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Poly(1,6-heptadiyne)/$NiFe_2O_4$ composite as capacitor for miniaturized electronics

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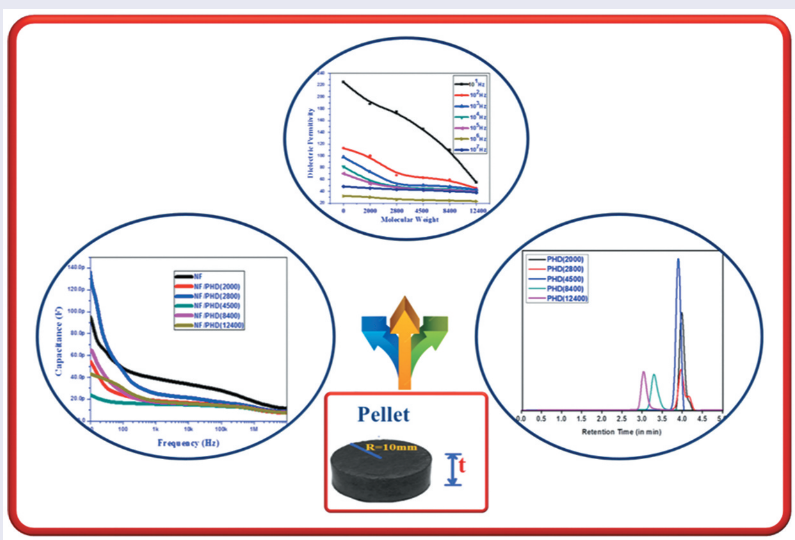
ABSTRACT

Impedance spectroscopy-based electrical measurements were conducted on different molecular weight (MW) Poly(1,6-heptadiyne)s (PHDs) embedded PHD/$NiFe_2O_4$ nanocomposites. Nanocomposites conductivity result demonstrated the conductivities of around $3.5 \times 10^{-6} - 16 \times 10^{-6} SCm^{-1}$ (nanocomposite Root mean square (RMS) current is 12–15 times greater than DC current of PHDs at 27° C). Additionally, dielectric loss and capacitance characteristics suggested the nanocomposite (4500 MW PHD) device quality factor is 35.7 at 1 kHz, which is ~13.89 times superior than that of $NiFe_2O_4$ alone sample, also 'Q' value for highest MW PHD nanocomposite is 50% enhanced than $NiFe_2O_4$. Moreover, the capacitance result suggested the 12400 MW PHD nano-composite nearly frequency-independent capacitance (15–20pF) over a frequency range of 500 Hz–500 kHz.



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1. Introduction

In accord with the manufacturing process, passive elemental systems are categorized into three categories such as discrete, integrated, and integral passives.[1,2] Integrated passives are interconnected on the surface of module, whereas integral passive elements are embedded within circuit board of a system. Among them, integrated passive associated with metal oxide nanocomposites has entrained reliable electronic system performance, enhanced design preference, and minimal economic burden.[1,3] The performance of electronic components has been improving quotidian, interconnected discrete capacitors were predominantly used for decoupling, bypassing, noise

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suppression, termination, frequency determination, filtering, and tuning.[1,16] The ratio of capacitors to other passive components presumably more than 60% which leads to an obstacle for miniaturization process.[1,3] Therefore, high-performance capacitors at reduced dimensions are quintessentially required for functional activities in miniaturized electronics.[2] This probability can be realized via decoupling capacitors with a high capacitance which renders increasing in integrated circuit (IC) speed that enables a shorter distance to serve components in a switching manner and makes them superior for digital electronics. Although many ceramic materials have achieved significantly high dielectric constant for embedded capacitor applications, they endure from low temperature processing compatibility. Consequently, ceramic materials alone are not reconcilable for embedded integrated electronics.[1,2] Recently researchers have experimentally explored on polymer ceramic composites for capacitors,[17,18] which exhibited combined qualities of low processing temperature of polymer, and high dielectric constant of ceramic fillers and made them as a promising candidate for embedded capacitors on organic substrates. Moreover, such organic ceramic capacitors provide frequency-independent characteristics which are more predominant for reliable embedded electronic industry.[1,2]

