

## INVESTIGATION OF THERMAL CURING EFFECTS ON HYBRID GNP/SILVER CONDUCTIVE INK PERFORMANCE AND PROPERTIES

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### ABSTRACT

*Thermal curing plays an important role in determining the final properties of conductive inks. This research presents an investigation of thermal curing effects on hybrid Graphene Nanoplatelets (GNP)/Silver conductive ink performance and properties. The GNP and silver were used to form a hybrid conductive ink with a different ratio of organic solvents of 1-butanol and terpineol. The GNP/Silver conductive ink was printed on the copper substrate and cured for five hours in the oven at 260 °C. The electrical performance of the hybrid conductive ink was observed in terms of average bulk resistance and average resistivity before and after the cyclic bending test. Results show the ratio of 60:40 of 1-butanol to terpineol gives the lowest initial average bulk resistance 0.8 Ω, and resistivity  $4.8 \times 10^{-5} \Omega \cdot \text{m}$  when cured at 260 °C, but after 16000 cycles of bending, the ink gives the highest average bulk resistance 1.18 Ω and resistivity  $7.08 \times 10^{-4} \Omega \cdot \text{m}$ . In conclusion, the GNP/Ag conductive inks' solvent ratio influenced in electrical performance of GNP/Silver conductive ink when cured at 260 °C. This study demonstrates that the solvent ratio of 1-butanol to terpineol significantly influences both the initial electrical conductivity and mechanical durability of thermally cured GNP/Silver conductive inks, revealing a performance trade-off critical for flexible electronic applications. For further investigation, the cyclic bending test should be conducted for a higher number of cycles.*

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## 1. Introduction

The fast growth of flexible and stretchable electronics has opened new potential in various application areas, including wearable health monitoring devices, smart textiles, flexible sensors, and next-generation photovoltaics. Among the numerous fabrication technologies, screen printing has emerged as a cost-effective and method for producing flexible electronic components, propose advantages such as material efficiency, pattern flexibility, and compatibility with different substrates. With the intention of meeting the performance and

durability requirements of these applications, conductive inks with high electrical conductivity, mechanical flexibility, and environmental stability are a must (Krzemiński et al., 2023).

Nanomaterial-based conductive inks have gained great attention in recent years, with formulations of combining the metal nanoparticles, carbon allotropes, or their hybrids. Graphene Nanoplatelets (GNP) become favorable because of the excellent electrical conductivity, thermal stability, and mechanical flexibility. The GNP, while combined with silver particles, offer enhance overall conductivity while maintaining structural flexibility (Htwe et al., 2021). This hybrid ink is typically composed of three main components which are conductive fillers GNP and silver, a polymeric binder for particle adhesion and film formation, and a solvent to regulate rheology for printability (Liang et al., 2018).

Thermal curing is a critical post-processing step that significantly influences morphology, interparticle connectivity, and electrical performance of printed conductive inks. While moderate curing temperatures 150 to 200 °C have been widely studied, higher temperature treatments may further improve conductivity by improving sintering process, reduce binder residue, and promote stronger interfacial bonding. However, excessive thermal exposure may also result in microstructural failure because of the rapid decomposition and gas evolution, thus reducing overall performance (Li et al., 2020) (Lepak-Kuc et al., 2024). Additionally, flexible electronic components often experience repeated mechanical deformation during operation, such as bending or stretching. This exposes the printed conductive networks to cyclic stress, which can lead to microcracks, particle detachment, or delamination and worsened electrical properties over time (Suhaimi et al., 2022). Therefore, it is essential to understand how thermal curing at elevated temperatures influences not only the conductivity but also the mechanical stability of the ink.

While several studies have reported on thermal curing below 200 °C, investigations at higher curing temperatures remain limited, particularly for hybrid GNP/silver conductive ink. Understanding how such conditions influence conductivity, morphology, and structural stability is essential for advanced high-performance printed electronics. This study addresses this gap by studying the curing behavior of a hybrid GNP/silver conductive ink at 260 °C. The findings aim to describe the development of thermally strong conductive inks suitable for advanced flexible electronics applications.

In this work, a hybrid conductive ink composed of GNP and silver microparticles was formulated, screen-printed on top of copper substrates, and thermally cured at 260 °C. The cured samples were then characterized to evaluate their electrical conductivity under cyclic bending conditions. The structure of this paper is as follows: Section 2 details the materials and methods used in ink formulation and characterization. Section 3 presents and discusses the results, including electrical performance and analyses. Section 4 concludes with key findings and suggestions for future research directions.

