure PANI has a lower capacity for generating power density and lacks N-active sites. Through the process of combining polymers with nano-additives like carbon materials, metal compounds, and other conducting polymers, we could fix this limitation [2]. Previous studies have reported that good interaction and distribution of GNPs as nanofiller and PANI as conductive polymers increased the morphological structure due to an effective change in the structure matrix [3].

Nowadays, there is no doubt about the efficacy of GNPs as nanofillers as it relates to their infinite molecule atomic layer properties. Although GNP has great potential, it has a zero-band gap and is inert in reaction. Therefore, several studies on the efficacy of organic and inorganic materials, chemical modification, and covalent and non-covalent surface modifications have been performed [4]. Compared with the covalent modification method, the non-covalent GNPs surface treatment shows the morphology of the conducting particles for attaining good mechanical performance and electrical conductivity. In the exploration of surface modification of GNPs nanofiller, there is a large effect on the interaction between hydrogen bonds and electrostatic forces, maintaining the bulk structure and excellent properties [5]. Modified GNPs molecular bonds are for attaching atoms or groups of molecules to sp2 carbon bonding. This functionalization is to maintain the graphene 2D lattice because of a loss in conjugated electronic. Ultrasonic treatment of GNPs nanofiller would enhance the exfoliation, dispersion and also increase the fragmentation and disordered degree of carbon atom distribution.

There are several drawbacks associated with the use of conducting polymers, such as the morphology of conducting polymers, which is limited by minimum diffusion. The conducting polymers can be inhomogeneous. Controlling the morphology during synthesis and processing is important to achieve a uniform distribution of conducting pathways, thus improving electrical performance. A hybrid combination between graphene nanoparticles and metal particles such as silver (Ag) has become a focus in this work suitable for different applications such as automotive, aerospace, and electronic packaging. The superior feature of Ag which demonstrates low contact resistance and helps minimize energy losses in electronic devices [6]. Nevertheless, processing techniques are not completely defined. For that reason, the relationship between features, a suitable matrix, and interphases is deeply investigated. However, the oxidation of Ag can decrease stability and increase resistance. In overcoming this problem, a combination of carbon-based and metallic nanomaterials could +help transfer electrons between molecules. This work aimed to characterize the morphological and physical characteristics of PANI/GNPs-Ag nanocomposite, whose surface was modified by a non-covalent approach with a focus on the effect of GNPs loading.

Materials and Methods

Raw Materials

Chemicals required for the synthesis of PANI and GNPs were purchased and used as is, without any purification steps. Anilinium chloride (AN), hydrochloric acid (HCL), and acetone were purchased from Merck KGaA (China); ammonium persulfate (APS) was purchased from R & M Chemicals (Semenyih Selangor, Malaysia). Dedocylbenzene sulfonic acid (DBSA) and polyethyleneimine (PEI) were purchased from Sigma Aldrich (USA), and graphene pure powder (10 microns) was purchased from BT Corp. Generique Nano, PVT Ltd. (India). Silver nanopowder (Ag) with a size of 60–150 nm was purchased from XFNANO (China).

Non-covalent Surface Treatment of GNPs Nanofillers

Non-covalent surface treatment of GNPs nanofiller was performed using the physical absorption method according to the previous report [7-8]. For modification, 750 ml of ethanol, 5.00 grams of GNPs and 7.50 grams of PEI were added to 250 ml of distilled water (DI). Thereafter, the solution was sonicated for 5 hours to form a homogeneous solution. At the same time, the suspension was mixed. under vigorous stirring at 500 rpm and 60 °C. The black precipitate obtained was washed for three times with distilled water to remove excess PEI and ethanol. Subsequently, the non-covalent treated GNPs were dried at 150 °C for 5 hours. The final product was obtained by grinding GNPs using a mortar and pestle to produce nanosized pieces of GNPs-PEI powder. This GNPs-PEI non-covalent treated powder was used as a raw material to synthesis the PANI/GNPs-DBSA nanocomposites.

