Microwave-assisted Synthesis of TiO2-based Transformer Nanofluid: Investigation on the perspective of Electrical and Thermal Properties

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Research Article

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Microwave-assisted Synthesis of TiO_2-based Transformer Nanofluid: Investigation on the perspective of Electrical and Thermal Properties

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Abstract

The study on dispersing nanoparticles in transformer oil to elevate their dielectric properties has evolved significantly in the recent decade. Alongside the conventional dispersion techniques (mechanical stirring & ultrasonic homogenization) in practice, this work experiments with the microwave energy of 2.45 GHz frequency radiated onto the TiO_2 nanoparticle-based nanofluid (TNF). The electrical and thermal properties of TNF (TiO_2 nanoparticle & surfactant dispersed in transformer mineral oil) are experimentally investigated & presented in this article. Effective synthesis procedure to enhance the dielectric properties with good dispersivity has been recognized from the superlative combination of dispersion techniques. Its effectiveness in enhancing the electrical and thermal properties is investigated by verifying its dielectric breakdown strength, dielectric constant ($\boldsymbol{\varepsilon_r}$), dielectric dissipation factor (tan $\boldsymbol{\delta}$), and flash & fire point. Results justified that the TNF synthesized by combining the processes of stirring, Ultrasonic homogenization, and microwave irradiation in a rational sequence exhibited better electrical & unaltered thermal properties when compared with samples prepared through stirring and sonication. TNF prepared through microwave synthesis improved the AC breakdown voltage (BDV) by 16.92% more than TNF prepared without microwave synthesis. Hence this could be an effective route to prepare the TNF with improved electrical properties.

Keywords: Micowave-assisted, titania nanoparticle, nanofluid synthesis, mineral oil, dielectric properties.

1 Introduction

The concept of nanofluids has found a broad range of applications ever since its conception by Choi [1], while many experimental studies and investigations justified its effectiveness in performance improvement of the nanofluid systems [2–6]. A well-dispersed nanofluid is an essential prerequisite that decides its added functionalities, improvised properties, and enhanced performances. Still, fluid stability is a fundamental long-standing issue yet to be addressed to realize its potential for industrial-scale real-time applications. Fan yu [7] has made an effectively decent review of the recent progress, underlying mechanisms, characterization techniques, research challenges, and strategies in improving the dispersion stability of the thermal nanofluids.

To overcome this issue to some extent, several researchers used conventional dispersion techniques like mechanical stirring, ultrasonic treatment, and chemical methods of surface modification either separately or in conjunction. Besides the application type and user's awareness of combining the techniques, no standards are defined yet to realize the optimal combination to attain the superlative dielectric BDV values with an elated dispersion strength. Yu-zhen Lv investigated the effect of dispersion techniques on the stability of functionalized TiO_2 nanoparticles in transformer mineral oil and proposed the combination of stirring and bath sonication found to have an efficient dispersion with the maximum value of dielectric breakdown strength. Despite the higher sonication energy, the ultrasonic probe immersed in the fluid is reported to affect the adsorption balance of the surfactant oleic acid [8]. Several other types of research were found to have an effective dispersion of functionalized nanoparticles through ultrasonic probe sonication combined with stirring [9–11]. The synthesis procedure of Titania-based Transformer Nanofluid (TNF) through mechanical stirring and probe sonication was experimented with the optimized levels of TiO_2 and surfactant Cetyl Trimethyl Ammonium Bromide (CTAB) dispersed in transformer mineral oil. And the quantity of titania nanoparticle and surfactant loading in mineral oil was optimized systematically by verifying its superior values of corona inception voltage and the AC & DC dielectric breakdown voltage [12].

Researches reveal that the microwave route of nanoparticle synthesis ensures a better dispersion in comparison to the conventional methods such as sol-gel, chemical vapour deposition, hydrothermal, solvothermal, etc. Accelerated reaction rates and altered synthesis yields such as particle size reduction, particle distribution, and enhanced purity & physiochemical properties are observed in microwave-assisted synthesis. Such improved parameters of microwave heating compared to the conventional heating processes at similar temperatures have raised the speculations on the existence of non-thermal effects in microwave synthesis chemistry [13].

