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ABSTRACT

The properties of traditional dielectric media have been a major limiting factor impacting the design and operation of many applications spanning from particle accelerators over x-ray radiography and radiotherapy to electrical power systems. Supercritical fluids (SCFs) combine the properties of high dielectric strength, low viscosity, and excellent heat transfer capability. Here, we show, for the first time, the anomalous breakdown strength characteristics of SCF mixtures, such as carbon dioxide (CO₂) and ethane (C₂H₆) mixtures and their azeotropic mixture under supercritical conditions. Our experiments suggest that the dielectric behavior deviates significantly from the established theory of gas discharge known by the work of Townsend and Paschen. Our results reveal that not only pure substances such as CO₂ exhibit a discontinuity of the dielectric strength near the critical point, but the supercritical mixture also manifests a discontinuity. The effect of random particle clustering in the pure substance and the mixture is observed, which impacts the mean free path of electrons. We present the measured breakdown voltage in a 0.1 mm gap with a uniform electric field over a wide range of mixture ratios and fluid densities and use a mathematical model by Stanley to show the density fluctuations that peak at around the critical point. By adjusting the mixing ratio, we prove that the mixture forms a useful combination of dielectric strengths and critical points and broadens the applicability of SC mixtures for a variety of purposes.

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Supercritical fluid (SCF), a state achieved when temperature and pressure are above the critical point of a substance, has drawn much attention because of its advantageous properties in chemical and material processing.^{1,2} It has also been studied for use as an insulating and arc quenching medium due to its combined strengths of liquid and gaseous dielectrics. SCFs show exceptional dielectric strength, high heat transfer capability, and low viscosity. Many experimental and theoretical works on electric discharge under supercritical (SC) conditions have been researched in the literature. Specifically, Zhang *et al.* demonstrated that SC nitrogen (N₂) shows excellent dielectric properties and recovery behavior from breakdown.^{3,4} Kiyon *et al.* conducted research on the pre-breakdown and breakdown in SC carbon dioxide (CO₂) with different electrode geometries. They also revealed the polarity effect in SC CO₂.^{5–10} Ito *et al.* performed measurements of breakdown voltages in SC CO₂, SC water (H₂O), and SC xenon (Xe) with 1 μm gap tungsten electrodes and discovered the decrease in the breakdown voltage near

the critical point.^{11,12} In our previous study, a theoretical model was developed to describe the mechanism of electrical breakdown in SC CO₂ with molecular clusters formation.¹³

In all the investigations of the electric discharge plasmas generated in SC CO₂ and SC N₂ described above, there is no consideration of many other highly promising supercritical substances or mixtures even though the outstanding dielectric performance of supercritical fluids is demonstrated. Moreover, to the best of the authors' knowledge, there are no data on the dielectric performance of supercritical carbon dioxide–ethane (CO₂–C₂H₆) mixtures, including their azeotropic mixture. Figure 1 summarizes the heat capacity, viscosity, and thermal conductivity of CO₂ and C₂H₆ from 0 MPa to 20 MPa absolute pressures at a temperature of 308 K, and water at the same temperature is also added in this figure for comparison.¹⁴ As shown in Fig. 1, properties such as viscosity, thermal conductivity, and heat capacity of SCFs are intermediate between those of liquids and gases. These properties are important for a dielectric medium,