Synthesis of PANI/GNPs/Ag Hybrid Nanocomposite

The synthesis of PANI/GNPs-DBSA hybrid nanocomposites was done by a similar oxidation polymerization route with four different non-covalent treated GNPs nanofiller loadings, which are 0.25 wt.%, 0.50 wt.%, 0.75 wt.% and 1.00 wt.% as shown in the following Table 1. In the routine PANI synthesis process, two aqueous solutions of surfactant were prepared separately. The first solution contains 15.54 grams of AN and the second solution contains 34.26 grams of APS. Both solutions were mixed with 300 ml of DI water and refrigerated at 9 °C for 12 hours. After that, an appropriate amount of non-covalent treated GNPs nanofillers was added based on their weight percentages and about 2.0 mmol of DBSA was added to the prepared surfactant. Then, the mixed solution was stirred for 1 hour and transferred to a chiller at 9 °C for 24 hours. The synthesized material was separated out using a vacuum filtration method utilizing filter paper of 11-micron sizes. After that, the filtered surfactant was washed with 300 ml of 0.20 M HCL and 300 ml of acetone. Finally, it was dried in a vacuum oven at 60 °C for 24 hours and crushed into a fine powder. Surfactant without GNPs loading was left as a control sample. However, for PANI/GNPs/Ag sample preparation, the synthesis process was repeated with various Ag loadings, with a constant GNPs non-covalent content of 1.00 wt.%.

Table 1: Sample formulation for untreated PANI/GNPs (OP) and treated PANI/GNPs by non-covalent method and hybrid PANI/GNPs(NV)-Ag nanocomposites

Specimen	Untreated GNPs (wt%)	Treated GNPs (NV) (wt%)	Ag
PANI/GNPs (OP)			
S1	0		
S2	0.25		
S3	0.50		
S4	0.75		
S5	1.00		
PANI/GNPs (NV)			
S1		0	
S2		0.25	
S3		0.50	
S4		0.75	
S5		1.00	
PANI/GNPs (NV)-Ag			
S1		0.75	0
S2		0.75	0.25
S3		0.75	0.50
S4		0.75	0.75
S5		0.75	1.00

Characterization and Testing

SEM Observation

The morphological image was captured by using the scanning electron microscope (SEM) of a model ZEISS EVO 50 system which operated at an accelerating voltage of 15 kV. To prevent the sample from charging, it was first coated with a gold-palladium sputter coating material. The powder samples were observed under magnifications of 10.00 KX, 5.00 KX, 1.00 KX and 500 X for better comparison.

TEM Analysis

TEM measurements were made using a Talos L 120 from Thermofisher, which can expand research requirements for 2D and 3D imaging and multi-modality imaging experiments such as polymer imaging. This high-resolution transmission electron microscope operates at 20–120 kV and has a point imaging platform uniquely designed for the performance and productivity of samples and applications.

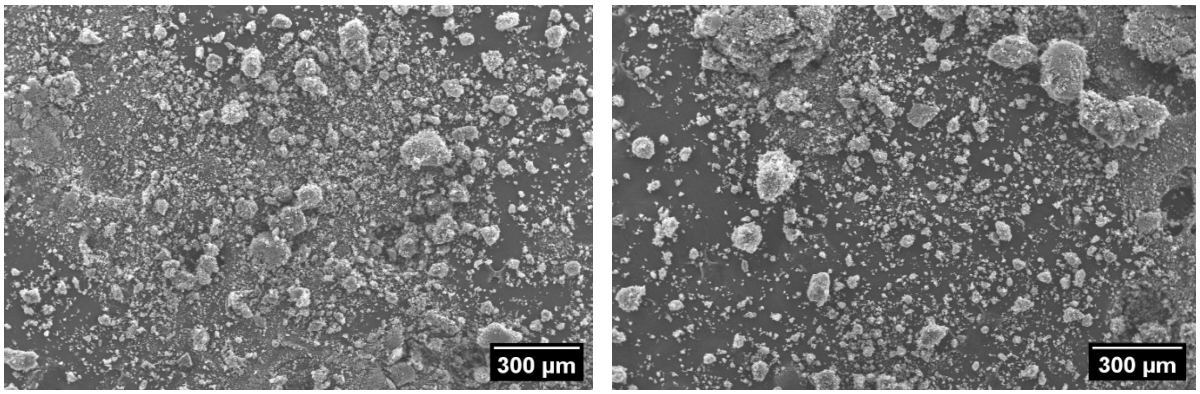
FTIR Analysis

A fourier transform infrared (FTIR) spectrometer was used to evaluate the absorption of the infrared spectrum. It was collected at room temperature by a JASCO model FT/IR-6000 spectrometer machine. The test was performed at a rapid scan up to 20 Hz (50 msecs) with a wavelength range of 600 to 1800 cm^{-1} spectral changes.

