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Tailoring the properties of epoxy/silicone blends for high-voltage capacitor applications

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Abstract

In recent decades, high dielectric constant (*k*) polymer nanocomposites have proved excellent potential in dielectric and energy storage applications. Epoxy/silicon rubber composite materials have shown promising properties in applications such as high-voltage insulation. Three types of nanomaterials (SiO_2, TiO_2, and TiO_2@SiO_2) with distinct intrinsic properties are carefully chosen to build high-*k* epoxy/silicone polymer nanocomposites in this study. To raise the value of *K*, this work tailored the polarizing ability or permittivity of dielectric material by inserting different weight ratios of nano-fillers (SiO_2, TiO_2, TiO_2@SiO_2) in the base matrix of dielectric material in epoxy and silicon rubber blends. With a base matrix containing 75% epoxy and 25% liquid silicone rubber concentration, the maximum value of *K* obtained is $K = 158$ for 5% TiO_2 and there is an increase in the dielectric strength to 398 kV/mm. The obtained results indicate that, among the three different kinds of epoxy/silicone, TiO_2@SiO_2 has the most potential in enhancing the energy storage capabilities of the proposed nanocomposites, owing to the largest increase in *k* while maintaining low dielectric loss and leakage current.

Keywords: Permittivity, Polarization ability, Dielectric strength, Nanocomposites, Silicone rubber, Capacitance, Silicone rubber, Titanium dioxide-coated over silicone dioxide (TiO_2@SiO_2)

Introduction

Dr. Pierre Castan and Dr. Sylvan Greenlee developed epoxy for the first time in the 1930s. Epoxy compositions used to be predominantly solid, although liquid variants were produced subsequently [1]. Various researches on advanced capacitor dielectrics have been underdeveloped for the last half-century. The demand for smaller and lighter capacitors with high dielectric constant, energy density, breakdown strength, and higher temperature capability increases in the high-voltage industry. Dielectrics are insulator materials with some leaky characteristics. The polarization ability of the dielectrics depends upon their internal chemistry. Materials with higher permittivity have charges that can be more easily displaced. Epoxy resin and silicone rubbers are considered for capacitor dielectrics in high-voltage applications [2]. The properties which make its use attractive are biocompatibility, environmentally friendly, flame resistance, and long shelf-life [3]. With the new developments in nanotechnology, it has been anticipated that the combination of nanoparticles like TiO_2 and SiO_2 with traditional resin systems can create

Table 1 Various curing agent groups

	Aliphatic amines	Polyamides	Cycloaliphatic amines	Aromatic amines	Anhydrides	Imidazoles	Lewis acids
Dielectric constant 23 °C 60 Hz–1 MHz	3.22–3.83	2.91–4.17	3.42–3.97	3.85–4.65	2.89–4.27	2.95–3.15	3.13–4.34

Table 2 Properties of silicone rubber

Property	Min value (S.I)	Max value (S.I)
Density	1.1 Mg/m^3	2.3 Mg/m^3
Tensile strength	2.4 MPa	5.5 MPa
Compressive strength	10 MPa	30 MPa
Thermal conductivity	0.2 W/m k	2.55 W/m k
Breakdown potential	11 MV/m	28 MV/m
Resistivity	3.16e+19 Ω m	1e+22 Ω m

nanocomposite materials with enhanced electrical, thermal, and mechanical properties [4]. An epoxy nanocomposite system exhibits high permittivity at low frequencies and high electrical conductivity when dispersed with conductive fillers [5].

While silicone rubber (SiR) is a lightweight material that possesses high chemical resistance and flexibility with dielectric strength higher than 100 kV/mm [6]; today, SiR is used for volume insulation: high-voltage feed-throughs, generator stator coils, etc. Knowledge of the temperature dependence of dielectric properties from these materials is becoming important for future equipment designs [7]. Various studies show that the addition of nano-fillers (TiO_2, SiO_2, SiO_2@TiO_2) to SiR has significantly improved dielectric constant, dielectric strength, and thermal resistivity, hence leading to the formation of a good-quality dielectric material for capacitors. Thus, using epoxy and SiR can result in a material with enhanced resistivity and dielectric strength for usage at high frequencies and high-voltage capacitor applications.