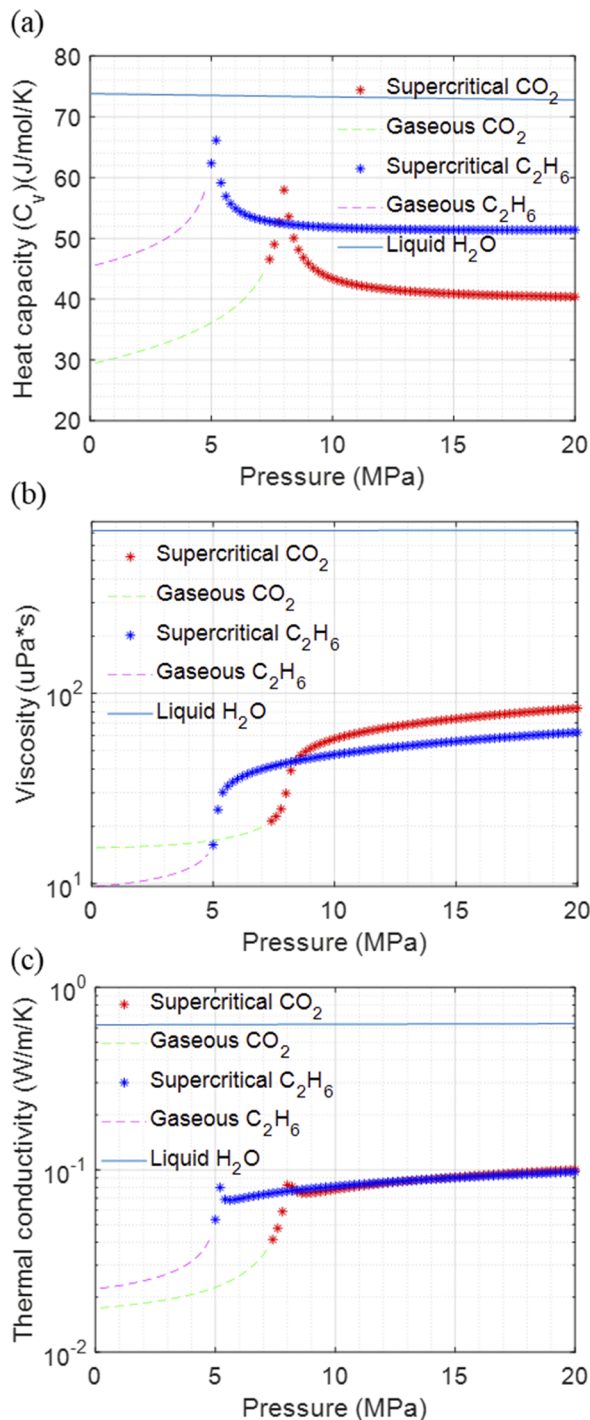


FIG. 1. Comparison of physical properties of carbon dioxide, ethane, and liquid water from 0 MPa to 20 MPa at 308 K: (a) heat capacity, (b) viscosity, and (c) thermal conductivity. Red stars represent carbon dioxide under the supercritical condition. Green dotted lines represent carbon dioxide in the gaseous phase. Blue stars represent ethane under the supercritical condition. Pink dotted lines represent ethane in the gaseous phase. Blue lines represent the thermodynamic properties of liquid water. Values are obtained from the NIST database.¹⁴

and hence, it is expected that such a medium can have many applications.

CO₂ has been one of the most frequently studied dielectric media due to its benign characteristics: high dielectric strength, non-toxic, low global warming potential (GWP), easily available, and low in cost. Furthermore, supercritical conditions are achievable with little effort ($T_c = 304.1$ K, $P_c = 7.38$ MPa).¹⁵ C₂H₆ was selected in this study for similar reasons. It is also non-toxic, has low GWP, and is easily available and low in cost. It also has a similar critical point to CO₂ ($T_c = 305.3$ K, $P_c = 4.87$ MPa),¹⁶ which results in a critical point that is technically easier to achieve compared to pairs of substances with very different critical points. A more significant reason why C₂H₆ was chosen in this study lies in the fact that C₂H₆ forms an azeotropic mixture with CO₂, which has a critical temperature that is lower than either of the constituents.¹⁷ This property is important because it allows for a wider range of applications. Some reports in the literature suggest that such a mixture could provide improved properties and more potential applications than the pure substance. Kravanja *et al.* evaluated the heat transfer performance of the CO₂–C₂H₆ azeotropic mixture under SC conditions. Their results revealed that the heat transfer coefficient of the CO₂–C₂H₆ azeotropic mixture fell between the values for pure CO₂ and C₂H₆, which proves that such a mixture has the capability to be used as an alternative fluid in heat power cycles.¹⁸ Considering its thermal properties, the investigation of the dielectric properties of such mixtures would be an imperative task in order to show the feasibility of a SCF as a promising dielectric medium.