## 2. Materials and Methods

The materials used in this study was GNP with a particle size of 25  $\mu\text{m}$  and a specific surface area of 150  $\text{m}^2/\text{g}$ , silver flakes with a particle size of 10  $\mu\text{m}$  and a purity of  $\geq 99.9\%$  trace metals basis, silver acetate 99.99 % trace metal basis acid silver salt, ethanol 99.9 % purity. All materials were obtained from Merck KGaA Darmstadt, Germany while terpineol approximately 65 %  $\alpha$ , 20 %  $\gamma$  and 10 %  $\beta$  and 1-butanol 99.9% purity was sourced from Gouden Sdn. Bhd. The ultrasonic bath was used as primary tool for combining the materials. The GNP and silver flakes performed as conductive fillers, silver acetate as a precursor and 1-butanol and terpineol were used as an organic solvent.

### 2.1 Preparation of GNP/Silver hybrid powder and conductive ink

The GNP/Silver hybrid powder and conductive ink were prepared according to previous technique conducted by (Syamsul Helmi, et al. 2024). The formulation of GNP/Silver hybrid powder as shown in Table 1. The GNP mixture was prepared by dispersing 0.005 g GNP in 5 ml of ethanol in a beaker, covered with aluminum foil to prevent the ethanol from evaporating (Norida, et al. 2024) and sonicated in an ultrasonic machine for 10 minutes to achieve initial dispersion. Then, 0.4292 g of silver flakes were added to the mixture, followed by an additional 60 minutes of sonication. Next, 0.042 g of silver acetate was added into the mixture and subjected to a further 60 minutes of sonication. After that, the mixture was transferred to a hotplate equipped with a magnetic stirrer and heated at 70 °C with continuous stirring at 200 rpm to facilitate solvent evaporation until most of the ethanol had evaporated, and the semi-solid mixture was collected. The mixture was dried in a reflow oven at 250 °C for 1 hour. The dried mixture was then allowed to cool down and then grounded into a fine powder using a mortar and pestle.

Table 1. Formulation for GNP/Silver hybrid powder and conductive ink

GNP (g)	Ethanol (ml)	Silver acetate (g)	Silver flake (g)
0.005	5	0.042	0.4292

Precisely, for every 0.1733 g of GNP/Silver powder, it is recommended to add approximately 1 drop of 1-butanol, which is roughly equivalent to 0.02 g, and 1 drop of terpineol, roughly equivalent to 0.03 g. The GNP/Silver hybrid powder was measured and mixed with organic solvent as in Table 2 to investigate the effects of varying ratio of 1-butanol and terpineol. Thinky mixer was used to mix the samples for three minutes at 2000 rpm.

Table 2. Weight and percentage of the solvent

Sample	1-Butanol			Terpineol			GNP Hybrid Powder
	Drop	%	g	Drop	%	g	
5-terpineol	10	67	0.200	5	33	0.140	1.733
10-terpineol	10	50	0.200	10	50	0.300	1.733
15-terpineol	10	40	0.200	15	60	0.420	1.733

The GNP/Silver hybrid conductive paste was printed on top of a copper substrates with dimension of 12 cm x 1 cm as illustrated in Figure 1. The manual stencil printing technique (Gholamalizadeh et al., 2022) was used to print the GNP/Silver conductive ink with the mesh stencil thickness of  $60\mu\text{m} \pm 2\mu\text{m}$ . The samples were cured at 260 °C for five hours.