In [8], it is proposed that the dielectric strength of some dielectric materials can be increased by adding nano-fillers TiO_2 coated with SiO_2.

A system may have a dielectric constant that increases with temperature (3.47 at 23 °C and 3.56 at 100 °C) for a 60 Hz application but changes with temperature (3.29 at 23 °C and 2.98 at 100 °C) for a 1 kHz application [9]. In general, the dielectric constant increases with increased temperatures and decreases with an increase in frequencies. In [10], characteristics are considered in Table 1.

SIR is a silicone-based polymer. The properties of silicone rubber in [11] are given in Table 2. It is an important electrical insulating material for nuclear power plant cables [12]. The dielectric constant value for silicone rubber ranges from 2.9 to 4.

Depending upon their properties and curing time, there are different types of silicone rubber. There is a way to modify dielectric elastomers' properties by adding high-permittivity metal oxide fillers. Liquid silicone rubber possesses a relatively low viscosity favorable for loading inorganic fillers (TiO_2, SiO_2, Al_2O_3) [12–14]. Epoxy resin often adds fillers to enhance its mechanical, thermal, and chemical properties. The addition of fillers can deteriorate electrical performance. The dielectric properties and space charge behavior of epoxy resin/nanocomposites with nano-fillers SiO_2 and Al_2O_3 ions [13, 14]. The effect of epoxy species on the tensile and electrical insulation properties of epoxy/micro-silica composites using three species of epoxy resins for high-voltage insulators was investigated in [15]. Polymer nanocomposites are used in power capacitors for electrical energy storage [16–18]. Details of nano- and for Electrical Engineering Applications [19, 20]. There is the feasibility of using cassava cortex particles as reinforcement in an epoxy matrix for the development of composite materials with insulation properties (resistivity) [21].

The blend's appropriate weight percentage composition enhanced the dielectric strength and tracking resistance in different conditions [24]. Different dielectric behavior was observed depending on filler type, filler concentration, and temperature [25–27]. Variations in real (ε') and imaginary (ε'') parts of dielectric constants and loss tangent of material with frequency and temperature have been studied [28, 29]. The inclusion of silicone epoxy effectively improved the glass transition temperature (T_g), and the thermal insulation also improved the electrical properties like resistance and dielectric constant for using it as a capacitor at high frequencies and in high-voltage strength applications [26].

The prior work on $TiO_2@SiO_2$ by [7] was updated in [30] to focus on a limited range of 0.3–0.8% $TiO_2@SiO_2$ nanoparticles by weight in epoxy. To modify the dielectric characteristics of epoxy resin, a range of nanoparticles have been utilized, including ZnO, Al_2O_3, SiO_2, TiO_2, BN, AlN, and other fillers. In [14, 30–33], the study looked at the dielectric characteristics of epoxy nanocomposite with low inorganic nano-filler concentrations. The permittivity of the interphase between the polymer and nanoparticles in a nanocomposite has a major influence on the electric field distribution and AC breakdown strength of the nanocomposite, according to [30]. However, further study is needed to fully understand the impact of various nano-filler materials, their percentage loading, and dispersion on the dielectric characteristics of polymers.

Problem statement

For dealing with high-voltage capacitors, some materials have a dielectric between plates of a capacitor with very good dielectric strength. The greater the dielectric strength, the greater the ability to hold the charge between the plates of the capacitor, while a good dielectric means a material with high polarization ability but very low conductivity.

In 2016, Chen et.al [7] proposed that the dielectric strength of some dielectric materials can be increased by the addition of nano-fillers TiO_2 coated with SiO_2. It is proposed in this research to investigate the effect of some other nano-particles on different dielectric materials. Our target will be to tailor the properties of some novel materials like epoxy and silicone rubber for enhanced dielectric constant and dielectric strength. The increase in polarization ability leads to an increase in permittivity, and hence, there is the increase in the dielectric constant of the material.

