A Contribution to Environmentally-Compatible Energy Conversion: Characterization of Nano Structured Natural Ester Oil Decorated with Carbon Quantum Dots

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ONLINE FIRST

Journal of Electrical Engineering & Technology

ISSN 1975-0102

J. Electr. Eng. Technol.
DOI 10.1007/s42835-020-00531-5

Journal of Electrical Engineering & Technology
https://doi.org/10.1007/s42835-020-00531-5

ORIGINAL ARTICLE

A Contribution to Environmentally-Compatible Energy Conversion: Characterization of Nano Structured Natural Ester Oil Decorated with Carbon Quantum Dots

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Received: 27 March 2020 / Revised: 10 August 2020 / Accepted: 23 August 2020

Abstract

Performance of natural ester oils have not been investigated deeply yet, and concerns exist about their behavior under overstresses and partial discharges. The present work investigates the suitability of novel carbon quantum dots (CQD) decorated nanostructured natural ester based corn oil for transformer insulation applications, particularly focusing on collecting database for overstressed conditions due to lightning impulse (LI) and partial discharge (PD). CQD nanofluids prepared with SiO_2 and TiO_2 nanofillers are tested for its insulation properties such as PD, LI and AC power frequency breakdown voltage (BDV). The tests are carried out at different concentration of nanofillers with the mass fractions of 0.01, 0.05 and 0.1. Statistical analysis of test data is performed using two parameter Weibull distribution and the results are compared with and without CQD treatment. Results are also compared with conventional mineral oil. Addition of CQD treated insulative SiO_2 nanofillers show good insulation performance than semiconductive TiO_2 nanofillers. The overall results clearly indicate the positive influence of CQD treatment on SiO_2/TiO_2 nanoparticles at lower %wt (0.01%wt) concentration of nanofillers and clearly show the suitability for MV transformer insulation applications.

Keywords Carbon quantum dots · Nanofluid · Natural esters · Lightning impulse · Breakdown voltage · Partial discharge

1 Introduction

In recent times, natural esters are frequently preferred for distribution transformers, motivated by their environmental friendly characteristics [1, 2]. Beroual et al. [3] have shown that breakdown strength of natural esters before aging is close to mineral oils, whereas after aging natural esters keep the breakdown strength even better than mineral oils. Thien et al. [4] studied the positive and negative polarity lightning breakdown voltages of natural esters such as palm oil and coconut oil. Viet-Hung Dang et al. [5] have reported that since the relative permittivity of natural esters being closer to pressboard than mineral oil, the cress stress of natural ester impregnated pressboard is lower than mineral oil, which is positive especially for power transformers.

However, the resistance offered by natural esters to PD is considerably lower than mineral oils [1, 2]. Recent research works on nanofluids, particularly with natural esters, show positive impact of nano particles in its dielectric characteristics [6–8]. Agglomeration of nanoparticles and stability of the nanofluid is a major problem faced by researchers. Different techniques of surface treatment of nanofillers are being carried out by many researchers in order to improve the stability of the oil [9, 10].

It has been already reported that, [11, 12], mineral oil added with CQD treated silica nanofiller shows improved dielectric strength and enhanced PD properties. This is mainly because of the improved dispersion of nanoparticles in the base oil and improved stability of the medium. However, considering the environmental issues of mineral oil, the common focus of the electrical utilities is the usage

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of natural ester oils for high voltage applications. Hence it is important to accumulate the database of dielectric properties (such as BDV, PD and LI strength) of biodegradable natural ester oils mixed with CQD treated nanofillers for effective insulation design of transformers.

This work mainly focuses on the dielectric properties such as power frequency breakdown strength, PD and LI breakdown strength of corn oil modified with CQD treated silica and titania nanofillers at different concentrations. PD experiments are focused towards understanding the corona discharge and surface discharge characteristics through appropriate electrode arrangements. LI characteristics are evaluated at needle-sphere and needle-plane geometry. All the results reported are measured with reasonable size of samples to get 95% confidence level.

2 Experimental Studies

2.1 Preparation of CQD Covered Nanofluid

Carbon quantum dots were prepared by carbonization process using activated carbon black mixed with nitric and sulphuric acid at room temperature [11]. In order to obtain a homogeneous mixture of CQD and SiO_2/TiO_2 nanoparticles in a weight proportion of 1:1, appropriate amount of both components were weighed and mixed thoroughly for about 3 h at room temperature. Then the prepared CQD-SiO_2 and CQD-TiO_2 nanomaterials were mixed with corn oil and mechanically stirred for 30 min. In order to improve the homogeneity of suspension, test specimens were sonicated for another 30 min using ultrasonicator. Properties of SiO_2 and TiO_2 nanofillers are shown in Table 1.

The CQD nanofluids were prepared at different concentration of nanofillers with the mass fractions of 0.01, 0.05 and 0.1 as listed in Table 2. Further, in order to remove moisture content if any present in the oil, prepared sample was placed in a sealed steel container and kept in a temperature controlled oven for about 24 h at 75 °C.

Table 1 Properties of SiO_2 and TiO_2 nanofillers

Parameter	SiO_2	TiO_2
Relative permittivity	3.9	114
Density	2.5 g/mL	3.9 g/mL
Purity	99.5%	99.7%
Color	White	White
Type of materials	Insulator	Semiconductor
Size	10–30 nm	10–30 nm
Shape	Nanospheres	Nanospheres

Table 2 Summary of tested nanofluids

Base oil	Nano filler	% of filler added	Designation
Mineral oil	–	–	A1
Corn 0il	–	–	A2
	SiO_2	0.01%wt	B1
		0.05%wt	B2
		0.1%wt	B3
	CQD-SiO_2	0.01%wt	B11
		0.05%wt	B21
		0.1%wt	B31
	TiO_2	0.01%wt	C1
		0.05%wt	C2
		0.1%wt	C3
	CQD-TiO_2	0.01%wt	C11
		0.05%wt	C21
		0.1%wt	C31

2.2 Laboratory Test Procedures

Power frequency breakdown strength of oil samples were evaluated as per IEC 60156 with mushroom electrodes at gap distances such as 2.5 and 4.0 mm [8]. Mean value of BDV of each sample is computed by repeating the test 20 times. Slew rate of 2 kV/s is maintained throughout the experiments.

