

Study Committee D1
Materials and Emerging Test Techniques
Paper D1-PS2-10179

IMPORTANCE OF DISSOLVED OXYGEN CONTROL ON ACCELERATED AGING TESTS FOR MINERAL AND NATURAL ESTER INSULATING OILS

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Motivation

- Mineral insulating oil (MIO) oxidation produces acidic compounds responsible for accelerating insulating paper degradation. Advanced oil oxidation can lead to "sludge" formation, which is an oil-insoluble material and forms deposits that hinder transformer cooling.
- In spite of being less stable to oxidation, natural ester insulating oil (NEIO) forms degradation products less harsh to insulating paper, there is no sludge formation and acids are weaker than those released by MIO. The main effect of NE degradation is viscosity increase along with oxidation and, when viscosity rises above 10% compared to the initial value, reducing oil viscosity through regular regeneration process is unfeasible. Among factors that led to NE and MIO oxidation, oxygen concentration and temperature can be mentioned.
- To prevent early oil regeneration or change, controlling oxygen intake is very important. Regarding the differences between NEIO and MIO, it is assumed that oxygen level limits to maintain adequate condition of both fluids are different.

- NEIO and MIO aging at required oxygen content was carried out in stainless steel cells containing metallic copper (catalyst) under vacuum.
- Accelerated aging was performed at controlled temperature (200 °C) and oil conditions were evaluated at time intervals of 326, 768, 980 and 1175 h, identified as t1, t2, t3 and t4, respectively

Physicochemical analysis performed in aged MIO and NE samples

Analysis	Standard	MIO	NEIO
Color	Visual	x	x
Total acidity number – TAN (mg KOH g ⁻¹)	ABNT NBR 14348	x	x
Power factor (%)	ABNT NBR 12133	x ^a	x ^b
Interfacial tension (mN m ⁻¹)	ABNT NBR 11341	x	
Water content (ppm)	ABNT NBR 10710	x	x
Viscosity at 40 °C (cSt)	ABNT NBR 10441		x
Flash point (°C)	ABNT NBR NBR 10441		x
Sludge (%)	IEC 61125	x	
di-tert-butyl peroxide – DBPC (%)	ABNT NBR 12134	x	
Oxidation by Fourier transform infrared spectroscopy – FTIR (absorbance)	Internal method	x	
Dissolved gas analysis (DGA)	ABNT NBR 7070	x	x

^a Power factor measured at 90 °C; ^b Power factor measured at 25 °C.

Objective of investigation

To study the effect of different oxygen concentrations on MIO and NEIO in accelerated oxidation ageing tests.

Test results

Physicochemical analysis of non-used and aged MIO samples

Sample	Oxygen concentration (ppm)	Aging time (h)	Power factor at 90 °C (%)	Water content (ppm)	Interfacial tension (mN m ⁻¹)	DBPC (%)
Non-used MIO		0	0.05	11	43	0.28
t1MIO1	~3000	326	0.36	64	38	0.26
t2MIO1		768	0.34	44	45	0.24
t3MIO1		980	0.24	46	46	0.22
t4MIO1		1175	0.72	41	44	0.21
t1MIO2	~5000	326	0.25	65	37	0.26
t2MIO2		768	0.41	47	44	0.22
t3MIO2		980	0.17	48	46	0.22
t4MIO2		1175	0.09	33	47	0.21
t1MIO3	~10000	326	0.35	70	40	0.25
t2MIO3		768	0.26	49	43	0.24
t3MIO3		980	0.31	49	44	0.23
t4MIO3		1175	0.33	35	41	0.22
t1MIO4	~15000	326	0.16	69	44	0.25
t2MIO4		768	0.41	55	43	0.20
t3MIO4		980	0.36	46	41	0.19
t4MIO4		1175	0.74	38	38	0.19
t1MIO5	~20000	326	0.20	84	40	0.24
t2MIO5		768	0.29	40	43	0.25
t3MIO5		980	0.37	47	45	0.24
t4MIO5		1175	0.47	41	43	0.23

Method/Approach

- Physicochemical parameters of aged MIO and NEIO were compared with limits defined in Brazilian standards for NEIO and MIO immersed transformers (ABNT NBR 16518 for NEIO and ABNT NBR 10576 for MIO).
- A polyunsaturated NEIO was used.
- To assess effect of different oxygen concentrations on oxidation susceptibility MIO and NEIO samples were prepared with five different oxygen volume concentrations (~3000 ppm, ~5000 ppm, ~10000 ppm, ~15000 ppm and 20000 ppm). Dry nitrogen and pure oxygen bubbling were used to achieve desired gas concentration.

