

Impact of functionalised Silicon Carbide NPs on the mechanical and dielectric performance of Sago Starch based nanocomposites

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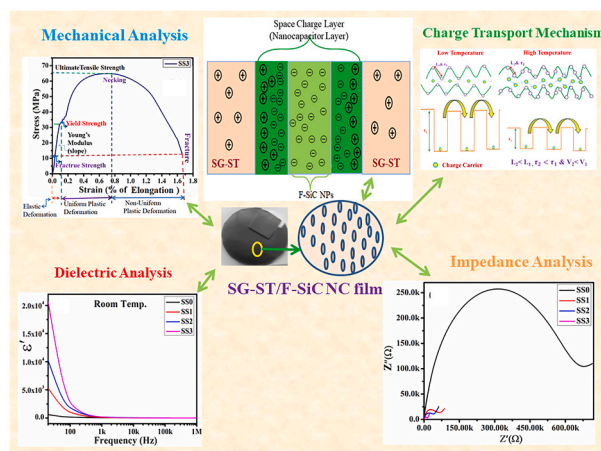
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HIGHLIGHTS

- 50 fold increment in ϵ' (at $\omega = 25$ Hz) for SG-ST after the addition of 6 wt% of F-SiC NPs.
- Young's modulus of SG-ST increases from 73.6 ± 2.9 MPa to 382.2 ± 8.9 MPa for SS3 NC film.
- R_G and R_{GB} of SG-ST decreases from 850.8 k Ω to 15.9 k Ω and 667 k Ω – 16.7 k Ω respectively for SS3.

GRAPHICAL ABSTRACT



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ABSTRACT

The current study elucidates the fabrication of self-supporting, flexible and bio-compatible polymer nanocomposite (NC) films based on sago starch (SG-ST) by utilizing the surface modified Silicon Carbide (F-SiC) nanoparticles (NPs). The fabricated SG-ST/F-SiC NC films were characterized using field emission electron microscopy (FESEM), X-ray diffraction (XRD), transmission electron microscopy (TEM), universal testing machine (UTM), Fourier transform infrared spectroscopy (FTIR) and dielectric spectroscopy. Analysis of UTM results demonstrated that the value of Young's modulus (E_{mod}) of SG-ST increases from 73.6 ± 2.9 MPa to 382.2 ± 8.9 MPa after the addition of 6 wt% of F-SiC NPs (SS3 NC film). Dielectric studies reveal that the dielectric permittivity (ϵ') exhibits a 50 fold increment ($f = 25$ Hz) for SG-ST ($\epsilon' = 348$) embedded with 6 wt% of F-SiC NPs ($\epsilon' = 17561$) at room temperature. Further, the value of relaxation time (τ_s) of SG-ST decreases from $692.3 \mu s$

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to $5.7\ \mu\text{s}$ (at room temperature) and further decreases to $0.8\ \mu\text{s}$ (at $100\ ^\circ\text{C}$) after the addition of 6 wt% of F-SiC NPs.

1. Introduction

Novel materials with high dielectric permittivity are in great demand as they are an essential component of green flexible electronic devices [1–5]. Moreover, fabrications of such devices require low weight, environment friendly and cost effective high performance multifunctional advanced materials [6–9]. Biopolymer based NCs comprising of nanofiller such as inorganic NPs embedded in host matrix are a class of materials that have inherent characteristics such as feasibility of processing, flexibility, biocompatibility, biodegradability etc. and can be investigated for application in electronic devices [10–13]. Among various biopolymers (sago starch, chitosan, cellulose, gelatin, sodium-alginate etc.) sago starch (SG-ST) is a naturally derived polysaccharide mixture of amylose and amylopectin with a repeating units of 1,4- α -D glucopyranosyl and it possess inherent features such as very low cost, easy availability, excellent film forming capability, hydrophilic nature, easy processing, biodegradability and biocompatibility [14–16]. Hence, SG-ST films can be examined for application in electronic devices such as transistors, flexible conductive electrodes, sensors, supercapacitors, nano-generators etc. [17,18]. Generally, bio-polymers possess low value of dielectric permittivity; hence, they are not preferred for application in energy storage devices. However, their dielectric permittivity can be enhanced by embedding inorganic nanofillers such as semiconducting NPs, metal NPs, graphene, carbon nanotube etc. Among various inorganic nanofillers, Silicon Carbide (SiC) a wide band gap semiconductors holds a special place as it is non-oxide ceramic exhibiting more than 250 polytypes [19,20]. Moreover, it has high thermal conductivity, high erosion resistance, smaller thermal expansion coefficient, excellent mechanical strength, high dielectric permittivity etc. These remarkable attributes makes it a suitable option for embedding into bio-polymeric film [21–23]. Thus, the synergetic combination of SG-ST and SiC NPs hold the promise of obtaining potential material with desired attributes such high dielectric permittivity and high mechanical strength etc. However, the properties exhibited by SG-ST/F-SiC NC depend upon the interfacial interaction and

dispersionability of SiC NPs in polymer host matrix. Thus, the major challenge faced during the synthesis of SG-ST/SiC NC is the tendency of SiC NPs to agglomerate inside the polymer matrix. To achieve high degree of dispersion of SiC NPs in SG-ST matrix, surface modification of SiC NPs via acidic oxidation method is an effective strategy leading to uniform dispersion and stabilization of SiC NPs within the polymer matrix [24–26]. Mollik et al. [27] examined the dielectric attributes of starch reinforced with rGO NPs and observed that addition of 1 wt% of rGO into starch reduces the resistance across the grain boundary from $1.6\ \text{M}\Omega$ to $0.8\ \text{k}\Omega$ and increases the capacitive element from $0.12\ \text{nF}$ to $2.24\ \mu\text{F}$. Alam et al. [28] investigated the dielectric permittivity of gelatine polymer and observed that it increases four times due on addition of just 0.02 wt % of MWCNT NPs. Ceballos et al. [29] examined the mechanical attributes Ag/starch NC and concluded that the value of ' E_{mod} ' of starch increases from $50 \pm 5\ \text{MPa}$ to $530 \pm 10\ \text{MPa}$ after the incorporation of 0.015 wt% of Ag NPs. Kaur et al. [30] investigated the mechanical, thermal and dielectric parameters of $\text{Au}^{\text{c}}\text{-Ag}^{\text{s}}$ /Starch- Sodium Alginate NC film and reported that the value of dielectric permittivity of Starch- Sodium Alginate polymer blend increases from 18.8 to 96.2 (at room temperature) and increases to 2227.4 at 353 K after the addition of 1.5 wt% of $\text{Au}^{\text{c}}\text{-Ag}^{\text{s}}$ NPs at 100 Hz. Saini et al. [31] fabricated PVA-grafted-SiC/PVA NC films and examined the electrical, optical and mechanical properties of resultant NC and reported that the value of refractive index of PVA increased from 1.5 to 2.0 after the addition of 0.015 wt% of PVA-grafted-SiC NPs at a wavelength of 550 nm. Pradhan et al. [32] examined the mechanical properties of SiC/Chitosan NCs and concluded that the fabricated NCs have application in packaging because of their improved tensile strength, thermal stability and low oxygen permeability. Alghunaim [33] investigated the dielectric and optical properties of SiC/PVC-PVK blend NC. Dash et al. [34] synthesized SiC/Starch NCs and observed that resultant NC exhibited higher thermal stability.

Therefore, the current work presents the investigation of mechanical and dielectric analysis of SG-ST NC film embedded with F-SiC NPs which have seldom been investigated at length to the best of our

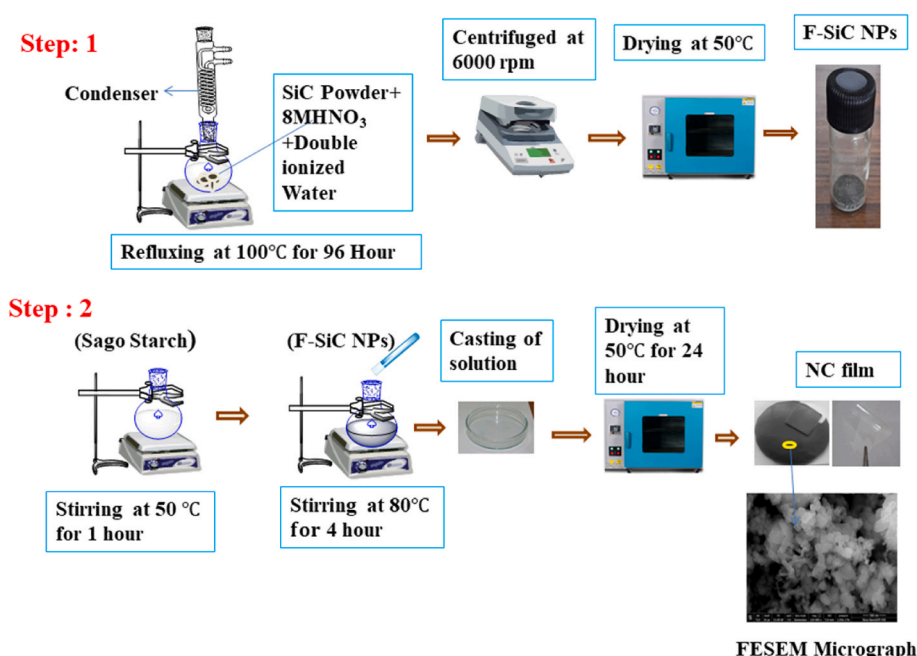


Fig. 1. Schematic diagram of functionalization process for SiC NPs and synthesis of SG-ST/F-SiC NC film.