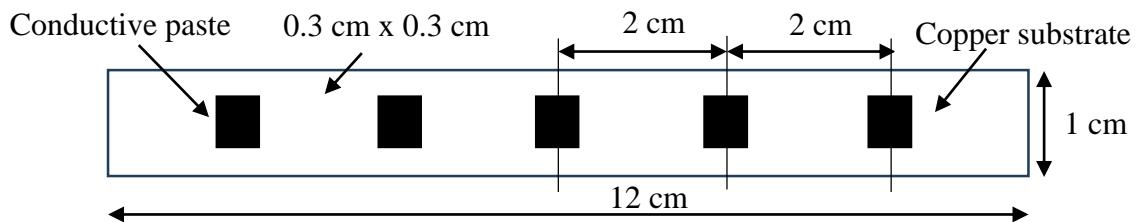


Figure 1. Dimension of the cooper substrate and conductive ink

## 2.2 Resistivity in GNP/Silver conductive ink

The cyclic bending test was conducted to printed GNP/Silver conductive ink using a cyclic bending test rig (Manaf et al., 2020) for 1000, 2000, 4000, 8000 and 16000 cycles. The resistance of the GNP/Silver conductive ink was measured using a Two-Point probe at four selected locations (1,1), (2,2), (3,3) and (4,4) as in Figure 2 and resistivity was calculated. To minimize measurement uncertainty, all measurements were carried out on five samples, and the standard deviation of the average bulk resistance and resistivity values was maintained below 5%.

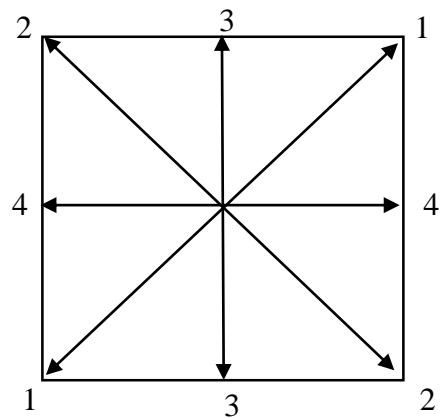


Figure 2. Four selected locations (1,1), (2,2), (3,3) and (4,4)

## 3. Results

Table 3 shows the average bulk resistance of GNP/Silver conductive ink at different ratio of organics solvent with the curing temperature of 260 °C. The average bulk resistance shows that, after the curing process ratio of 40:60, 1-butanol to terpineol exhibited the lowest value of bulk resistance  $0.8\Omega$ . Figure 3 illustrates the graph of average bulk resistance measurement of GNP/Silver conductive ink at different ratios of organic solvent.

Table 3. Average bulk resistance of GNP/Silver conductive ink at different ratio of organics solvent with the curing temperature of 260 °C

GNP/Silver conductive ink			
Ratio of 1-butanol: terpineol	67:33	50:50	40:60
Average bulk resistance ( $\Omega$ )	2.6	1.1	0.8

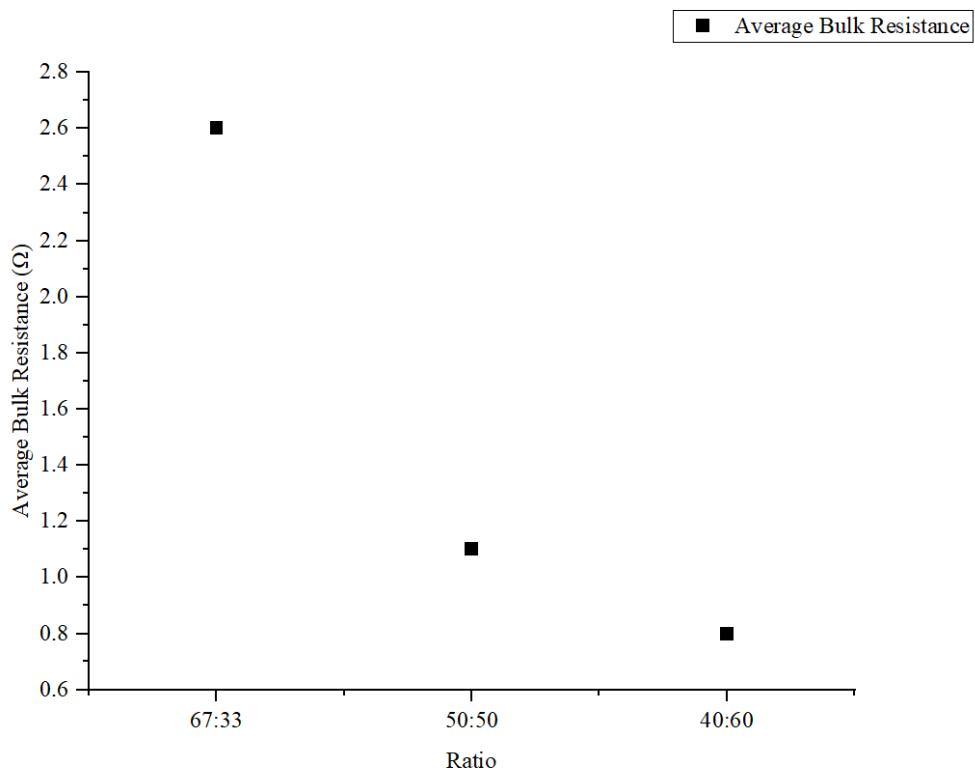


Figure 3. Graph of average bulk resistance measurement of GNP/Silver conductive ink at different ratios of organic solvent

