

# Dielectric Performance and Dissolved Gas Analysis of Natural Esters for Application in Power Transformers

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# ABSTRACT

This paper presents results of experimental investigations on natural esters regarding the pre-breakdown processes, breakdown strength and gassing behaviour under electric and thermal stresses. An intensive investigation of breakdown strength was done concerning the influence of gap distance, electrode shape, volume of stressed fluid and form of applied voltage. The conditions of investigated natural ester were new, thermally aged and contaminated with mineral oil. Generated fault gases were analysed by means of dissolved gas in oil analysis (DGA) with headspace technology.

The conducted experiments show that natural esters have comparable dielectric performance and gassing characteristics to mineral oil, but still requiring modification of design and operating parameters.

Key words: natural ester, dielectric strength, DGA, power transformer,

# 1. INTRODUCTION

Parallel with the worldwide constantly growing demand for the electrical energy an environmentally and safety aspect is raised. Power transformers being crucial elements in electric power transmission network also have to follow this trend, setting the challenge for power transformer manufacturers. For more than one century power transformers are filled with mineral oil serving as a heat transfer and insulating medium. Nowadays significant efforts are aimed in guest for environmentally friendlier replacement of this petroleum-based product. Some advantages offered by natural esters, are the faster biodegradability, no water hazard, higher flash/fire points and low thermal expansion coefficient. Nevertheless the different chemical and physical characteristics require special considerations. For the last 10 years natural esters have been successfully used in more than 45,000 low and medium power installations [1]. Since natural esters possess weak oxidation stability they require to be used in hermetically sealed transformers [2]. Dissimilarities between natural esters and mineral oil in chemical and physical characteristics require further considerations before they can be widely used in power transformers. Especially dielectric strength and gassing behaviour have to be taken into account for a reliable design and safe operation. The dielectric strength of natural esters being a crucial parameter of insulating fluid is extensively compared with mineral oil.

In this contribution results of breakdown strength measurements on two commercially available natural esters (NE1, NE2) and mineral oil Nynas Nytro 3000X (NN3000X) as a reference fluid are presented. The second part of the investigations is about dissolved gas in oil analysis. It has been used for many years successfully for diagnosis and condition monitoring of mineral oil filled transformers. In future the diagnostic interpretation schemes have to be verified for natural esters [4, 5]. In this article we present the results from experiments, which represent electrical and thermal faults. Where possible the amount and relation of the gases are compared to mineral oil gassing behaviour. The gas concentrations are measured



with a gas chromatograph with headspace technology. This is why there is an additional subsection about the determination of the gas-liquid partitioning coefficients (K-factors) of specific natural esters.

## 2. DIELECTRIC STRENGTH OF NATURAL ESTERS

## 2.1 Homogeneous Electrical Field

The characterization of natural ester dielectric strength in homogeneous AC electrical field is done by means of VDE calotte electrodes with a gap distance of 2 mm. This experiment is conducted according to IEC 60156 with certain modifications. Because of the higher viscosity of natural ester, the pause after breakdown was increased from 2 min for mineral oil to 5 min for natural esters. Natural esters require a longer pause in order to give enough time for all breakdown products to be removed from the stress area by stirring. The settling time after filling the vessel and before application of the voltage was 40 min. In order to obtain sufficient measured values for accurate statistical evaluation fluids are tested in series of 40 to 50 breakdowns. Furthermore, some fluids showed increasing tendency of the breakdown voltage for first 5 breakdowns. Therefore, the number of six breakdown values suggested by IEC 60156 had to be increased.

The two-parameter Weibull cumulative distribution function and Normal (Gaussian) cumulative distribution function are used for statistical evaluation of measured values. For each experiment the mean value ( $\mu$ ), the standard deviation ( $\sigma$ ), scale ( $\alpha$ ) and shape ( $\beta$ ) parameters are calculated.

After fitting of measured values to breakdown distribution functions, a 1 % breakdown probability (withstand) voltage  $U_{1\%}$  and 50 % breakdown probability (mean) voltage  $U_{50\%}$  were calculated (Table I). For homogeneous arrangement natural esters showed about 10 % higher mean breakdown voltages. The standard deviation of natural esters is smaller than for mineral oil. This leads even to stronger dissimilarity between the fluids for withstand voltage values. NE2 has about 35 % higher withstand voltage than mineral oil according to the Normal distribution.