Apart from reducing the processing time from hours to minutes with rapid and uniform heating, this microwave irradiation is proven to be a simple and effective method to synthesize nanostructures, with the commonly used operating frequency of 2.45 GHz [13–16]. Such attractive features of the microwave-based method insisted the authors to experiment the microwave irradiation on to the nanofluid synthesis procedure to investigate and verify the enhancement in the dielectric and thermal behaviour of the TNF with better dispersion strength. Hence the objectives are framed as follows. 1. To recognize an effective synthesis procedure with the application of microwaves to prepare the TNF, with the superlative combination of dispersion techniques in a rational sequence. 2. To investigate the electrical and thermal properties of TNF prepared through microwave synthesis procedure.

The electrical properties of an insulating liquid could be addressed by its dielectric behaviour, hence the dielectric breakdown strength, dielectric constant, and dielectric dissipation factor of the TNFs are investigated in this work. Flashpoint and fire-point are the physical property of an insulating liquid that determines its thermal volatility hence it is verified for TNFs in this work.

2 Experimental Procedure

2.1 Experimental Setup

2.1.1 TNF synthesis & Dispersivity Evaluation

The nanofluid samples used in this study for experimentation purposes are prepared by the two-step method of nanofluid synthesis. Initially, the TiO_2 nanoparticles are dried in a heating chamber for about 3 hours to get rid of the moisture from it. Before it is dispersed in mineral oil, the surfactant CTAB is mixed with mineral oil by mechanically stirring it in a magnetic stirrer for 30 minutes. Later the nanoparticles were added to it through mechanical stirring for 30 minutes and subsequently diffused using the ultrasonic homogenizer probe (750W, 20 KHz, Sonics Vibra-cell) for about 2.5 hours. Later then, it is irradiated with the microwave energy of 2.45 GHz frequency in a commercial microwave oven.

The UV absorption spectrum and the zeta-potential values are verified for evaluating the dispersion strength of the prepared TNF samples. Spectroscopy studies are carried out using a UV-Visible Double beam spectrophotometer (Analytical Technologies) and the zeta-potential is measured using Nanoparticle Analyzer (Horiba Scientific SZ-100). Cyclohexane was used as the solvent in both the measurement techniques, for the reference cuvette of the spectrophotometer, and the non-aqueous zeta-potential glass cell.

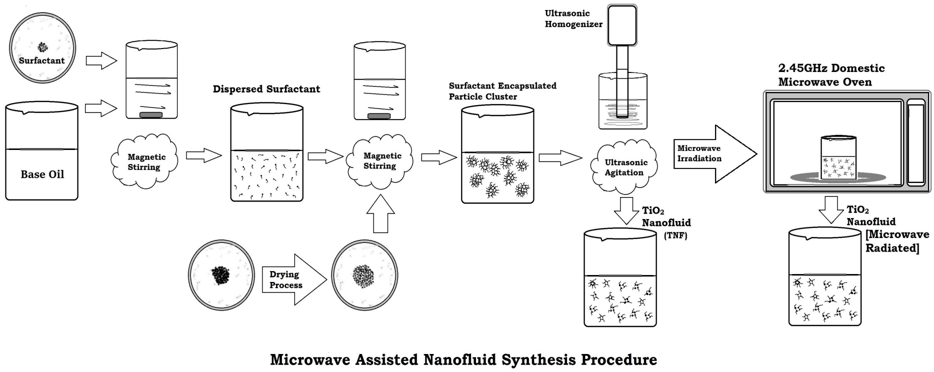

Fig. 1 Procedural flow hierarchy of microwave-assisted nanofluid synthesis

2.1.2 Dielectric & Thermal Studies

The dielectric breakdown strength, dielectric constant, and dielectric dissipation factor of the TNFs are investigated in this work to evaluate the electrical properties of TNF samples whereas the flash point and fire point are measured to assess their thermal volatility.

AC breakdown voltage test is performed using the Motorized Oil Breakdown Voltage Test System (LOBDV-100, Power Electronical, India), which has an AC high voltage (maximum up to 100KV) generating unit and an oil test cell as per the standards. Mushroom electrodes with 2.5mm spacing are used in the test cell. Dielectric constant and Tan δ of the oil samples are measured using the Automatic Dielectric constant, Tan delta & resistivity test set (ADTR-2K PLUS, Eltel Industries, India). The oil sample is filled in a three-terminal test cell (IEC-60247), and the test is performed under a pre-programmed test sequence. Flashpoint and fire point of the oil samples are conducted in the Pensky Martens Flash point apparatus with a temperature rising rate of 5 to 6 degrees Celsius per minute.