Lightning impulse BDV was carried out as per IEC 60897 (Fig. 1a). Both needle-sphere and needle-plane electrode geometry was used during the test maintaining gap distance

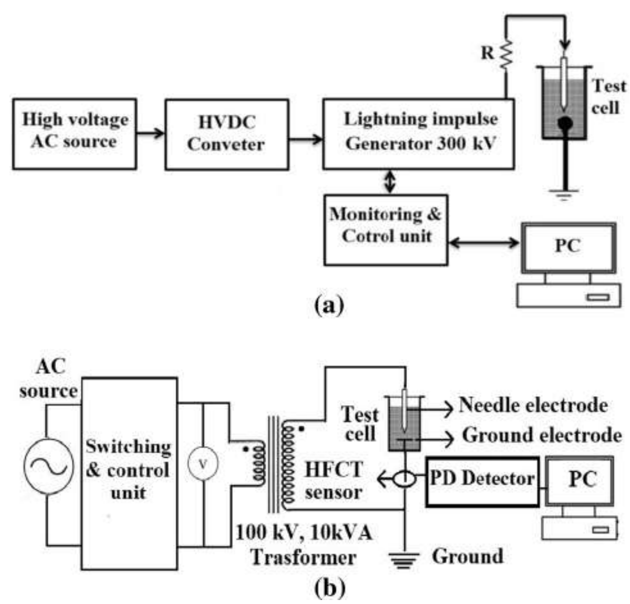

Fig. 1 Schematic of **a** LI, **b** PD test setup

of 5 mm and 10 mm. Impulse generator (300 kV rating) controlled by PC is able to supply standard LI waveform of time period 1.2/50 µs to the sample.

PD measurements were carried out at needle-plane and rod-plane electrode geometry in order to simulate corona and surface discharges respectively as shown in Fig. 1b [1]. Gap distance between the electrodes is maintained as 5 mm throughout the experiments. Needle electrode with a radius of curvature 1.5 µm is used and it is replaced for every 3 test specimen in order to maintain uniform test condition. High frequency current transformer of 50 MHz bandwidth was adopted to measure PD signals which is interfaced with a PD detector (sampling rate 100 MS/s) and PC for storing the data. Figure 2 shows the photograph of needle-plane, needle-sphere and rod-plane electrode test cell used in the experiments. Figure 3 shows the photograph of PD setup used in laboratory.

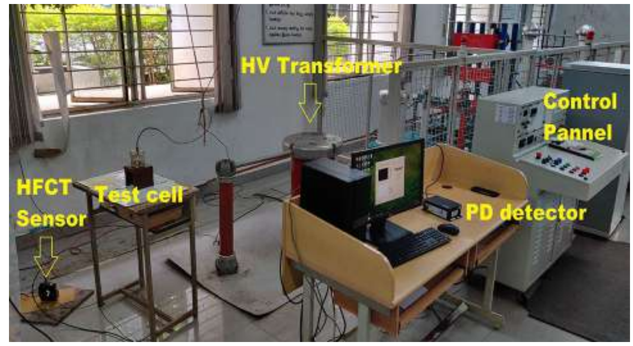

Fig. 3 Photograph of PD test setup

3 Test Results

3.1 AC BDV Tests

Figure 4a and b shows the Weibull probability distribution results of BDV values of tested corn oil nanofluids with and without CQD treatment at 2.5 mm and 4 mm gap distance of electrodes respectively. Two parameter Weibull analysis is carried out as follows,

$$F\left(V_{bdv}\right)_{=}\exp[-(V_{bdv}/\alpha)^{\beta}] \tag{1}$$

where the F (V_{bdv}) is the cumulative probability of breakdown voltage, V_{bdv} is the AC breakdown voltage, α and β are the scale and shape parameter respectively [13]. It is observed that irrespective of the concentration of nanofillers and electrode gap distance, CQD treated insulative SiO_2 filled corn oil nanofluid shows higher BDV values. For comparison purpose, BDV results of conventionally used