Figure 2 shows the density vs pressure diagram of both CO₂ and C₂H₆ by two isothermal lines. Experiments were conducted at a constant temperature of 308 K, and the breakdown voltages were measured from gaseous to SC conditions. Besides the pure substances, it is also necessary to know the critical points of the mixtures with different mixing ratios so that the thermodynamic

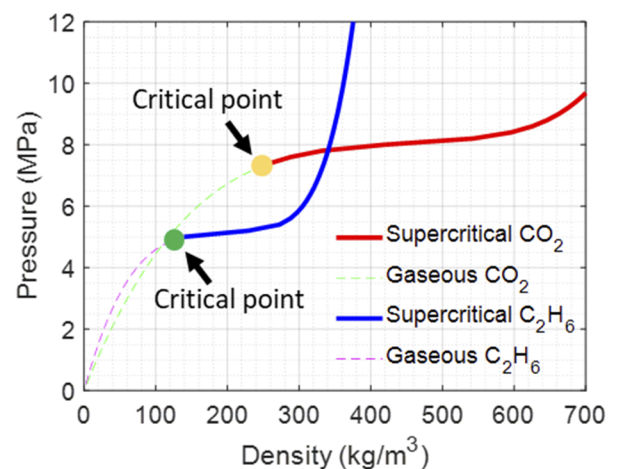


FIG. 2. Phase diagram of carbon dioxide and ethane. Experimental conditions represented by isothermal lines of CO₂ and C₂H₆ density–pressure diagram at 308 K ($T_r = T/T_c = 1.012$). The red line represents carbon dioxide under the supercritical condition. The dotted green line represents carbon dioxide in the gaseous phase. The solid blue line represents ethane under the supercritical condition. The pink dotted line represents ethane in the gaseous phase.

phase inside the high pressure chamber can be confirmed. A high pressure optical vessel was used to observe the phase transition of the mixtures visually. The critical points of the mixtures from the optical diagnostics were in good agreement with the critical points calculated from the PSRK (predictive Soave–Redlich–Kwong) model.¹⁹ The comparison of the critical points of the mixtures with respect to the mass fraction will be discussed in the following paragraphs.

The breakdown voltage measurements of pure CO₂ were carried out at a temperature of 308 K from the gaseous phase to SC condition, as shown in Fig. 2. Figure 3(a) shows the measured breakdown voltages as a function of the pressure, and Fig. 3(b) shows the measured breakdown voltages as a function of the density. The average breakdown voltage of 15 measurements and their scattering data under one experimental condition are represented by an open circle and a vertical error bar, respectively. The density values were calculated by using the equation of state (EoS)²⁰ from pressure and temperature during the experiment and confirmed by weighing the mass of the substance inside the high pressure chamber.

Clearly, the measured breakdown voltage increases with the density of CO₂ and scatters more in the supercritical region. An obvious discontinuity of the slope can be observed near the critical point where the substance experiences a phase change. Under the SC condition, the composition of the fluid is characterized by inhomogeneity in the molecular distribution due to the distinct clusters of molecules.²¹ Especially, under the conditions close to the critical point, the density fluctuation F_D increases substantially due to repeated aggregation and dispersion of clusters, which influences the breakdown strength significantly. The density fluctuation F_D is defined by²²

$$F_D = \frac{\langle (N - \langle N \rangle)^2 \rangle}{\langle N \rangle} = \frac{(n_s V)^2}{n_{ave} V} = \frac{k_T}{k_T^0}, \quad (1)$$

where N is the total number of particles in a given volume V , $\langle N \rangle$ is the average of N , n_s is the standard deviation of the local number density, n_{ave} is the average number density, k_T is the isothermal compressibility, and k_T^0 is the value of k_T for an ideal gas. A larger F_D means larger density fluctuations, and F_D reaches local maxima at the critical point.

