EXPERIMENTAL CHARACTERIZATION OF ESTER BASED OILS FOR THE TRANSFORMER INSULATION

Renza Berti
CESI RICERCA – Italy

Franco Barberis
CESI RICERCA – Italy

renza.berti@cesiricerca.it

ABSTRACT

With the aim to verify and compare the performances of more fire resistant and highly biodegradable dielectric fluids, such as the ester based oils, with conventional transformer oil, an experimental activity on a natural ester oil, a synthetic ester fluid and a naphthenic mineral oil was carried out, financed by the public Research Fund for Italian Electrical System.

In this paper the results of an accelerated thermal ageing cycle, at a hot-spot temperature in the range 140÷143°C on single-phase devices insulated with these fluids and paper, are presented and discussed. The most meaningful outcomes are:

1. *an extension of the paper and pressboard life using the ester based oils, also when these fluids are used to retro-fill a mineral oil device, measured through the degree of polymerization (DP) of the cellulosic insulation*
2. *an oxidation high in mineral oil but little evident in ester based fluids, when samples of the copper conductors of HV windings are compared.*

INTRODUCTION

The greatest majority of subtransmission transformers are paper-oil insulated and were designed for a 25÷30 years service life, however, many of them are operating well over these limits. The cost of their replacement is very high, but for utilities, in a case of failure, the costs could be enormous when related to the damages caused to the environment, to the users, to the adjoining equipment and to the general public image.

The risk of fire and of water and soil contamination in the substations can be reduced by replacing the mineral oil with fire resistant and highly biodegradable dielectric fluids, such as the ester based oils, both in the operating and in new transformers. This should also allow a saving of non renewable resources, such as crude oil, and the overcoming of the problems due to the oil availability and fluctuations in price.

With the aim to verify and compare their performances, an experimental activity was carried out on the following transformer fluids:

(a) a natural ester oil, manufactured in US, derived only from seeds with no gene manipulation technique, certified by EPA for its biodegradability and UL and FM approved as a less flammable fluid for transformers;

(b) a synthetic ester fluid, manufactured in UK, derived from alcohol pentaerythritol and a branched organic acid, adopted in compact railroad traction transformers for its excellent lubricity and FM approved as less flammable fluid for transformers;

(c) a mineral oil obtained from naphthenic crude oil severely refined, PCB and corrosive sulphur free, belonging to I and II classes both of IEC 296 and CEI 10-1 Standards.

The measurement of the main chemical-physical and dielectric properties of these fluids and the compatibility test, carried out in qualified laboratories according to national or international Standards, confirmed a very high value of flash point, aquatic biodegradability, breakdown voltage and high viscosity of the ester based oils and their good compatibility with mineral oil [1].

To study and to compare the behaviour of these fluids in service, an accelerated thermal ageing was carried out on five single phase devices: three filled with mineral oil, one with synthetic ester oil and one with natural ester oil.

The temperature is surely the most critical parameter because it can change the mechanical and electrical properties of the insulation materials. In pyrolysis of the cellulose macromolecules the supplied thermally vibrational energy cleaves the single bonds, C-H, C-C, C-O, producing CO_2, CO, H_2 and H_2O. At the end of the chain, the final molecule can be separated and converted to sludge or acids, by interacting with the oil components. Also the thermal ageing of the mineral oil is characterizated by chemical reaction and by production of H_2, CH_4, C_2H_4 and C_2H_6, meanwhile the ester based oils pyrolysis produce also a lot of CO e CO_2, due to the cleaving of the bonds in COO groups.

To measure the degradation of insulation, many techniques were used:

1. the routine analysis, with the evaluation of appearance, colour, acidity, particles, dissipation factor tg δ at 90°C, water content, breakdown voltage of the oil [2,3], the Dissolved Gases Analysis (DGA) [4] and the determination of furan compounds by High Performance Liquid Cromatography (HPLC) [5],
2. the degree of polymerization (DP) indicating the cracking of cellulose chains [6] and the determination of water in paper and pressboard by automatic coulometric Karl Fischer titration [7],
3. Polarization and Depolarization Current analysis (PDC) for determination of the moisture content in pressboard and the electrical conductivity of oil [8],

using a PDC Analyzer 1-MOD of ALFF Engineering.
This paper reports on the results of these tests performed on the paper-oil insulation of the experimental devices during the thermal ageing cycle and on a possible extension of the paper and pressboard life using the ester based oils for retro-filling two mineral oil filled devices at the mid ageing cycle.