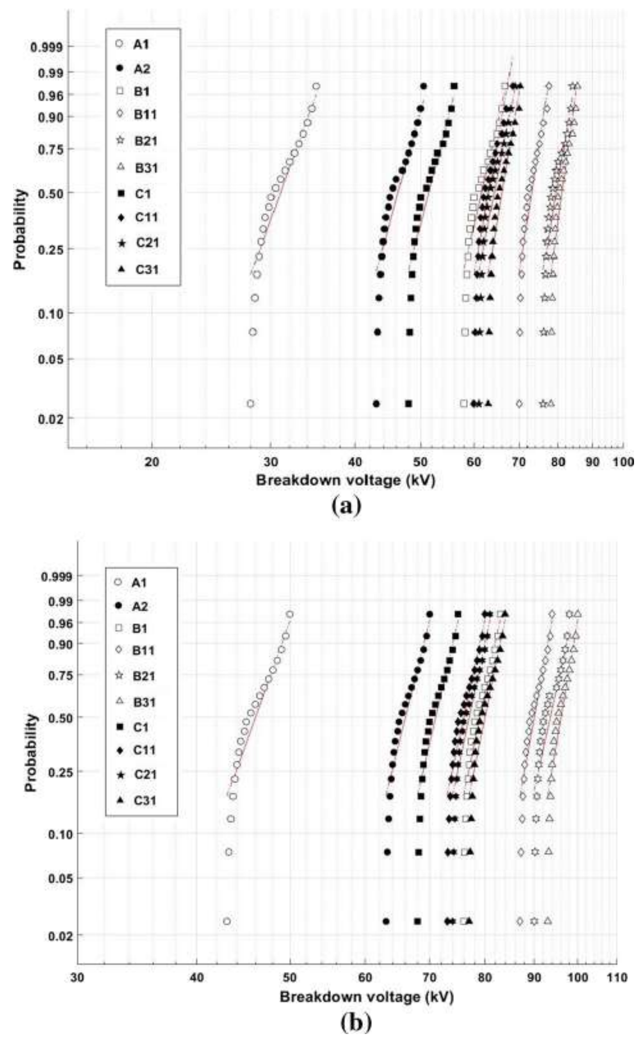

Fig. 4 Probability distribution of BDV values of tested nanofluids at **a** 2.5 mm **b** 4.0 mm gap distance

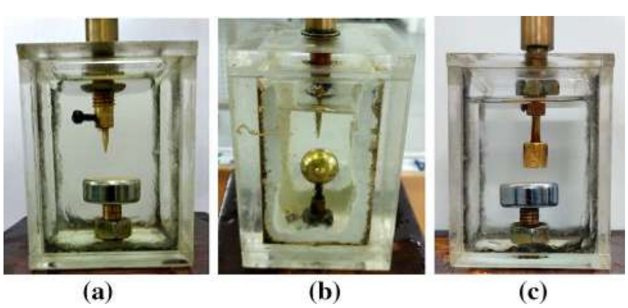

Fig. 2 Photograph of electrode geometry **a** needle-plane, **b** needle-sphere, **c** rod-plane

mineral oil are also shown. Breakdown strength of tested corn oil specimens is relatively higher than mineral oil.

Figure 5 shows the 50% probability BDV results of tested nanofluids at different electrode gap distance. It clearly shows the enhancement in BDV values of CQD treated nanofluids with respect to corresponding untreated nanofluids. It is also observed that there is a considerable reduction in $V_{50\%}$ values of semiconductive TiO_2 nanofluids compared with insulative SiO_2 nanofluids. However, treatment of TiO_2 nanofillers with CQD clearly shows significant improvement in BDV values than untreated samples. Irrespective of the type of filler, increase in CQD concentration above 0.05%wt does not show any significant improvement in BDV results. Table 3 shows the summary of shape parameter, standard deviation and coefficient of variance of BDV data from Weibull analysis, which clearly shows the advantages of CQD treated nanofluids with increase in β and reduction in standard deviation and s(%).

3.2 PD Tests

During PD measurements, threshold value of PD signal was kept above noise level of 0.2 mV. Figure 6 shows the PD inception voltage results of tested samples.

During PDIV measurements, supply voltage is slowly increased in steps of 0.5 kV until the first PD pulse is observed over a time interval of 10 min. It is understood that PD inception starts at much higher voltage for CQD treated samples than untreated one. In particular, insulative SiO_2 nanofillers treated with CQD shows higher PDIV values at lower %wt concentrations (B11) than CQD treated semiconductive TiO_2 nanofillers (C11).

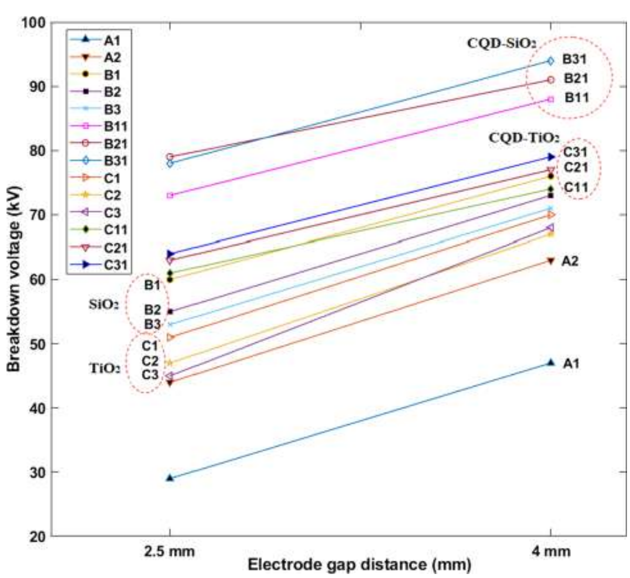

Fig. 5 $V_{50\%}$ results of tested nanofluids at different electrode gap distance

Table 3 Weibull parameter analysis of BDV data

Sample	Shape Parameter (β)	STD (kV)	s (%)
A1	14.31	2.26	7.33
A2	19.86	2.48	5.40
B1	22.65	2.88	4.69
B11	31.96	2.35	2.99
B21	31.19	2.65	3.35
B31	30.73	2.42	3.23
C1	20.49	2.67	5.22
C11	24.39	2.53	4.01
C21	26.20	2.51	3.93
C31	29.07	2.33	3.54

3.2.1 PD Test Results of Needle-plane Electrode

In order to understand the magnitude of PD above PDIV, test voltage was slowly increased above PDIV and phase resolved PD patterns and corresponding PD signals were captured at each voltage. Test was carried out upto 30 kV with needle-plane electrode. When the voltage magnitude is increased above 30 kV for the present electrode gap arrangement, strong and more intense PD pulses were observed which leads to breakdown with a further marginal increase in applied voltage. Figures 7 and 8 shows the typical PRPD patterns of tested samples at 25 kV (left) and 30 kV (right). In general, PD activity is stronger in the positive half cycle than negative one and dispersion of PD increases with rise in applied voltage.