2.2 Material Used

For experimentation purposes, the commercially available TiO_2 nanoparticle (Anatase, spherical, the average particle size of 10-20 nm, 99.9% pure) was purchased from Nano Research lab (NRL) India, and an analytical grade cationic surfactant CTAB (extra pure) was procured from Sisco Research Laboratories, India. Commercial purpose transformer mineral oil was procured from Venlub Petro products Chennai, and the solvent Cyclohexane (for HPLC, 99.9%) was used with that for the UV spectroscopy studies & the zeta-potential measurement is procured from Nano Research lab (NRL) India.

2.3 Methodology

The TNF sample is prepared by dispersing the optimized quantity of Titania nanoparticles and surfactant CTAB in mineral oil that is used for insulation

Table 1 Naming convention of the sample types used

Name of sample	Sample type
Virgin MO	Virgin Mineral Oil
TNF without Surfactant	Virgin Mineral Oil + TiO_2 nanoparticles
TNF without Microwave	Virgin Mineral Oil + Surfactant CTAB + TiO_2 nanoparticles
TNF with Microwave	Microwave irradiated TNF

and cooling the transformers in high voltage applications. Different types of TNF samples used in this study and the naming conventions given for this article's write-up purpose are listed in Table 1 The pictorial representation of microwave-assisted TNF synthesis or Microwave synthesis is shown in Fig. 1.

The sample type 'TNF without Surfactant' is prepared by dispersing 0.00562 wt. % (0.05 grams/ Litre) of TiO_2 nanoparticles in mineral oil through magnetic stirring for 30 minutes and then sonicated for 2.5 hours with the help of an Ultrasonic probe homogenizer. For the case of 'TNF without Microwave', the surfactant CTAB with 1% of Titania weight was mixed with mineral oil through stirring for 30 minutes, before adding TiO_2 nanoparticle in it. Such obtained nanofluid is irradiated with microwave energy of 2.45 GHz frequency in a commercial microwave oven, to prepare the 'TNF with Microwave'.

A series of 'TNF with Microwave' samples were prepared with different irradiation times of 60, 90, 120, 180, and 240 seconds keeping in mind that the temperature of the sample does not exceed the transformer's top-oil temperature of 65º C. Moreover the rise in temperatures will affect the alignment of electric dipoles and reduce the orientation polarization of the sample and hence the dielectric permittivity. For all 'TNF with Microwave' samples with different time duration, the microwaves are applied intermittently onto the sample with 60 seconds ON time and 15 seconds OFF time to avoid rapid heating of the sample.

3 Result Discussion

3.1 Dispersivity Evaluation

In this work, the dispersion strength of nanofluid is evaluated by measuring the zeta-potential and analyzing the UV-visible absorption spectrum of TNF samples. Zeta-potential measurement is a widely used and significantly effective technique to assess the dispersion strength of nanofluid quantitatively whereas, UV-visible spectral analysis is a simple and reliable technique to analyze the nanofluid dispersion. The optical absorption intensity of the sample when diluted appropriately with a suitable solvent is linear to the nanoparticle concentration in the fluid, and the absorption peak decreases correspondingly as the fluid gets destabilized. Zeta-potential is an electric potential generated in the interfacial layer (shear plane) formed between the stern layer around the particle surface and the diffuse layer in bulk solution due to electro-kinetic

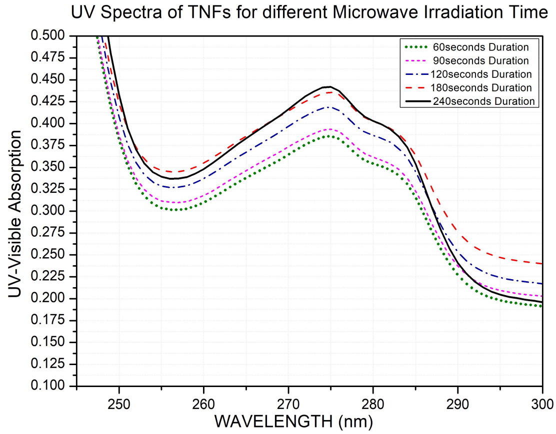

Fig. 2 UV-Visible Absorption spectra of TNFs for different Microwave irradiation time