Work aims to provide the following key benefits by undergoing this research:

1. Increasing the permittivity of some common dielectrics.

2. Enhancing the dielectric constant of epoxy/SiR blends beyond the values already reported in the literature.
3. Increasing the breakdown inception voltage.

Energy storage devices are always required while dealing with high voltages. For that purpose, high-voltage capacitors are designed. But there is always a space limitation that limits the energy storage capacity. Therefore, to cope with this problem, various types of research were made to tailor the dielectric medium between the plates of a capacitor. No further change can be made in the internal material chemistry up to a certain limit. New dielectric materials by introducing three different types of nanoparticles the targeted factors are dielectric constant (K) and dielectric strength. It will be to tailor the properties of some novel materials like epoxy and SiR for enhanced dielectric constant and dielectric strength. The increase in polarization ability leads to an increase in permittivity, increasing the material's dielectric constant. The dielectric properties of epoxy and SiR are tailored by adding three different nanoparticles, e.g., SiO_2, TiO_2, and SiO_2@TiO_2. The applications of this work will be high-voltage (HV) capacitor dielectric materials, compact electric power circuits, HV capacitors used in Marx generators, pulse generators, etc. A pulse duration of a hundred microseconds at a megavolt level to achieve high-energy pulsed power conditioning is used. Examples of loads that require currents in the 100 KA range are electrochemical guns, active armor, directed energy weapons, electric launch platforms, and all-electric warships. Chemical and electrical characterization of the formed nanocomposite was carried out using scanning electron microscopy (SEM), energy-dispersive X-ray (EDX), leakage current measurement, relative permittivity, and electrical resistivity measuring methods. The characteristics of epoxy nanocomposites and raw epoxy were then compared. The paper is divided into the following sections which are presented as follows: "Experimental methodology", "Methods of analysis", "Results and discussions", "Conclusion" sections.

Experimental methodology

After deeply going through the literature review, a schematic methodology is designed for carrying out experiments. The base materials are chosen based on their dielectric strength, inertness, and a variety of other factors. Then focus comes on selecting nanoparticle fillers to enhance the strength and properties of sample dielectrics. A proper ratio of materials and filler is defined for each sample composition, leading to nanocomposites' unique characteristics. The detailed view of the research methodology is as follows:

Design of experiment

Experiment design will be accomplished by following steps:

Selection of raw materials

After developing a deep understanding of the nature and properties of materials, it is decided that the base matrix of laminates will consist of epoxy and liquid silicone rubber RTV-528 blends. Epoxy with hardener TETA (Tri-ethylene tetra amine) is selected, being inert and having good dielectric strength and mixing ability. Epoxy/SiR blends are

Table 3 Properties of YD128

Properties	Values
Color	Clear, light yellow liquid
Viscosity at 25 °C	11,000–14,000 cP
Density at 25 °C	1.16 g/ml
Moisture content	0.1% max
Non-volatile component	100%
Flashpoint	> 150 °C
Epoxide value	5.15–5.40

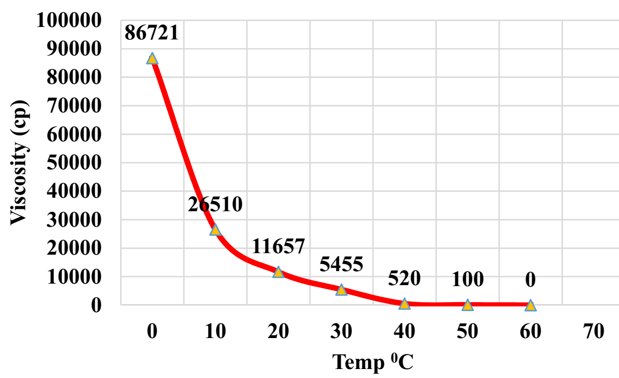

Fig. 1 Viscosity and temperature relation

prepared in this research by mixing epoxy and silicon rubber weight ratios. Three different nano-filler types are added to increase the permittivity and hence the dielectric constant of sample blends. Base polymer materials include liquid silicone rubber (LSR-528) and epoxy YD 128 + TETA resin. Nano-fillers SiO_2 nanoparticles, TiO_2 nano-particles, and TiO_2@SiO_2 nano-particles are incorporated.