EXPERIMENTAL SETUP

With the aim to reduce costs and dimensions, five 600 kVA single phase devices were designed and manufactured with the following features:

- 1090 mm in height and 860 mm in diameter,
- the high voltage HV winding wound on the low voltage LV winding, without ferromagnetic core,
- a large quantity of pressboard between the windings as in a 150 kV, 17,6A dielectric dimensioning, suitable for reliable measurement of oil and paper degradation with the PDC technique,
- a hermetically and non corrugated iron tank, equipped with an oil level indicator, a ground connection, a drain valve in the bottom, LV and HV bushings to connect the electric cables to the windings,
- a freely running oil flow inside the ducts and around the active part owing to the power to the HV winding resistance,
- four T-type thermocouples, inside the Kraft paper between the turns, at the top and at the bottom of every winding, for the temperature monitoring during the ageing cycle,

To reduce heat losses the devices were wrapped in an insulating material, 50 mm in thickness, and placed in the open air on a vat, to avoid soil contamination due to an oil spill. Some details of the devices are shown in Fig.1.

a) b) c)

Fig.1 Single phase device for thermal ageing: a) the tank, b) the cover with the active part and c) experimental area with insulated devices

The paper-oil insulation of each device, at the beginning of the thermal ageing, is described in Table 1.

Table 1: Insulation of the single phase devices at the beginning of the ageing cycle

Device number	*1*	*2*	*3*	*4*	*5*
Type of oil	mineral	mineral	mineral	synthetic ester	natural ester
Paper impregnation oil	mineral	mineral	mineral	synthetic ester	natural ester

An electric circuit was set-up to feed the HV winding of every device in parallel, with a constant current of 34 A at 20°C through a rectified voltage of ~ 190 V, and to allow the PDC Analyzer 1-MOD connection. Furthermore software was developed to acquire the temperature values of every device, to adjust them via the opening or closing the feed circuit and to control of the alarms in case of failures and in presence of hot points in windings.

ACCELERATED THERMAL AGEING CYCLE

The accelerated thermal ageing cycle lasted 118 days and was divided into the following steps :

- *Step 1*, lasted 40 days with the devices at an average temperature of 132 °C ± 3°C in steady conditions: at the beginning and at the end of the step, the moisture content in their solid insulation and the oil conductivity were determined with the PDC technique.
- *Step 2*, lasted 15 days with the devices at an average temperature of 130 °C ± 3°C in steady conditions: at the end of the step, the PDC measures were repeated and an oil sample was drawn for routine analysis, DGA and the determination of furan compounds by HPLC. Then the devices were disconnected from the electric circuit and opened. Every cover with active parts was raised for examination of the components and a photographic documentation of their condition. Pressboard samples were drawn from the upper and the lower angle ring for the DP measurement (see Fig.2).

Fig.2 Sampling of pressboard from the upper and lower angle ring of the active part of a device

Devices 4 and 5 were closed, whereas the tank of devices 1, 2 and 3 were filled with respectively unused synthetic ester oil, unused naphtenic oil and unused natural ester oil. Only mineral oil underwent a hot treatment before the filling operation. The devices were again insulated and connected to the electric circuit and PDC measurements were made on device 1, 2 and 3 with new oils.
The paper-oil insulation of each device, at the end of step 2 is described in Table 2.

Table 2: Insulation of the single phase devices at mid ageing cycle

Device number	1	2	3	4	5
Type of oil	synthetic ester	mineral	natural ester	synthetic ester	natural ester
Paper impregnation oil	mineral	mineral	mineral	synthetic ester	natural ester

- *Step 3,* lasted 63 days with the devices at an average temperature of 130 °C ± 3°C for 30 days and after at 122 °C ± 3°C in steady conditions: at the end of the step, the PDC measures were repeated at 20°C, and an oil sample was drawn for routine analysis, DGA, HPLC and aquatic biodegradability test [9]. Then the devices were definitively disconnected from the electric circuit and opened again, raising the active parts for an examination of the components and a photographic documentation of their condition. For DP measurement and the determination of water by Karl Fischer method, samples of pressboard were cut again in the upper and in the lower angle ring and in the middle of the pressboard cylinders pulled off the LV and HV windings. Paper and copper samples were cut from the HV and LV winding connections to the bushings.

Fig.3 shows the highest temperatures, measured at time intervals Δt = 600s in every device, during the accelerated thermal ageing.