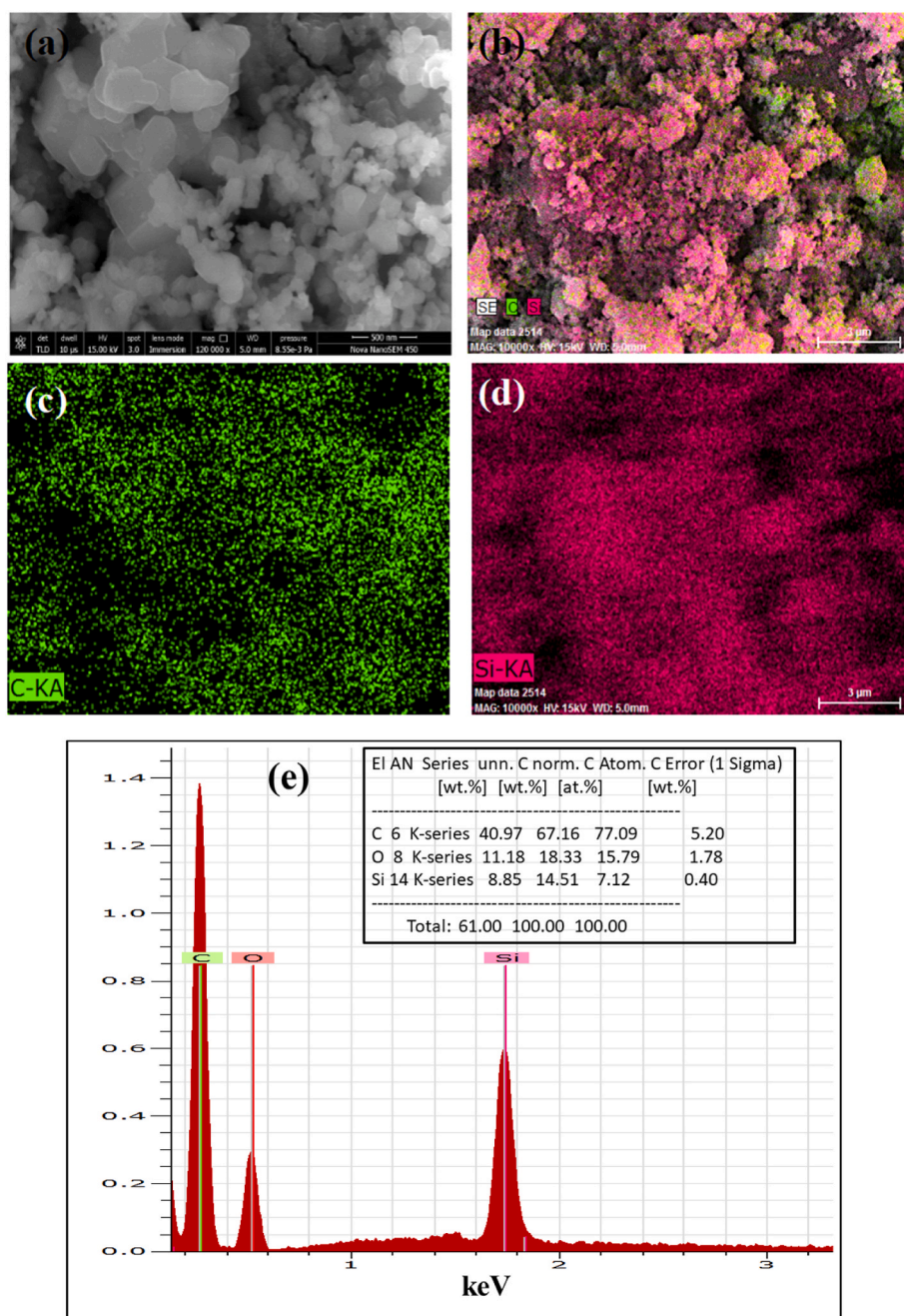


Fig. 2. (a) FESEM image, (b–d) elemental mapping and (e) EDAX spectra of F-SiC NPs.

knowledge. Firstly, the surface modification of SiC NPs was carried out via acidic oxidation treatment by using nitric acid and subsequently SG-ST/F-SiC NC films embedded with different wt.% of F-SiC NPs were fabricated utilizing solution casting approach. The fabricated SG-ST/F-SiC NC films were characterized using TEM, FESEM, XRD, FTIR, UTM and impedance analyser to investigate the surface morphological, structural, mechanical and dielectric properties. The improved dielectric and mechanical attributes makes them strong condenser for utilization in biofriendly flexible electronics devices.

2. Experimental section

2.1. Materials

SG-ST was procured from local market. Nitric acid (HNO_3) and

glycerol were procured from Rankem, India. SiC NPs were synthesized by using the solid phase reaction of carbon with molten silicon as discussed in detail by Dhiman et al. [35]. Double ionized (DI) water was used to prepare all the solutions.

2.2. Experimental procedure

2.2.1. Functionalization of SiC NPs

In the first step, functionalization of as synthesized SiC NPs was done by acidic oxidation treatment using HNO_3 . As a result, the functional group were introduced on the surface of SiC NPs. For functionalization, 1 g SiC NPs were mixed with 8 M HNO_3 in an idometric flask. The prepared solution was refluxed for 96 h at 100 °C with constant stirring using magnetic stirrer. For uniform heating, idometric flask was put on an oil bath.

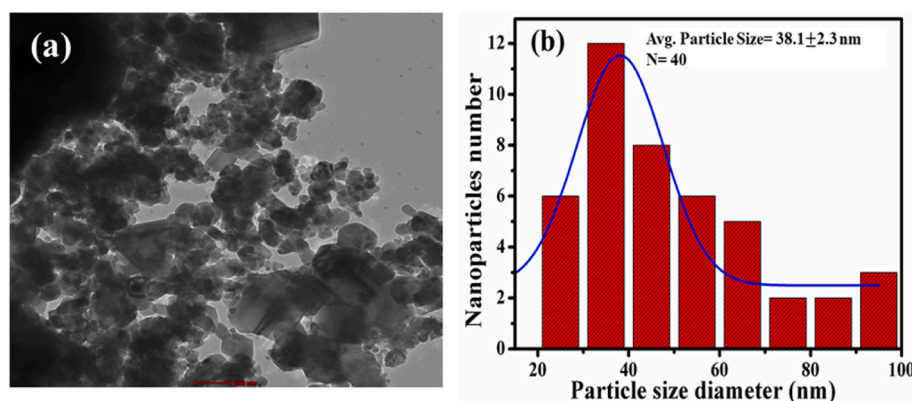


Fig. 3. (a) TEM image and (b) particle size distribution of F-SiC NPs.

After refluxing, SiC NPs were centrifuged using centrifuge machine at 6000 rpm for 15 min. The resulting SiC NPs were transferred into crucible and dried in an oven for 48 h. The dried F-SiC NPs were collected from oven and analysed by XRD and FTIR spectroscopy. Schematic diagram of functionalization of as synthesized SiC NPs has been depicted in Fig. 1(step 1).

2.2.2. Synthesis of SG-ST/F-SiC NC films

After the surface modification of SiC NPs, biopolymer based NC films

embedded with F-SiC NPs were synthesized via solution casting technique. 0.5 g of SG-ST was dissolved in DI water in a 50 ml flask and heated up to 50 °C with magnetic stirring for 1 h. Different proportions of F-SiC NPs (2 wt% (10 mg of F-SiC NPs in 0.5 g of SG-ST), 4 wt% (20 mg of F-SiC NPs in 0.5 g of SG-ST) and 6 wt% (30 mg of F-SiC NPs in 0.5 g of SG-ST) represented as SS1, SS2 and SS3 respectively) were dissolved in DI water at 65 °C under constant ultrasonication until a homogenous solution was obtained. After that, F-SiC NPs and SG-ST were mixed together with glycerol (used as a plasticizer) under constant stirring