Epoxy YD 128

YD 128 is an unmodified liquid epoxy resin produced from bisphenol-A and epichlorohydrin and has a medium viscosity. A wide variety of curing agents are available to cure this liquid epoxy resin at ambient conditions. When cured with a suitable hardener, epoxy YD 128 gives excellent chemical, mechanical, and electrical properties. In [30], properties are given in Table 3.

The relationship between viscosity and temperature for YD128 is given in Fig. 1 from [30].

Silicone rubber RTV-528

Mold-making SiR, generally named two-part room temperature cured, has exceptional fluidity and good operability. It consists of two parts, flowable liquid silicon, and a curing agent. It gets cured at room temperature 25 °C within 5–7 h in the demolding process. RTV-528 bears good tensile strength, tear strength, and low shrinkage, which is suitable for pouring methods of operation. In [35], the detailed properties are given in Table 4.

Table 4 Properties of RTV-528

Properties	Values
Appearance	White
Mixing ratio	3~4%
Operating time (25 °C)	30~50 min
Curing time (25 °C)	4~6 h
Hardness	28 ± 2 Å
Density	1.08 g/cm^3
Viscosity (25 °C)	13,000~17,000 Mpas
Tensile strength	≥ 30 kgf/cm^2
Tear strength	≥ 10 kgf/cm
Elongation break	≥ 300%
Refractivity	≤ 0.25%

Table 5 Curing time of different curing agents

Curing agent	Curing time
2%	8–10 h
3%	6–8 h
4%	4–6 h
5%	45–50 min

The amount of curing agent added decides the time required for silicone rubber to get cured. The approximate values of curing time are given in Table 5.

Nano-fillers are a class of materials in which at least one component has a nano-sized dimension. By varying the shape, size, and concentration of nanofillers, the macroscopic properties of nanocomposite can be controlled easily [36]. Different nano-fillers will be added to the epoxy/silicone rubber base matrix to improve the permittivity, dielectric strength, and dielectric constant. SiO_2, TiO_2, and TiO_2@SiO_2 are added in varying 1–5% ratios to prepare 45 different composition samples.

TiO_2 is widely used because of its easy availability, nontoxicity, chemical stability, dielectric and optical-electronic properties, low cost, and high photocatalytic properties [37]. Titanium dioxide in ore form possesses a high dielectric constant. The dielectric constant of blend materials depends on the dielectric properties of their components, that is, base polymer matrix and inorganic filler. So, the nanoparticles of TiO_2 with elevated dielectric strength values and low dispersity are strongly recommended for application in high-voltage dielectric materials.

The presence of SiO_2 nanoparticles reduces the conjugation length and thus lowers the conductivity compared to the base matrix alone. Due to the excellent electrical and dielectric properties of SiO_2, it is extensively used in HV capacitors and field-effect transistors. The addition of SiO_2 nanoparticles into epoxy resin enhances the electrical, thermochemical, and optical properties. The addition of SiO_2 nanoparticles into epoxy resin enhances the electrical, thermochemical, and optical properties which are SiO_2 powder and nanoparticle [38–40].

Titanium dioxide coated with silicon dioxide

SiO_2-coated TiO_2 nanoparticles are prepared by a sol–gel process. A silica layer is deposited chemically over titanium dioxide nanoparticles in this process. TiO_2 alone possesses high permittivity values, but due to the silica layer above it, the value of permittivity decreases comparatively. This decrease in permittivity is due to the presence of a hydroxyl group in the deposited silica layer. A view of TiO_2@SiO_2 nanoparticles taken from a transmission electron microscope can be seen in the image below. However, this surface modification increases resistivity and polarization ability value.