In most of the oil samples, rise in applied voltage show a reduction in PD phase inception delay. When compared with mineral oil, it is noted that PD repetition rate of corn oil samples are slightly higher, which may be due to the hygroscopic nature of vegetable oils due to which PD propagates faster under AC stress [2]. Figure 9 shows the

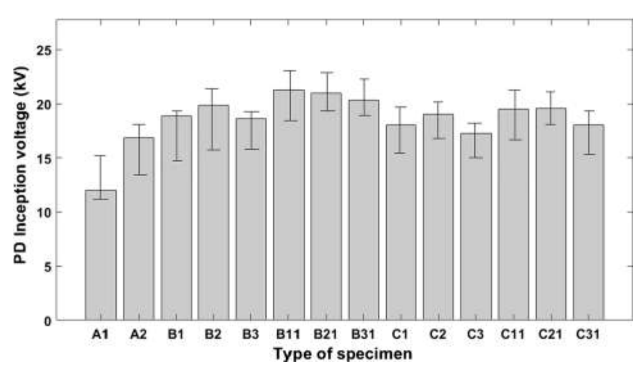

Fig. 6 PD inception voltage of tested oil samples

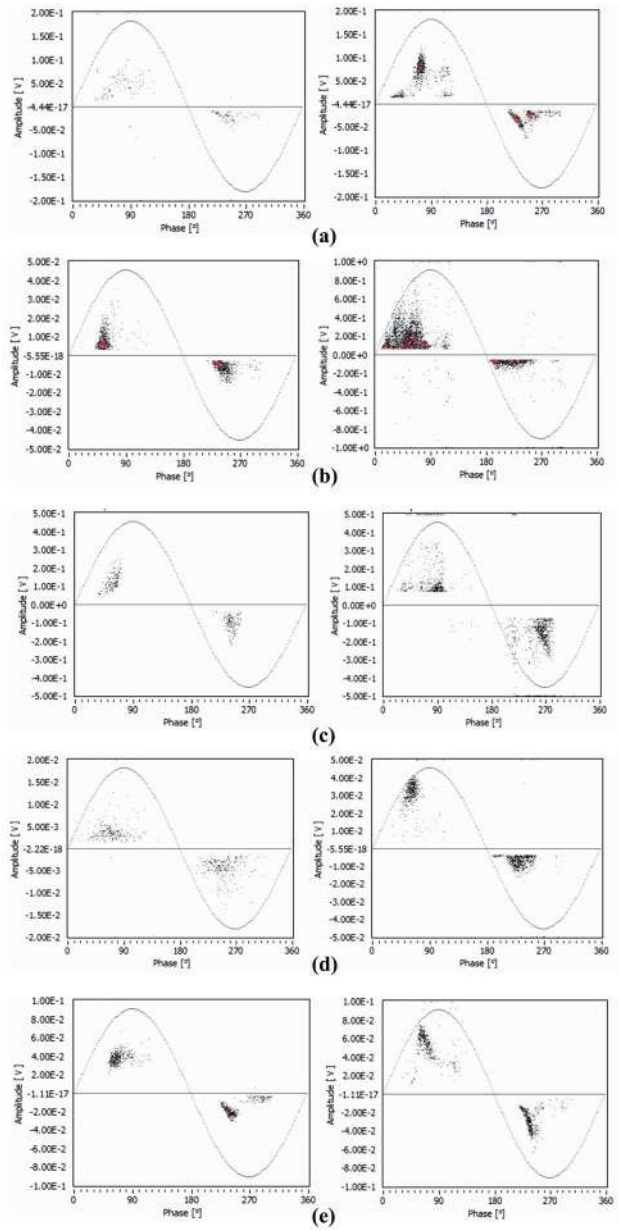

Fig. 7 PRPD pattern of SiO_2 samples at needle-plane geometry. Test voltage 25 kV (left) and 30 kV (right) **a** A1, **b** A2, **c** B1, **d** B11, **e** B31

variations in PD amplitude, PD repetition rate and beta value (shape parameter) of PRPD pattern of tested nanofluids at needle-plane electrode with respect to increase in test voltage. Increase in amplitude, repetition rate and decrease in beta value of PD pattern with voltage is clearly visible from the results. However, the CQD treated nano silica filled corn oil samples show slightly good performance than other samples.

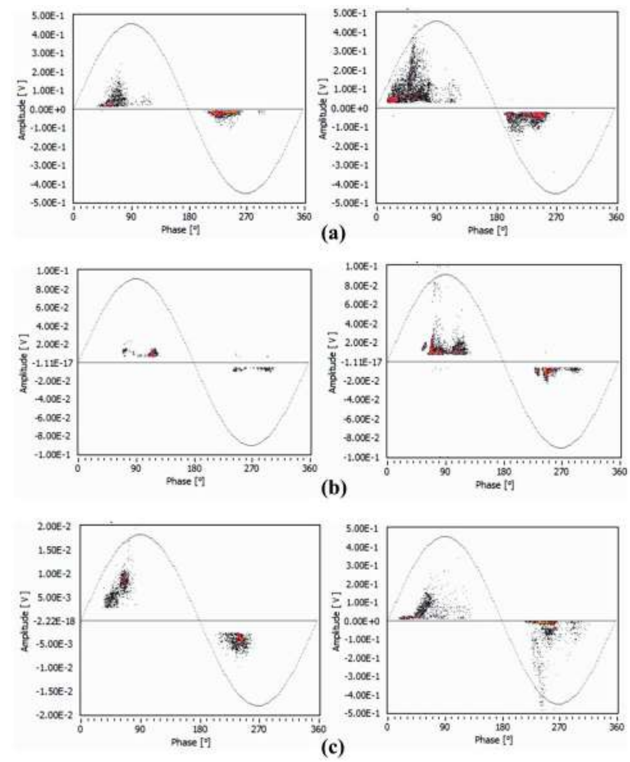

Fig. 8 PRPD pattern of TiO_2 samples at needle-plane geometry. Test voltage 25 kV (left) and 30 kV (right) **a** C1, **b** C11, **c** C31

