



Preparation of a New Formulation of Hybrid GNP/Ag Conductive Ink with a Specific Ratio of Organic Solvent

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ABSTRACT

This paper presents the preparation of a new formulation of hybrid GNP/Ag conductive ink with a specific ratio of organic solvent to optimize the properties of hybrid conductive ink. The GNP/Ag hybrid conductive ink was formed with a different ratio of organic solvents which are 33:67, 60:40, 67:33 and 40:60 of 1-butanol to terpinol which the ratio of conductive filler is higher than the organic solvent. The performance of each ink was examined in terms of bulk resistance and resistivity where the conductive ink was prepared on the copper substrate and cured for five hours in the oven at a temperature of 250°C and underwent a cyclic bending test. Results indicate that the 40:60 ratio of 1-butanol to terpinol is the best ratio for GNP/Ag hybrid conductive ink with a bulk resistance of 0.600Ω and resistivity of $3.6 \times 10^{-4} \Omega \cdot m$. For further exploration, the curing time should remain five hours at a different curing temperature.

1. Introduction

Graphene has emerged as a leading material for formulating electroconductive ink in flexible electronics because of the excellent chemical and thermal stability, incredible stretch ability, flexibility and superior charge carrier mobility of the material according to Ahmed *et al.*, [1]. Graphene was classified by Etri *et al.*, [2] as a carbon allotrope, consisting of one-atom-thick planar sp^2 layers of carbon atoms tightly packed within a crystal honeycomb lattice and graphene acts as a coating to enhance other materials and can be integrated into composite materials. Graphene consists of carbon atoms bound in sp^2 configurations, forming a perfect two-dimensional lattice with exceptional properties in line with Mohammad *et al.*, [3]. The bond length between C-C atoms measuring 0.142 nanometres, graphene exhibits sp^2 hybridization and demonstrates high thermal and electrical conductance. The thermal and electrical conductivity of graphene is approximately 5300 W/mK and 106 S/m, respectively, representing the highest values recorded for any solid

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materials obtained by Kumar *et al.*, [4]. Graphene particles have improved thermal conductivity, mechanical strength and electrical conductivity as stated by Lewis *et al.*, [5] and it is an excellent conductor of electricity and has exceptional sensitivity to changes in the surrounding environment according to Mohd *et al.*, [6]. Even though graphene is regarded as the revolutionary material of the 21st century, graphene synthesis is complex and produced in small quantities. Therefore, graphene nanoplatelets (GNP) become the alternative with the potential for large quantities of production. The GNP has a few graphite layers with thicknesses from 0.7 to 100 nm and is widely used as nanofillers with different matrices which enhance the mechanical and tribological behaviour, increasing the barrier properties and thermal conductivity, transforming insulating matrices into electrical conductors and acting as flame retardant. The dispersion of GNP with the achieved exfoliation degree affects the properties of manufactured composites was determined by Suarez *et al.*, [7].

Conductive ink is a technology that merges the advantage of ink with the ability to conduct electricity through formulation by depositing the conductive nanomaterial with a combination of a non-conductive material which enables the flow of electrons to pass through the ink according to Dmitriev *et al.*, [8]. The GNP and silver flake (Ag) particles are commonly used in stretchable conductive inks because of their unique electrical properties as determined by Mohd *et al.*, [9]. Conductive inks are composite materials that are combined of a binder matrix, conductive filler, solvent and additives. Many kinds of conductive nanomaterials, including metals-based (silver (Ag), gold (Au), copper (Cu)), conducting polymers and other conductive organic materials like carbon-based inks (graphene, graphene oxide, carbon nanotube) have been employed for various types of printed flexible and wearable electronic applications consistent with Syed *et al.*, [10]. Ag-based conductive inks are the most common type in the market, however, carbon-based conductive inks are progressively accepted. Conductive inks prepared using metal nanoparticles such as gold nanoparticles (AuNPs), silver nanoparticles (AgNPs), and copper nanoparticles have a great influence on electrical conductivity, but these metal nanoparticle materials (AuNPs and AgNPs) are expensive and require high sintering conditions that restrict their application to flexible electronics. Besides the electrical conductivity of polymer inks is comparatively lower than metal nanoparticles in accordance with Amit *et al.*, [11] and these limitations are overcome after introducing graphene-based ink.

Solvents are the main liquid component in graphene conductive ink preparation to ensure proper dispersion and desired viscosity for different printing methods. Solvents are added to the formulation to dissolve the resins and control the properties of ink rheological was introduced by Wang *et al.*, [12]. Graphene conductive inks traditionally rely on organic solvents like N-methyl-2-pyrrolidone (NMP) and dimethylformamide (DMF) according to Tran *et al.*, [13], but these solvents suffer from a high boiling point (>150°C) and low-temperature treatment is required restricted them from being used with plastic substrate besides highly expensive and toxic consistent with Bhat *et al.*, [14].

Solvents with lower boiling points such as acetone, isopropanol and ethanol were used for the dispersion of graphene effectively while maintaining suitable surface tension according to Tran *et al.*, [15]. However, these lower boiling point solvents typically require extended sonication periods to achieve optimal dispersion, which may reduce sheet size and introduce defects affecting graphene properties. A solvent exchange technique is frequently employed to lessen toxicity concerns and this technique involves a heat ultrasonication process utilizing two solvents with different boiling points, facilitating solvent exchange via the heat ultrasonication process conducted by Jiantong *et al.*, [16].