The present scrutiny for the first time explored on the fabrication of cyclopolymerized different MWs of conjugated PHDs embedded nanocomposite (Ni-$NiFe_2O_4$ /PHDs) capacitor for miniaturized electronic applications. As fabricated nanocomposites, impedance spectroscopy-based electrical measurements were conducted to reveal dielectric constant, capacitance, and Quality factor. Surprisingly, observed frequency-independent capacitance with low dielectric loss conversely quality factor suggests that this capacitor could be a functional component as an alternative to eliminate the use of traditional semiconductor polymer nanocomposite capacitor materials in industrial embedded electronic applications.

2. Instrumentation techniques

2.1. Gel permeation chromatography (GPC)

Molecular weights (MWs) and polydispersity index (PDIs) were measured via gel permeation chromatography system: Waters alliance GPC system equipped with Waters Model 515 HPLC pump and Waters Model 2414 Refractive Index Detector.

2.2. I–V Characteristics

The poly(1,6-heptadiyne) different molecular weight samples were coated on a dry glass substrate to evaluate the electrical DC conductivity via a two-probe technique (Keithley Instruments, India) under laboratory conditions.

2.3. Broadband impedance analyzer

Developed samples (different molecular weight poly (1,6-heptadiyne) embedded metal-oxide nanocomposites) room temperature dielectric properties were scrutinized within the frequency range 1–10 MHz by utilizing broadband impedance dielectric spectroscopy (Nova-control – ALPHA, Germany).

3. Experimental section

3.1. Synthesis of diacetylene and PHDs

Diacetylene and PHD polymer experimental synthesis has been demonstrated by Magisetty et al.[2] In accord with the reported research, cyclopolymerization was performed to yield different MW PHDs, the MW distribution was confirmed via GPC technique, and then developed different MW PHDs were utilized during fabrication of nanocomposite capacitor to facilitate for miniaturized embedded electronic applications.[2]

3.2. Different MW PHD nanocomposite fabrication

Atlas hydraulic pellet press was implemented to fabricate pellets as per the ASTM-D150-11 standard for electrical measurements. During pellet fabrication, to segregate the mixture, nano-nickel ferrite compound particulates were crushed with different MW PHDs powder separately, in a mass ratio of about 1:20 (PHDs: $NiFe_2O_4$) via an agitate mortar. Finally, Atlas: GS03100 high-end evacuated die (∅10mm) was utilized to press 0.4 g of mixture under 15 tons of applied force load for 30 sec via microprocessor-controlled hydraulic press to fabricate dense pellet (Thickness: 1.5 mm). The fabrication of pellets has been pictorially illustrated in Figure 1.

4. Results and discussions

4.1. Gel-permeable-chromatography (GPC) analysis

Figure 2a illustrates the distribution of MWs of PHDs was performed via GPC. Though Figure 2a,b,

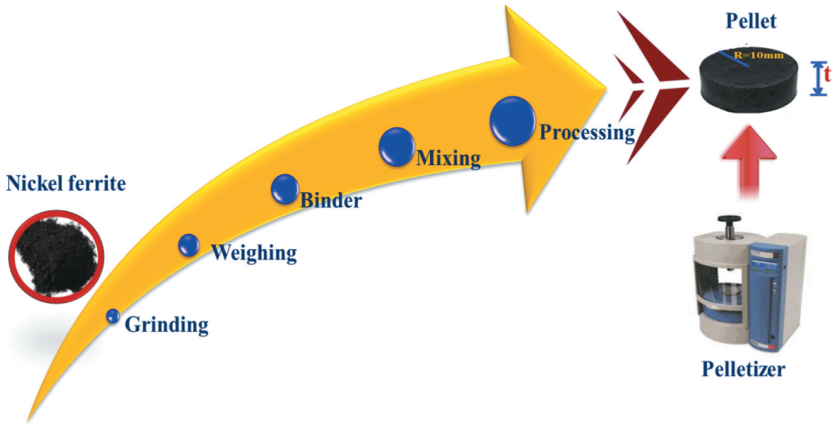

Figure 1. Schematic representation of pellet preparation.