The density inhomogeneity is caused by the clustering effect, which forms a large mean free path where electrons could gain enough energy and ionize particles. Although the phenomenon of the discontinuity in breakdown vs density near the critical point in the experiment is pronounced, the decrease in the breakdown voltage near the critical point reported by Ito *et al.* was not observed,^{11,12} which was expected because the gap length in this study is relatively large. This is because when the gap distance decreases, the volume of the locally low-density domain at the breakdown V also decreases. This causes n_s/n_{ave} to increase, as shown in Eq. (1). This indicates that the effect of the density fluctuations on the breakdown voltage becomes more significant with a decrease in the gap distance.²³ Moreover, in the case when discharges happen under the condition of being close to the critical point, clustering and density fluctuation F_D decrease due to the increase in the local temperature caused by discharges. If the gap length is smaller than 1 μm , the cluster structure can be preserved because more effective heat dissipation

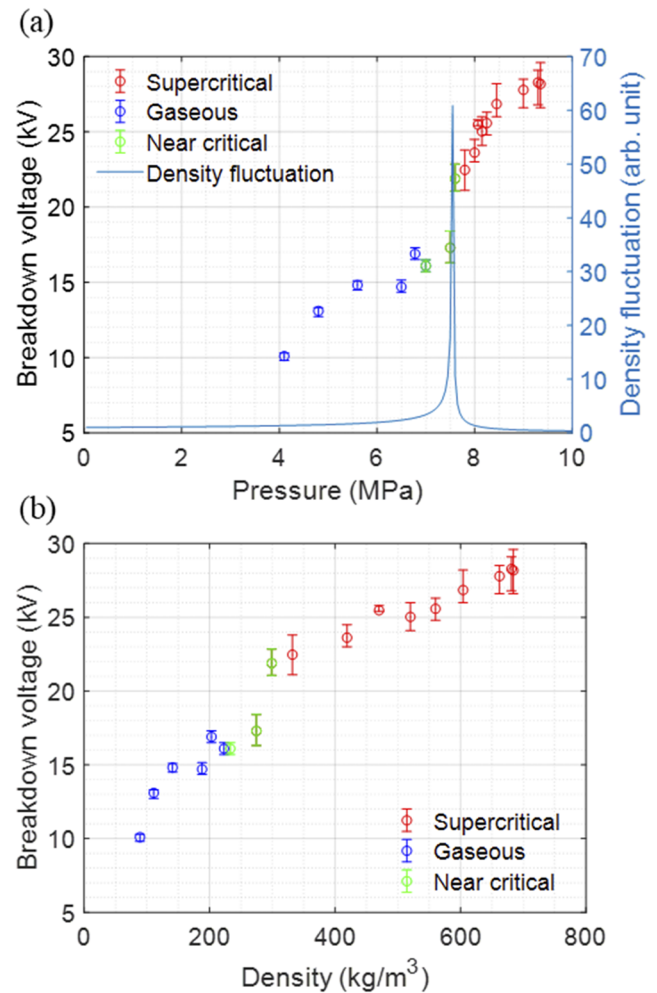


FIG. 3. Experimental results for the dielectric strength of supercritical carbon dioxide up to 10 MPa. (a) Breakdown voltage as a function of the pressure of CO₂ in a uniform electric field at a 0.1 mm gap. The right y-axis indicates the density fluctuation F_D , which quantifies the molecular clustering. F_D values are calculated by Eq. (1), and the isothermal compressibility $k_T = k_s C_p / C_V$, where k_s is the isentropic compressibility, C_p is the heat capacity for the isobaric process, and C_V is the heat capacity for the isochoric process. (b) Experimental values of the breakdown voltages as a function of the density of CO₂. Red circles represent the average breakdown voltage for carbon dioxide under the supercritical condition. Blue circles represent the average breakdown voltage in the gaseous phase. Green circles represent the average breakdown voltage measured near the critical point of carbon dioxide. Vertical error bars represent the corresponding experimental scattering data.

is enabled by the large specific surface area.²⁴ Thus, a reduction in the breakdown voltage can be observed.