Graphene conductive inks can be prepared with a solvent exchange process that may utilize DMF with terpineol, allowing for improved dispersion while addressing toxicity issues. Lateral graphene flake inks were printed and fired at 400 °C for several hours to achieve a surface resistance of around 200 kΩ/sq. In another study, graphene-based conductive inks were formulated through a solvent exchange process involving DMF and a blend of terpineol and cyclohexanone. The resulting

conductive film revealed a sheet resistance of 1-2 kΩ/sq following annealing at 400 °C was determined by Jiangtong *et al.*, [17]. Table 1 shows past reviews on solvent-exchanged graphene conductive inks employing various printing methods. The DMF is chosen in these formulations because of its low viscosity, enabling shorter sonication durations. Nevertheless, the high annealing temperatures ranging from 100 °C to 400 °C are necessary to improve electrical conductivity. Therefore, there is a pressing need for fewer toxic and lower annealing temperature conductive inks to enable the production of flexible electronic circuits. Despite this need, a comprehensive literature review has revealed a lack of research exploring the performance of conductive inks prepared via solvent exchange techniques employing different solvents.

Table 1

Summary of previous studies on solvent exchange graphene conductive inks reviewed by Htwe *et al.*, [18]

Types of solvent exchange	Printing method	Substrate	Annealing conditions	Sheet resistance
DMF exchange to terpineol/ethanol	Inkjet	Glass	400°C for 60 min	200 kΩ/sq
NMP/vinyl acetate in isopropanol	Screen	PET	100°C for 5 min	30 kΩ/sq
DMF to cyclohexanone, terpineol	Inkjet	Glass	400°C	1-2 kΩ/sq

Previous research has extensively explored hybrid conductive ink; however, there has been a significant lack of detail concerning the formulation of organic solvent or capping agent in the production process of conductive ink and complete investigation into this matter remains limited. This paper concentrates on the specific ratios of organic solvent with the formulation of GNP/Ag hybrid as a conductive ink. The organic solvents discussed are 1-butanol and terpinol and the performance is observed in terms of the condition of the ink and electrical performance.

2. Materials and Method

The materials utilized in this research are GNP with a particle size of 25 μm as in Figure 1 and a surface area of 150 m²/g, as well as Ag flakes (10 μm, ≥99.9% trace metals basis) in Figure 2, Ag acetate as in Figure 3 and ethanol 99.9% as in Figure 4, where all materials are sourced from Merck KGaA, Darmstadt, Germany. In addition, terpinol was acquired from Gouden Sdn. Bhd. The primary fabrication process throughout the experiment relied on the sonication technique using an ultrasonic bath. The GNP 25 μm powder and Ag flakes are used as a filler material whereas 1-butanol and terpinol are used as binder exchange techniques and hardener.



Fig. 1. GNP



Fig. 2. Ag flakes



Fig. 3. Ag acetates



Fig. 4. Ethanol 99.9%

2.1 Preparation of Hybrid Powder

A solution containing 0.3 wt% of GNP was prepared by dissolving the GNP in 5 ml of ethanol. The GNP solution (protected with aluminium foil) as in Figure 5 underwent sonication in ethanol using an ultrasonic machine in Figure 6 for ten minutes. Following this, 0.4292 g of Ag flakes were added to the solution and sonication continued for one hour. Subsequently, 0.042g of Ag acetate was added to the mixture, which was then sonicated for an additional one hour. The dispersibility between the GNP/Ag in ethanol was monitored during the sonication process.

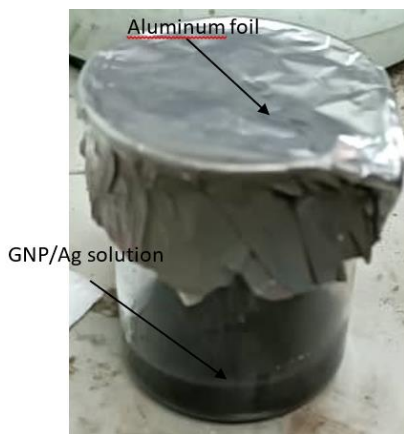


Fig. 5. GNP/Ag solution



Fig. 6. Ultrasonic machine

The mixed solution was then heated on a hotplate under constant stirring (200 rpm) at 70 °C using a magnetic stirrer as in Figure 7 until most of the ethanol had evaporated into the environment as in Figure 8.