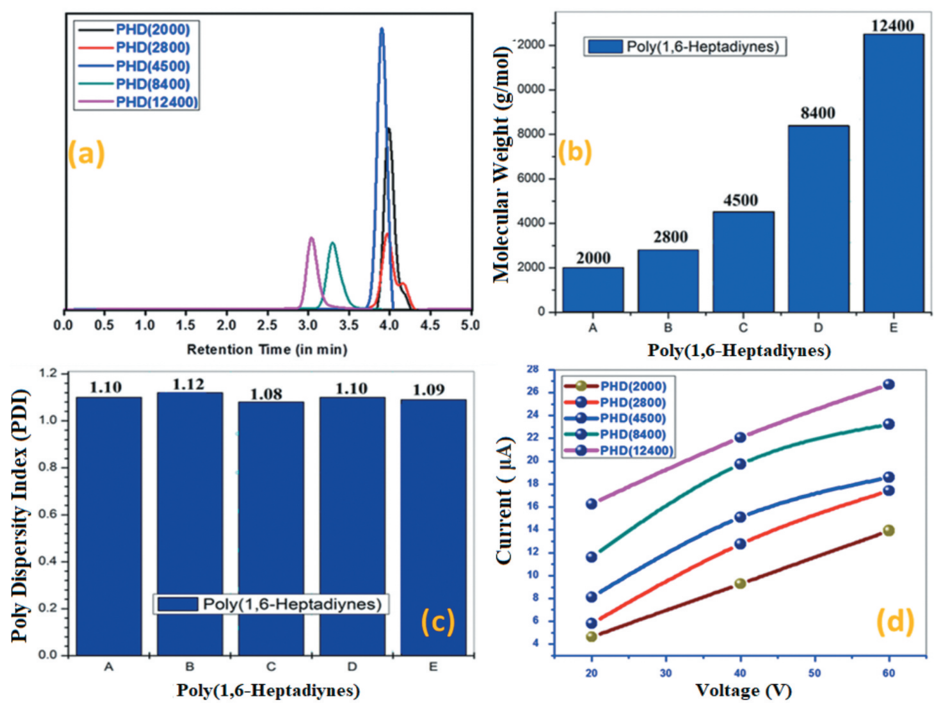

Figure 2. (a) GPC, (b) MW distribution in g/mol, (c) PDI distribution, (d) I–V characteristics of PHDs at MWs (2000, 2800, 4500, 8400, 124000 MW in g/mol).

illustrating variation in its peak intensity, unimodel GPC chromatogram curves smoothly shifted toward the shorter retention time with increases of the degree of polymerization, indicating that the increased MWs of synthesized PHDs.[19] Importantly these molecular-chain-extended species showed relatively narrow PDIs (1.08–1.12) (Figure 2c) with reasonable agreement with the theoretical MWs suggested well-controlled MW distribution of cyclopolymerized PHDs.[19]

4.2. I-V Characteristics of PHDs

Two-probe technique was used to perform electrical DC conductivity measurement under laboratory conditions. Conductivity was determined for $\tilde{3}0$ thickness of different MW PHD coated films and indicated results ranging from $1.2 \times 10^{-5} - 2.3 \times 10^{-5} SCm^{-1}$, and these comparable to the conductivity of synthetic organic nanostructures such as polyaniline and Au-Polyaniline nanocomposite polyacetylene.[20] However, in order to reveal a prominent conductivity mechanism within the different MW PHDs, we considered the following current density mathematical expression.