Results and Discussion

Morphological Observation using SEM

In this study, the detailed morphology of unfilled PANI-DBSA and PANI/GNPs-DBSA nanocomposite was produced by oxidation polymerization of aniline and the addition of non-covalent surface-treated GNPs nanofiller. Figure 1(a) shows the micrograph representation of the PANI powder sample in the absence of GNPs nanofiller. The DBSA addition affects the polymer active surface through the total contact area. This play an important role in the arrangement of pores in the polymer matrix, thus increased surface activity is desired for adsorption processes [9]. After the addition of untreated GNPs, the homogeneous nanofiller distribution seen in Figure 1(b) indicates multi-point electrostatic interaction between the carboxylate groups on GNPs and the ammonium group on PANI, which in turn can avoid self-aggregation of GNPs. This improved GNPs dispersion within the PANI matrix has enabled the electrons to link the gap between PANI components and pass through between the nanofillers.

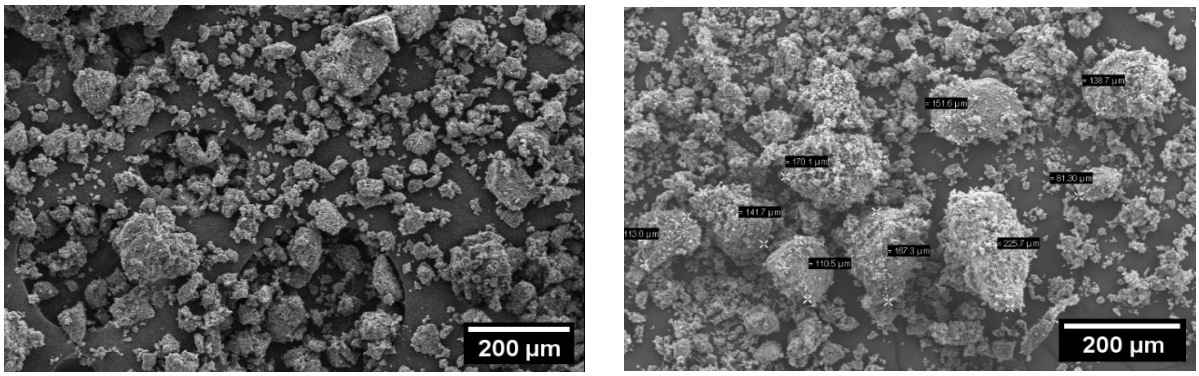


(a) (b)

Figure 1: SEM images (a) PANI/DBSA-without GNPs nanofiller addition used as a control sample at 100x magnification and (b) PANI/GNPs-DBSA at 1.00 wt.% GNPs nanofiller loading at 1000x magnification.

The covalent method can destroy the surface structure of graphene by modifying the bonding network or π-conjugation defect, resulting in a reduction in mechanical strength. Therefore, the non-covalent surface treatment approach was applied in this work. As seen in Figures 2(a), the non-covalent functionalization performed on GNPs nanofiller produced a strong bond between the GNPs nanofiller and the PANI polymer conductive matrix through hydrogen bonding and van der Waals forces. Furthermore, the reduction in lateral sizes of GNPs had occurred due to shear action during the sonication process of GNPs layers. However, the physical absorption of π-π stacking between the aromatic surfaces of GNPs could result in agglomeration [10]. It was obviously showing a higher loading of GNPs (1.00 wt.%). In Figure 2(b), the addition of Ag nanoparticles that form clusters on the surface of PANI/GNPs

introduces a new dimension to the overall morphology. These Ag nanoparticles have acted as active catalysts, which have negative charges on their surfaces to absorb the positively charged aniline cations. The electrostatic interaction has formed an aniline silver complex [11].