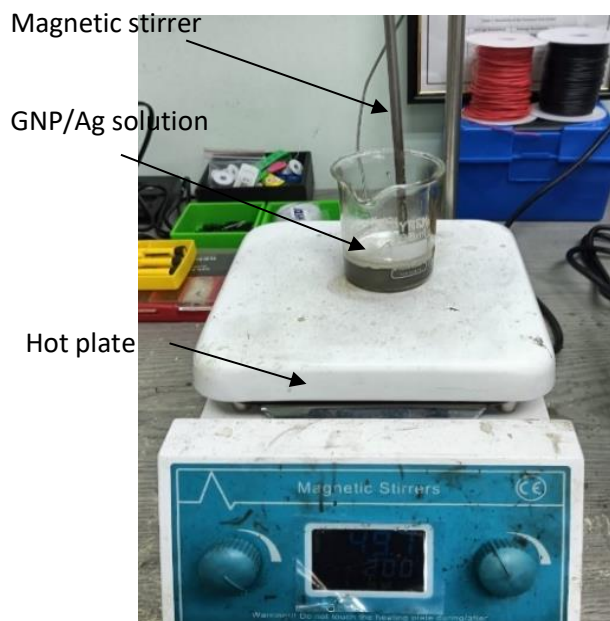


Fig. 7. Magnetic stirrers

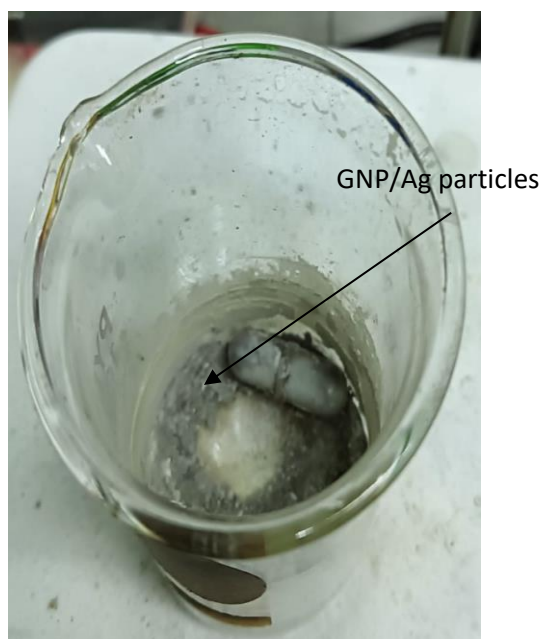


Fig. 8. GNP/Ag particles after stirring process

The mixture was then collected as in Figure 9 and then dried in an oven for one hour at 250 °C using a reflow oven in Figure 10. Finally, the dried hybrid material was collected, cooled down and pounded into a fine powder using a mortar.



Fig. 9. GNP/Ag particles collected



Fig. 10. Reflow oven

2.2 Organic Solvent in Fabrication Hybrid Conductive Ink

For this experiment, a blend of 1-butanol and terpinol was selected as the organic solvent combination for generating conductive ink. The GNP/Ag hybrid powder was measured as in Figure 11 and mixed with varying ratios of 1-butanol to terpinol as in Figure 12 and then mixed by using a Thinky mixer in Figure 13.

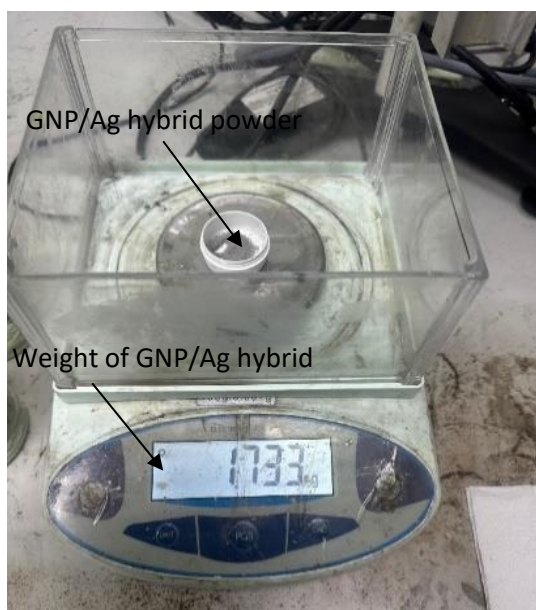


Fig. 11. Weight scale

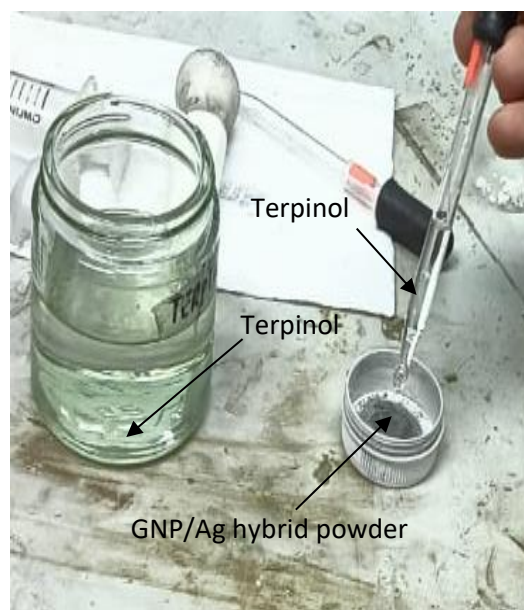


Fig. 12. Solvent addition

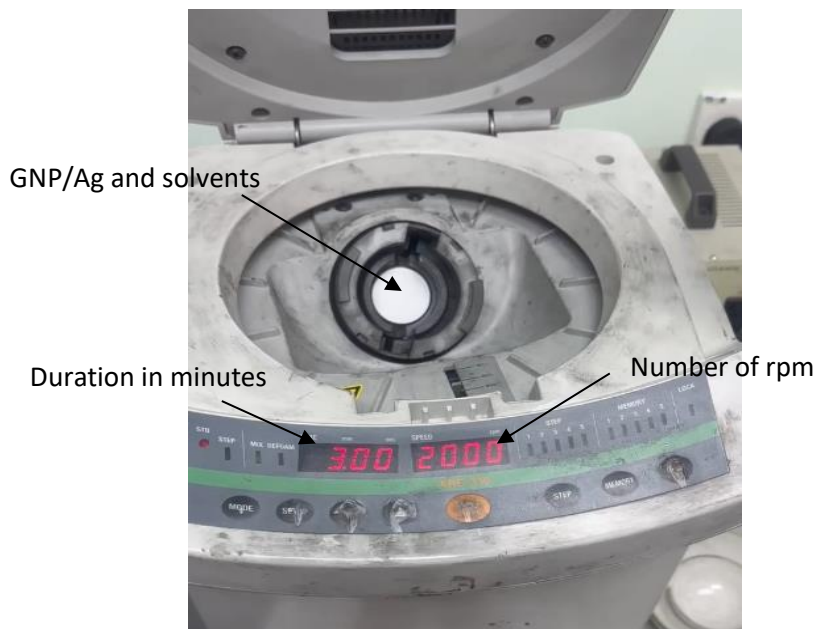


Fig. 13. Thinky mixer

The ratios employed were 33:67, 60:40, 67:33 and 40:60 of 1-butanol to terpinol, as summarized in Table 2.