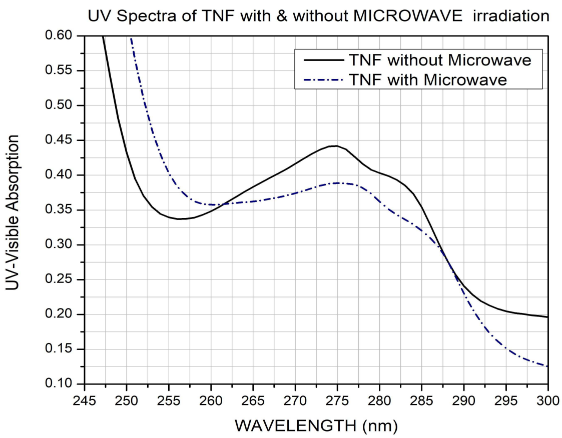

Fig. 3 UV-Visible Absorption spectra of TNFs with & without Microwaves irradiation

phenomena. Direct assessment of colloidal dispersion strength can be made from the zeta-potential magnitude in millivolt (mV), which should be $> +30$ mV or < -30 mV for a nanofluid to be reasonably stable [12] and [17].

As mentioned in the previous section, the dispersion studies are conducted with the help of UV-visible absorption spectroscopy which is aided by the zeta-potential measurement. The UV-visible spectra of the effect of microwave irradiance duration on 'TNF with Microwave' samples are shown in Fig. 2. It is seen that all the 'TNF with Microwave' samples exhibit its characteristic spectra with a typical absorption peak at the wavelength of 275nm. It is also observed that the higher the irradiation time, the higher the intensity of the absorbance peak, which could be a measure to observe the dispersion process. A substantial increase in the UV absorbance observed as the irradiance time changes from 60 seconds to 240 seconds, evidences a better dispersion of nanoparticles in the colloid [6], [18] and [19]. So, the longer duration of microwave irradiation leads to an enhanced homogenized suspension is inferred from the results, and hence a better homogeneity is achieved with 240 seconds of Microwave irradiance.

The absorption spectrum and its peak of the 240sec 'TNF with Microwave' sample and the 'TNF without microwave' sample are given in Fig. 3. The significant difference in the absorbance peaks infers that the particles are dispersed well in TNF irradiated with microwaves. The absorbance spectra of these two samples give a better realization of the impact of microwaves in the dispersion process and its potential as an effective dispersion mechanism for nanofluids. The zeta-potential values obtained for the 'TNF without Microwave' & the 'TNF with Microwave' samples are 41mV and 50mV respectively. The latter one is found to have a better dispersion strength and hence the 'TNF with Microwave' sample has good dispersivity. So the UV absorption spectrum & Zeta-potential values of the two samples ascertain the difference in the dispersivity and establishes a conclusiveness on the effect of the microwaves in the dispersion process.

3.2 Microwave Polarization Phenomena

The common electro-dynamic properties of matter like the dielectric permittivity (ϵ), magnetic permeability (μ), and conductivity (σ) have a strong interaction with microwaves. Although its interaction with matter is overall represented well by Maxwell's equations, it does have a microscopic level of interaction with its constituent atoms, and free electrons & magnetic dipoles if any are present.

Electric permittivity holds the information on the polarization of a dielectric material that is correlated with the number of electric dipoles and the molecular polarizability of the medium. Microwave interaction with dielectric medium induces the polarization phenomena that physically originate from the local reorientation of the free charges and the interconnected charges (dipole moment). Electric dipoles greatly absorb microwaves as they execute damped oscillations as a function of microwave frequency. The storage of microwave energy inside the dielectric medium, and the thermal conversion with respect to the microwave frequency are the two major facts of the polarization phenomena that the microwaves induce when interacting with the dielectric medium. So the polarization takes a complex form with the real part being in phase with oscillations, while the imaginary part has the phase lag from that which causes the thermal conversion of microwave energy. Such phenomena quantify the microwave interaction with a dielectric medium as a complex physical quantity of dielectric permittivity (ε) with the real part ($\varepsilon^{'}$) expressing the stored microwave energy and the imaginary part (ε") in proportionate to the thermal conversion [20] and [21] as shown in Eq.1.