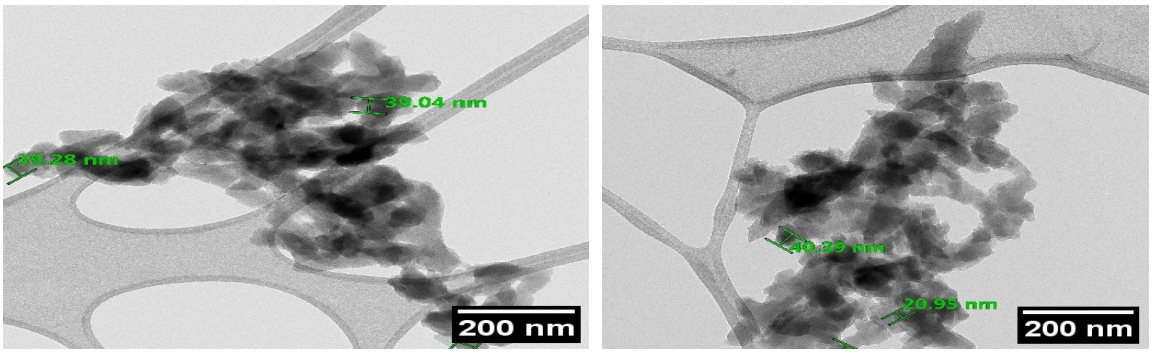


(a) (b)

Figure 2: SEM images (a) PANI/GNPs(non-covalent)-DBSA (0.75 wt.%) and (b) PANI/GNPs (NV)-Ag with 0.25 wt% Ag nanofiller loading at 100x magnification.

Microscopy Characterization using TEM

Transmission electron microscopy (TEM) observation that utilized a particle beam of electrons was used to visualize the nanocomposites structure of PANI/GNPs-DBSA nanocomposite and PANI-DBSA without GNPs nanofiller specimens. Figure 3(a) shows the PANI/DBSA morphology, which indicates the formation of nanorods in the structure of the produced PANI-DBSA [12]. The range diameter of the PANI/DBSA particle size was about 39.04 nm-39.28 nm. In Figure 3(b), the addition GNPs nanofiller to the PANI surfaces revealed that the different morphologies could maintain the strong adsorption that is responsible for causing no GNPs agglomerates depending on the range of particle size in this study of pure GNPs which is from 16.74 nm- 19.86 nm as depicted in the following Figure 3(b).



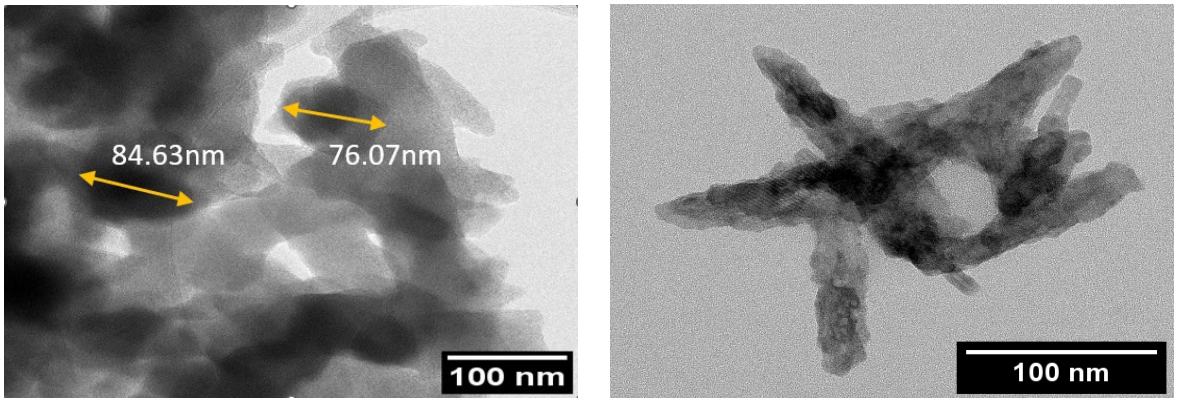
(a) (b)

Figure 3: TEM images of (a) PANI-DBSA without GNPs at 57 kx of magnification and (b) PANI/GNPs (Untreated)-DBSA with 1.0wt% GNPs loading at 57 kx of magnification

The uniform distribution of nanofiller in the PANI matrix implies good interaction between them. Homogeneous dispersion of GNPs in the PANI-DBSA matrix further improves with increasing GNPs content which has a great influence on the hardness and density of the

product. The GNPs sheet was visible in a few layers. It was found that GNPs shapes were irregular and rounded rather than sharp [13]. After multilayer accumulation of GNPs some of their sizes become smaller such as 20.95 nm and some of their sizes become larger reaching up to 40.39 nm. The porous structure and larger number of cavities entangled on the PANI surface can reduce the uniformity and structural quality of the polymer. The diameter of 12–20 nm nanomaterials facilitate electrolyte diffusion deep into the electrode material.