3.2.2 PD Test Tesults of Rod-plane Electrode

Figures 10 and 11 shows the typical PRPD patterns of tested samples at 25 kV (left) and 30 kV (right) with rod-plane electrode geometry. In similar line with needle-plane geometry, there is a considerable reduction in PD magnitude and PD repetition rate noticed with respect to addition of CQD treated nanofillers irrespective of the type of filler, particularly dominant in the case of CQD treated insulative silica samples. This is most likely due to the strong bonding between the CQD treated nanoparticles and base oil.

3.3 LI Test

Lightning impulse tests were carried out at both needle-sphere and needle-plane electrode geometry. Figures 12 and 13 shows the 2-parameter Weibull distribution of LI BDV values at needle-sphere and needle-plane geometry for tested oil samples. In the case of positive LI test results, it is noted that all corn oil samples show higher BDV values than mineral oil. However, under negative LI polarity, addition of untreated SiO_2/TiO_2 nanofillers in the corn oil shows considerable reduction in BDV than pure sample. When the nanofillers are treated with CQD, then significant improvement in negative LI BDV is noticed in corn oil samples than

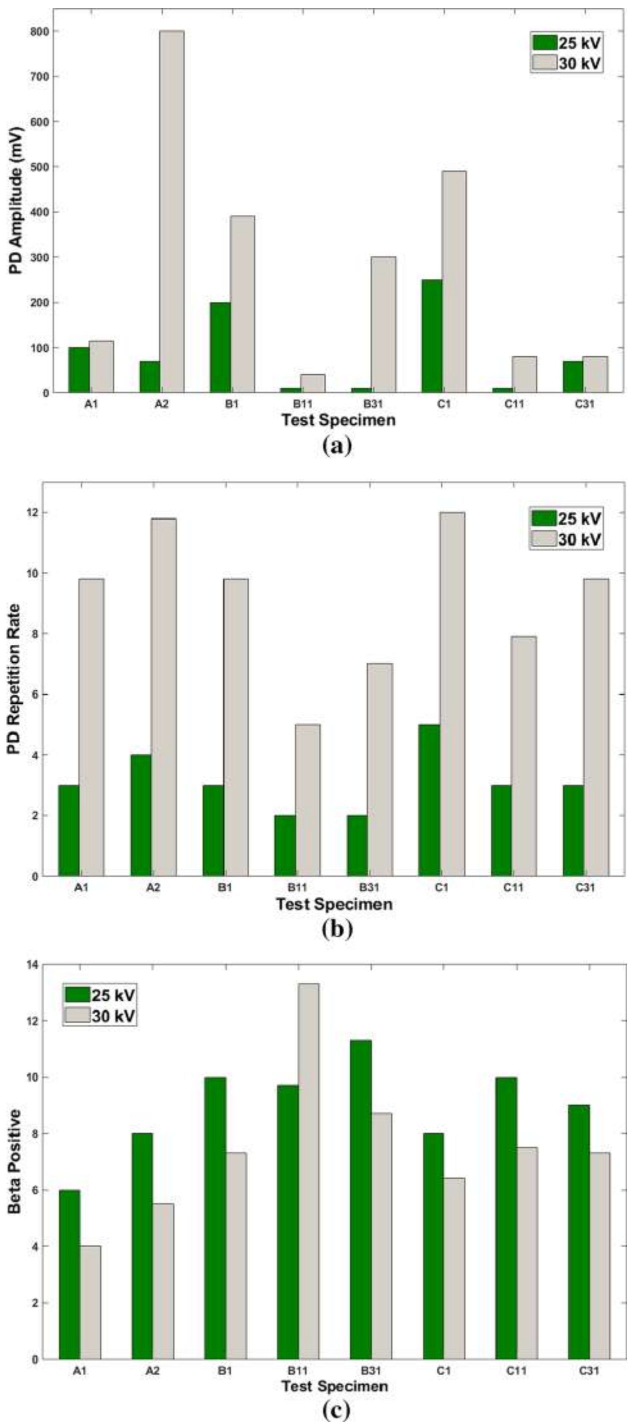

Fig. 9 Variation in **a** PD amplitude **b** PD repetition rate **c** Beta parameter of tested samples at needle-plane geometry with respect to test voltage