$$\varepsilon = \varepsilon^{'} - j\varepsilon^{"} \tag{1}$$

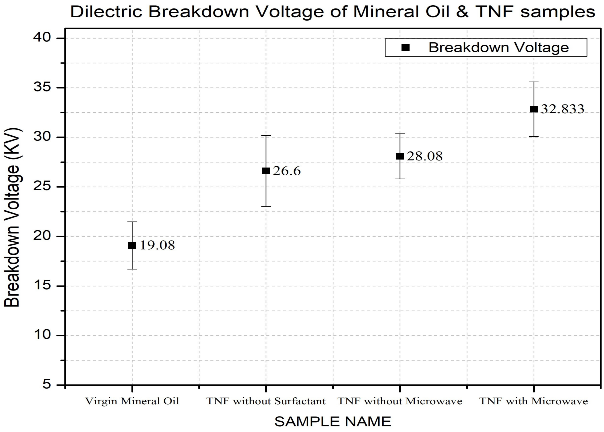

Fig. 4 Dielectric Breakdown voltage of different sample types

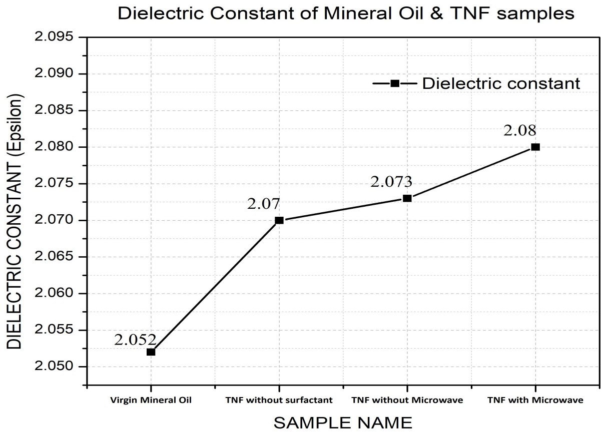

Fig. 5 Dielectric constant of different sample types

3.3 Dielectric & Thermal Studies

AC Breakdown voltage magnitudes of the TNF samples are shown in the Fig. 4 and the Fig. 5 shows the graph plotted for the measured values of the dielectric constant. It depicts that the TNF prepared through microwave synthesis procedure exhibits the maximum values of AC-BDV and dielectric constant.

Mean BDV increased with the addition of TiO_2 nanoparticles and further improved with the addition of the cationic surfactant CTAB. The higher values of BDV with the addition of Titania nanoparticle is attributed to the suppression of corona propagation in the fluid. The electronegative property of Titania nanoparticles can attract the electrons propagated with relatively low velocity, under AC voltages. Also, the charge transport process observed in the semi-conductive nanoparticle aids the BDV enhancement by trapping