After selecting raw materials, the standard mixing ratios of the base matrix are calculated based on experiments. Different formulations, *F1*, *F2*, and *F3*, are decided to prepare dielectric discs. It is the weight ratios of two base materials that can be varied by 25% epoxy/75% RTV 528, 50% epoxy/50% RTV 528, and 75% epoxy/25% RTV 528 to form these different kinds of materials.

Filler ratios

After deciding the base matrix ratios, the filler concentration is decided based on previous studies and experiments. Three different filler nanoparticles SiO_2, TiO_2, and SiO_2@TiO_2 are added in 1–5% in base matrix ratio decided above. Thus, 15 unique samples are prepared for each type of filler. For better testing and results, three samples per type are prepared.

Preparing blends

Epoxy/silicone rubber blends are prepared by hand lay-up method. The hand lay-up method is the oldest technique for preparing woven composites. There are following steps involved in preparing the blends. The block diagram of preparation is shown in Fig. 2. At first, a weighted amount of epoxy resin is taken in a beaker. Then, a measured amount of ethyl acetate is added to it with hand blending, followed by 10 mins of magnetic mixing to form a smoothie. Then, a weighted amount of RTV-528 is also taken in a separate beaker to prepare its smoothie with a small amount of ethyl acetate. Ethyl

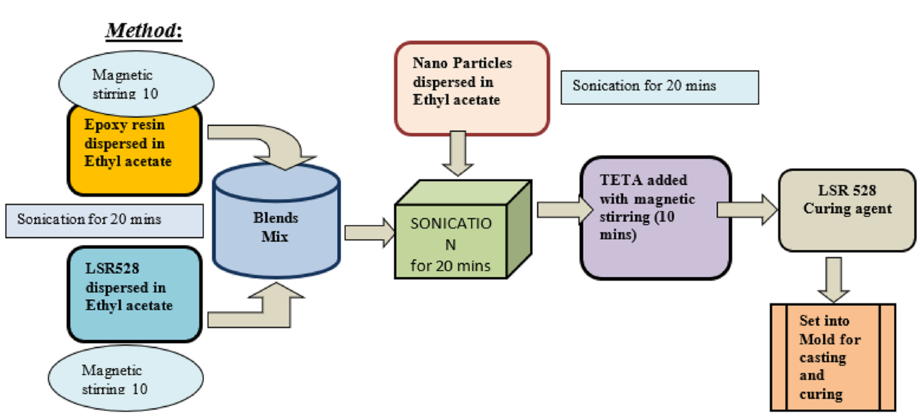

Fig. 2 Block diagram for preparation of blends

Fig. 3 Formulation of blends

Fig. 4 Dielectric between plates of a capacitor

acetate is added to these base materials to decrease their viscosity so that they can blend up with each other to form a uniform mixture. Lowering the viscosity, better and easier the mixing of materials gives a uniform mixture. The liquids (epoxy resin + LSR) are recently mixed and set for ultra-sonication for 20 mins. There is the blend of two materials with uniform mixing at the particle level.

The next step is the dispersion of nanoparticles for the reinforcement of blends. A very carefully weighted amount of nanoparticles is added to a small amount of ethyl acetate to prepare a dispersion. These dispersed nanoparticles are then added to the base matrix mixture and set for ultrasonication for 20 mins. The prepared mixture is ready to be cast in molds. Figure 3 describes the formulation blends steps.

Methods of analysis

Characterization of specimens

The prepared dielectric disks are passed through various analysis methods for their characterization, capacitance measurement, dielectric strength measurement, leakage current measurement, breakdown current measurements, etc. Depending upon these characteristics, it will be determined how good our prepared dielectric material is.