Fluid	Ν	Iormal Distri	ibution		Weibull Distribution				
i iuiu	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β	
NN3000X	45.1	66.4	66.36	9.14	43.7	67.5	70.02	9.72	
NE1	53.0	70.6	70.59	7.59	47.4	71.4	74.00	10.30	
NE2	61.1	74.2	74.25	5.77	55.9	75.0	76.84	14.41	

Table I: Breakdown probability values (AC) for 2 mm uniform fluid gap

### 2.2 Inhomogeneous Electrical Field

In order to generate inhomogeneous electrical field the Perspex test vessel equipped with brass point-plate electrodes is mounted on the Baur DTA 100E device. The point electrode was with tip radius of 100  $\mu$ m and the gap distance was 30 mm. The experiment procedure was similar to homogeneous electrical field experiment. Fitted AC breakdown results of this experiment are given in Table II. In this case, tested fluids showed similar mean breakdown voltages. As well, having similar standard deviations the withstand voltages are in the same relation.

Table II: Breakdown probability values (AC) for 30 mm non-uniform (100 µm tip radius) fluid gap

Fluid	Ν	ormal Distri	bution		Weibull Distribution				
i lulu	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β	
NN3000X	43.8	56.0	56.00	5.21	39.7	56.6	58.35	11.96	
NE1	45.4	55.6	55.63	4.39	41.6	56.1	57.65	14.07	
NE2	44.1	53.0	53.00	3.78	38.4	53.3	54.84	12.96	

In order to obtain different electrical field intensities at the tip of point electrode, the gap distance and point electrode tip radius are altered. Results of measurement with point tip radius of 3  $\mu$ m and 100  $\mu$ m and electrodes gap of 30 mm and 40 mm are given in Tables II-V.



By reducing the tip radius (increased electrical field intensity) mineral oil kept similar breakdown values and natural ester obtained about 10 % lower mean breakdown value and unaltered withstand voltage (Table II, III). Reiterative decrease of electrical field by increasing the gap distance has similar effect on both fluids. The mineral oil kept slightly higher mean breakdown voltage but withstand voltages are in the same range (Table III, IV).

Table III: Breakdown probability values (AC) for 30 mm inhomogeneous (3 µm tip radius) fluid gap

Fluid	Ν	Iormal Distri	bution	Weibull Distribution				
	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β
NN3000X	44.2	55.5	55.48	4.88	40.2	56.05	57.65	12.72
NE1	43.0	48.4	48.35	2.32	40.1	48.6	49.47	21.81

Table IV: Breakdown probability values (AC) for 40 mm inhomogeneous (3 µm tip radius) fluid gap

	Fluid	Ν	ormal Distri	bution		Weibull Distribution					
	Tulu	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β		
	NN3000X	56.1	63.0	63.00	3.21	51.2	63.9	65.17	19.07		
-	NE1	53.3	57.0	56.96	1.57	51.0	57.2	57.74	36.85		

*Table V:* Breakdown probability values (AC) for 40 mm inhomogeneous (3 μm tip radius) aged fluid gap

Fluid	Ν	lormal Distri	bution		Weibull Distribution				
Fiulu	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β	
NN3000X	53.0	56.5	56.54	1.53	51.1	56.8	57.29	40.23	
NE1	52.8	55.5	55.55	1.18	51.8	55.7	56.09	57.67	

Table VI: Breakdown probability values (AC) for natural ester contaminated with 10 % of mineral oil in40 mm inhomogeneous (3 μm tip radius) gap

Fluid	١	Iormal Distri	bution		Weibull Distribution				
i iuiu	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β	
Cont. NE1	54.2	55.8	55.77	1.18	51.4	55.9	56.36	49.35	

Table VII: Breakdown probability values (LI) for 60 mm inhomogeneous (3 µm tip radius) fluid gap