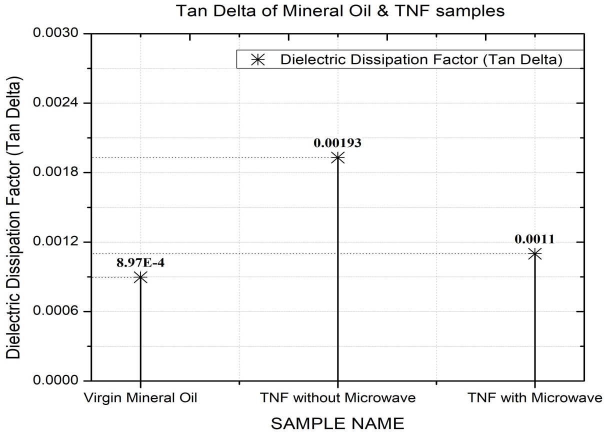

Fig. 6 Tan δ value of different sample types

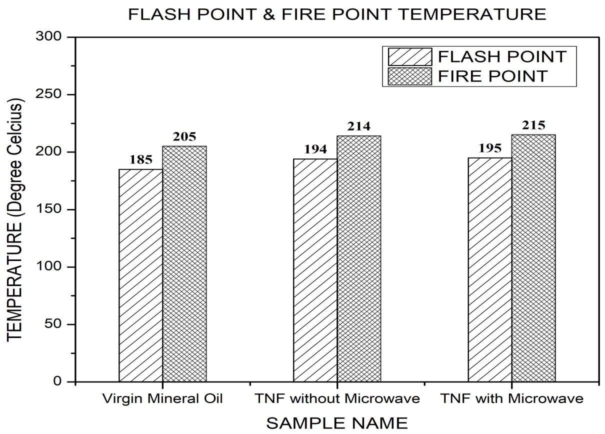

Fig. 7 Flashpoint and Firepoint temperature of different sample types

and de-trapping the fast-moving electrons [12] [22–24]. The BDV improvement for the surfactant addition is mainly contributed by the interfacial layer that the CTAB molecules form with the oil-particle layer [25]. The slight increment observed in the dielectric constant of 'TNF without microwave' is caused by polarization due to surfactant encapsulation. Farade et al. also proposed the relative permittivity model with surfactant aiding the orientation polarization of the nanoparticle [26].

The permittivity of the 'TNF without surfactant' sample is higher than the virgin mineral oil due to the presence of Titania nanoparticles in it. A nanoparticle dispersed in the dielectric liquid will acquire the charge through several mechanisms and the net charge creates an interfacial polarization in the particle fluid interface. So the increase in polarization of the bulk fluid increases its dielectric constant [27–29]. Interaction of microwaves with the dielectric medium was briefed in the earlier section. TNF when irradiated with microwaves tends to attain polarization due to the orientation of its dipole moments and free electrons [20]. This results in the dielectric constant improvement could further impact the enhancement in BDV magnitude [30].

Fig. 6 shows the measured values of the dielectric dissipation factor of different TNFs for the power frequency (50Hz) at 90^o, which is comparatively less for 'TNF with microwave' with that of 'TNF without microwave'.

The increment in Tan delta value for the surfactant addition in TNF is attributed to the interfacial layer that it forms normal to the oil molecules. The torque created from the applied electric field required to orient the dipole moments of oil molecules present in this 'stiff to polarize' layer is a bit larger. So, higher potential energy is required to orient these dipole moments of the interfacial layer. More energy lost in the medium causes the increment in tan delta values [31]. Whereas in the case of microwave irradiated in TNF orients such dipole moments, and hence the corresponding reduction in energy loss could slightly decrement the loss factor value of the medium.

Fig. 7 shows the flashpoint and fire point temperature values of the oil samples. It shows the higher values for the TNFs when compared with virgin mineral oil, and remains unchanged for the microwave irradiated sample. Enhancement in thermal characteristics for the addition of TiO_2 nanoparticles is attributed to the heat transport mechanism through phonons, created with in the nanostructures due to the Brownian motion of oil molecules. Flashpoint and fire point remains unchanged for the TNF with and without microwave irradiation, as the Brownian motion of oil molecules tends to disrupt the polarization effect induced by microwaves [22] and [32].

4 Conclusion

The microwave route of the TNF synthesis procedure has been experimented in this work and the results are articulated in this paper. Five different samples were prepared by irradiating the microwave on the TNF for different time durations. Dispersivity of prepared TNF samples was verified systematically from its UV-visible spectra, and 240 seconds was found to be an optimal irradiation time to attain a better dispersion. TNF with microwaves irradiated for 240sec exhibited better dispersion comparatively with that of 'TNF without microwave' sample was ascertained by verifying their UV absorption spectrum & Zeta-potential values.

Dielectric and thermal properties of 240 seconds irradiated 'TNF with microwave' sample were investigated, and found to have higher BDV & dielectric constant values compared with that of 'TNF without microwave'. Enhanced AC breakdown voltage of 16.92% is attributed to its improved dielectric permittivity, explained in terms of polarization phenomena induced by the microwaves interacting with the dielectric medium. Tan delta of TNF decreases with the application of microwaves as it tends to orient the dipole molecules held in the interface layer formed by CTAB addition. Flashpoint and fire point temperature values of the TNF didn't show any increment with the irradiation of microwave onto it.

Hence the proposed microwave method is proved to be efficient, and could effectively contribute to framing a standard two-step synthesis procedure for the TNF synthesis with good dispersivity and incremented dielectric behaviour.

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