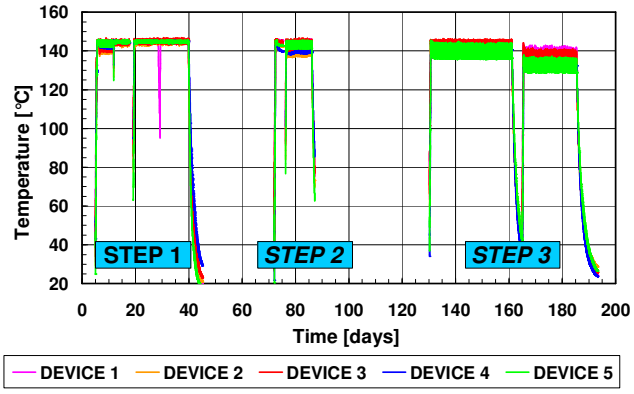


Fig.3 The highest temperatures measured during the accelerated thermal ageing cycle.

These temperatures, in the range of 140÷143°C, were used as the winding hot-spot temperatures T_{hsi} to calculate the loss of life L of each device insulation , according to IEC 60076-7 Standard [10]:

$$L = \int_{t_1}^{t_2} 2^{(T_{hs}-98)/6}\, dt \approx \sum_{i=1}^{N} V_i \times \Delta t \qquad (1)$$

where $V_i = 2^{(T_{hsi}-98)/6}$ is the ageing rate referred to T_{hsi} in °C, measured at Δt and considered constant in this interval. The value of hot-spot factor H was assumed 1.

L represents the operating time at normal thermal condition (90°C) to produce the degradation of the paper-oil insulation like the degradation due to accelerated thermal ageing. Table 3 shows the values of L, calculated with the equation (1): in the steps 2 and 3, the differences among the L values of device 1 and 3 and the L values of the other devices are due to a progressive detachment of the thermocouple positioned at the bottom of LV winding. This could be verified only at the end of the cycle.

Table 3: Operating times to produce the same degradation of the paper-oil insulation as in the thermal accelerated ageing

Cycle step	*Length step(days)*	*Loss of life L (days)*				
		1	2	3	4	5
1	40	18.10	18.20	18.90	18.60	18.90
2	15	8.20	6.80	7.70	6.10	6.50
3	63	19.00	13.00	16.70	13.50	13.20
Total	118	45.30	38.00	43.30	38.20	38.60

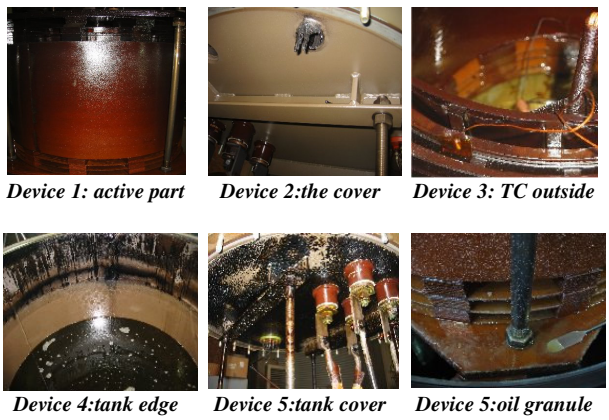
TEST RESULTS AND DISCUSSION

The main results at the end of the accelerated thermal ageing carried out on the single phase devices can so be resumed:

Inspection of the opened devices

At mid ageing cycle devices 1, 2 and 3, filled with mineral oil, did not show deposits along the free internal surface of the tank, meanwhile devices 4 and 5 presented blackish deposits on the internal surface of the cover and along the internal edge of the tank. At the end of the ageing, these deposits were much more remarkable and were present along the internal edge of the tank also in devices 1and 3, retrofilled with ester based oils. Only in device 2, aged in mineral oil for the whole cycle, there were deposits only under the valve for inspection on the cover, as if the mineral oil was less sensitive to oxidation. To avoid the presence of air in the tank, the ester base oil manufacturers suggest to use a dry nitrogen cushion. Moreover in devices 1, 3 and 4 a dark sludge layer, more dense in device 4, was present on the angle rings and on the wood base. In device 5 there were a lot of little granules on all free surfaces, due to the precipitation of the saturated fatty acids of vegetable oil when temperature lowers. In all the devices the pressboard cylinders around the active part showed a thermal gradient from top to bottom.