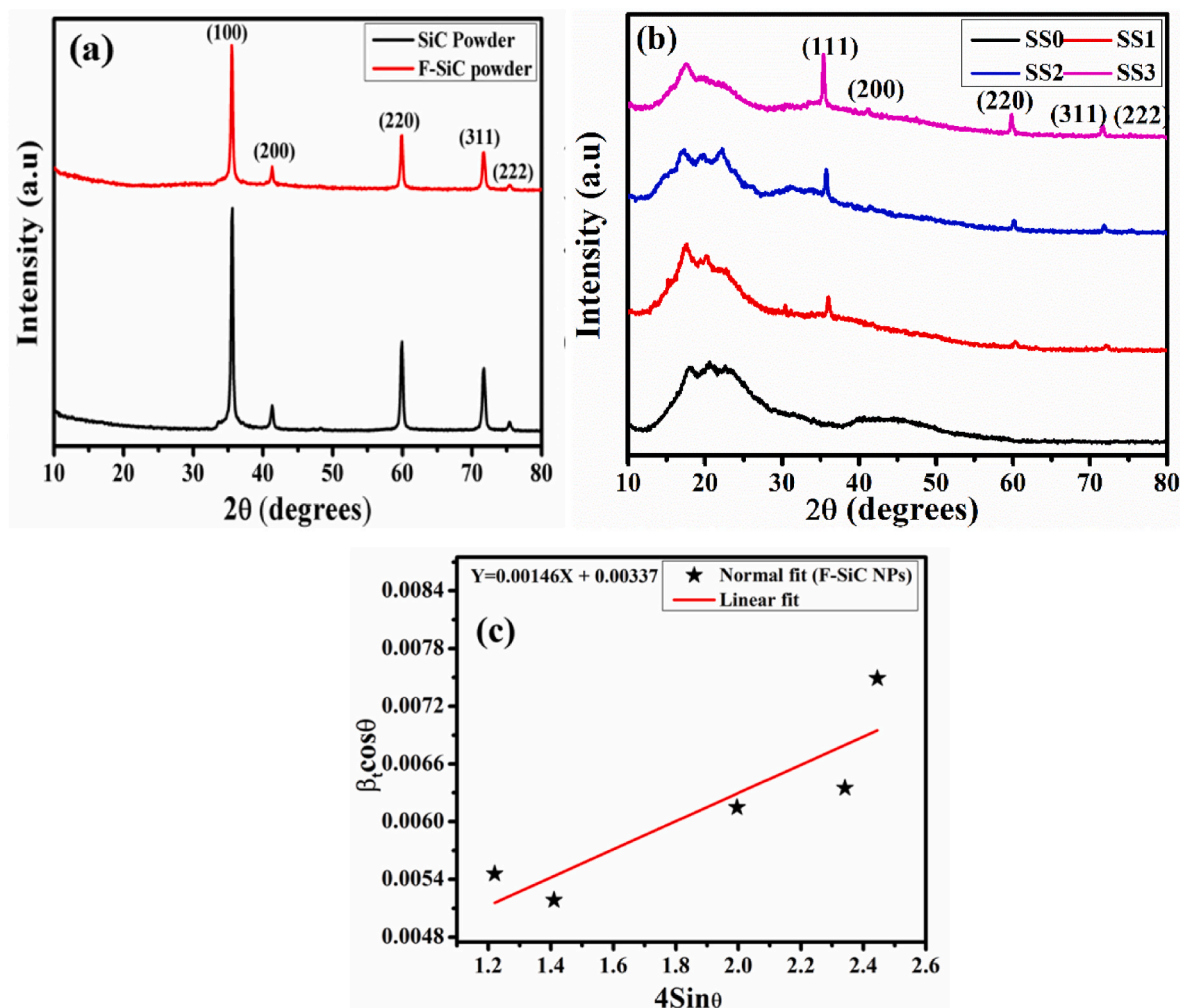


Fig. 4. XRD pattern of (a) SiC NPs and F-SiC NPs, (b) SG-ST/F-SiC NC films and (c) plot of $\beta_i \cos \theta$ versus $4 \sin \theta$ for F-SiC NPs.

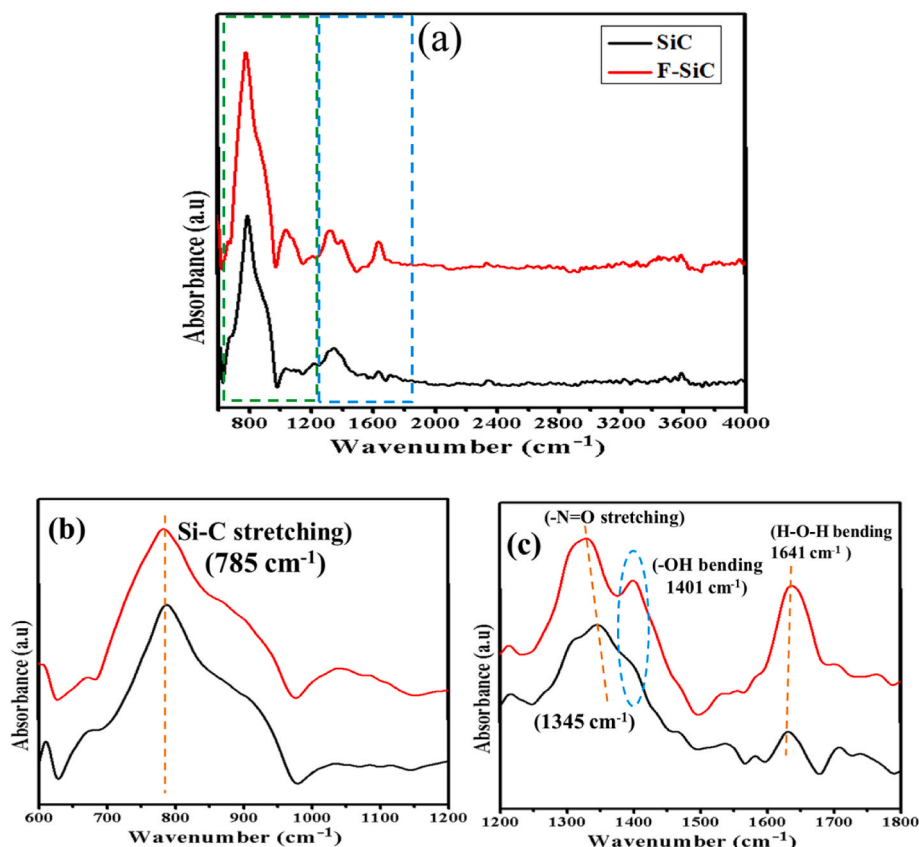


Fig. 5. FTIR spectra of SiC NPs and F-SiC NPs in wavenumber region between (a) 600–4000 cm^{-1} , (b) 600–1200 cm^{-1} and (c) 1200–1800 cm^{-1} .

using magnetic stirrer at 80 °C for 4 h. Then, resulting prepared solution was poured into petri dishes and marked as SS1, SS2 & SS3 respectively and dried in vacuum oven at 50 °C until the solvent evaporated. After that, the fabricated NC films with thickness of 180 – 220 μm were peeled off for further characterizations. Virgin SG-ST (SS0) film was also fabricated by the similar process. Schematic diagram of synthesis of SG-ST/F-SiC NC film has been depicted in Fig. 1 (step 2).

2.3. Characterization

Surface modification and interfacial interaction of F-SiC NPs with SG-ST matrix was confirmed by utilizing the FTIR spectroscopy (FTIR-ABB MB 3000) within the wavenumber region of 600 cm^{-1} to 4000 cm^{-1} . The surface morphology and elemental composition of F-SiC NPs was investigated with the help of Nova Nano FE-SEM 450 FEI equipped with EDS (energy dispersive X-ray spectrophotometer). For FESEM analysis, F-SiC NPs were coated with a layer of gold via sputtering technique. The size distribution of F-SiC NPs was analysed utilizing TEM (Talos) operated at 200 kV. For TEM measurements, a solution of F-SiC NPs dissolved in deionised water (5 mg/mL) was dropped on carbon-coated copper grid and dried in air. XRD measurements of as synthesized SiC NPs, F-SiC NPs, and SG-ST/F-SiC NC films was carried out by using Bruker AXS D8 Advance Diffractometer equipped with $\text{Cu-K}\alpha$ X-ray source ($\lambda = 0.1541 \text{ nm}$) in the wide angle region of 10° to 80° with a step size of 0.02° operated at 40 mA and 40 kV. The tensile properties of SG-ST and SG-ST/F-SiC NC films (cut into strips of 11 mm $\times$ 33 mm) were measured by using UTM (Static UTM Z010) with a load of 10 kN scanned at a fixed speed of 10 mm/min. The value of mechanical parameter such as 'E_{mod}', ultimate tensile strength (U_{TS}), % elongation at break (E_B), and fracture value (F_V) were obtained from the stress-strain curve. The various dielectric parameters such as ϵ' , tangent loss ($\tan \delta$), relaxation time (τ) of fabricated SG-ST/F-SiC NC films were

computed using Wayne Kerr 6500B Precision Impedance Analyser in the frequency regime of 20–1 MHz at room temperature (30 °C) and in the temperature range of 40 °C to 100 °C with a step size of 20 °C.

3. Results and discussion

3.1. FESEM

Fig. 2(a–e) depicts the surface morphology, elemental mapping and chemical composition of functionalised F-SiC NPs. According to FESEM image of F-SiC NPs, F-SiC NPs have irregular shape. Elemental mapping (shown in Fig. 2(b)) was also utilized to study the distribution of constituent elements of F-SiC NPs and Fig. 2(c) and 2(d) represents mapping in green and pink colour corresponding to Carbon and Silicon elements respectively. The chemical composition of F-SiC NPs was analysed by EDAX (shown in Fig. 2(e)) and found that F-SiC NPs were composed of 67.09 % of Carbon (C) and 32.10 % of Silicon (Si).

3.2. TEM analysis

Fig. 3(a) represents the TEM image of F-SiC NPs. Particle size distribution of F-SiC NPs is presented in Fig. 3(b) and the crystallite size of F-SiC NPs comes out to be $38.1 \pm 2.3 \text{ nm}$.