$$J\alpha \exp^{-\beta d} \quad (1)$$

Here 'd' represents barrier width, and 'β' stands for tunneling decay coefficient. For different PHDs, various values of voltages corresponding current densities are shown in Figure 2d. The results suggest that the current densities are in incremental proportion to the MW of PHDs, this increment from Figure 2d, elucidates 'J' probably be an exponential state to the applied voltages and that corresponds to the potentially induced charge transfer in conjugated PHD polymer chain length. Moreover, 'J' probably demonstrates the existence of non-resonant bond tunneling transport mechanism (intercharge-carrier mechanism) in between metal-polymer-metal junction.[21] This significant characteristic can be interpreted with the aid of tunneling coefficient 'β', if β= 1, which means that the total charge-carrier mechanism is only the tunneling charge-carrier mechanism, if it reduces, this means that the possibility of hoping charge-carrier mechanism, i.e., the existence of non-resonant bond tunneling-transport mechanism. 'β' is attained by the slope of I–V plot of Figure 2d, suggest that the predominant mechanism is non-resonant bond tunneling-transport mechanism (Table 1), also, results reveal that as the $\tilde{\beta}$ value decreases with increasing bias voltage, PHDs (different MWs) would expect incremental current density; thus, large area molecular junction induced voltage dependence current (Figure 2d).[21] This similar concept of tunneling-decay coefficient decrease with increasing bias voltages was reported by wang et al.[22] and is in good agreement with the reported result. As the MW increases conductivity characteristics progress due to its MW dependent chain distribution induced increase in charge-carrier mean free path, as a result, an increase in intercharge carriers. This significant increase in conductivity characteristics with different MW PHDs attributable to inter-charge-carrier mechanism is an effective consideration in order to reduce losses in nanocomposite capacitors.

Table 1. Tunneling Decay Coefficient 'β' at Different Bias Voltages.

Molecular weight of PHD	Bias Voltage V=20-40	Bias Voltage V=40-60
2000	$\beta = 0.2320$	$\beta = 0.2320$
2800	$\beta = 0.3480$	$\beta = 0.2325$
4500	$\beta = 0.3484$	$\beta = 0.1741$
8500	$\beta = 0.4066$	$\beta = 0.1741$
12400	$\beta = 0.2901$	$\beta = 0.2321$

4.3. Electrical and electronic characteristics of PHD nanocomposites

Reorientation of existing permanent dipoles in an electric field does not contribute to the quantitative dielectric permittivity over a wide frequency range,[23] thus evidence the dielectric permittivity decay response value as observed in Figure 3a–c. MW of PHD polymer increases consequently increase in molecular chain, the direct dependence of different MWs with intra/inter-chain tunneling charge-carrier conduction mechanism renders static decay in the dielectric permittivity of nanocomposites which holds a good agreement with the Clausius–Mossotti relation.[1,19] The tendencies of non-bonding interactions in nanocomposites are also known as electrostatic Van der Waal forces[24] which facilitate electron-hoping instigated charge transport in between inter-molecules, the major influencing factors for electron-hoping are the surface of nickel ferrite and PHD polymer, their size, density, and chain-length proportions.[1,19,23] This significant property is probably attributable to the static decay in the dielectric permittivity of nanocomposites. This momentous property suggests that the dielectric permittivity characteristics are contingent on the type of polymer chain length, charge-carrier capacity, and applied frequency,[17,18] thus predominantly revealing that the static decay was due to the conjugated conducting PHD polymer molecular weights. The frequency-dependent characteristics have been illustrated in Figure 3a–c, as the frequency intensifies the dielectric permittivity spectra relaxes in its state due to molecular-chain resonance mismatch.[25,27] This type of permittivity characteristic mechanism is observed in nickel ferrite/PHD polymer nanocomposite attributable to their space charge and dipolar mechanisms, where the typical relaxation time of ~10^{-9} s has been ascertained.[1,2]

From Figure 3a–c, it is observed that the permittivity diminishes with respect to molecular chain, which in turn depends on molecule density and intermolecular forces. Since the permanent dipoles subsist in a PHD polymer, these dipoles are incompetent to follow-up with elevated-frequencies,[5,28] which is perchance due to MW dependent molecular chain resonance and nano-nickel ferrite enveloping the molecule, that restricts the angular momentum of molecule as a result of reduced self-diffusing coefficient while increasing PHD MW.[1,19,23] Studies appertaining to self-diffusing coefficient relative to molecular weight interaction suggests that the non-bonding inter-potentials and its factors