In Fig.4 are shown some images of the internal surface of the tank and the active part of the devices.



Device 1: active part ***Device 2:the cover*** ***Device 3: TC outside***

Device 4:tank edge ***Device 5:tank cover*** ***Device 5:oil granule***

Fig 4 Images of the internal part of the devices at the end of cycle

Examination of copper samples, cut from HV winding connections

The copper oxidation was high in samples cut from the HV winding of devices 1, 2 and 3, aged in partially or entirely in mineral oil, but little evident in samples aged only in ester based fluids, specially in the synthetic ester oil (see Fig.5). Moreover, the surface aged only in mineral oil was dull, while the other samples presented a bright surface, particularly the sample aged only in synthetic ester oil, probably due to their high lubricity.

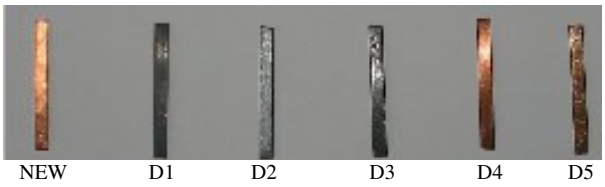


Fig 5 Comparison of the copper samples cut from HV winding of each device

Tests on oils

The main results of the routine analysis, the DGA and HPLC are reported in Table 4. Referring to the limits given by Standards for a mineral oil in service, the fluid in device 2 showed only a higher water content and a colour change due to a further paper degradation. The ester based oils in devices 1 and 3 showed the values of tgδ and acidity much higher than Standard limits owing to the contamination coming from pressboard and to the oil oxidation. The synthetic and natural ester oils respectively in device 4 and 5, presented a very high degradation as they suffered the whole ageing cycle.

The DGA for oils of devices 1÷4 showed a very high value of CO_2/CO ratio, but smaller than the values obtained in the analysis made at mid ageing cycle: this indicated no faults in the devices. On the contrary in device 5 the value of CO_2/CO ratio was much higher than the old value, indicative of a thermal fault: this was confirmed by carbonization traces on the surface of the laminated paper near the HV winding. The value of O_2/N_2 <0.3 and the high value of CO_2/CO in all fluids indicated a high cellulose degradation, confirmed by HPLC tests.

Mineral oil showed a much higher 2FAL value in comparison to the other fluids, in spite of its ageing was shorter than the ageing of the ester based oils in devices 4 and 5 (whole cycle) and of the ester based oils in devices 1 and 2 (step 3 of the cycle). The data confirmed a greater production of compounds, related to paper ageing in mineral oil, and their considerable reduction when the devices were retrofilled with the ester base oils.

The aged fluids of devices 4 and 5 confirmed again their high values of aquatic biodegradability.

Table 4: Results of measurements on oils at the end of thermal ageing The value with (*) are measured at mid cycle

Device number	*1*	*2*	*3*	*4*	*5*
Colour (ASTM color unit)	2.5	L 1,5	L 1,5	DIL L1,5	DIL 0,5
Acidity(mg KOH/g)	7.46	0.03	6.56	10	12.1
Particles (%)	0.01	0.01	0.02	0.03	0.04
Water content (ppm)	50	15	49	77	77
Breakdown voltage (kV)	56	49	86	47	57
tg δ at 90°C	1.745	0.0039	0.3869	2.855	1.398
O_2/N_2 (*)	0.04	0.06	0.08	0.01	0.01
O_2/N_2	0.02	0.03	0.03	0.02	0.02
CO_2/CO (*)	13.63	15.35	18.62	15.59	9.62
CO_2/CO	8.22	13.01	13.21	12.47	15.02
2FAL (mg/kg) (*)	27.63	33.23	39.44	5.01	9.55
2FAL (mg/kg)	2.69	26.28	9.74	4.32	5.25

Tests on paper and pressboard

The graphic in Fig.6 shows the comparison of the DP values of all paper and pressboard samples, drawn from the angle rings and the cylinders between LV and HV windings in every device (see Fig.6). All DP values confirmed a very high degradation and the end of life of cellulose insulation, just as that one expected in a thermal ageing at 140°C for ~ 3.5 months.