3.3. XRD analysis

Fig. 4(a) depicts the XRD pattern of SiC NPs and F-SiC NPs. The XRD pattern of as received SiC NPs display a major diffraction peak at 2θ value of 35.56° which corresponds to (111) plane and four minors peaks at 41.31°, 59.89°, 71.68° and 75.39° which are associated with (200), (220), (311) and (222) planes of SiC NPs respectively and matches with JCPDS file # 29-1129 [36,37]. Further, the XRD pattern of F-SiC NPs

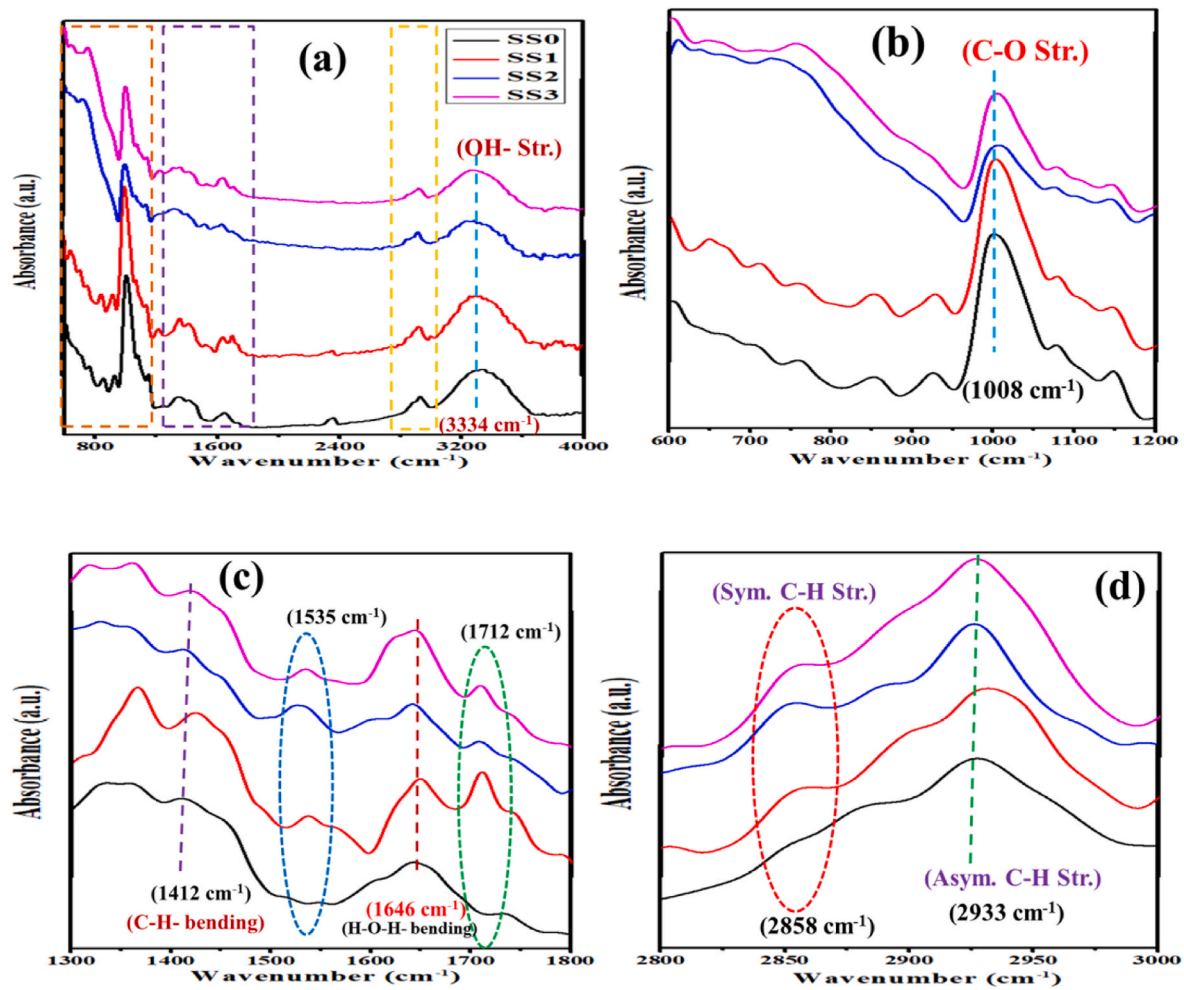


Fig. 6. FTIR spectra of pristine SG-ST and SG-ST/F-SiC NC films within wavenumber region (a) 600-4000 cm^{-1} , (b) 600-1200 cm^{-1} , (c) 1300- 1800 cm^{-1} and (d) 2800- 3000 cm^{-1} .

also display characteristic peaks at same 2θ values suggesting that the crystallographic planes of SiC NPs are not altered after the functionalization process.

Fig. 4 (b) depicts the XRD pattern of SS0, SS1, SS2 and SS3 NC films. The XRD pattern of virgin SG-ST (SS0) displays characteristic diffraction peaks at 2θ values of 16.84° , 19.54° , 21.81° corresponding to C-type crystalline structure (mixture of A- and B- type crystalline form) of SG-ST [15,38]. Further, in the XRD pattern of SG-ST/F-SiC NC films, the characteristic peaks at 2θ values of 16.91° , 19.35° and 21.81° are associated with SG-ST and at 2θ values of 35.41° , 41.14° , 59.81° , 71.62° and 75.22° correspond to (111), (200), (220), (311) and (222) planes of F-SiC NPs. The presence of F-SiC NPs characteristic peaks in XRD pattern of composites confirms the NC formation.

Further, the crystallite size (t) and lattice strain (ϵ) of F-SiC NPs was calculated utilizing Williamson-Hall method [39] and is given in eq. (1):

$$\beta_t \cos \theta = \frac{0.9\lambda}{t} + 4\epsilon \sin \theta \quad (1)$$

Where, ' β_t ' is the full width at half of the maximum intensity diffraction planes of F-SiC NPs and ' λ ' is the wavelength of incident $\text{Cu K}\alpha$ X-rays.

A graph between $4\sin \theta$ and $\beta_t \cos \theta$ has been plotted and the results are presented in Fig. 4 (c). The values of ' ϵ ' and ' t ' were computed using slope and intercept of best fit line. The value of ' t ' of F-SiC NPs comes out to be 40.62 nm which agrees well with TEM results. Further, intrinsic lattice strain (ϵ) comes out to be 1.46×10^{-3} and the positive value of slope indicates the expansion of lattice.

Inter-planar spacing (d) of F-SiC NPs was calculated using the Bragg's formula given in eq. (2):

$$2d \sin \theta = n\lambda \quad (2)$$

Where ' n ' is the order of reflection.

The calculated value of ' d ' corresponding to the (111), (200), (220), (311) and (222) planes comes out to be 0.25 nm, 0.22 nm, 0.15 nm, 0.13 nm and 0.12 nm respectively. Further, the value of lattice constant (a) of F-SiC NPs (simple cubic system) has been determined using the value of inter-planar spacing (d) by using eq. (3):

$$a = d(h^2 + k^2 + l^2)^{1/2} \quad (3)$$

Where h , k and l represents the miller indices of the diffraction planes. The calculated value of ' a ' of F-SiC NPs comes out to be 4.36 Å and agrees well with the experimentally measured value of 4.34 Å [40].

3.4. FTIR analysis

3.4.1. FTIR analysis of SiC NPs and F-SiC NPs

To confirm the functionalization of SiC NPs, the detailed analysis of FTIR results was carried out and Fig. 5 depicts FTIR spectra of SiC NPs and F-SiC NPs within the wavenumber region 600-4000 cm^{-1} . For highlighting the shifting in peak position with variation in intensity, FTIR spectra of SiC NPs and F-SiC NPs has been re-plotted in two different regions i.e 600-1200 cm^{-1} (Fig. 5(b)) and 1200-1800 cm^{-1}

Table 1
FTIR absorption band assignments of fabricated NC films.

FTIR absorption peaks of SS0	FTIR absorption peaks of SS3	FTIR absorption band assignment	References
1002 cm^{-1}	1008 cm^{-1}	C–O stretching vibration due to glycosidic bond linkage	[41,42]
1412 cm^{-1}	1416 cm^{-1}	C–H bending vibration	[41,42]
	1535 cm^{-1}	N=O stretching vibration	[41,42]
1642 cm^{-1}	1646 cm^{-1}	H–O–H bending vibration due to the presence of tightly bound water in the SG-ST	[41,42]
	1712 cm^{-1}	C=O stretching vibration	[41,42]
	2858 cm^{-1}	C–H symmetric stretching vibration	[41,42]
2927 cm^{-1}	2933 cm^{-1}	C–H anti-symmetric stretching vibration	[41,42]
3329 cm^{-1}	3334 cm^{-1}	O–H stretching vibration of glycosidic bond	[41,42]

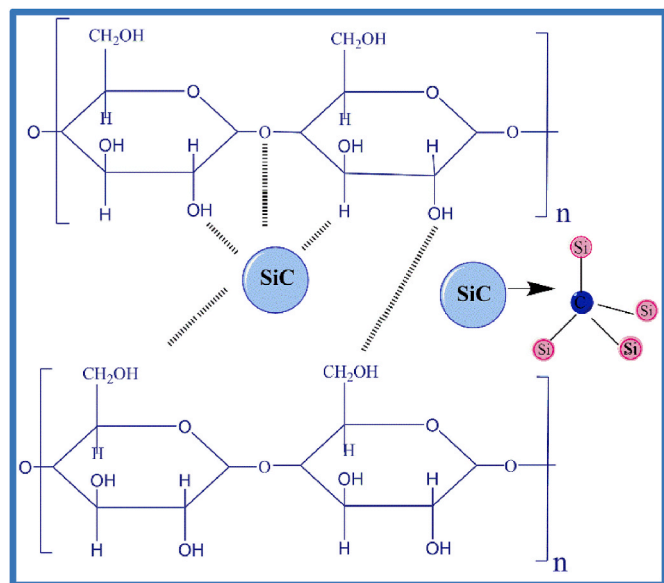


Fig. 7. Schematic representation of possible interaction of F-SiC NPs with SG-ST.

(Fig. 5(c)). The peak observed at 785 cm^{-1} in FTIR spectra of SiC NPs (Fig. 5(b)) is the characteristic peak of Si–C stretching mode of vibration and it remains intact even after functionalization suggesting that the crystal structure of SiC NPs remains same [24]. Further, the absorption peak observed at 1345 cm^{-1} (Fig. 5(c)) in SiC NPs representing the –

Table 2
 E_{mod} , U_{TS} , E_B and F_V of SS0 and SS3 NC films.