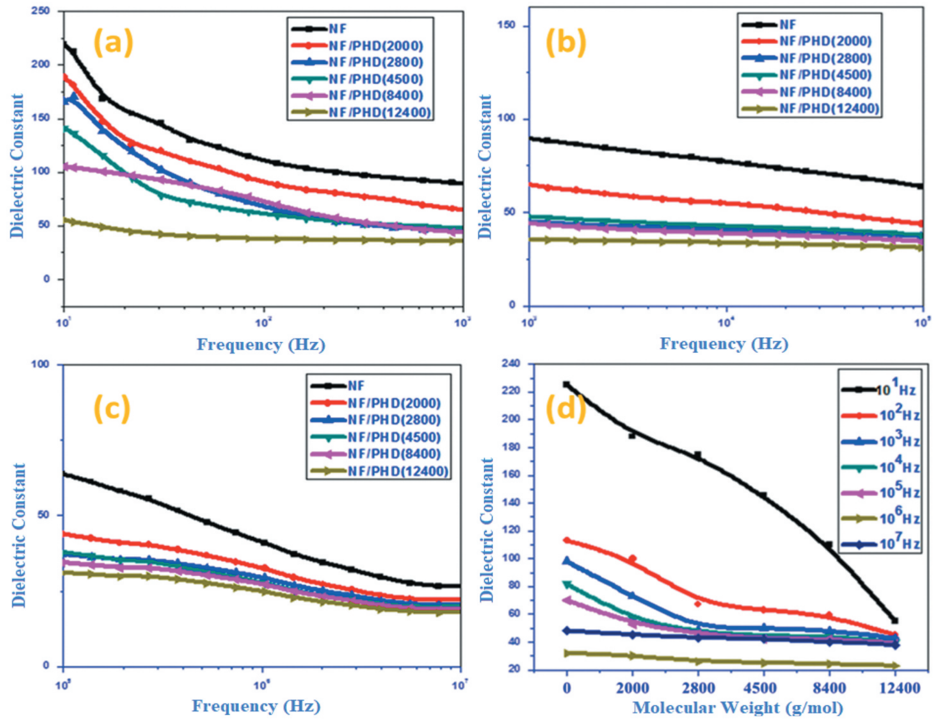

Figure 3. (a–c) Dielectric constant (ε′), and (d) Dielectric constant (ε′) as a function of frequency at different MWs (2000, 2800, 4500, 8400, 12400 MW in g/mol).

furthermore influence on its final property. The relative energy induces negligible non-bond potentials as the frequency increases, hence the permittivity falls at the higher frequency.[5,28,29] Moreover, this can be elucidated as the molecule behaves qualitatively like a nonpolar substance in between nickel ferrite nanoparticles which leads to higher charge transport capability, and thus nanocomposite attains sustainably excellent conductivity (Figure 2d). The ratiocination for abatement in dielectric permittivity intensity is the consequence of superfluity addition of different molecular weights (Figure 3).[1] Electronic conduction is also a frequency-dependent hypothesis, as frequency increases there is more proclivity at the atomic level electron diffractions to induce greater flow of charge carriers in interconnected space charge path,[30] which further increases as the MW chain distribution increases,[1,3] this was probably a reason for characteristically observed conductivity in Figure 3a, and holds fine agreement with the universal Jonscher's power law (JPL).[1,2,28] As well as, MW dependent intra-chain conduction because of the immobility of polymer molecule rendered with minimum path resistance is also the reason for conductivity[1,3] The stemming conductivity values ranging from 1.67×10^{-4}SCm^{-1} to 2.47×10^{-4}SCm^{-1} attributable to the reported MWs were obtained, and these results propose that the conductivity characteristics are superior to formerly indicated PANI at CNT/PU nanocomposites,[23] and as well as, comparable to nearer quantitative approximations of reported $MoSe_2$ metal-oxide semiconducting capacitors.[1] In Figure 4a, interestingly conductivity profile shifts toward higher frequency side with increment in MW of PHD in nanocomposite (Table 2 and S1),[1,3] which probably elucidates relaxing dipoles rendered relaxation times as a consequence of molecular weight addition indicates the peak shift from lower frequencies to higher frequencies.[2] Additionally, this consequence can be explained with the aid of inter-chain charge-carrier probabilities, wherein the inter-chain relaxation time probably instigates the peak shift.[2]