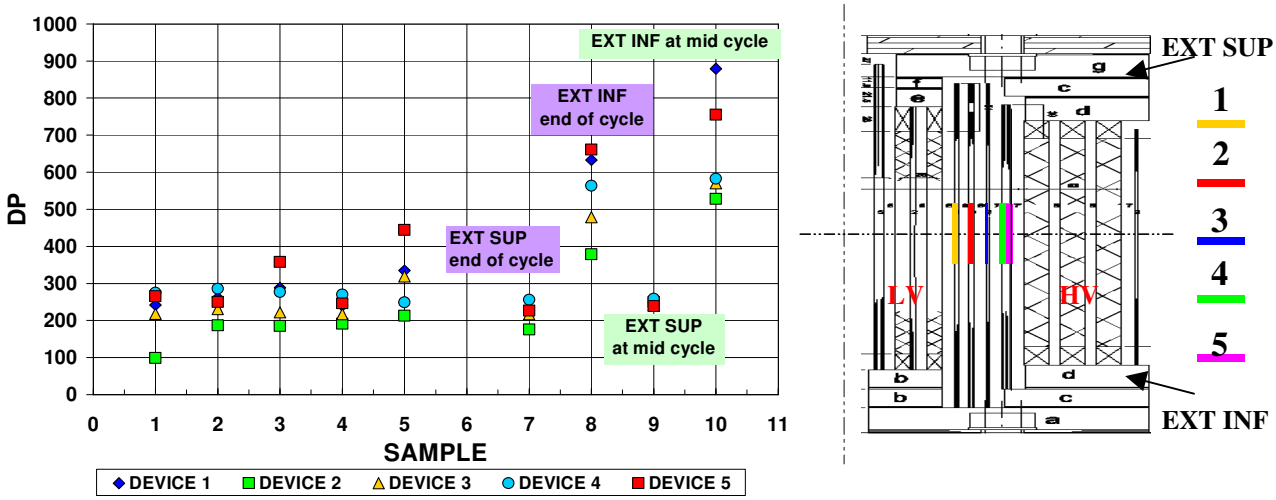


Fig6 Comparison of the DP values evaluated for the samples, drawn and numbered according to the device active part scheme on the left

Owing to the thermal gradient due to the oil natural convection, DP values reduced from the top to the bottom of the windings in every device. DP of device 2 were the smallest, while the samples, aged for much longer time in device 1 and 3, presented DP values a little higher than the device 2 ones. The retrofilling operation, with the ester based oils, could have to slow down the cellulose degradation, in spite of their greater water content. Some authors [11,12] propose that the natural ester oil has a function of water scavenging: at elevated temperature the oil can undergo hydrolysis, consuming the available water from the cellulose with a paper drying effect.

The water content values, measured according to the Karl Fischer method are showed in Table 5. The pressboard samples 1 to 4 of every device gave values of moisture <1%. The sample 5, in laminated paper, near the HV winding, presented moisture values ranging from 0.76% for device 5 to 2.5% for device 1. This was probably due to a non-equilibrium of the water content inside the solid insulation when the samples were drawn. In any case there was a good agreement between the DP and water content values for all the samples.

Table 5: Water Content in % of the solid insulation between LV and HV windings

Sample	*Thickness (mm)*	*Device*				
		1	*2*	*3*	*4*	*5*
1	2.5	0.92	1.11	0.52	0.86	0.6
2	3	0.55	0.76	0.46	0.49	0.46
3	2	0.86	1.14	0.58	0.86	0.48
4	4	0.39	0.71	0.49	0.6	0.45
5	0.6	2.49	1.12	1.45	1.12	0.76

PDC measurements on paper-oil insulation

The tests were performed with a PDC Analyzer 1-MOD, applying a DC voltage step of 500 V between HV an LV windings for a polarization time T_p of 10000s. The charging current, flowed in the insulation system of the device, was acquired and then the windings were short-circuited for a depolarization time T_d of 10000s during which the current goes gradually towards zero. A model of the experimental device insulation was used (see Fig.7) according to the linear dielectric theory and from a best fit between measured and calculated relaxation currents, the values of moisture content UR% in the solid insulation and of the oil conductivity σ were obtained.

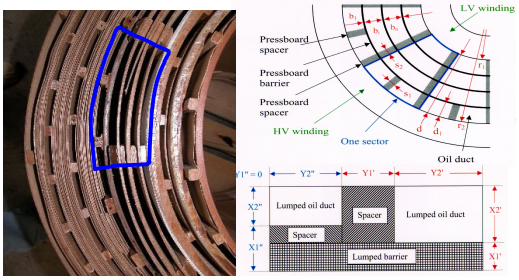


Fig.7 Cross section of the insulation system between HV and LV windings of each device and its simplified geometry model

The results are reported in Table 6, were the red line indicate the replacement of oil in device 1, 2 and 3.