Sample	E_{mod} (MPa)	U_{TS} (MPa)	E_B	F_V (MPa)
SS0	73.6 $\pm$ 2.9	7.6	0.9	2.8
SS3	382.2 $\pm$ 8.9	65.4	1.7	13.2

N=O stretching mode of vibration and it shifts towards lower wave-number (1328 cm^{-1}) in F-SiC NPs [41,42]. Moreover, a new peak appears at 1401 cm^{-1} (Fig. 5(c)) and is attributed to –OH bending/C–H bending vibration [41,43]. The absorption peak appearing at 1631 cm^{-1} (Fig. 5(c)) in FTIR spectra of SiC NPs representing H–O–H bending vibration (chemically bound water present in SiC NPs) shifts towards higher wavenumber (1641 cm^{-1}) in F-SiC NPs [41,42]. Thus, the considerable shifting in peak positions along with the appearance of new peak after the acidic treatment of SiC NPs suggests the successful functionalization of SiC NPs with acidic oxidation treatment via HNO_3 .

3.4.2. FTIR analysis of SG-ST/F-SiC NC films

Fig. 6 depicts the FTIR absorbance spectra of SS0, SS1, SS2 and SS3 NC films. The characteristic absorption peaks observed at 1002 cm^{-1} and 3329 cm^{-1} in FTIR spectra of SS0 are associated with the C–O and O–H stretching vibration due to glycosidic bond linkage of SG-ST and confirm the monomer structure of SG-ST. For highlighting the variation in peak intensity and peak position shifting, the FTIR spectra of fabricated NC films has been re-plotted in three different regions, viz. 600–1200 cm^{-1} (Fig. 6 (b)), 1300–1800 cm^{-1} (Fig. 6 (c)) and 2800–3000 cm^{-1} (Fig. 6 (d)). With increasing wt.% of nanofiller in SS0, shifting in the peak's position towards higher wavenumber was observed and is listed in Table 1. Additionally, new absorption peaks are observed at 1535 cm^{-1} , 1712 cm^{-1} and 2858 cm^{-1} after the addition of F-SiC NPs and these peaks correspond to the N=O, >C=O and C–H (symmetric) stretching vibrations respectively [41,42]. Fig. 7 describes the possible molecular interaction of SG-ST with F-SiC NPs. Thus, above results suggests the strong interaction of F-SiC NPs with SG-ST; confirming the successful fabrication of SG-ST/F-SiC NC film.

3.5. Mechanical parameter analysis

The various mechanical parameters such as E_{mod} , U_{TS} , E_B and F_V of SS0 and SS3 NC films were computed utilizing stress versus strain curves presented in Fig. 8 (a). The stress-strain curves of fabricated NC films display a linear elastic behaviour (elastic deformation region) and a yield point followed by necking (uniform plastic deformation) before the final fracture (non-uniform plastic deformation region). Fig. 8 (b) depicts that the value of E_{mod} , U_{TS} , E_B and F_V SS0 increases with the addition of F-SiC NPs. The value of E_{mod} has been estimated from the slope of linear region (elastic region) of stress-strain curves and it

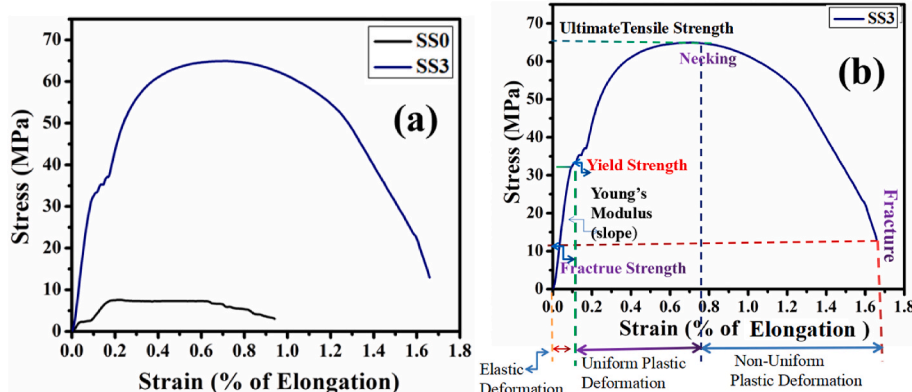


Fig. 8. (a) Stress-strain curve for SS0 and SS3 NC films and (b) stress-strain curve with detailed analysis of SS3 NC film.

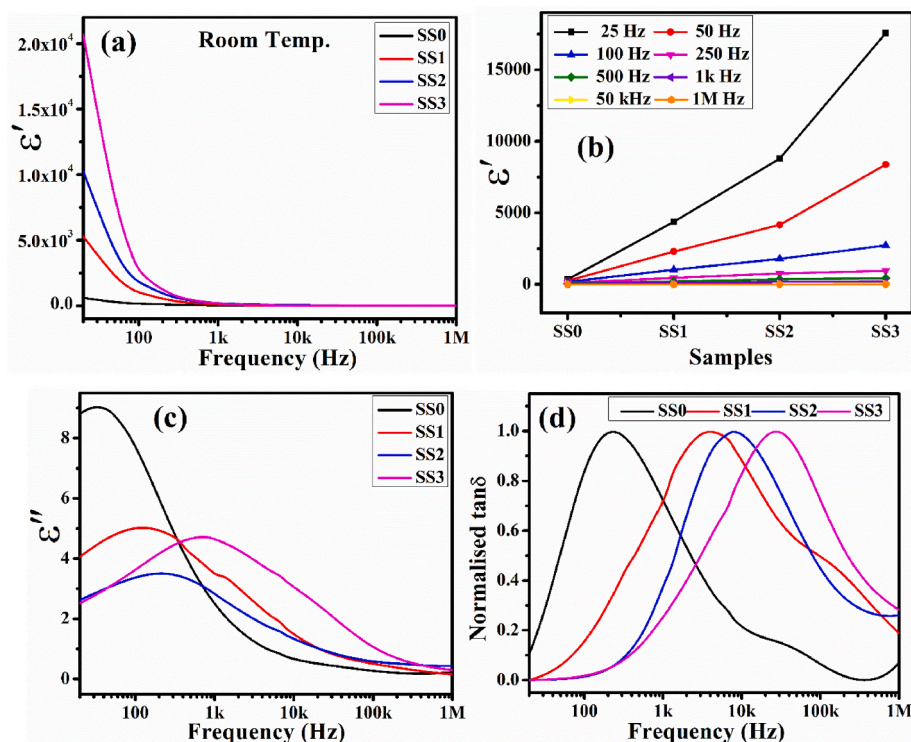


Fig. 9. Variation of (a) ϵ' with frequency, (b) ϵ' with concentration of F-SiC NPs, (c) ϵ'' , and (d) normalized $\tan \delta$ with frequency for fabricated NC films.

increases from 73.6 ± 2.9 MPa (SS0) to 382.2 ± 8.9 MPa for SS3. Further, the value of U_{TS} of SS0 increases from 7.6 MPa to 65.4 MPa for SS3. The value of F_V of SS0 increases from 2.8 MPa to 13.2 MPa and E_B increases from 0.9 to 1.7 for SS3. The enhanced mechanical parameters of SG-ST/F-SiC NC film (listed in Table 2) indicates that the F-SiC NPs has good interfacial interaction with SG-ST matrix and is also supported by FTIR analysis. Ceballos et al. [29] also presented similar results i.e E_{mod} and U_{TS} of thermoplastic starch increases from 50 MPa to 530 MPa and 2.8 MPa–9 MPa respectively, after the incorporation of 0.015 wt% of Ag NPs and attributed this increase to the good dispersionability and adhesion of nanofiller with the host matrix.

3.6. Dielectric parameter analysis

3.6.1. Frequency dependent behavior of dielectric parameters of SG-ST with varying wt.% of F-SiC NPs

Fig. 9 (a) represents the variation of dielectric permittivity (ϵ') with frequency for fabricated NC films. According to Fig. 9 (a), ϵ' of SS0 increases with the addition of F-SiC NPs, all the NC films shows high value of ϵ' in low frequency region and it gradually decreases and finally attains a constant value in high frequency region. In low frequency region, charge carriers are accumulated at the interfaces in SS0 due to difference in conductivity of nanofiller and host matrix. This separation of a pair of conducting F-SiC NPs by a thin layer of SG-ST, acts as a capacitors having nano-dimensions [44] (depicted in Fig. 14). Additionally, the high value of ϵ' at low frequency region may be ascribed to the

contribution from all type of polarization mechanism such as interfacial polarization also known as Maxwell- Wagner Sillars (MWS) polarization, dipolar, ionic, and electronic polarization. However, as the frequency increases, the contribution of interfacial polarization fades out followed by orientational (dipolar) and ionic polarization and, only electronic polarization contributes towards ϵ' at high frequencies thereby attaining a constant value. The value of ϵ' of SS0 (at $f = 25$ Hz) increases from 348 to 17561 with the addition of 6 wt% of F-SiC NPs. Thus, for SS3 NC film ϵ' increases drastically (50 times) in comparison to SS0. Alam et al. [28] observed similar behavior of ϵ' for gelatin embedded with varying concentration of MWCNT as nanofiller and concluded that ϵ' increases from 0.5 to 12000 with the addition of 0.02 % of nanofiller in gelatin matrix at 100 Hz and attributed this increase to interfacial polarization effect. Mollik et al. [27] also reported similar variation of ϵ' for starch after the addition of rGO and observed that ϵ' of starch increases from 195 to 4000 and finally to 60000 after embedding 0.5 wt% and 1 wt% of nanofiller in starch and concluded that interfacial polarization is the prime mechanism responsible for drastically enhanced value of ϵ' . Fig. 9 (b) represents the variation of ϵ' for all NC films at different values of frequency and it was observed that the value of ϵ' increases with increase in concentration of nanofiller which is prominent at low value of frequency (refer Table 3).

Fig. 9(c–d) depicts the variation of dielectric loss (ϵ'') and normalized $\tan \delta$ (given by relation $\tan \delta = \frac{\epsilon''}{\epsilon'}$) with frequency for all NC films and the value of ϵ'' and $\tan \delta$ increases, initially, attains a maxima and finally decreases with further increase in frequency, exhibiting Debye type

Table 3

Value of ϵ' at various frequencies and τ_s for SS0, SS1, SS2 and SS3 NC films.