Table 2
 Weight and percentage of the solvent

Sample	1-butanol			Terpinol			GNP hybrid powder g
	Drop	%	g	Drop	%	g	
5-butanol	5	33	0.100	10	67	0.280	1.733
15-butanol	15	60	0.300	10	40	0.280	1.733
5-terpinol	10	67	0.200	5	33	0.140	1.733
15-terpinol	10	40	0.200	15	60	0.420	1.733

The GNP/Ag hybrid conductive ink paste in Figure 14 was applied onto copper substrates in Figure 15 using manual stencil printing as shown in Figure 16. The mesh stencil, with a thickness of $60 \mu\text{m} \pm 2 \mu\text{m}$, was carefully positioned over the substrate measuring 12 cm x 1 cm, aligning with a 0.3 cm x 0.3 cm selected grid at the desired location. The GNP/Ag hybrid conductive ink was then placed on top of the surface of the stencil. Employing a squeegee, consistent pressure was exerted to evenly spread and force the ink through the holes of the stencil onto the substrate. Once the pattern was successfully transferred, the stencil was carefully lifted off. The ink was printed at five designated points, as depicted in Figure 17.

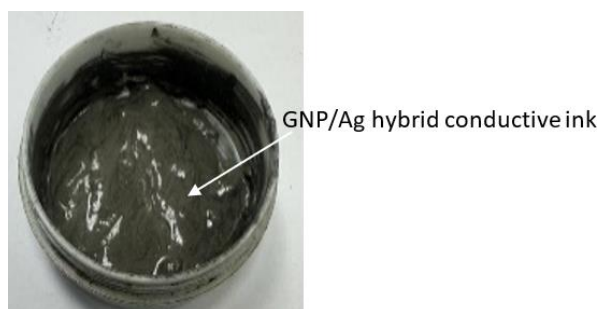


Fig. 14. GNP/Ag hybrid conductive ink paste

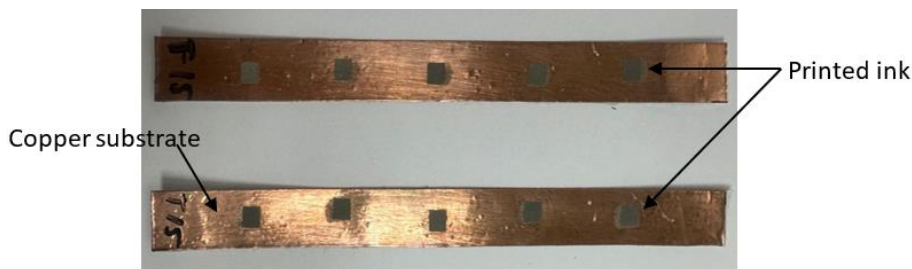


Fig. 15. Copper substrate

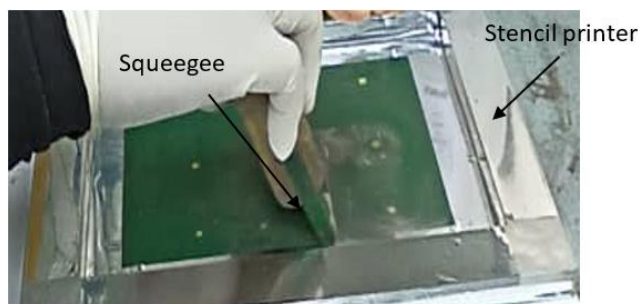


Fig. 16. Manual stencil printing

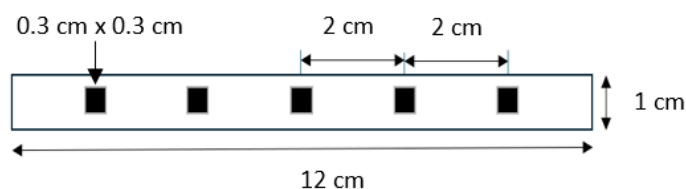


Fig. 17. Dimension of the copper substrate

The printed substrate underwent thorough inspection for uniformity and accuracy, with adjustments made where necessary. The mesh stencil was cleaned and the procedure was repeated for the next samples. The printed conductive ink as in Figure 18 was cured in Reflow oven for five hours at a temperature of 250 °C.



Fig. 18. GNP/Ag conductive ink for curing process

2.3 Resistivity in GNP/Ag Hybrid Conductive

The resistance of each conductive ink was assessed using a Two-Point probe at four designated points (1,1) (2,2) (3,3) (4,4) as depicted in Figure 19. Subsequently, the printed paste underwent a

cyclic bending test across 1000, 2000, 4000, 8000 and 16000 cycles to simulate real conditions. The surface structure of the paste was then observed using a light microscope.

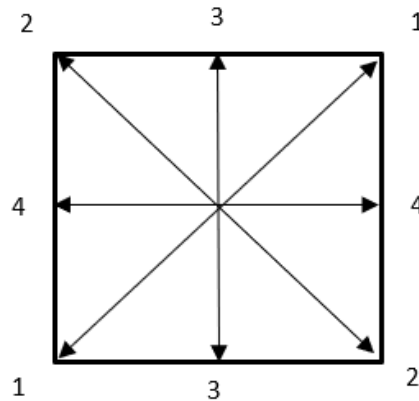


Fig. 19. Four Points measurement of resistance of GNP/Ag hybrid conductive ink

The printed ink goes through a cyclic bending test as in Figure 20 for 1000, 2000, 4000, 8000 and 16000 cycles to simulate real conditions which measure the durability of the sample.