Table 6: Moisture content in the solid insulation between LV and HV windings and oil conductivity (B=before the step, E=at the end of the step)

Parameter	*Cycle Step*	*Device*				
		1	*2*	*3*	*4*	*5*
UR%	1 B	1.5	1.5	1.5	2.5	2.3
	1 E	1.7	2	2	3	4
	2 E	1.7	2	-	3.5	4.5
	3 B	3	2.4	3	-	-
	3 E	3.5	2	3.3	4.5	4.5
σ oil (pS/m)	1 B	0.29	0.26	0.36	4.36	6.7
	1 E	0.125	0.045	0.14	32	93
	2 E	0.27	0.19	-	76	179
	3 B	5.4	1.2	5.6	-	-
	3 E	0.9	0.24	130	280	201

The data confirmed the increase of the moisture content in solid insulation and of the oil conductivity, during the thermal ageing, in all the devices. But at the end of the ageing cycle, UR% values of the ester oil impregnated paper were already higher than the values determined by Karl Fisher method. Moreover, when the mineral oil was replaced by ester based oils in device 1 and 3, PDC measurements were carried out before the replacement (2E) and before the start of step 3 (3B): the moisture of solid insulation turned out higher than the one measured before the retrofilling, even if the devices were not energized between the two PDC measurement. This could be due to the higher water content admitted by the Standards for the syntethic ester oils (used here also for natural ester oil), not suitable for the diagnostic tool of PDC Analyzer, setup for in service mineral oil with a much lower water content.

CONCLUSION

The main results at the end of the accelerated thermal ageing carried out on the single phase devices, were:

- the degradation of the cellulose insulation, impregnated ad aged in mineral oil was more accelerated than the degradation of the pressboard impregnated and aged in the ester based oils, in spite of their much higher water content. Because of their higher water saturation level the ester based oils can absorb more water than mineral oil and have a greater paper drying effect;
- an extension of life of the paper and pressboard life using the ester based oils was proved through the measurement of the polymerization degree (DP) of the cellulosic insulation, also when these fluids are used to retro-fill the mineral oil device;
- mineral oil showed a much higher 2FAL value in comparison to the other fluids, in spite of its ageing was shorter than the ageing of the ester based oils. The data confirmed a greater production of compounds,

related to paper ageing in mineral oil, and their considerable diminution when the devices were retrofilled with the ester base oils;

- the copper samples of the HV winding conductors, showed an oxidation, high in mineral oil, hardly to be seen in ester based fluids. The surface aged only in mineral oil was dull, while the other samples presented a bright surface, particularly the sample aged only in synthetic ester oil, probably due to their high lubricity;
- PDC technique could not be suitable to assess the insulation condition in presence of ester based oils owing to the diagnostic tool of PDC Analyzer, setup for in service mineral oil with a much lower water content;
- European Standards, setup for synthetic and natural ester oils, could allow a higher diffusion of these transformer fluids in European electrical market.

A synthetic description of the results is shown in Table 6.

Table 6: Visual comparison of the performances of the paper-oil insulation at the end of accelerated thermal ageing (M=mineral, SE=synthetic ester, NE= natural ester)

Device number	*1*	*2*	*3*	*4*	*5*
Oil/Paper impregnation oil	SE/M	M/M	NE/M	SE/SE	NE/NE
Oil loss of life *(days)*	19.00	13.00	16.70	38.20	38.60
Paper loss of life *(days)*	45.30	38.00	43.30	38.20	38.60
DP values					
Water content in pressboard					
Copper oxidation					
Deposits and sludge					
2FAL values					
LOW PERFORMANCE	**MIDDLE PERFORMANCE**			**HIGH PERFORMANCE**	

Acknowledgments

This work has been financed by the Research Fund for Italian Electrical System established with Ministry of Industry Decree DM 26/1/2000.

The authors sincerely thank all persons and the companies who made this research possible: J.Borghetto, R. Passaglia, P.Mazza (CESI RICERCA SpA), M.Saottini, (CESI SpA) H. Athanassatou, C.P. Mc Shane (Cooper Power Systems), M. Molteni (Erba Isolanti Srl), V.Milesi, R.Reboldi (TES Srl), J.O'Neill (M&I Materials Ltd), F.Mauri (ENEL Distribuzione Roma)

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