Sample	ϵ' at different value of frequency								τ_s
	25 Hz	50 Hz	100 Hz	250 Hz	500Hz	1 kHz	50 kHz	1 MHz	
SS0	348	252	183	112	74	46	11	7	692.3 μs
SS1	4372	2297	1029	455	207	104	14	9	36.2 μs
SS2	8799	4156	1789	760	360	187	16	12	19.2 μs
SS3	17561	8383	2719	950	436	205	26	13	5.7 μs

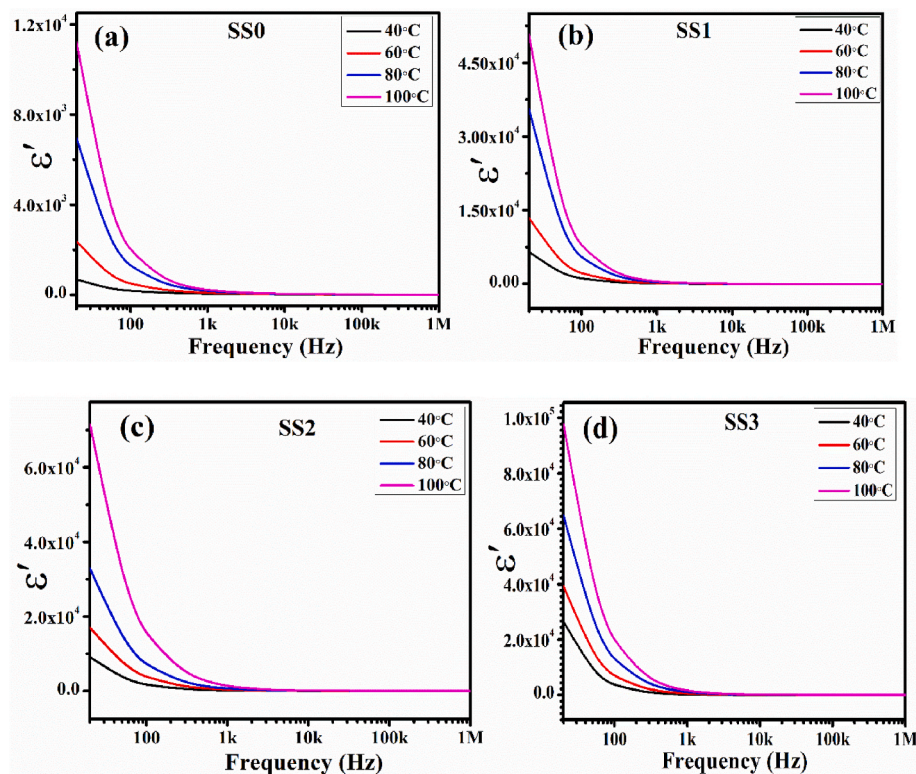


Fig. 10. Variation of ϵ' with frequency for all NC films at temperature range from 40 °C to 100 °C.

relaxation behavior (relaxation peak). In the low frequency region, all types of polarization mechanism are active; as a result, the value of $\tan \delta$ increases [45]. In the mid frequency region, resonance occurs among the resultant dipole movement of the polymeric chain and frequency of applied electric field, as a result, peak (resonance peak) appears. At

higher frequencies, dipoles are unable to follow the rapidly varying applied electric field as a result $\tan \delta$ decreases. The frequency, at which resonance condition occurs, gives the value of relaxation time (τ_s) and can be calculated utilizing the relation $\tau_s = 1/2\pi f$ where ' f ' is the frequency of applied electric field. Moreover, the resonance peak shifts

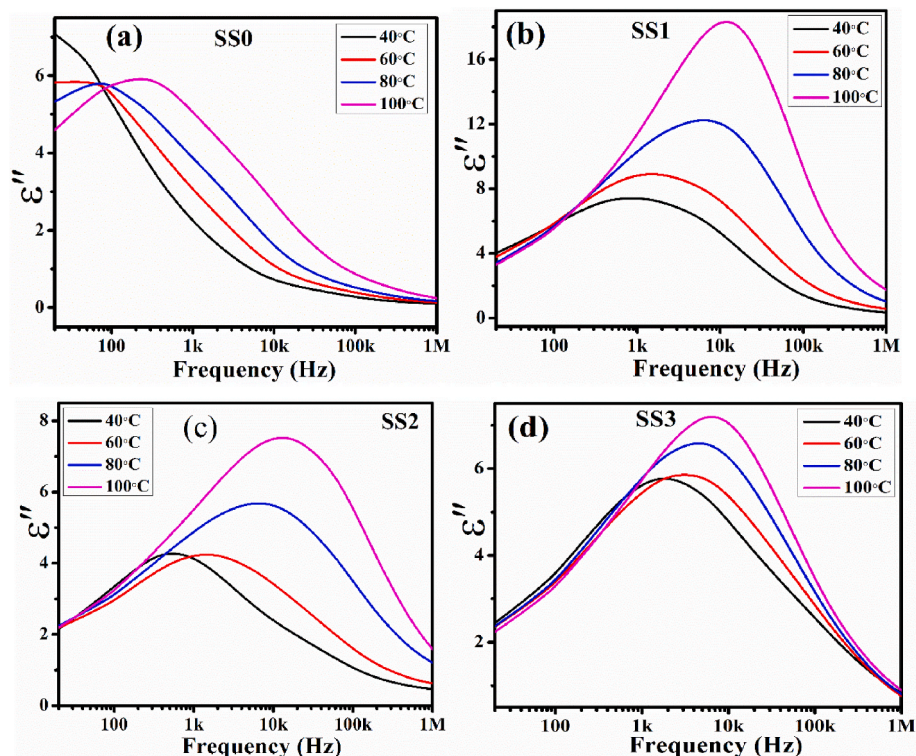


Fig. 11. Variation of ϵ'' with frequency for all NC films at temperature range from 40 °C to 100 °C.

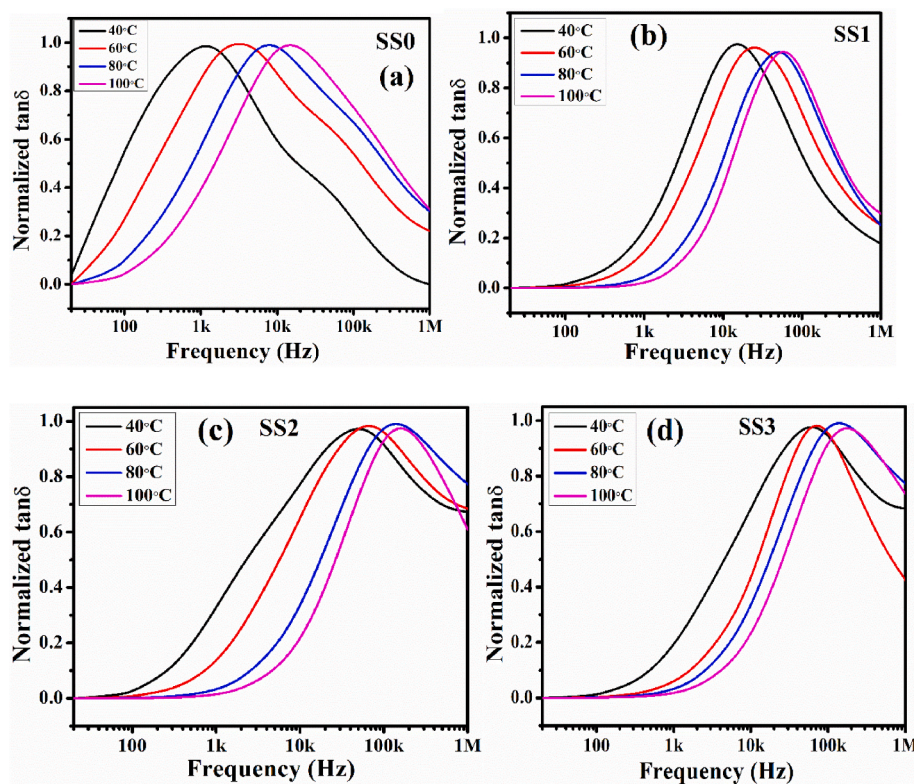


Fig. 12. Variation of normalized $\tan \delta$ with frequency for fabricated NC films from 40 °C to 100 °C.

Table 4

Value of ϵ' ($f = 25$ Hz) for synthesized NC films at temperature range from 40 °C to 100 °C.

Sample	$\epsilon' (f = 25 \text{ Hz})$			
	40 °C	60 °C	80 °C	100 °C
SS0	602	1987	5904	9534
SS1	5480	11433	30392	42518
SS2	10361	19355	38677	61381
SS3	22099	33874	55740	85176

towards the higher frequencies (as represented in Fig. 9 (d)) with increase in wt.% of F-SiC NPs in SG-ST. The calculated value of τ_s of SS0 with increasing concentrations of nanofiller is listed in Table 3 and it decreases from 692.3 μ s to 5.7 μ s for SS3 and this decrease in τ_s is attributed to faster energy transfer from one regulating site to another [46].

Thus, the fabricated NCs exhibiting high value of ϵ' even at room temperature with low value of ϵ'' and τ_s are a potential material for applications in energy storage devices [47].