Fluid		Normal Disti	ibution	Weibull Distribution				
i iuiu	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	μ	σ	U <sub>1%</sub> [kV]	U <sub>50%</sub> [kV]	α	β
NN3000X	123.1	139.0	139.00	6.82	114.3	139.8	142.30	21.00
NE1	108.1	125.4	125.40	7.47	91.9	125.8	129.31	13.45

Since the natural esters are very new products in power transformers, there are no records of the dielectric behaviour over longer period available. Therefore, a thermal ageing experiment is conducted. Metal vessels, having a total volume of 5 litres, are filled with the same proportional mass ratio of fluid and materials that can be found in real power transformers (cooper, aluminium, iron, zinc and cellulose). The vessels are sealed preventing the contact of heated fluid with the atmosphere and put in an oven under temperature of 150 °C for 63 days.

The AC breakdown voltages of the aged fluids are measured for point-plate electrodes. The point electrode was an Ogura fine needle with the tip radius of 3  $\mu$ m. The oil gap is adjusted to 40 mm. Fitted AC breakdown results of this experiment are given in Table V. It can be seen that thermal aging does not affect breakdown strength of natural ester. For mineral oil there is only a slight decrease of breakdown strength (Table IV, V).

For retro fill of power transformers, which were previously filled with mineral oil, another aspect to be considered is the breakdown strength of natural ester contaminated with mineral oil. For this purpose the test vessel with point-plate electrodes is filled with a ratio of 90 % natural ester and 10 % of mineral oil. Fitted AC breakdown results of this experiment are given in Table VI. Results show that contamination of natural ester with mineral oil does not influence the breakdown strength of natural ester (Table IV, VI).

In the next experiment the lightning impulses are applied in steps starting from about 80 % of expected withstand voltage. Used electrodes were point-plate with gap distance of 60 mm and point tip radius of 3  $\mu$ m. At each step three impulses are applied until the breakdown



occur. Regarding lightning impulses natural ester has a 12% lower withstand voltage (normal distribution) than mineral oil (Table VII).

By investigating pre-breakdown processes it is observed that PDs in natural ester are initiated at slightly lower voltage than in mineral oil (Table VIII).

The point-plate electrode system with a gap distance of 40 mm is used in order to investigate the streamer propagation for lighting impulse voltages. Starting at expected withstand voltage value the applied impulse voltage is continuously increased. In this manner, a decrease of the time to breakdown is achieved. Assuming that the streamers are initiated directly after applying the voltage impulse, with known gap distance and measured time to breakdown, the streamer propagation velocity for lightning impulse is calculated as gap distance divided by time to breakdown.

As in some previously done investigations on mineral oil [3] three different propagation modes are identified in both fluids (Figure 1). Mode 1 is related to the overvoltages directly above breakdown value and involves slight increase of streamer propagation velocity in the range from 2 to 3 mm/µs. In mode 2, streamer velocity increases strongly with slight increase of applied voltage. Mode 3 involves again slight increase of streamer velocity over applied voltage in rage of 10 mm/µs. The natural ester reached fast Mode 3 of the streamer propagation velocity at lower voltages than mineral oil. Also, the velocity in Mode 3 of natural ester is higher than of mineral oil. This indicates that streamers propagate faster in natural ester than in mineral oil, having more potential to bridge larger gaps and to create a breakdown.

PD Ir	ception	Tip Radius			
Vo	oltage	3 µm	100 µm		
30 mm	NE1	18.8	22.4		
50 11111	NN3000X	20.6	23.2		
10	NE1	21.7	23.9		
40 mm	NN3000X	22.5	24.7		

Table VIII: Partial discharge inception volvage



*Figure 1*: Streamer propagation velocity over 40 mm point-plate gap under lightning impulse

# 3. DISSOLVED GAS IN OIL ANALYSIS

Oil samples from the experiments are analysed with a gas chromatograph involving headspace technique. It means that the gas concentrations above the analysed liquid are measured. With these values it is possible to calculate back to the original dissolved gas amounts in the insulating fluid. Because of this, it is necessary to sample the vials carefully in a glove box with argon atmosphere in order to avoid contamination with the gases from the atmosphere. The used gas chromatograph detects H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, C<sub>2</sub>H<sub>2</sub>, C<sub>2</sub>H<sub>4</sub>, C<sub>2</sub>H<sub>6</sub>, C<sub>3</sub>H<sub>6</sub> and C<sub>3</sub>H<sub>8</sub>.