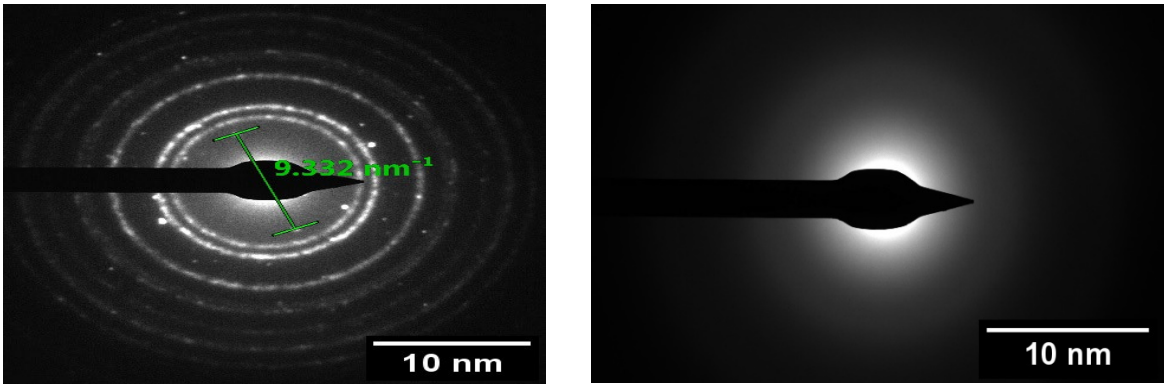
The treated PANI/GNPs-DBSA nanocomposite was prepared by a non-covalent surface treatment method. PANI polymer was densely distributed on GNPs nanofiller that could result from polymer grown by aniline monomer physisorbed. PEI adsorption onto PANI/GNPs flakes was influencing polymer chain dispersion in suspensions as shown in Figure 4(a). The equivalent sphere size distribution measurement in the size range 74.07 nm-84.63 nm maintains nanoparticle dispersion and prevents re-agglomeration. The obtained TEM results agree well with the SEM micrograph image of the GNPs distribution. GNPs untreated were composed of dual sheets with intact edges. After surface treatment, the flakes tend to wrinkle, fold, and crumple at the edges due to the prior acid treatment [14]. Figure 4(b) indicates the TEM image of an Ag coated GNP sheet. PANI/GNPs (treated)/Ag-DBSA detailed analysis showed coral reef-like structure and extensively agglomerated morphology on Ag nanoparticles. Agglomeration also reduced the even distribution of Ag filler nanoparticles in the PANI/GNPs-DBSA polymer matrix leading to a decrease in conductivity due to a lack of electron flow.



(a) (b)

Figure 4: TEM micrographs of (a) PANI/GNPs(treated)-DBSA with 1.0 wt% loading of GNPs in noncovalent treatment at 120 kx magnification and (b) PANI/GNPs(treated)/Ag-DBSA with 1.00 wt% loading of GNPs in noncovalent treatment at 120kx magnificant. The Ag loading is 1.00 wt%.

The selected area of electron diffraction pattern (SAED) of PANI-DBSA and PANI/GNPs non-covalent treated is observed. The four crystal diffraction planes display the typical polycrystalline nature [15]. Some diffuse and blurry rings proved the amorphous phase of the external structure of HCl doped PANI [16]. The bending of electron beams was surrounded by granular particulate material that was associated with the hydroxide phase, which shows oxygen and hydrogen compounds in Figure 5(a). A core–shell morphology is mainly observed whether the particles are isolated, or part of a chain-like configuration as represented in Figure 5(b). In PANI/GNPs-Ag, the particles' chain-like configuration shows a passivating layer when exposure to small amounts of oxygenated groups is affecting metal oxide.