3.6.2. Impact of temperature on ϵ' , ϵ'' and $\tan \delta$

Fig. 10 presents the variation of ϵ' with frequency at various temperature (40 °C to 100 °C) for all prepared NCs and shows that ϵ' increases with an increase in temperature from 40 °C to 100 °C. The value of ϵ' ($f = 25$ Hz) for SS3 NC film increases from 17561 (room temperature) to 22099 (40 °C) to 85176 (100 °C) suggesting that the polarization of synthesized NC films is thermally active. This several order of increment in the value of ϵ' for prepared NC films with increase in temperature may be attributed to the redistribution of F-SiC NPs in the SG-ST which results in formation of nanocapacitive layer between nanofiller and host matrix. With increase in temperature, segmental motion of molecular chains and flexibility of SG-ST increases, moreover, the accumulation of charge carriers at the interface increases (interfacial

polarization), as result, the value of ϵ' increases [46,48]. The calculated value of ϵ' ($f = 25$ Hz) at different value of temperature for SS0, SS1, SS2 and SS3 NC films are listed in Table 4.

Fig. 11 represents the behaviour of ϵ'' with frequency at 40 °C to 100 °C for all NC films. It depicts that as temperature increases ϵ'' also increases. Increasing temperature changes the space charge region, which results in an increased induced leakage current. Fig. 12 represents the behaviour of $\tan \delta$ with frequency for all NC film at 40 °C to 100 °C. It depicts that NC films displays Debye type relaxation peak which shifts towards the higher frequency region with increase in temperature resulting in the decreased relaxation time (τ_{temp}) [49]. The calculated values of τ_{temp} for all NC films are listed in Table 5.

3.7. Impedance analysis

Fabricated NC films were examined by impedance spectroscopy to ascertain the bulk resistivity and have an in-depth understanding of the exact conduction mechanism active in the NC material [50]. The impedance data (in terms of θ and Z) obtained from impedance analyzers was utilized to evaluate the real part (Z') and imaginary part (Z'') of complex impedance function ($Z = Z' + iZ''$) using eq. (4) and eq. (5):

$$Z' = Z \cos \theta \quad (4)$$

$$Z'' = Z \sin \theta. \quad (5)$$

Fig. 13(a–b) presents the variation of Z' and Z'' with frequency for fabricated NC films at room temperature. It depicts that the value of Z' for SS0 increases while the value of Z'' decreases with increasing wt. % of F-SiC NPs. The Nyquist plot (Z' vs Z'') for prepared NC films at room temperature are presented in Fig. 13 (c). These plots display a semi-circular curve followed by an inclined line, and radius of curvature of semicircular arc of SS0 specimen decreases as the concentration of F-SiC NPs in SG-ST matrix increases, suggesting that resistivity decreases while capacitive nature of the SG-ST/F-SiC NC films increases. This observed decrease in resistivity of the SG-ST/F-SiC NC films with

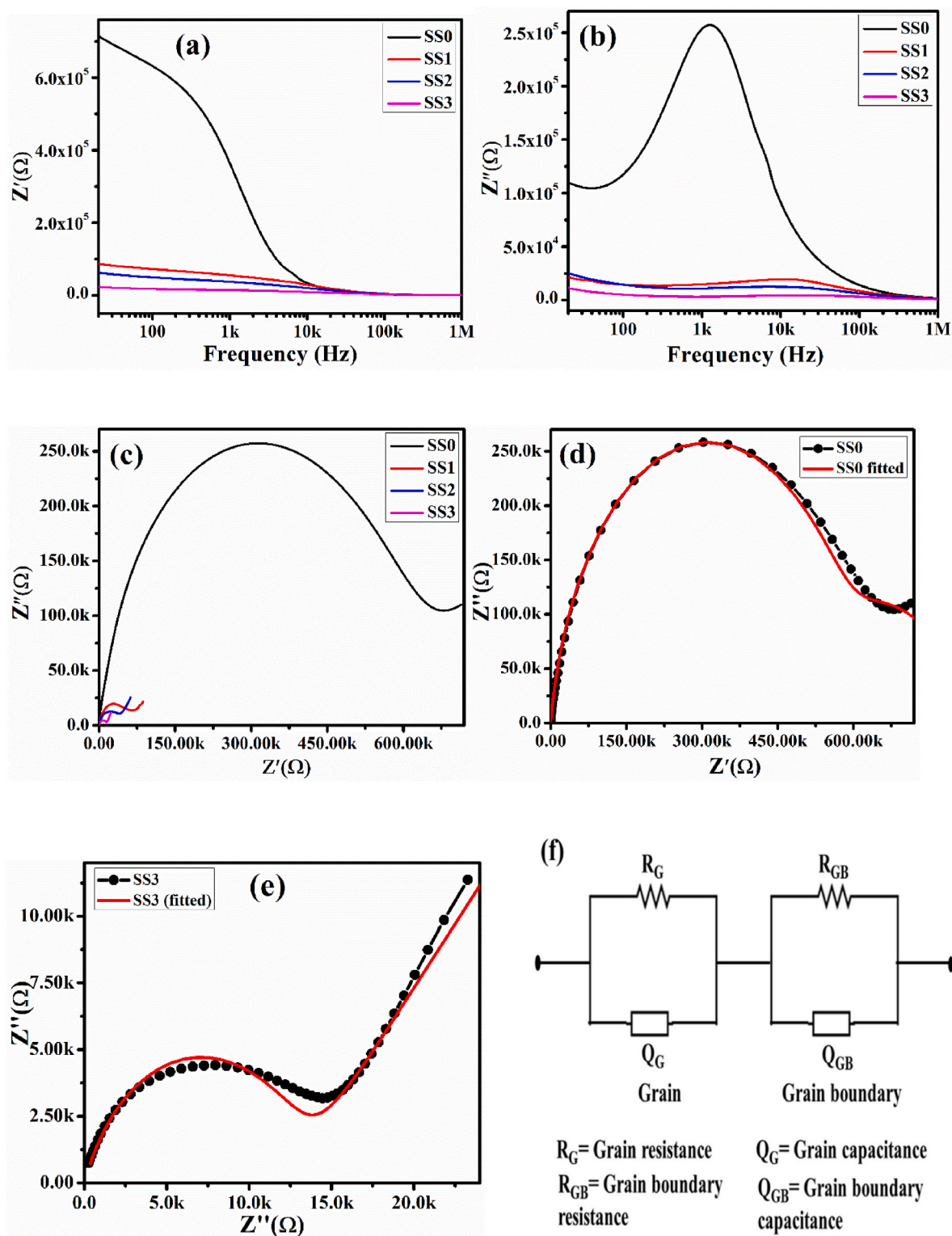


Fig. 13. (a) Variation of Z' , (b) Z'' with frequency, (c) Nyquist's plot for all NC films, (d-e) fitted Nyquist's plot of SS0, SS3 NC film, and (f) equivalent circuit.

increasing content of F-SiC NPs may be attributed to the formation of partly continuous conducting pathways throughout the SG-ST matrix resulting in conduction primarily via inter-filler tunneling and hopping mechanisms [51,52].

For quantitative analysis, Nyquist plots of fabricated NC films were also simulated by equivalent circuits using Z_{SIMPWIN} software. The Nyquist plots for SS0 and SS3 (Fig. 13(d-e)) were best fitted with the equivalent circuit (depicted in Fig. 13(f)) comprises of combination of

two RC parallel circuits, each having resistance (R), and constant phase element (CPE) denoted by Q (capacitive element) and is given in eq. (6):

$$Z_{\text{CPE}} = \frac{1}{Q(j\omega)^n} \quad (6)$$

Where ' Z_{CPE} ' is the impedance of the constant phase angle component, 'n' is the exponential factor and ' ω ' is the angular frequency. For $n = 0, 1$,

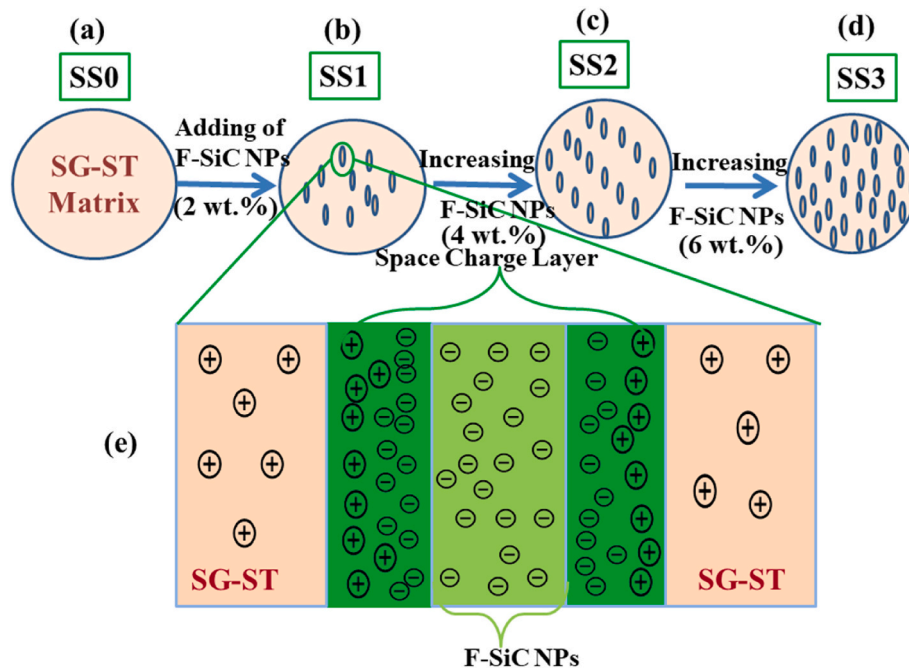


Fig. 14. Schematic mechanism of formation of nanocapacitor layer in SG-ST/F-SiC NC.

Table 5

Value of τ_{temp} for all the samples at temperature range from 40 °C to 100 °C.