The reason for the deviation between the breakdown voltage in SCFs and the gaseous state is that the gaseous discharge theory cannot explain the situation when the mean free path of electrons in SCFs starts to decrease. In this circumstance, SCFs deviate considerably from the ideal gas behavior, and a small change in the pressure causes a large change in the density. Paschen's law

cannot precisely predict the breakdown voltage in high pressure gases and SCFs. Specifically, Paschen's law fails to estimate the breakdown characteristics in the compressed medium when the electric field is of the order 10–20 kV/mm. The deviation from Paschen's law depends on the material, separation and area of the electrodes, and particles of dusts near the electrodes. Similarly, the failure of the Townsend mechanism for SCFs is that the Townsend theory does not take the field emission of electrons from the cathode in a high density situation into consideration.²⁵

To identify the state of the mixture inside the high pressure chamber, the critical points of the mixtures with different mixing ratios need to be determined. Horstmann *et al.* conducted the experimental determination of the critical line for CO₂–C₂H₆ mixtures and compared their results with the PSRK model.¹⁹ The PSRK model is able to reliably predict the thermodynamic properties of carbon dioxide and alkanes by using one pair of temperature-dependent group interaction parameters. Such a model has been widely used in process simulators because it combines the advantages of the EoS, the local composition concept, and the group contribution approach. Optical diagnostics were conducted by using an optical chamber to replicate the experimental conditions considering mixing ratios and densities so that the critical points of the mixtures can be measured. Table I compares the critical points observed from the optical cell and calculated from the PSRK model with respect to the mass fraction. The error between two methods, as shown in Table I, is expected to be caused in part due to the presence of impurities.

To ensure consistency in the experimental results with the pure CO₂ data, the gap between the copper electrodes was also set to 0.1 mm. C₂H₆ mass percentages of 10%, 25% (azeotropic),¹⁸ 30%, 40%, and 50% were tested in the breakdown experiment. An oily substance between two electrodes was observed after the first breakdown when the C₂H₆ mass percentage (ω) reaches beyond 60%. Rosocha *et al.* conducted experiments to determine the decomposition of C₂H₆ under dielectric-barrier discharges at 77.3 kPa and a temperature range of 293–323 K. The primary decomposition products they found were molecular hydrogen (H₂), methane (CH₄), acetylene (C₂H₂), and ethylene (C₂H₄).²⁶ Oro *et al.* also observed a similar phenomenon that organic compounds are formed under the influence of electric discharges with C₂H₆ at a pressure range of 10–30 kPa at 303 K. Their results indicate that the product caused by the electric discharge could be a highly cross-linked polyethylene-type polymer.²⁷

TABLE I. Comparison of the critical temperature T_c and critical pressure P_c for $\omega\text{C}_2\text{H}_6 + (1 - \omega)\text{CO}_2$.

ω	Optical diagnostics		PSRK model	
	T_c (K)	P_c (MPa)	T_c (K)	P_c (MPa)
0.1	300.0	6.75	297.5	6.73
0.25	294.5	6.50	292.0	6.35
0.3	294.0	6.05	291.2	5.94
0.4	293.5	5.82	291.5	5.70
0.5	293.0	5.55	292.0	5.52

A similar anomalous breakdown behavior near the critical point of pure CO₂, as shown in Fig. 3, was also observed in the CO₂–C₂H₆ mixture at the azeotropic mixing ratio, as shown in Fig. 4. It should be noted that the discontinuity of breakdown voltages happens at the locus of the states where the scale and the magnitude of the density fluctuation reach maxima on the isothermal line. It also represents a boundary that divides the supercritical region into liquid-like and gas-like phases.^{28,29} According to the experimental condition in this study, since experiments were conducted under the isothermal condition of 308 K, the density fluctuation F_D is expected to peak slightly above 6.5 MPa, which is the observed critical pressure.