(a) (b)

Figure 5: Selected area of electron diffraction pattern (SAED) of (a) PANI-DBSA and (b) PANI/GNPs (non-covalent)-DBSA

FTIR Analysis

FTIR was used to evaluate the presence of functional groups on PANI-GNPs nanocomposites. The spectroscopy of PANI-DBSA, PANI/GNPs (untreated) and PANI/GNPs (treated) with various GNPs nanofiller loadings was evaluated to further confirm the characteristics and related chemical properties of the produced nanocomposites. In Figure 6, it is nearly identical that the bands in the range of 1200–1400 cm^{-1} (1220 cm^{-1} and 1370 cm^{-1}) are the C-N stretching band of an aromatic amine. These have clearly indicated the existence of PANI on the surfaces of GNPs. The polymer chain has grown and broadened the interlayer gap of graphene platelets surfaces. The 550 cm^{-1} peak which refers to C-H out of plane bending vibration. In Figure 7, for PANI/GNPs with the addition of non-covalent treated GNPs the peak at 1745 cm^{-1} of PANI/GNPs nanocomposites had shown the disappearance of the carboxylic acid (-C=O-OH) functional group. The process of removing hydrogen from the carbon atom adjacent to the carbonyl group, which producing formation of the carboxylic acid [17]. The several new peaks like 1523 cm^{-1} and 1434 cm^{-1} have appeared in the IR spectrum of PANI/GNPs-OP which are attributed to C=C stretching vibrations in the quinoid and benzenoid rings. This common feature of PANI can highly improve the electrical conductivity since emeraldine salt has been successfully grafted into the GNPs surface. As for PANI/GNPs (0.75 wt.%) surface treatment, the vibration at 1677 cm^{-1} has indicated the existence of the C=C stretching vibration of the pyrene ring [18]. The stretching peaks at 1556 cm^{-1} and 1108 cm^{-1} are belong to the -NH2 bending vibration and the C-N stretching vibration, respectively [19]. The deformation peak at 877 cm^{-1} is due to the N-H deformation vibration of primary amine, which indicates the chemical state of nitrogen in PANI [20]. The FTIR results confirmed the effectiveness of grafting GNPs functional groups onto the PANI surfaces, which in turn can improve the stability of the produced nanocomposites.

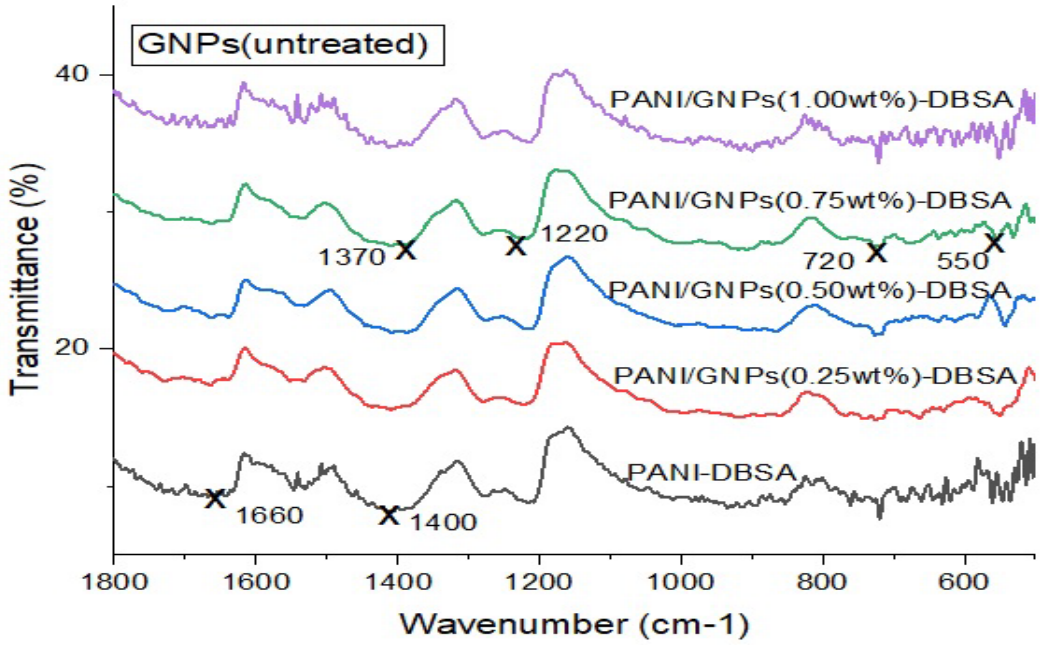


Figure 6: PANI-DBSA, PANI/GNPs (untreated)-DBSA with various GNPs nanofiller loadings