Table 4 shows the average bulk resistance and average resistivity of 5-terpineol cured at 260 °C for five hours. The electrical performance of the GNP/Silver conductive ink demonstrates a significant improvement in conductivity with cyclic bending cycles. At first, the resistance and resistivity were  $2.6 \Omega$  and  $1.5 \times 10^{-4} \Omega \cdot \text{m}$ , respectively. After 1000 cycles, both values decreased, indicate enhanced conductive pathways possible because of the rearrangement and compaction of the filler network under mechanical stress. Although a temporary increase was observed at 2000 cycles, further cyclic cycles led to a consistent decline, with the lowest resistance  $1.5 \Omega$  and resistivity  $0.9 \times 10^{-4} \Omega \cdot \text{m}$  recorded at 8000 cycles. This trend suggests the formation of a more stable and interconnected conductive structure over time. Even after 16000 cycles, the values remained significantly lower than the initial state, indicates excellent electrical stability and durability, which are critical for long-term applications in flexible or printed electronic devices. Figure 4 displays the graph of 5-terpineol average bulk resistance and average resistivity versus cycle.

Table 4. The average bulk resistance and average resistivity of 5-terpineol cured at 260 °C for five hours

Cycle	Average Bulk Resistance (Ω)	Average Resistivity (Ω.m)
0	2.6	$1.5 \times 10^{-4}$
1000	2.1	$1.2 \times 10^{-4}$
2000	2.6	$1.6 \times 10^{-4}$
4000	2.1	$1.3 \times 10^{-4}$
8000	1.5	$0.9 \times 10^{-4}$
16000	1.6	$1.0 \times 10^{-4}$

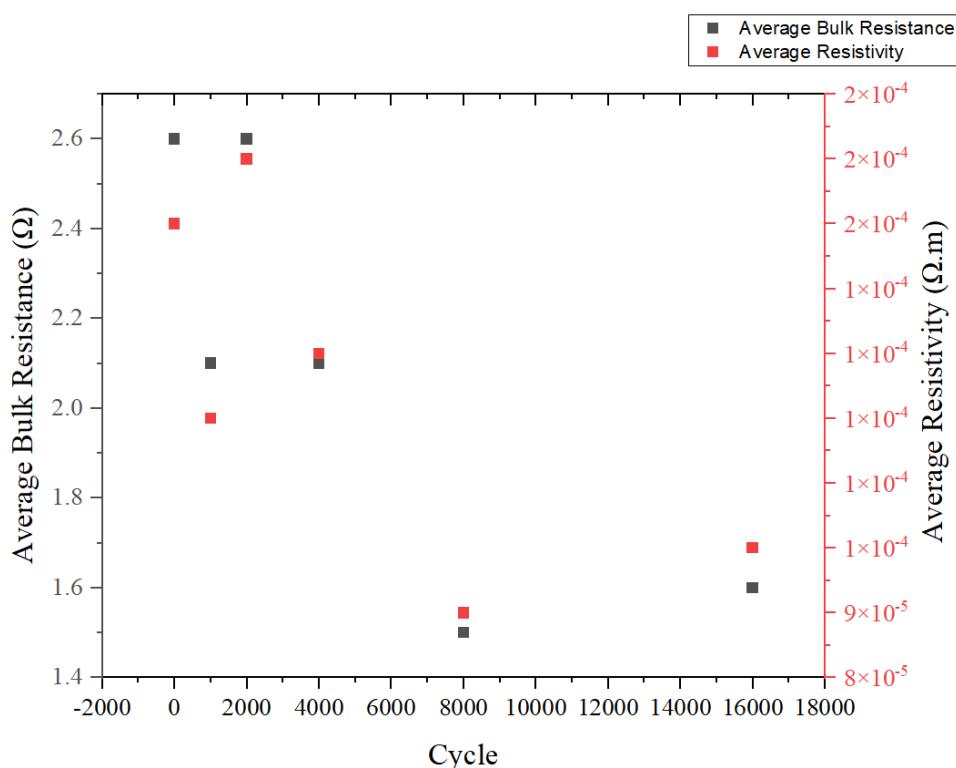


Figure 4. Graph of 5-terpineol average bulk resistance and average resistivity versus cycle