Figure 5(a) shows a 2D plot of the measured breakdown voltage at different mixing ratios as a function of the density. A solid

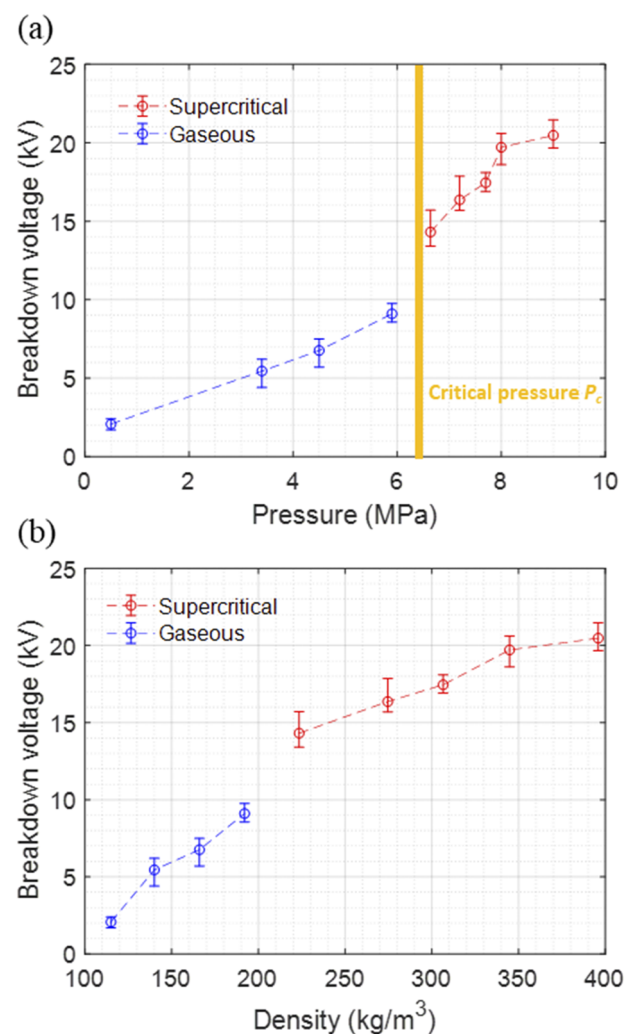


FIG. 4. (a) Breakdown voltage as a function of the pressure of the CO₂–C₂H₆ mixture at the azeotropic mixing ratio (25% mass fraction of C₂H₆) in a uniform electric field, at a temperature of 308 K in (a) 0.1 mm gap. (b) Breakdown voltage as a function of the density of the CO₂–C₂H₆ mixture at the azeotropic mixing ratio.