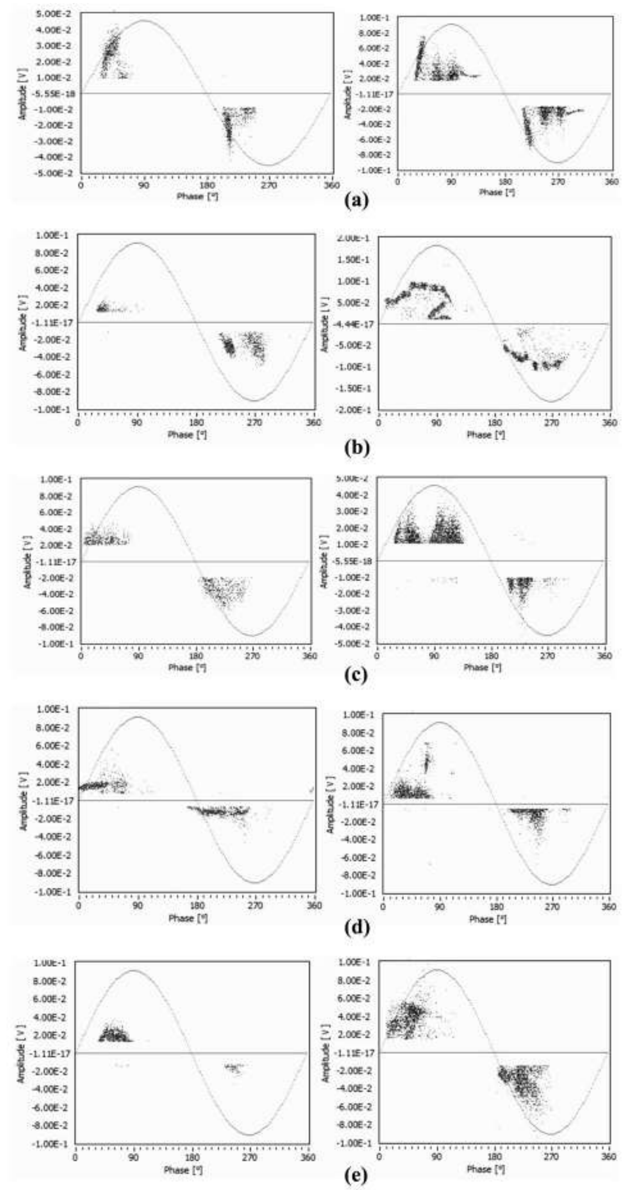

Fig. 10 PRPD pattern of SiO_2 samples at rod-plane geometry. Test voltage 25 kV (left) and 30 kV (right) **a** A1, **b** A2, **c** B1, **d** B11, **e** B31

untreated one. In general, negative LI BDV strength of corn oil seems to be higher than positive LI and similar results are reported in earlier paper [14].

Table 4 shows the summary of shape parameter (β), standard deviation and coefficient of variance of LI data at needle-sphere electrode under both positive and negative polarity. It also confirms the merits of CQD treated samples with higher β values and reduced standard deviation and s(%) values. Figure 14 shows the mean LI BDV values of tested oil samples at needle-sphere electrode geometry. It clearly shows the advantages of addition of CQD treated nanofillers in the corn oil samples. In particular, insulative silica treated with CQD shows better performance than other samples.

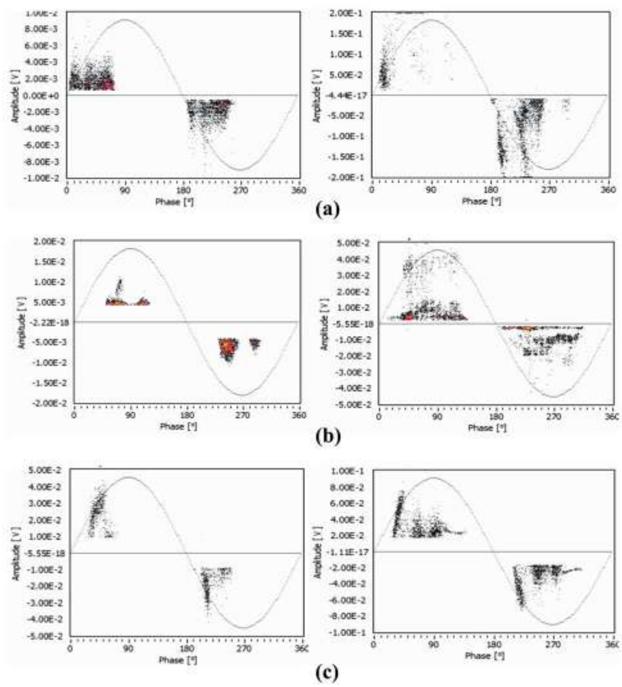

Fig. 11 PRPD pattern of TiO_2 samples at rod-plane geometry. Test voltage 25 kV (left) and 30 kV (right) **a** C1, **b** C11, **c** C31

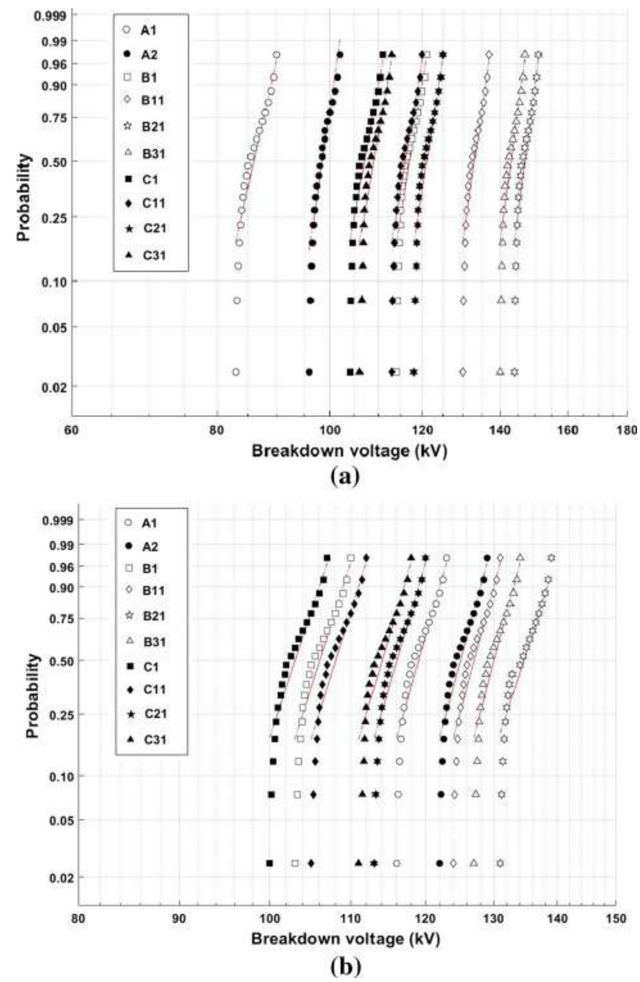

Fig. 12 Weibull probability distribution of LI BDV values of oil samples at needle-sphere electrode **a** positive LI, **b** negative LI