Measurement of capacitance

The prepared dielectric disks are 5 cm in diameter. To form a capacitor, two 6-cm-diameter metal discs are placed on both sides of the dielectric disk to form a capacitor as

Fig. 5 Capacitance measurement

shown in Fig. 4. The mode is set to capacitance measurement, and the scale is adjusted at nF. The measurement process is shown in Fig. 5.

Resistivity is the intrinsic property of a dielectric material. It is the measure of opposition offered by a material to the flow of electric charges in a particular dimension [24]. Resistivity can be measured easily with digital multi-meter (DMM). The unit is set at giga-ohm, and the resistivity of all sample dielectric disks is measured.

Dielectric constant

The dielectric constant is a factor that determines the capacitance of a dielectric material. It is a ratio of two capacitances: capacitance of material and capacitance of free space, given by:

$$K = \frac{C_{\text{material}}}{C_0} \tag{1}$$

$$C = KC_0 \tag{2}$$

$$C = \frac{KA\varepsilon_0}{d} \tag{3}$$

$$K = \frac{Cd}{A\epsilon_0} \tag{4}$$

Here the value of $\epsilon_0 = 8.854 \times 10^{-12}\,\mathrm{CV^{-1}\,m^{-1}}$. The dielectric constant values are calculated by putting the values $A = 19.63\ \mathrm{cm}^2$ and $d = 2$ mm in (4). The energy stored in a capacitor depends on the dielectric constant—greater the stored energy for greater values of dielectric constant K.

Our prepared dielectric disks are tested at a high-voltage laboratory to determine their maximum dielectric strength. The apparatus is installed in the laboratory setup as shown in Fig. 6. A 220 V source supply is provided to the control unit, where two sections: low voltage measuring input and regulated voltage. Then, a 100 KVA transformer is connected in series. A current limiting resistor is connected in series with the transformer, and then, a diode rectifier is connected. The sample to be tested is clamped after the rectifier, another end is grounded, and a high-voltage reading comes to the display at the HV measuring unit.

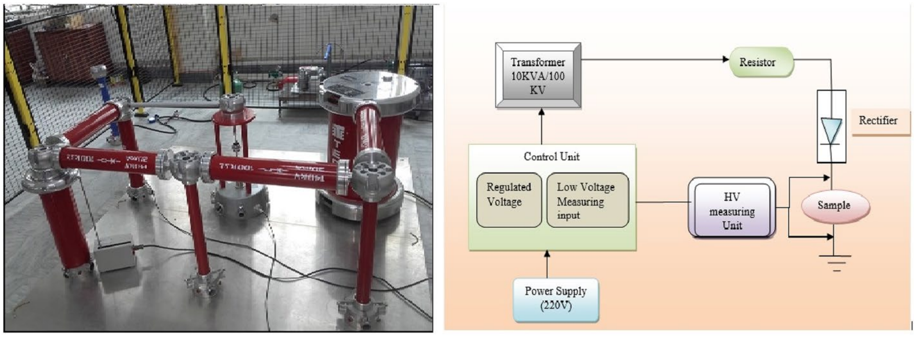

Fig. 6 Dielectric strength measurement

Fig. 7 Testing thermal conductivity

Thermal conductivity

Thermal conductivity measures how much heat energy the sample conducts in case of capacitor heat-up. The greater the thermal conductivity, the higher the heat dissipation rate of the material. Good thermal conductivity prevents the equipment from being damaged by dissipating the generated heat in the surroundings. The sample is placed above heated plates, as shown in Fig. 7. The temperature is raised to 200 °C, and conduction is measured at the other end. The assembly for heat measurement is shown below:

The dielectric disks are exposed to fire flame for 60 s approx. A slight black carbon layer was deposited over the spot where the flame touched the sample.

Results and discussion

Different behavior was observed for three different types of reinforced nanoparticles upon measuring the capacitance. The maximum capacitance values were found for TiO_2 nanocomposites. At a 5% wt ratio of TiO_2, the maximum value was achieved, which is 1372.4 PF, while minimum values were recorded for SiO_2 nanoparticles.