In a closed system the gas concentration of gas and liquid phase reach equilibrium according to Henry's law. In this state the ratio of the two phases' concentrations is a constant for each specific gas – given a constant temperature and pressure. These partitioning coefficients  $K_i$  depend on the investigated insulating liquid.

The gas chromatograph measures  $C_G$  (Figure 2). The desired initial gas-in-oil concentration  $C_{L,0}$  can be determined with the conservation of mass law (formula 2 to 6). One can see that with the knowledge of the oil volume  $V_L$ , the headspace volume  $V_G$ , the K-factors and  $C_G$ , it is possible to compute the original dissolved gas-in-oil  $C_{L,0}$  for further diagnostic statements.





$$K = \frac{C_L}{C_G} \tag{1}$$

$$M_0 = M_L + M_G \tag{2}$$

$$\mathcal{C}_{L,0} \cdot \mathcal{V}_L = \mathcal{C}_L \cdot \mathcal{V}_L + \mathcal{C}_G \cdot \mathcal{V}_G \tag{3}$$

$$C_{L,0} \cdot V_L = K \cdot C_G \cdot V_L + C_G \cdot V_G \quad (4)$$

$$K = \frac{C_{L0}}{C_G} + \frac{V_C}{V_L} \tag{5} \quad or$$

$$C_{L,0} = C_{\mathcal{G}}(K + \frac{V_{\mathcal{G}}}{V_{L}}) \tag{6}$$

Figure 2: Vial with gas concentrations

#### 3.1 Experimental determination of the K-factors

The partitioning coefficients can be measured in different ways, e.g. with direct vapour phase calibration method or indirect phase ratio variation method and others [6, 7]. In this experiment gas-inoil standards were produced by dissolving a defined volume (6 ml) of reference gas in a glass syringe (90 ml) filled with dried and degassed oil. In the glovebox with controlled argon atmosphere 20 ml vials were filled with 10 ml gas-in-oil standard. Additionally gas samples from the glovebox atmosphere were taken, in order to subtract the still existing small contamination with N<sub>2</sub> and O<sub>2</sub>. The vials' exact volumes were measured by weighting with and without distilled water at a known temperature. The exact oil volume V<sub>L</sub> was also calculated by weighting and factoring in the temperature dependant density. The K-factors then can be computed with formula (5) (Table IX).

Table	IX: K-factors of	/VE

gas	K-factor
H <sub>2</sub>	0,08
O <sub>2</sub>	0,14
N <sub>2</sub>	0,19
CO	0,03
CO <sub>2</sub>	1,59
$CH_4$	0,08
$C_2H_6$	1,37
$C_2H_4$	1,16
$C_2H_2$	1,58
$C_3H_8$	3,67
C₃H <sub>6</sub>	4,07

#### 3.2 Gas generation due to partial discharges

The aim of these experiments was to generate partial discharges (PDs) of different intensities and durations and to analyse the generated gases. The used test cell had the same configuration as already described in subsection 2.2 and table IV. Additionally a double pressboard barrier between electrodes is used to generate stable and strong partial discharges. The distance between barriers was 5 mm and the distance between nearest barrier and plate electrode was 10 mm. The test cell has been proven as PD-free by measurements. The measurement system was a conventional one with a coupling capacitance. The intensity of the PDs is quantified in apparent charge in coulombs.



Figure 3: Gases generated under 700-1000pC (NE1)



Figure 4: Gases generated under 1000pC PDs (NE1)



Three different PD intensities were generated: a low intensity with 700-1000 pC, a medium with 1000 pC and a high one with 2000 pC. The inception voltage was between 34 and 58 kV. The experiments lasted three hours. Every hour a pause was made and an oil-sample of 50 ml was taken with a syringe.