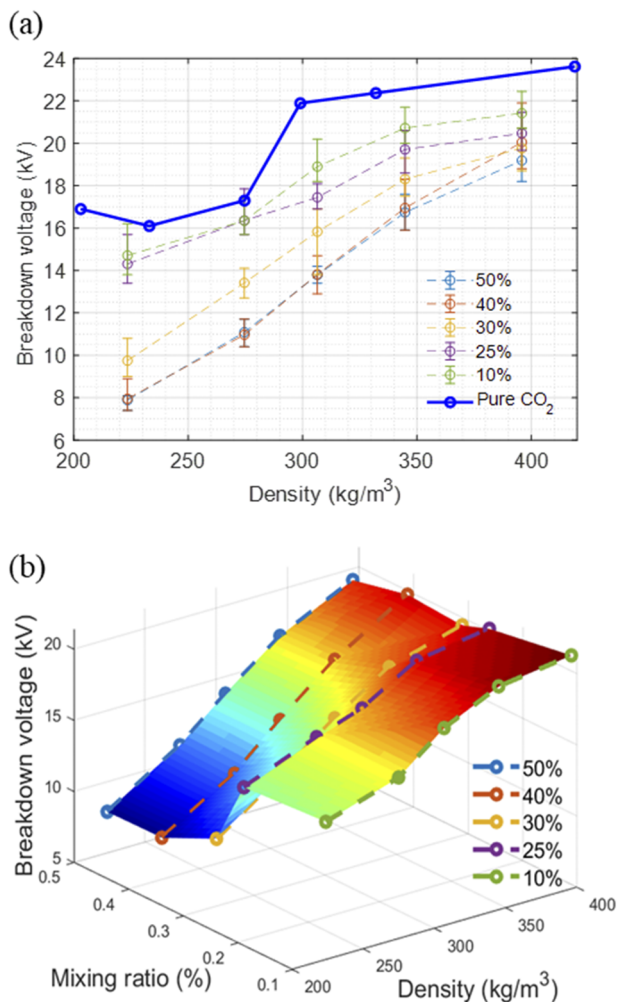


FIG. 5. (a) A 2D plot of the breakdown voltage at different mixing ratios as a function of the density. The solid blue line indicates the average of the measurement data of pure CO_2 . (b) Breakdown voltage at different mixing ratios as a function of the density at a temperature of 308 K. ω : the mass percentage of C_2H_6 . The C_2H_6 mixing ratio varies from 10% to 50% by mass into CO_2 .

blue line indicates the average of the measurement data of pure CO_2 . Figure 5(b) presented a 3D plot of all the measured breakdown voltages of supercritical CO_2 - C_2H_6 mixtures at different mixing ratios as a function of the density. All the breakdown values presented in Fig. 5(b) were measured under the supercritical condition. For all the mixtures at different mixing ratios, the highest dielectric strength is shown at the highest density of 396 kg/m^3 and the lowest at 223 kg/m^3 . In addition, for all five SCF mixtures, the breakdown voltage decreases with an increase in the C_2H_6 mass fraction. The transition between 25% and 30% of the C_2H_6 mass fraction in Fig. 5 shows a steep decrease, especially in the lower density region. Such a transition can be attributed to the fact that under different C_2H_6 mass fractions, the locations where density fluctuations reach maxima can also be shifted. In this situation, the large inhomogeneity in the molecular distribution caused by the rapid and repeated

aggregation and dispersion of clusters can be shown from the unstable breakdown voltages. Specifically, we speculate that our experimental result indicates that the samples of C_2H_6 concentration at 25% and 30% are closer to the supercritical liquid-gas boundary line. Also, the measured breakdown voltage of the mixtures scatters more widely compared to the values of pure CO_2 . In the lower density region between 220 kg/m^3 and 300 kg/m^3 , the difference in the breakdown voltages of the mixtures tends to be more obvious than those in the higher density region. The data also indicate that the breakdown voltages of different mixing ratios saturate at higher densities. The breakdown voltage of the mixtures in the lower density region also shows a more pronounced reduction compared to pure CO_2 . For the azeotropic mixture of CO_2 and C_2H_6 (25% mass fraction of C_2H_6 and 75% mass fraction of CO_2), the breakdown voltage shows an average of 20.5% reduction compared to the dielectric strength of pure CO_2 in the vicinity of the critical point of CO_2 . In the higher density region far away from the critical point, the reduction in dielectric strength of the mixture drops to about 13.5% compared to pure CO_2 .

Our study found that the anomalous breakdown characteristics discovered in the pure SCF are also observable in binary mixtures of supercritical fluids at various azeotropic mixture ratios. The similar behavior suggests that the unstable molecular clustering could significantly affect the discharge phenomenon observed in both pure SCFs and supercritical mixtures. Unique properties of SCF mixtures with respect to the dielectric strength, viscosity, specific heat capacity, and tunable critical point are expected to attract interest for a wide range of applications. Besides the applications in power and energy, where they could be used for switchgear and electrostatic machines,^{30–32} SCF mixtures could also enable affordable van-de-Graaff generators for particle accelerators used in high energy physics and medical applications, such as radiation therapy.^{33,34}

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