4 Discussion

Laboratory test results found in this work with corn oil nanofluids clearly shows considerable improvement in BDV, LI and PD properties of CQD treated nanofillers than untreated one. In earlier paper consisting of the results of CQD treated mineral oil [11], it has been reported that surface treatment of silica nanofillers with CQDs considerably improved the insulation strength because of the improved dispersion and stability of the oil. In Refs. [15–17], it is shown that hydroxyl, epoxide and carboxylic acid functional groups present in the CQD treated nanofillers improve the electrostatic repulsive force. In the present work, with natural ester corn oil, it is found that similar trend of results are obtained with both semiconductive (TiO_2) and insulative nanoparticles (SiO_2) treated with CQD. This may be due to the reduction in surface energy and interfacial tension between filler/oil interface which helps in improving the stability of the medium. Improvement in electrostatic repulsive force between nanoparticles in turn helps in avoiding the agglomeration of nanoparticles and hence improves the insulation characteristics of oil medium.

From the experimental results, it is found that CQD treated insulative SiO_2 nanofillers show better insulation properties than CQD treated semiconductive TiO_2 samples. Qi Wang et al. [14] have reported that the relaxation time constant of SiO_2 nanofillers is much lower than TiO_2 nanofillers, which helps in fast capturing of free electrons on the surface of nanoparticles. Similar trend of results are obtained in the present work that corn oil showed better breakdown strength with addition of CQD-SiO_2 fillers than CQD-TiO_2 fillers. This may be due to the fast capturing of free electrons by silica nanofillers than titania nanofillers, by which the growth of ionic constituents responsible for streamer development is very much reduced. In addition, since the nanofillers are partially covered with CQDs, the original hydrophilic property of silica (which helps in absorption of moisture content in the medium) is not affected and it also supports in improving the BDV when compared with TiO_2 nanofillers.

Yuzhen Lv et al. [18], have shown that positive LI streamer propagation length is much shorter in nanofluids forming a bush like structure which slows down the propagating process. From the positive LI results of present study, it is observed that there is a considerable enhancement of positive LI breakdown voltage of CQD treated samples.

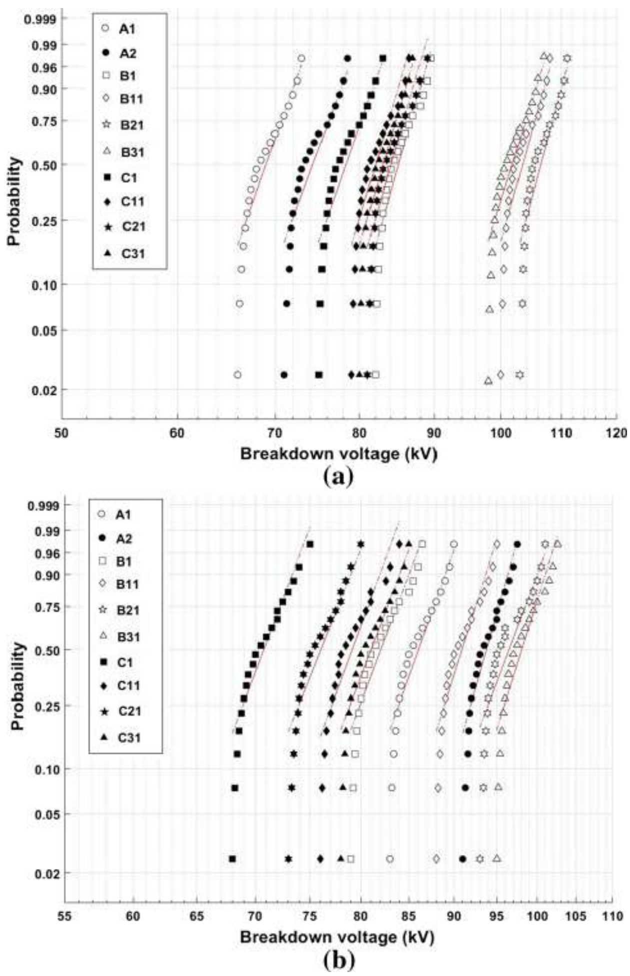

Fig. 13 Weibull probability distribution of LI BDV values of oil samples at needle-plane electrode **a** positive LI, **b** negative LI

Since the CQDs possess high electron transport characteristics, free electrons generated from the needle electrode are quickly captured by CQD treated nanoparticles leading to the formation of more number of negative ions near the positive electrode and reduction in the distortion of electric field. This in turn prohibits the growth of streamer and improves breakdown strength.

In the case of negative LI studies, Qi Wang et al. [14] have reported that breakdown strength of oil samples reduces under negative polarity of LI waveform due the presence of large number of negative charged nanoparticles near the top electrode leading to the weakening of the electric field. Negative LI results obtained in this work with untreated SiO_2 and untreated TiO_2 samples are in similar line. However, it is also seen that even under negative LI polarity, CQD treated nanofillers sufficiently improved the breakdown strength in both silica and titania, which may be because of the restructuring of electric field in the CQD/oil interface due to the stably suspended CQD nanostructures.