The 10-terpineol GNP/Ag conductive ink sample described in Table 5, also cured at 260 °C for five hours, exhibited a different performance profile compared to 5-terpineol. The initial average bulk resistance and average resistivity were  $1.1 \Omega$  and  $0.66 \times 10^{-4} \Omega.m$ , respectively. Both parameters peaked at 1000 cycles, with average bulk resistance increased to  $2.3 \Omega$  and average resistivity to  $1.38 \times 10^{-4} \Omega.m$ , which indicates disruption of conductive pathways in the early stages of cyclic bending. However, the following cycles showed a clear recovery and improvement. Apparently, in 2000 cycles, the values dropped significantly, and from 4000 cycles onward, a consistent decrease was observed. At 16000 cycles, the average bulk resistance and average resistivity reached the lowest values of  $0.7 \Omega$  and  $0.42 \times 10^{-4} \Omega.m$ , respectively demonstrating a 36% reduction compared to the initial state. This performance

suggests that while initial cyclic may introduce temporary disorder in the conductive network, prolonged mechanical stress facilitates reorganization and compaction of the fillers thus improving electrical conductivity and stability over time. Figure 5 displays the graph of 10-terpineol average bulk resistance and average resistivity versus cycle.

Table 5. The average bulk resistance and average resistivity of 10-terpineol cured at 260 °C for five hours

Cycle	Average Bulk Resistance ( $\Omega$ )	Average Resistivity ( $\Omega \cdot \text{m}$ )
0	1.1	$0.66 \times 10^{-4}$
1000	2.3	$1.38 \times 10^{-4}$
2000	1.2	$0.72 \times 10^{-4}$
4000	1.1	$0.66 \times 10^{-4}$
8000	0.8	$0.48 \times 10^{-4}$
16000	0.7	$0.42 \times 10^{-4}$

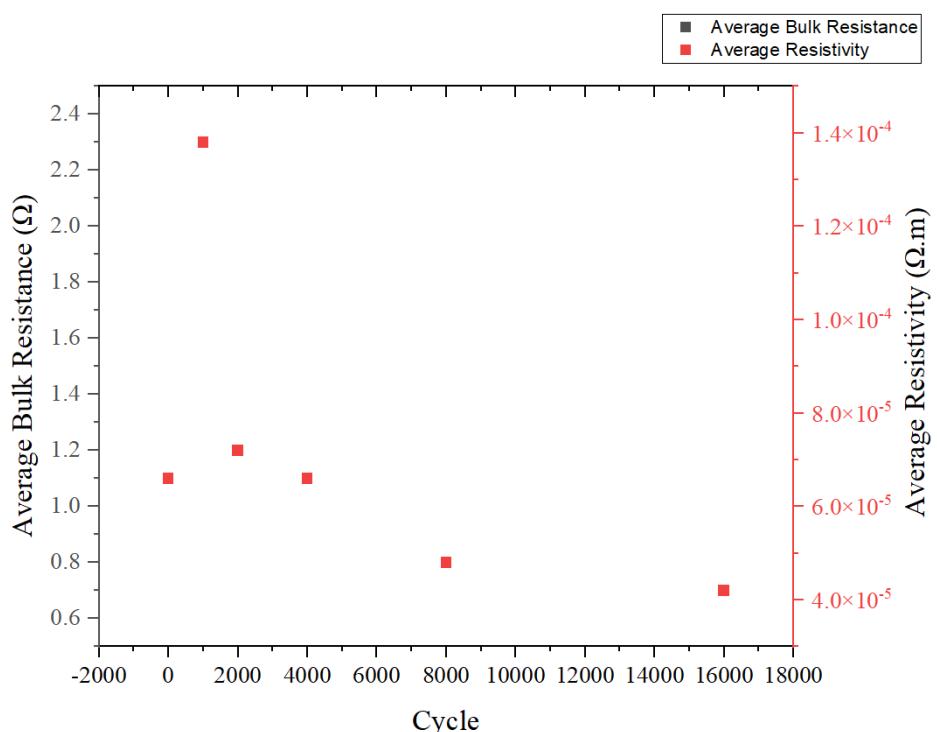


Figure 5. Graph of 10-terpineol average bulk resistance and average resistivity versus cycle