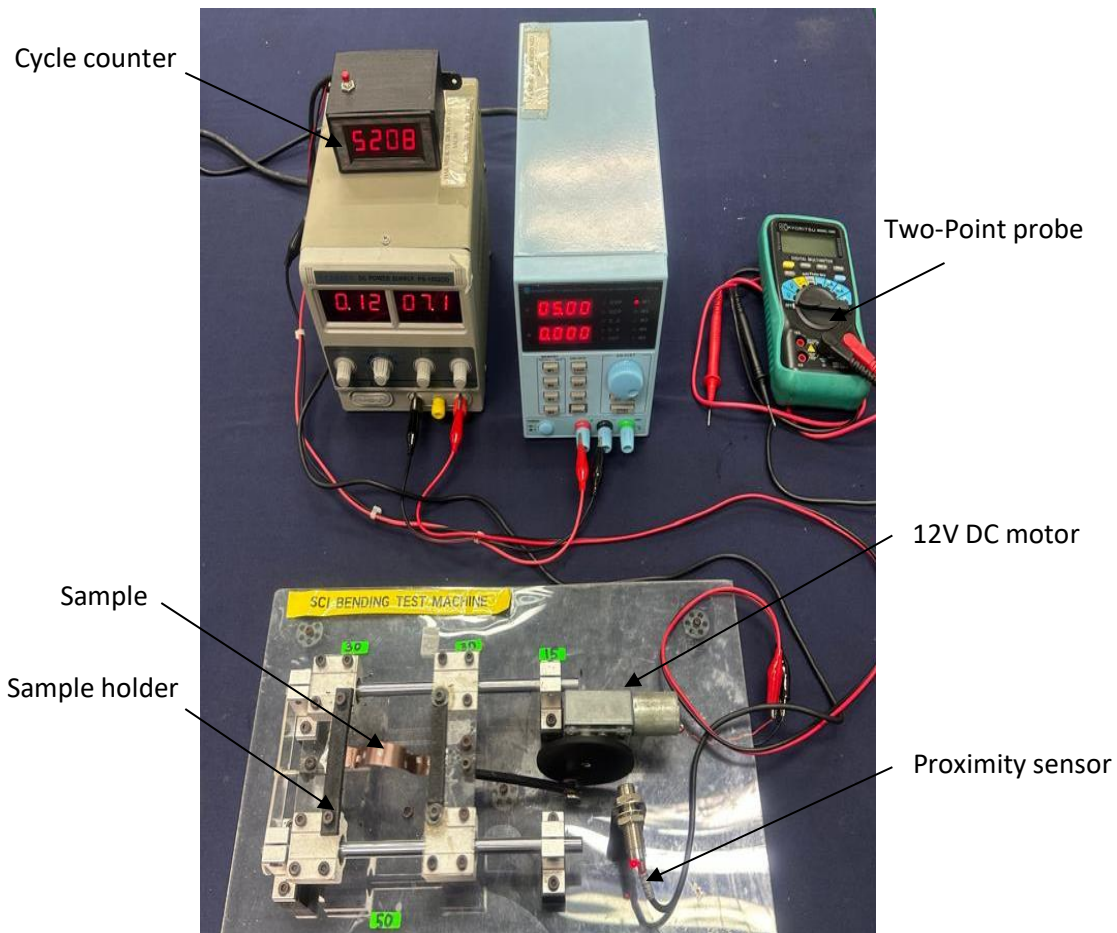


Fig. 20. Cyclic bending test equipment

3. Results and Discussion

The GNP/Ag hybrid solution was prepared by adding 5 ml ethanol into the GNP in a beaker. Before the curing process was done, the GNP/Ag hybrid solution was subjected to 10 minutes of sonication in ethanol using the ultrasonic machine. After the sonication process, 0.4292g of Ag flakes was added into the solution, which was then sonicated again for another one hour. The observation was done and it showed that the GNP/Ag hybrid solution dispersed thoroughly in ethanol after one hour of sonication and a few sediments appeared after 24 hours of preparation. These results suggest that GNP/Ag can form a relatively stable dispersion in ethanol, consistent with findings from previous research conducted by Xiaoyan *et al.*, [19].

After the sonication process, GNP/Ag solution was heated on a hotplate under a constant stirring (200 rpm) at 70 °C using a magnetic stirrer to dissolve the ethanol then dried in the oven for one hour at 250 °C to make sure the ethanol dissolved completely. Figure 21 shows the dried GNP/Ag material after being heated. The materials are stuck together and coarse which need to be pounded until fine before being used to fabricate a conductive ink.