It is well reported in earlier papers [19, 20] that nano silica filled mineral oil show increased partial discharge inception voltage and reduction of total discharge magnitude compared with unfilled mineral oil. In the present work, PD results of corn oils clearly show reduction in PD magnitude of CQD treated samples than untreated one, which is in similar line with already reported works. This can be attributed to the electron scavenging, i.e. the free electrons coming out of electrodes due to ionization are well captured by the CQD treated nanofillers, thus preventing the growth of further discharge. As reported in Refs. [1, 2], in the case of untreated corn oil samples, occurrence of slightly higher amplitude PD is more effective in breaking the chemical bonds within liquid molecules leading to the formation of fault gases. However, in the case of CQD treated samples, they are more stable and occurrence of lower amplitude PD has less influence in molecular chain scission and chemical degradation of oils. When compared with mineral oil results, CQD treated corn oil showed much reduction in PD amplitude. This can be attributed to the formation rate of dissolved gases in corn oils which is slightly less than mineral oils [2], which may be the reason for reduction in magnitude of PD pulses.

Table 4 Summary of Weibull analysis of LI BDV data at needle-sphere electrode

Sample	Positive LI			Negative LI		
	β	STD	s (%)	β	STD	s (%)
A1	39	2.26	2.63	84	2.26	1.90
A2	55	2.29	1.98	56	2.26	1.81
B1	54	2.24	1.91	49	2.24	2.11
B11	67	2.21	1.54	60	2.25	1.72
B21	65	2.26	1.57	58	2.7	2.00
B31	61	2.24	1.69	54	2.24	1.77
C1	49	2.25	2.11	47	2.25	2.19
C11	53	2.26	1.95	50	2.24	2.07
C21	56	2.23	1.84	53	2.26	1.95
C31	50	2.25	2.03	53	2.21	1.92

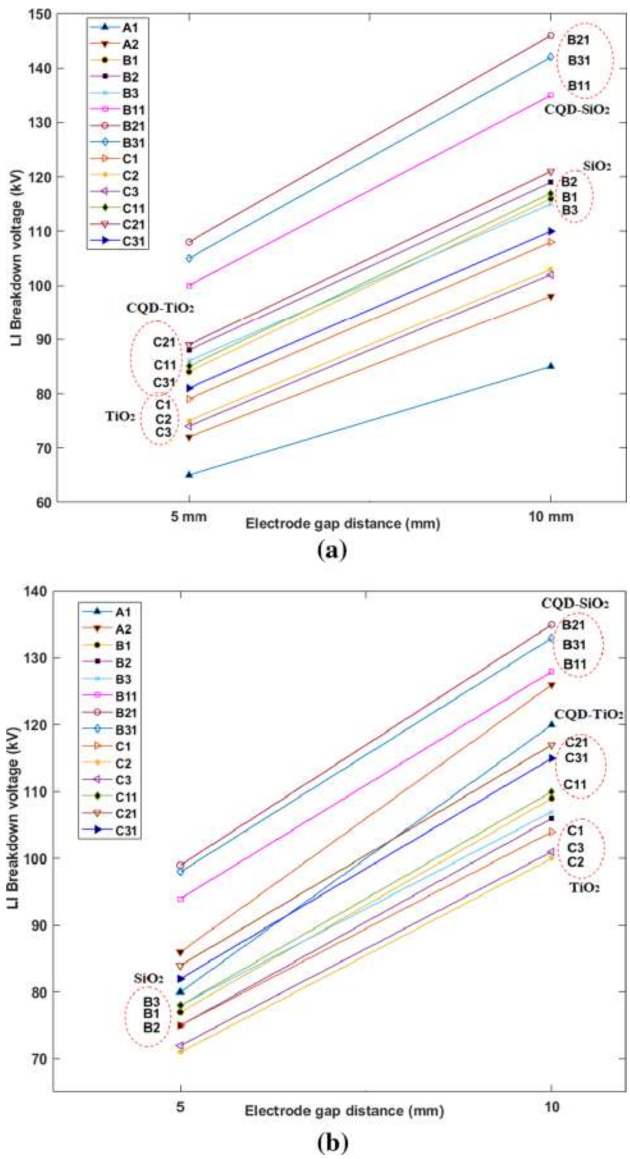

Fig. 14 Mean LI BDV values of tested oil samples under **a** positive and **b** negative polarity at needle-sphere electrode with different electrode gad distance

From the overall experimental studies, it is noticed that when the nanofiller concentration is above 0.05%wt, slight reduction in performance is noticed in PD, LI and BDV tests irrespective of the type of nanofiller. This is mainly because of the high surface area of nanoparticles and the strong attractive interaction between particles. Hence molecular ionization and streamer initiation starts at relatively lower voltage because of the formation of chain of particles near the electrode. Further thermal aging studies are planned on these samples to collect the database for understanding long term thermal stability of the proposed oils for transformer insulation applications.

5 Conclusion

Present study demonstrates that the addition of carbon quantum dots treated SiO_2 nanofillers in the corn oil can improve the AC breakdown voltage, LI breakdown strength and PD resistance. CQD treated SiO_2 nanoparticles in mass fractions between 0.01 and 0.05 have significant effect on the insulation properties of corn oil, because of the improved dispersion and stability of the oil.

Since the CQDs possess high electron transport characteristics, free electrons generated in the oil medium are quickly captured by CQD treated nanoparticles leading to the formation of more number of negative ions near the positive electrode and reduction in the distortion of electric field. This in turn prohibits the growth of streamer and improves breakdown strength.

CQD treated SiO_2 nanofluids show enhanced dielectric characteristics when compared with CQD treated TiO_2 nanofluids which is mainly because of the difference in relaxation time constants of nanofillers. In summary, findings of the present work suggest that the carbon quantum dots covered silica nanofluids with corn oil may be an alternate in place of the conventionally used mineral oil and it will impact positively on the design and reliability of power transformers.

Acknowledgement Author (S.Chandrasekar) would like to sincerely thank the Department of Science and Technology (DST), Nano Mission Scheme, New Delhi, India for providing financial support for carrying out this research work.

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