Electrical parameters of materials are compared in Fig. 8. A comparison of capacitance values is made based on the graphs shown in Fig. 8a. It is observed that the capacitance value usually increases with the increase in nano-filler ratios. This trend was not found in the case of SiO_2, where capacitance values decreased after a 3% wt ratio. At the same time, the highest capacitance values can be seen for 5% TiO_2 and 5%TiO_2@SiO_2.

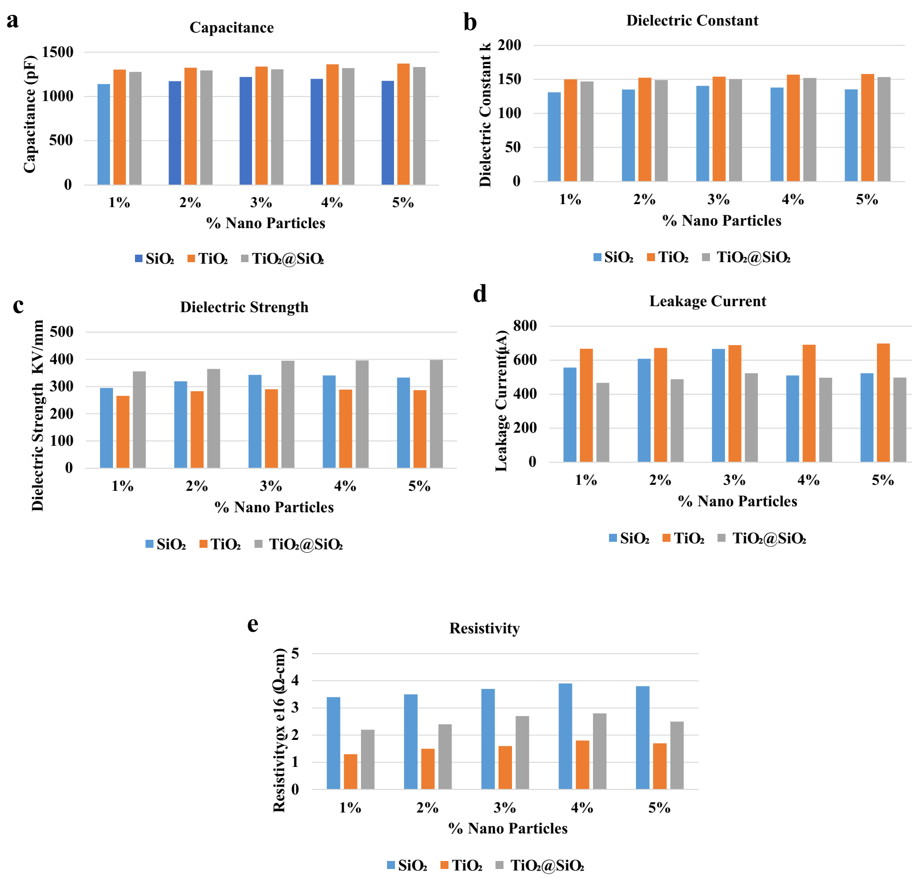

Fig. 8 **a** Capacitance comparison of different materials, **b** dielectric constant comparison of different materials, **c** dielectric strength comparison of different materials, **d** leakage current comparison of different materials, **e** resistivity comparison of different materials

The dielectric constant values are calculated using Eq. (4) described above. Dielectric constant K is a direct measure to determine the quality of a dielectric material. Higher K values represent the greater energy storage capacity of the dielectric medium. The maximum value achieved by our research is $K = 158$ for a 5% wt the ratio of TiO_2. It was also observed that $TiO_2@SiO_2$ samples also possess good values of K as compared to SiO_2 dielectric samples. A comparison made for K values is given in Fig. 8b. The maximum value for $TiO_2@SiO_2$ samples is $K = 153.4$. K values for three types of particles are represented below. It is found that SiO_2 has more insulative properties and less polarization ability as compared to TiO_2 nanoparticles. Because of less polarization ability, SiO_2 has less permittivity, which leads to less dielectric constant values. At the same time, TiO_2 nanoparticles possess more polarization ability and thus higher K values.