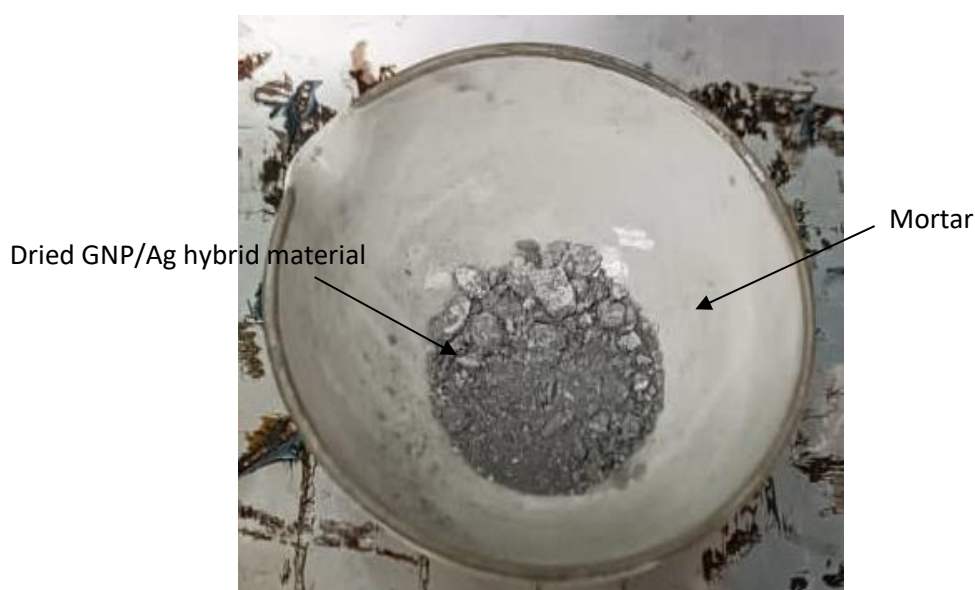






Fig. 21. GNP/Ag Hybrid after 1 hour cured at 250 °C

For this experiment, 1-butanol and terpinol were selected as an organic solvent for conductive ink fabrication, based on a previous study conducted by Shaojia *et al.*, [20]. Initially, optimization of the organic solvent ratios was performed separately for GNP and Ag. The primary challenge involved determining the appropriate ratio of organic solvent when the quantity of GNP/Ag flake was held constant. However, for this research, the solvents used are combined.

Table 3 shows the results of GNP/Ag hybrid conductive ink fabricated with a different ratio of organic solvents which were 33:67, 60:40, 67:33 and 40:60 of 1-butanol to terpinol and all the ratios of conductive filler are higher than the organic solvent. The GNP/Ag hybrid conductive ink fabricated are shown in ink form and all conductive paste look metallic in appearance and more to dark grey in colour. For 5-terpinol and 15-terpinol conductive ink are slightly thicker than 5-butanol and 15-butanol, however, all conductive inks are more viscous than regular ink.

Table 3

Result of GNP/Ag hybrid conductive ink with different organic solvent ratios of 1-butanol to terpinol with the value of 33:67, 60:40,67:33 and 40:60

Sample	GNP/Ag(g)	1-butanol	Terpinol	Ratio (1-butanol: terpinol)	Ratio (filler: solvent)	Observation
5-butanol	1.733	0.100	0.280	33:67	77:23	
15-butanol	1.733	0.300	0.280	60:40	65:35	
5-terpinol	1.733	0.200	0.140	67:33	80:20	
15-terpinol	1.733	0.200	0.420	40:60	62:38	

The surface structure of the GNP/Ag conductive ink was observed using a light microscope. Table 4 shows the GNP/Ag hybrid conductive ink under the light microscope. For all conductive inks once applied and dried onto the copper substrate, the ink has a smooth or slightly grainy texture. The conductive particle forms a conductive path that can be used to circuit. However, the level of electrical conductivity depends on the type and concentration of the conductive material used. After drying or curing, the conductive ink adheres well to the copper substrate and can withstand various environmental conditions, although this can depend on the specific formulation and intended application.

Table 4

Image of GNP/Ag hybrid conductive ink with different organic ratios of 1-butanol to terpinol under a light microscope





Sample	GNP/Ag (g)	1-butanol	Terpinol	Ratio (1-butanol: terpinol)	Ratio (filler: solvent)	Observation
5-butanol	1.733	0.100	0.280	33:67	77:23	
15-butanol	1.733	0.300	0.280	60:40	65:35	
5-terpinol	1.733	0.200	0.140	67:33	80:20	
15-terpinol	1.733	0.200	0.420	40:60	62:38	

Table 5 shows the bulk resistance of different formulations of organics solvent with a curing temperature of 250 °C. The bulk resistance shows that after the curing process ratio of 33:67, 1-butanol to terpinol gives the lowest value of bulk resistance 0.500 Ω and a ratio of 40:60, 1-butanol to terpinol gives the highest value of bulk resistance 0.600 Ω.

Table 5

Bulk resistance of GNP/Ag conductive ink at different ratios of organics solvent with the curing temperature of 250 °C

GNP/Ag conductive ink				
Ratio	33:67	60:40	67:33	40:60
Bulk resistance (Ω)	0.500	0.510	0.525	0.600

Figure 22 illustrates the graph of bulk resistance measurements for different ratios of GNP/Ag hybrid conductive ink. Lower resistivity values signify enhanced electrical conductivity, thus describing the material as more effective for electronic applications and electrical performance. The electrical conductivity of the resulting ink can be influenced by the solvent ratio. The right

combination can enhance the contact between GNP and silver particles improving the overall conductivity of the printed ink.