The electrical behavior of 15-terpineol GNP/Ag sample in Table 6 presents a more dynamic trend during cyclic bending test. At first, the sample exhibited low resistance and resistivity values of  $0.8 \Omega$  and  $4.8 \times 10^{-5} \Omega \cdot \text{m}$ , respectively. A clear increase occurred at 1000 and 2000 cycles and peaked at  $1.4 \Omega$  and  $8.4 \times 10^{-5} \Omega \cdot \text{m}$ . This early rise reflects mechanical disturbance or partial degradation of conductive pathways. However, a sharp drop followed at 4000 cycles, where resistance and resistivity decreased to  $0.4 \Omega$  and  $2.4 \times 10^{-5} \Omega \cdot \text{m}$ , the lowest values in the

series. This suggests a reconfiguration or reconnection of the conductive network, because of better alignment or compaction of the conductive fillers under cyclic stress. The subsequent mild increase in values up to 16000 cycles ( $1.18 \Omega$ ,  $7.08 \times 10^{-5} \Omega\text{m}$ ) might indicate slight fatigue or microstructural rearrangements that moderately disrupted the conductive continuity. Overall, the material shows a capacity for self-restructuring, with alternating phases of disruption and enhancement in electrical conductivity over prolonged cycles. Figure 6 displays the graph of 15-terpineol average bulk resistance and resistivity versus cycle.

Table 6. The average bulk resistance and average resistivity of 15-terpineol cured at 260 °C for five hours

Cycle	Average Bulk Resistance ( $\Omega$ )	Average Resistivity ( $\Omega\text{.m}$ )
0	0.8	$4.8 \times 10^{-5}$
1000	1.1	$6.6 \times 10^{-5}$
2000	1.4	$8.4 \times 10^{-5}$
4000	0.4	$2.4 \times 10^{-5}$
8000	0.6	$3.6 \times 10^{-5}$
16000	1.18	$7.08 \times 10^{-5}$

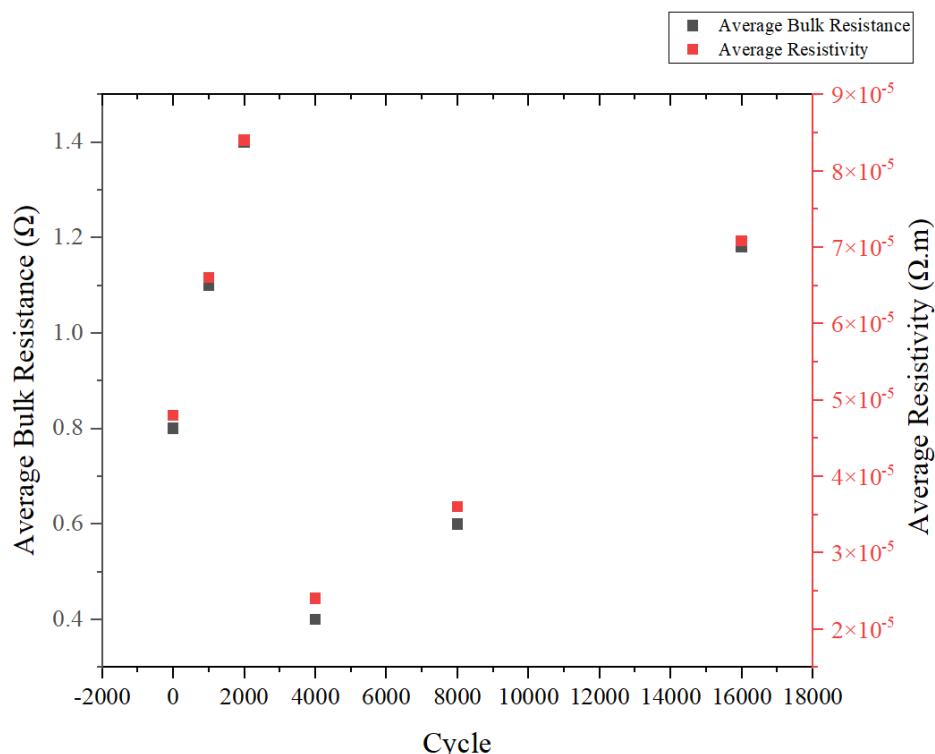


Figure 6. Graph of 15-terpineol average bulk resistance and average resistivity versus cycle