The recorded values of dielectric strength, dielectric constant, leakage currents, and resistivity of three types of particles are given in Table 6.

When designing the dielectric for a high-voltage capacitor, you must take into account the higher voltage by using a material with high dielectric constant and dielectric strength values. high-voltage capacitor dielectric, deal with high voltage, so such as a material with high values of both dielectric constant and dielectric strength. It is observed that TiO_2 particle samples had more dielectric constant than the two other

Table 6 Electrical properties of different materials at different concentrations

Dielectric strength (KV/mm)				Dielectric constant (K)			Capacitance (pF)		
Concentration	SiO_2	TiO_2	$TiO_2@SiO_2$	SiO_2	TiO_2	$TiO_2@SiO_2$	SiO_2	TiO_2	$TiO_2@SiO_2$
1%	295	266	356	131	150	147	1138.416	1303.53	1277.46
2%	319.5	283	365	135	152.5	149	1172.647	1324.657	1294.255
3%	343	290	395	140.5	154	150.5	1220.422	1337.686	1307.284
4%	341	289	396.5	138	157	152	1198.706	1363.745	1320.314
5%	333	287	**398**	135.4	**158**	153.4	1176.122	**1372.431**	1332.475

Leakage current (μA)				Resistivity ϱx e16 (Ω cm)		
Concentration	SiO_2	TiO_2	$TiO_2@SiO_2$	SiO_2	TiO_2	$TiO_2@SiO_2$
1%	557	667	467	3.4	1.3	2.2
2%	608	672	488	3.5	1.5	2.4
3%	666	688.5	523	3.7	1.6	2.7
4%	510	691	497	3.9	1.8	2.8
5%	523	698	498	3.8	1.7	2.5

It shows the high amount of capacitance

types of samples. But at the same time, the dielectric strength values of TiO_2 samples were less than $TiO_2@SiO_2$. Therefore, the best option available was $TiO_2@SiO_2$ nanofiller samples, with a high K value of 153.4 and a high dielectric strength of 398 kV/mm. So, it was found that using $TiO_2@SiO_2$ nanocomposites gave a high value of dielectric constant K more than SiO_2 and dielectric strength more than TiO_2. Therefore, using 5 wt% of $TiO_2@SiO_2$ gave the best possible choice for HV capacitor dielectrics. These samples also had the least leakage current and comparatively less resistivity.

Conclusions

The addition of a minimal amount of $TiO_2@SiO_2$ nanoparticles to epoxy resin improved the dielectric characteristics of the nanocomposite compared with pure epoxy resin. There was an increase in dielectric constant, resistivity, dielectric strength, and leakage current densities. It was also discovered that a low ratio of nanoparticles produces an important influence on the dielectric characteristics of the base material due to the high surface energy of nanoparticles. The highest capacitance value was observed for TiO_2 nanoparticle sample dielectrics. While looking at the dielectric strength of TiO_2, that was the least as compared to the other two types of samples. Leakage current and resistivity values are also low for TiO_2.

Furthermore, due to the high surface-to-volume ratio and high surface energy of nanoparticles, filler concentration plays a crucial impact in defining the dielectric characteristics of nanocomposites. As a result, it is proposed that increasing the filler concentration in fractional phases is explored to discover the optimal loading concentration for the best dielectric characteristics.

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Nanocomposite samples were prepared and Tested in the "High Voltage Lab," Electrical Engineering Department UET, Taxila, Pakistan.

Author contributions
AK wrote the manuscript and provided data for all tables and figures, MH conducted the interviews, and MSS, MF, and AU conducted all statistical analyses. All authors have reviewed and approved the manuscript.

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Declarations

Competing interests
The authors declare that they have no competing interests.

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