Temp. (°C)	Relaxation time (τ_{temp}) (μ s)			
	SS0	SS1	SS2	SS3
40	142.2	10.4	3.6	2.6
60	49.1	6.5	2.8	1.4
80	20.1	3.4	1.2	1.0
100	10.8	2.7	0.9	0.8

Table 6

Parameters of the equivalent circuit of the fitted Nyquist's plot for SS0 and SS3 NC films.

Sample	R_G (Ω)	Q_G (F)	n_G	R_{GB} (Ω)	Q_{GB} (F)	n_{GB}
SS0	850.8 k Ω	37 μ F	0.6018	667 k Ω	0.5945 nF	0.8784
SS3	15.9 k Ω	14.36 nF	0.7238	16.73 k Ω	341.6 nF	0.999

or -1 , Q represents pure resistance, ideal capacitor, or an inductance, respectively [30]. The corresponding parameters of equivalent circuits for the SS0 and SS3 NC films are listed in Table 6. The first parallel component of the equivalent circuit refers to the grain i.e. the conductive regions formed by F-SiC NPs in the SG-ST whereas the second parallel circuit indicates the grain boundary regions i.e. the SG-ST dominated gaps among the comparatively densely F-SiC enclosed regions [27]. It was observed that the value of grain resistance (R_G), grain boundary resistance (R_{GB}) as well as grain capacitance (Q_G) decreases after the incorporation of F-SiC NPs while grain boundary capacitance (Q_{GB}) increases with the incorporation of F-SiC NPs. The value of R_G and Q_G for SS0 decreases from 850.8 k Ω to 15.9 k Ω and from 37 μ F to 14.4 nF after the addition of 6 wt% of F-SiC NPs. Further, the value of Q_{GB} for SS0 increases from 0.5945 nF to 341.9 nF while the value of R_{GB} decreases from 667 k Ω to 16.7 k Ω after the addition of 6 wt% of F-SiC NPs. The value of Q_{GB} increases with increase in the concentration of nanofiller in SG-ST matrix, because density of charge carriers is higher at the grain boundary in comparison to grains, as a result, capacitance increases at the grain boundary. Therefore, the incorporation of F-SiC NPs into SG-ST decreases its total resistance ($R_G + R_{GB}$). Hence the enhanced

capacitance of SG-ST matrix after the addition F-SiC NPs results in the enhanced value of ϵ' and hence supports the experimental results (as presented in Table 6).

3.8. Charge carrier transport mechanism of SG-ST/F-SiC NC film

3.8.1. Effect of F-SiC NPs

The charge transport mechanism active in fabricated NC film is elucidated using a hypothetical model (proposed utilizing the results obtained from dielectric parameter analysis) is depicted in Fig. 14. In Fig. 14, SS0 represents the pristine SG-ST matrix which does not have significant charge deposition area however, on addition of F-SiC NPs in SS0, resultant NC offers a large surface area for collection of charge (creation of space charge layer between the F-SiC NPs and the SG-ST matrix which act as a nanocapacitor layer) as depicted in Fig. 14 (e) and as a result, the net capacitance increases. This increase in capacitance further increases the ϵ' of SG-ST/F-SiC NC films. Further, the increase in wt.% of the nanofiller in the host matrix offers more surface area for the transport of charge carriers with in the polymer via hopping and generates a continuous conductive pathway [46,50]. Thus, the transport of charge carriers from one coordinating site to another occurs via hopping and tunneling process.

3.8.2. Effect of temperature

As it has been discussed in earlier results that the segmental motion of SG-ST chains and hopping of charge carriers increases with increase in the temperature, and as a result, the value of ϵ' of SG-ST/F-SiC NC enhances. Thus, the segmental motion of biopolymer chains and hopping of charge carriers are interlinked. On the basis of these dielectric results, a model has been proposed to apprehend the role of temperature on charge carrier transport mechanism of SG-ST/F-SiC NC. At low temperature, SG-ST/F-SiC NC has a semi rigid structure and charge carriers migrate from one coordinating site to another occurs with a hopping length L_1 , hopping potential barrier V_1 and relaxation time τ_1 . However, as the temperature increases, NC system transforms into a more flexible system, as a result, hopping length ($L_2 < L_1$), relaxation time ($\tau_2 < \tau_1$) and hopping potential barrier ($V_2 < V_1$) decreases [46] as depicted in Fig. 15. The reported decrease in relaxation time with increase in temperature is evident in loss tangent analysis also (refer Table 5). Further,

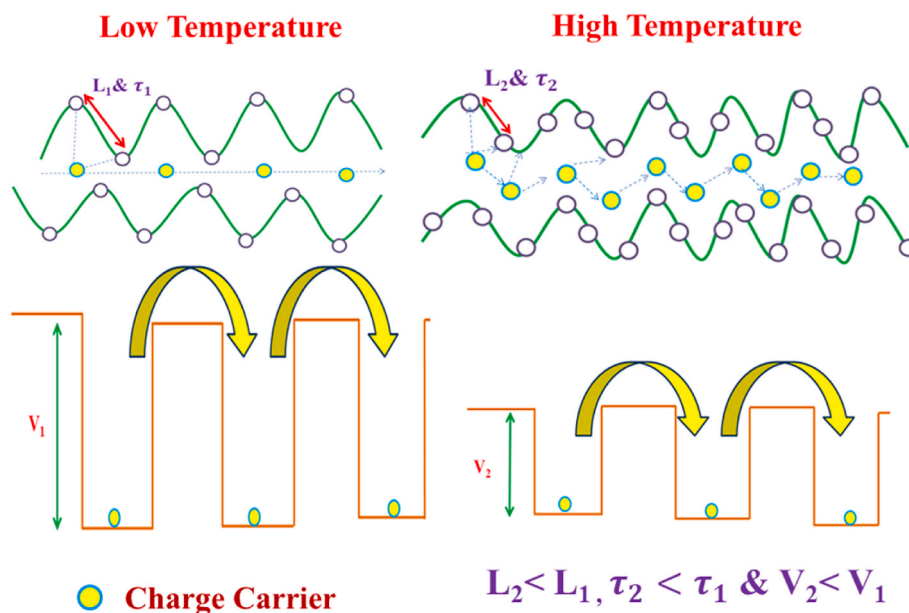


Fig. 15. Schematic representation of temperature dependent charge transport mechanism.

the observed results are also supported by Arrhenius relation. According to this relation, the temperature (T) is related to the relaxation time (τ) by eq. (7):

$$\tau = \tau_0 \exp(E_a/k_B T) \quad (7)$$

where ' τ_0 ' is the pre-exponential factor, ' E_a ' is the activation energy for hopping and ' k_B ' is Boltzmann's constant. According to eq. (7), τ is very sensitive to variation in T as T appears in an exponential term. Thus, higher is the temperature, lower is the relaxation time. Hence, the observed results i.e. the decrease in the value of the relaxation time with increase in temperature indicates that the hopping length reduces as a result, the time taken by the charge carriers to hop from one coordinating site to another decreases [48,53,54]. The results i.e. the decrease in hopping length with increase in temperature is further supported by diffusion-based model [53] also.

According to this model, the hopping length (L) and the relaxation time (τ) are related by eq. 8:

$$\tau = \frac{L^2}{2D} \quad (8)$$

Where, ' D ' is the diffusion coefficient.

According to eq. (8), for a given diffusion coefficient, a shorter hopping length is associated with a lower relaxation time and vice versa [46,53,54]. Thus, the Arrhenius relation and diffusion models validate the experimentally obtained results.

These results indicate that the value of ϵ' increases due to an increase in the polarization of the SG-ST/F-SiC NC films with increase in temperature. The chief mechanisms responsible for the observed increases in dielectric parameters of the SG-ST/F-SiC NC films with temperature are enhanced flexibility, reduced hopping length, segmental motion of the polymeric chains, and lowering of hopping potential barrier of the SG-ST/F-SiC NC films [46,53,54].

4. Conclusions

Self-supporting, flexible, and biodegradable polymer NC films based on SG-ST with the incorporation of F-SiC NPs were successfully synthesized via solution casting technique. The surface morphology and structural analysis of F-SiC NPs were investigated by utilizing the FESEM, TEM and XRD technique. The average crystallite size ($38.1 \pm$

2.3 nm) of F-SiC NPs from TEM analysis agrees well with XRD measurements (40.62 nm). The value of ϵ' ($f = 25$ Hz) for SS3 NC film increases from 17561 (room temperature) to 22099 (40°C) to 85176 (100 °C) suggesting that the polarization of synthesized NC films is thermally active. The value of relaxation time for SS0 at room temperature decreases from 692.3 μ s to 5.7 μ s with increase in concentration of 6 wt% of F-SiC NPs which further decreases to 0.8 μ s at 100 °C. Impedance studies shows that the value of R_G and R_{GB} of SS0 decreases from 850.8 k Ω to 15.9 k Ω and 667 k Ω –16.7 k Ω respectively after the addition of 6 wt% F-SiC NPs. The value of Q_{GB} increases from 0.5945 nF to 341.9 nF with the incorporation of 6 wt% of F-SiC NPs in SS0. Thus, the prepared NC films with outstanding mechanical and dielectric attributes can be suitable for future green flexible electronics and energy storage devices.

CRedit authorship contribution statement

Savita: Writing – original draft, Formal analysis, Data curation, Writing – original draft, Formal analysis, Data curation. **Jaspreet Kaur:** Validation, Data curation. **Navneet Kaur:** Methodology, Formal analysis. **Anil Arya:** Writing – review & editing, Validation, Investigation. **Kiran Siwach:** Resources, Formal analysis, Data curation. **Rajnish Dhiman:** Resources, Data curation. **Pawan K. Sharma:** Validation, Methodology, Investigation. **Annu Sharma:** Writing – review & editing, Validation, Supervision, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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