#### 4. Discussion

The sample fabricated with a 67:33 ratio of 1-butanol to terpineol showed a gradual decrease in resistance from  $2.6 \Omega$  to  $1.5 \Omega$  and a corresponding decrease in resistivity from  $1.5 \times 10^{-4}$  to  $0.9 \times 10^{-4} \Omega \cdot \text{m}$  by 8000 cycles. This consistent improvement can be attributed to the high instability of 1-butanol, which allows fast solvent evaporation, which led to close packing of the conductive fillers. The presence of GNP, which possesses high thermal conductivity, supports the uniform dissipation of thermal energy during curing at  $260^\circ\text{C}$ , therefore promotes silver flake connectivity and minor sintering. This network becomes mechanically reinforced under cyclic loading, indicates filler rearrangement and compaction that improves conductivity. The moderate amount of terpineol maintains film integrity during evaporation, balancing flow and structure during ink deposition.

In the 50:50 formulation, resistance at first peaked at  $2.3 \Omega$  at 1000 cycles but then declined to  $0.7 \Omega$  by 16000 cycles. This intermediate solvent balance seems to produce uniform ink dispersion and controlled drying. The initial increase in resistance may reflect disruption of weak connected filler sites during early cycles. Because of microscopic changes in morphology or increase in distance between the conductive fillers, the electrical resistance changes under mechanical strain (Ameeruz Kamal, et al. 2022). However, following cyclic loading stimulates the realignment of silver flakes and GNP and improves connectivity. The moderate evaporation rate supports a more uniform decomposition of silver acetate and allows silver to integrate into the developing filler network. GNP enables stable reconfiguration and resistive decline over time. The overall behavior indicates stress-assisted compaction and filler combination enhanced by thermal conductivity and sustained silver relocation.

The 40:60 formulation demonstrated a more complex, dynamic behavior: resistance increased initially to  $1.4 \Omega$  at 2000 cycles, dropped sharply to  $0.4 \Omega$  at 4000 cycles, then showed a mild increase through 16000 cycles. The higher terpineol content characterized by a higher boiling point and viscosity, slows solvent evaporation and may delay uniform filler alignment during initial curing. However, this also allows silver ions, generated from the thermal decomposition of silver acetate, to transfer and form metallic silver bridges within the matrix. The elevated terpineol fraction may impart plasticity, allowing stress-induced self-restructuring of the conductive network. Thermal energy, supported by GNP's conductivity, assists localized softening and continued silver ion mobility, allowing the network to heal and reconfigure under cyclic loading. At higher cycles there is a tendency for resistance to drop because of a phenomenon known as self-healing where the conductive pathways might reestablish themselves because of further compaction or realignment under continuous stress or elimination of weak pathway (Sánchez-Romate et al., 2021).

Several recent studies demonstrate that achieving high electrical conductivity in metal-based inks often requires thermal treatments at or above  $\sim 200^\circ\text{C}$ . For example, according to (Corrales-Pérez et al., 2024), nanoparticle-based inks were heated at  $220^\circ\text{C}$  for 30 minutes to induce sintering and remove residual organics, which significantly improved conductivity. Similarly, silver-based inks used in industrial printing typically require drying or curing in the  $150\text{--}300^\circ\text{C}$  range in order to meet performance benchmarks (Lepak-Kuc et al., 2024). In contrast, this formulation achieves comparable resistivity after curing at  $260^\circ\text{C}$ , thereby

improving substrate compatibility.

## 5. Conclusion

The thermal decomposition of silver acetate at curing temperatures ( $>200^{\circ}\text{C}$ ) plays a critical role in network formation by generating elemental silver, which integrates with silver flakes and GNP to enhance conductivity. The efficiency of this process is affected by solvent behavior: fast-evaporating systems, high 1-butanol help rapid drying and early silver nucleation, while high-terpineol systems support extended silver ion mobility and late-stage reduction. The thermal conductivity of GNP not only supports heat distribution during curing but also assists in dissipating stress-induced localized heating during cyclic loading, reducing thermal hotspots and promoting filler rearrangement. Together, these thermal and chemical effects contribute to the observed electrical behaviors and highlight the critical role of solvent ratio engineering in hybrid conductive ink design. This study demonstrates that the solvent ratio of 1-butanol to terpineol significantly influences both the initial electrical conductivity and mechanical durability of thermally cured GNP/Silver conductive inks, revealing performance trade-off critical for flexible electronic applications.

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