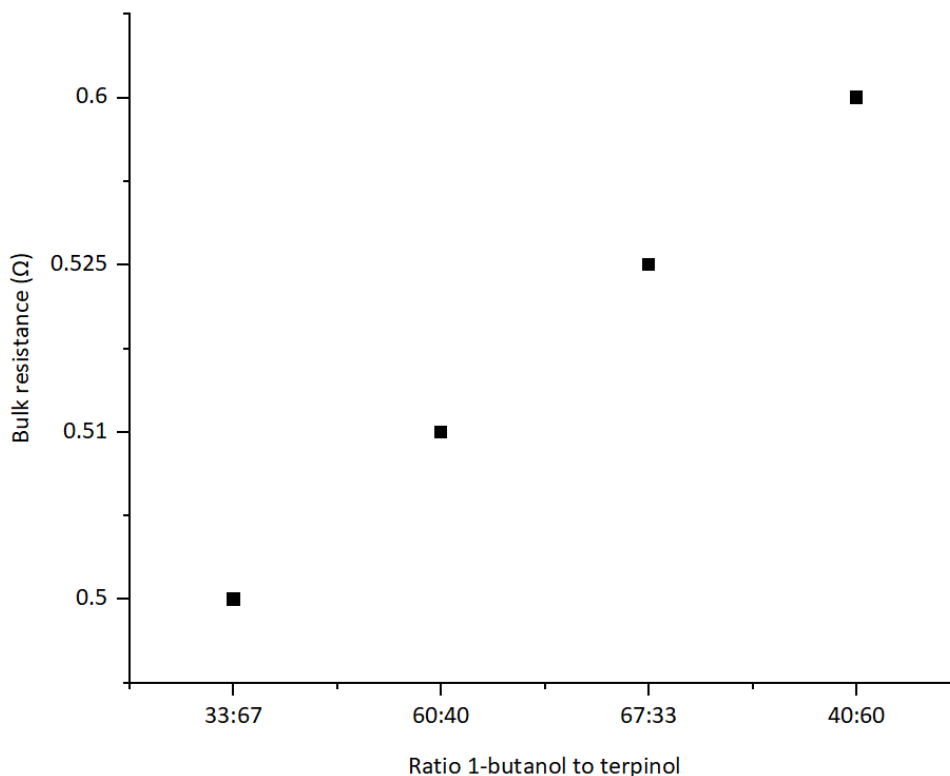


Fig. 22. Bulk resistance measurement of different ratios of GNP/Ag hybrid conductive ink

Table 6 shows the average resistance and resistivity measurements for 5-Terpinol GNP/Ag hybrid conductive ink before and after the cyclic bending test. The average resistance and resistivity value of the GNP/Ag hybrid conductive ink is higher before the test at 0 cycles and decreases after 1000 cycles. During the initial phase of bending, the conductive ink undergoes a form of settling or restructuring. The mechanical stress causes better alignment or compaction of the GNP and Ag particles leading to improved electrical pathways and decreased resistance and resistivity.

However, the average resistance and resistivity increase after 2000 and 4000 cycles and tend to drop at 8000 and 16000 cycles. As the number of bending cycles increases beyond 1000 cycles, the material starts to experience microcracking and fatigue. These structural damages disrupt the conductive network within the ink, causing an increase in resistance and resistivity. This phase reflects the deterioration of the conductive properties of the material because of repeated mechanical stress. The increase in average resistance and resistivity is associated with the deformation of the conductive pathway of the ink, which can lead to cracks formation or gaps in the ink layer as Kausar *et al.*, [21] found in the findings.

At higher cycles (8000 and 16000) there is a tendency for the resistance and resistivity to drop again because of a phenomenon known as 'self-healing' where the conductive pathways might re-establish themselves because of further compaction or realignment under continuous stress or elimination of weak pathways according to Sánchez *et al.*, [22].

Table 6
 The 5-terpinol average resistance and resistivity

Cycle	Resistance (Ω)	Resistivity (Ωm)
0	0.525	3.15×10^{-5}
1000	0.425	2.55×10^{-5}
2000	0.550	3.30×10^{-5}
4000	1.175	7.05×10^{-5}
8000	0.625	3.75×10^{-5}
16000	0.350	2.10×10^{-5}

Figure 23 displays the graph of 5-Terpinol average resistance and resistivity. It shows a trend of increasing average resistance and resistivity after certain cycles (4000 cycles) and decreasing after certain cycles (8000 and 16000 cycles). Resistance may increase caused by several factors. Temperature changes can lead to increased resistance in materials because of the heightened atomic vibrations, which impede the flow of electrons. Additionally, mechanical stress can alter the structure of a material, hindering electron movement and resulting in higher resistance. Contamination from foreign substances or impurities can disrupt electron flow pathways, causing increased resistance. Chemical reactions induced by exposure to certain substances can also change the conductivity of the material, causing resistance to rise. Moreover, because of time, materials may degrade or change properties, resulting in increased resistance. Finally, defects or damage to the structure of the material could impede electron flow, causing resistance to increase.