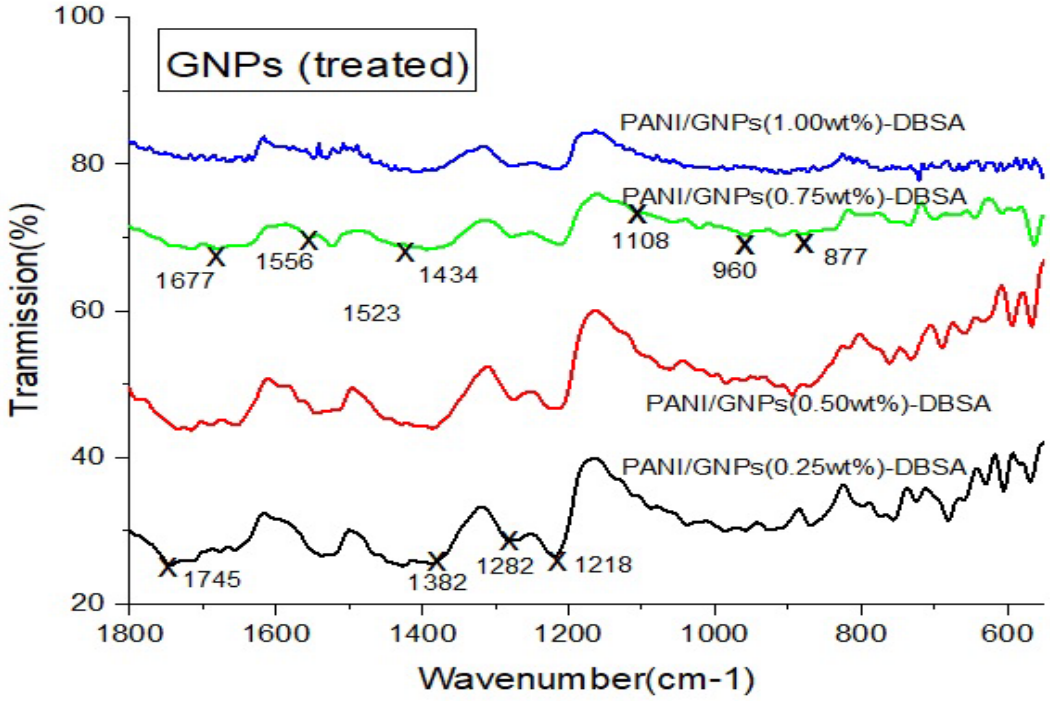


Figure 7: Result of PANI/GNPs (treated)-DBSA with various GNPs loading non-covalent surface treatment GNPs.

Conclusions

In the present study, PANI/GNPs-DBSA was synthesis by the oxidation polymerization of aniline method that provides good interaction between the GNPs nanofiller and the PANI polymer matrix. The morphological characteristics have clearly shown a homogeneous distribution of GNPs nanofiller along the PANI matrix, which enables the electrons to link the gap. In TEM, the surface treatment of GNPs flakes tends to wrinkle at the edges and maintains nanoparticle dispersion. Ag nano-additive shows a coral reef-like structure and is extensively agglomerated. In the FTIR analysis, it was explained non-covalent treated GNPs sample has indicated new peak formation and physical structure compared to PANI/GNPs without surface treated of GNPs nanofillers. Basically, by increasing of 0.25 wt.%, 0.50 wt.%, 0.75. wt% and 1.00 wt.% loading GNPs conductive polymers could have modified the microscopy properties of composites.

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Author Contributions

N.A. Khalid: Conceptual, data analysis, writing original; J. Abd Razak: conceptual, investigation, writing, review & editing, supervision; M.M. Ismail: conceptual, investigation, writing, review & editing, supervision; M.H. Flaifel: conceptual, investigation, writing, review & editing; P. Puspitasari: conceptual, investigation, writing, review & editing.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

Reference

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