In contemporary electronic technology, the motivation to utilize the capacitors for inter-digital electronic applications are based on their Quality factor or 'Q' factor value.[1,2] Quality factor or 'Q' factor at operating frequency 'ω' is defined as the ratio of the reactance of a capacitor to its resistance. The value of quality factor or Q-factor can also be interpreted by dissipation factor

Table 2.

Sample	Conductivity at 10^7Hz
NiFe2O4	2.47E-4 S/cm
NiFe2O4/PHD(2000MW)	1.17E-4 S/cm
NiFe2O4/PHD(2800MW)	2.03E-4 S/cm
NiFe2O4/PHD(4500MW)	1,47E-4 S/cm
NiFe2O4/PHD(8400MW)	1.83E-4 S/cm
NiFe2O4/PHD(12400MW)	1.67E-4 S/cm

$NiFe_2O_4$ and $NiFe_2O_4$/poly(1,6-heptadiyne) nanocomposite conductivity characteristics at 10^7 Hz.

(DF) value (Figure 4c), DF is inversely related to the 'Q' factor, it can be mathematically expressed as $\frac{1}{\tan(\delta)} = \frac{1}{DF} = Q$.[1,2,18,31] From, Figure 3b, the quantified quality-factor of 35.7 at 1 kHz was obtained for nanocomposite (4500 MW PHD) device, which is ~13.89 times higher than the $NiFe_2O_4$, similarly the 'Q' value for highest MW of PHD nanocomposite is 50% higher than $NiFe_2O_4$ alone (Figure 4c). This significant accretion in Q factor is attributed to the MW rendered reduced intrinsic resistance as a consequence of conductivity.[1,2,23] Similarly, in Figure 4c, capacitance value almost nearly frequency-independent/constant in the frequency range of 1 K to 10 kHz for all MW PHD nanocomposites except 2800 MW sample. It is observed that, at this intercept frequency, capacitance alters with increasing MW of PHD which is probably due to the MW dependent electronic charge distribution, thus preponderantly impeling frequency-independent charge-separated capacitance mechanism.[1,2] Frequency-dependent capacitance can be explained by various kinds of semiconductor-interface states with a different lifetime, this interface state capacitance is parallel to space charge depletion capacitance,[1,2,23] and superior capacitance value can be perceived because of the response of the interface state to the AC signal at curtailed frequencies.

In summary, 4500 MW poly(1,6-heptadiyne)/$NiFe_2O_4$ nanocomposite capacitor is providing efficient performance among the all MW dependent nanocomposite capacitors. Among all composites, 4500 MW nanocomposite capacitor exhibiting low loss component with optimum dielectric constant $\varepsilon = 45$ (100 Hz-10 kHz). Moreover, the improved Q-factor value of 4500 MW poly(1,6-heptadiyne)/$NiFe_2O_4$ nanocomposite is ranging from 8.1 to 104 at 1 kHz. Therefore, 4500 MW poly(1,6-heptadiyne)/$NiFe_2O_4$ nanocomposite capacitor could be considered as an efficient component for integrated electronic circuits, also, this could be used as a low impedance path to the ground