The observed key gases for the natural ester (NE1) are  $H_2$  and CO. Acetylene ( $C_2H_2$ ) and methane are typical secondary gases (Fig. 3-5). This behaviour is different to mineral oil, which has a considerable lower gas generation and CO is missing (Fig. 6).





*Figure 5: Gases generated under 2000pC PDs (NE1)* 

Figure 6: Gases generated under 2000pC PDs in mineral oil

# 3.3 Gas generation due to arcing

In this experimental setup a similar test cell was used, but a point-point electrode configuration was used. The distance between the electrodes was 10 mm. The breakdowns were generated with a 10 stage Marx lightning impulse generator. The peak voltage value of the standard lightning impulse (1.2/50  $\mu$ s) was 140 kV. The energy per breakdown was 580 J. Series of 10, 20, 30, 50, 70 and 90 breakdowns were conducted, where again after each series an oil sample was taken for analysis.

As for mineral oil the key gas for arcing in NE1 is acetylene (Fig. 7). The gas generation ratio is similar for both insulating liquids. Although in mineral oil more  $H_2$  is produced.



Figure 7: Gases generated by lightning impulses in NE1



## 3.4 Thermal stress with hotspot

The goal of this experiment is to emulate a hotspot inside the transformer. A glass cylinder with a volume of 15 litres is used. Inside the cylinder is a resistherm wire, which depending on the flowing current settles at the desired temperature. The 600 mm long wire is connected to the cylinder covers and lies in the middle of the oil filled cylinder. It is heated to three different temperatures: 300°C, 500°C and 700°C. The experiments lasted, depending on the temperature, several hours to several days.

Key gases in this experiment are CO,  $CO_2$  and propylene -  $C_3H_6$ . The experiments at the highest temperature had to be stopped, because the gas generation ratio was too high resulting in unacceptable big gas bubbles in the vessel. Detectable and linearly accumulated were also hydrogen, ethane and ethylene (Table X).

	Gener	ated ga	ses in p	pm for N	IE1 at 30	0° 00	•		Generated gases in ppm for NE1 at 700 °C					
h	CO <sub>2</sub>	CO	H <sub>2</sub>	$C_3H_6$	$C_2H_6$	$C_2H_4$		h	CO <sub>2</sub>	СО	$H_2$	$C_3H_6$	$C_2H_6$	$C_2H_4$
24	988	923	248	3122	806	271	-	0.5	183	160	924	192	165	12
42	1448	1362	377	4247	1156	375		1.0	447	446	2415	443	588	38
72	2219	2251	495	5342	1363	537		1.5	5048	4205	3037	3037	2026	755
139	2789	2506	724	7146	1877	706		2.0	5318	4423	3417	3417	2345	778
144	3249	3994	1168	8709	2625	873		2.5	13705	11835	3742	3742	3819	2267
192	7228	5769	1619	8767	3831	1242		3.0	14063	13428	4065	4065	3870	2429
								3.5	14968	15072	4469	4469	3957	2514

Table X: gassing of natural ester 1 at hotspot temperature of 300°C and 700°C

# 4. CONCLUSION

Natural ester showed for gap distances up to 40 mm the same or better AC dielectric properties than mineral oil. The determination of withstand voltage of natural esters by means of a Weibull distribution leaded to a lower value than by Normal distribution. The lower PD inception voltage and higher streamer propagation velocity indicate that natural esters in complex non-uniform electrical field stress conditions can manifest poorer breakdown strength properties than mineral oil. This is going to be investigated in conditions involving higher voltages and larger oil gaps.

Concerning the dissolved gas-in-oil analysis it was shown, that typical faults and stresses in transformers filled with natural esters lead to the generation of detectable fault gases. In case of partial discharges the gassing of natural ester is higher and for arcing lower than for mineral oil. The gas production in natural ester for thermal faults is high enough to be used for diagnostic methods too. With these findings it becomes evident, that for the existing faultdiagnostic interpretation schemes (Duval triangle, Doerenburg ratios, MSS, etc.) the limits have to be adjusted for natural esters.

In future other natural esters have to be investigated and compared. The influence of paper and pressboard and the effect of the dielectric fluids' aging on the gassing behaviour (and Kfactors) also have to be considered.

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