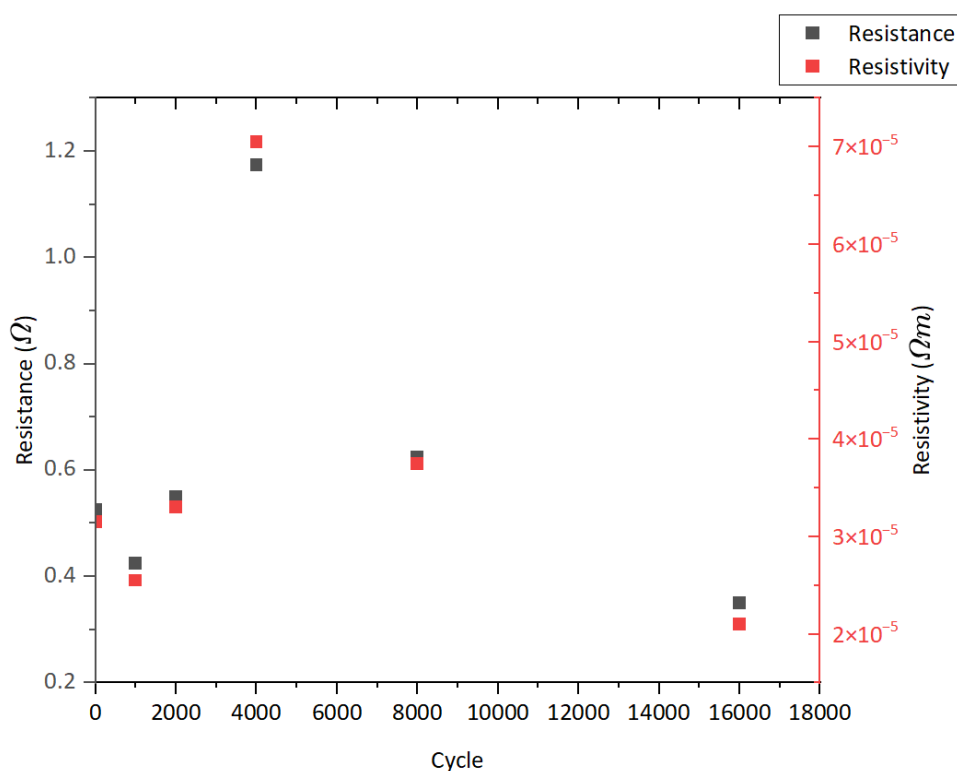


Fig. 23. The 5-terpinol average resistance and resistivity

Table 7 shows the average resistance and resistivity for 15-Terpinol GNP/Ag hybrid conductive ink before and after the cyclic bending test. The average resistance and resistivity value of the GNP/Ag hybrid ink is lower before the test and decreases after 1000 cycles. However, the average resistance and resistivity continuously decrease with increasing cycles. The reason for this condition is because of a reduction in temperature, as lower temperatures typically result in fewer atomic

vibrations, thus facilitating electron flow and decreasing resistance. Additionally, removing mechanical stress or strain on a material can restore the original structure of the conductive ink, allowing electrons to move more freely and reducing resistance and mitigating defects or damage to the structure of the material could restore the conductivity of the ink and lower the resistance. The decrease in resistivity with the increasing temperature can attributed to a decrease in the number of impurities or defects in the conductive ink at higher temperatures which leads to improved electron transport and reduced resistivity as determined by Zuhilmi *et al.*, [23].

Table 7

The 15-terpinol resistance and resistivity

Cycle	Resistance (Ω)	Resistivity (Ωm)
0	0.600	3.6×10^{-4}
1000	0.925	5.6×10^{-4}
2000	0.875	5.3×10^{-4}
4000	0.700	4.2×10^{-4}
8000	0.600	3.6×10^{-4}
16000	0.450	2.7×10^{-4}

Figure 24 displays the 15-Terpinol Average Resistance and Resistivity graph after the cyclic bending test. It also shows a trend of increasing average resistance and resistivity after certain cycles (1000 cycles) and decreasing after certain cycles (2000 cycles, 4000 cycles, 8000 cycles, 16000 cycles).

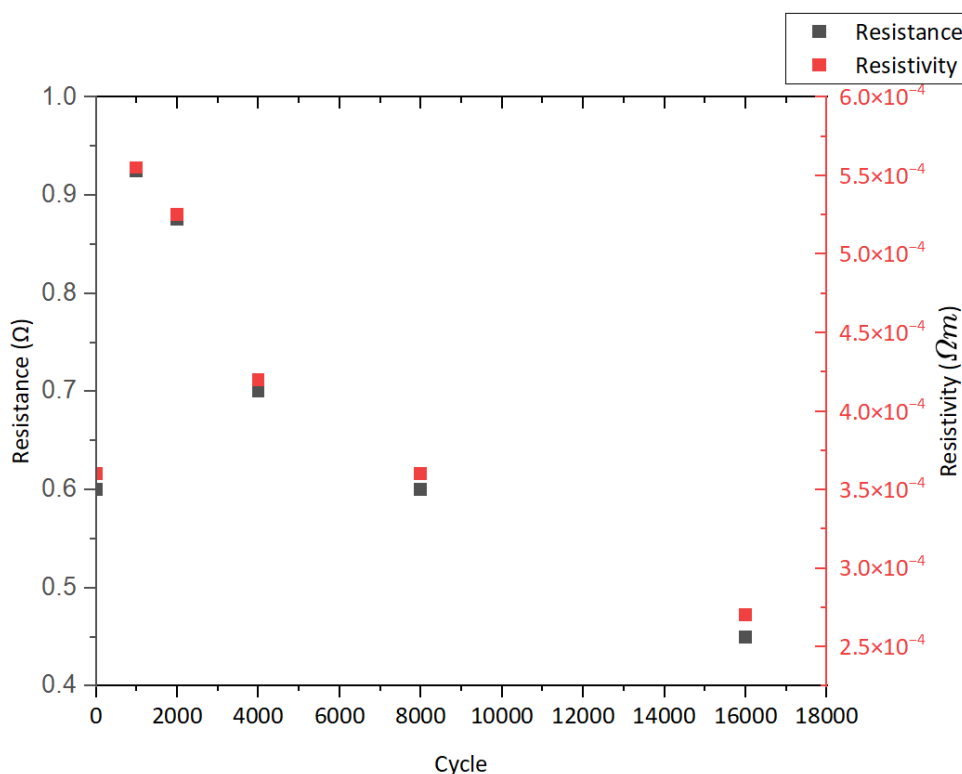


Fig. 24. The 15-terpinol average resistance and resistivity

4. Conclusions

Based on this study, it was found that the ratio 1-butanol to terpinol 40:60 provides a better average resistance and resistivity. The GNP/Ag hybrid ink was considered stable when its average

resistance and resistivity decreased after the cyclic test. It shows that 250 °C curing time for 5 hours improved the resistance and resistivity of the GNP/Ag hybrid conductive ink.

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