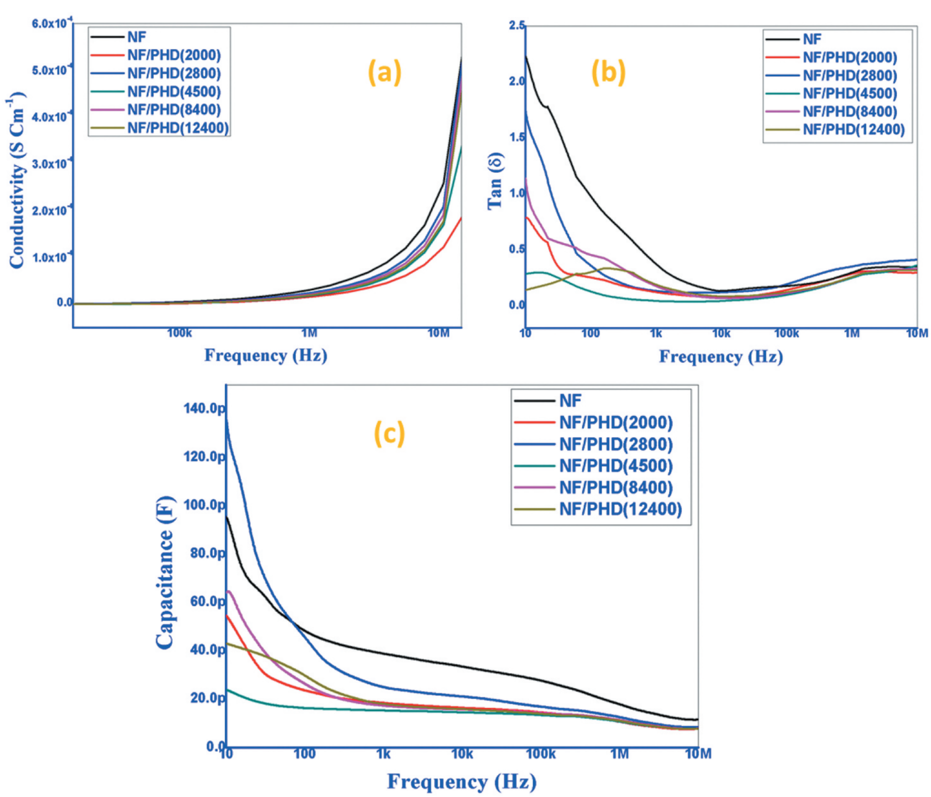

Figure 4. (a) Conductivity, (b) Dielectric loss and (c) Capacitance as a function of frequency at Different MWs.

for radiation-induced high-frequency currents at the shielding enclosures for efficient protection in electronic circuits.

5. Conclusion

Two-probe measurement was adopted for PHDs to demonstrate one-dimensional electrical DC-conductivity which is of around $1.2 \times 10^{-5} - 2.3 \times 10^{-5} SCm^{-1}$. These different MW PHDs enabled nanocomposite's measurement result suggesting that the inherent variation in conductivity characteristics of PHD polymer is probably attributable to different MWs, wherein, intra, intermolecular chain, nonresonant, and non-bond interactions are preeminent parametric features. Thus, evidencing the highest MW (12400 MW) of PHD nanocomposite 'Q' which is 50% higher than $NiFe_2O_4$ alone, also, capacitance results suggesting the nearly frequency-independent capacitance, which is 15–20pF over 500 Hz–500 kHz frequency range. Moreover, among all composites, 4500 MW nanocomposite capacitor exhibiting low loss component with optimum dielectric constant $\varepsilon = 45$ (100 Hz–10 kHz). The improved Q-factor value of 4500 MW poly(1,6-heptadiyne)/$NiFe_2O_4$ nanocomposite is ranging from 8.1 to 104 at 1 kHz. Therefore, 4500 MW poly(1,6-heptadiyne)/$NiFe_2O_4$ nanocomposite capacitor could be considered as an efficient component for integrated electronic circuits, also, this could be used as a low impedance path to the ground for radiation-induced high-frequency currents at the shielding enclosures for efficient protection in electronic circuits.

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Highlights

- Poly(1,6-heptadiynes)/$NiFe_2O_4$ nanocomposite capacitor
- Molecular weight dependent electronic properties
- Quality factor 'Q' for highest MW PHD nanocomposite is 50% enhanced than $NiFe_2O_4$
- 12400MW PHD nanocomposite demonstrated frequency independent capacitance of 15–20pf (500Hz–500 kHz).

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