Experimental setup

Identification of MIO and NE samples according to their initial oxygen concentration

Identification	Oxygen concentration (ppm)
MIO1	~3000
MIO2	~5000
MIO3	~10000
MIO4	~15000
MIO5	~20000
NE31	~3000
NE32	~5000
NE33	~10000
NE34	~15000
NE35	~20000

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(continued)

Test results

Physicochemical analysis of non-used and aged NE samples

Sample	Oxygen concentration (ppm)	Aging time (h)	TAN (mg KOH g ⁻¹)	Power factor at 25 °C (tan δ)	Water content (ppm)	Viscosity at 40 °C (cSt)
Non-used NE		0	0.10	0.08	150	34.11
e1 NE31	~3000	326	1.08	0.42	24	41.02
		768	1.37	0.48	49	49.25
		980	1.61	0.74	101	50.36
		1175	1.79	0.61	46	43.00
e1 NE32	~3000	326	0.97	0.60	21	37.79
		768	1.42	0.82	54	45.70
		980	1.42	0.94	81	46.11
		1175	2.35	1.11	71	46.83
e1 NE33	~10000	326	1.11	0.52	263	36.72
		768	1.43	0.48	63	41.90
		980	1.54	0.94	51	44.45
		1175	1.39	0.70	29	43.16
e1 NE34	~15000	326	1.80	0.43	325	47.90
		768	2.33	0.58	61	62.23
		980	1.99	0.54	116	59.91
		1175	2.17	0.82	33	53.85
e1 NE35	~20000	326	1.15	0.41	31	37.31
		768	1.73	1.10	43	42.93
		980	1.42	0.14	56	44.63
		1175	1.66	0.60	36	44.39

DGA analysis of non-used and aged MIO samples

Sample	Aging time (h)	H ₂	CO	CO ₂	C ₂ H ₄	C ₂ H ₂	C ₂ H ₆	C ₃ H ₈
Non-used MIO	0	17	0	90	0	0	0	0
e1 MIO1	326	27	242	2986	92	42	19	0
	768	17	116	2525	47	22	14	0
e1 MIO1	980	49	285	7362	150	56	29	0
	1175	26	310	5138	130	57	34	0
e1 MIO2	326	29	263	3598	121	56	24	0
	768	28	160	3302	93	44	18	0
e1 MIO2	980	43	81	6917	124	42	23	0
	1175	52	93	8139	122	42	27	0
e1 MIO3	326	25	165	3441	107	114	20	0
	768	19	185	3600	83	42	19	0
e1 MIO3	980	21	284	3741	109	55	24	0
	1175	22	219	4737	109	60	27	0
e1 MIO4	326	18	148	3634	65	27	12	0
	768	54	394	12743	347	111	54	0
e1 MIO4	980	84	360	13796	300	77	49	0
	1175	61	372	5720	165	72	36	0
e1 MIO5	326	45	498	6096	253	106	39	0
	768	23	203	3819	111	57	26	0
e1 MIO5	980	41	279	3847	128	61	23	0
	1175	44	375	5557	175	71	33	0

DGA analysis of non-used and aged NEIO samples

Sample	Aging time (h)	H ₂	CO	CO ₂	C ₂ H ₄	C ₂ H ₂	C ₂ H ₆	C ₃ H ₈
Non-used NE	0	24	5	417	0	31	1	0
T1 NE1	326	106	399	4236	34	585	67	0
	768	64	256	9536	61	524	63	0
	980	46	297	6424	36	561	60	0
	1175	56	168	14463	75	622	74	0
T2 NE1	326	323	274	3827	21	549	53	0
	768	166	271	6033	34	623	63	0
	980	49	250	5909	32	543	62	0
	1175	21	219	7069	8	785	33	0
T3 NE1	326	66	286	7143	42	618	58	0
	768	177	364	8741	65	609	68	0
	980	222	394	8196	38	704	76	0
	1175	41	170	3935	27	417	49	0
T4 NE1	326	90	363	9276	74	792	75	0
	768	46	448	7523	58	658	56	0
	980	58	583	11182	60	851	71	0
	1175	66	598	9034	48	643	67	0
T1 NE4	326	71	447	10598	67	783	98	0
	768	225	410	7575	38	686	71	0
	980	224	402	15463	100	832	88	0
	1175	24	5	417	0	31	1	0

Conclusions

- This study allowed to conclude that different concentrations of oxygen did not affect MIO behavior during testing as shown by unchanged physicochemical characteristics, with exception of **water content** that was increased in all aged samples.
- NEIO aged oils presented significant increase on TAN and viscosity under all oxygen concentration.
- Laboratory accelerated aging tests are a powerful tool for insulating oils properties evaluation such as thermal class. To establish a useful standard procedure for NEIO is necessary to define a range of acceptable oxygen concentration for this oil, moisture and maximum test temperature as well. Therefore, a standard aging test procedure for NEIO with oxygen, water and aging temperature limits must be defined. Further studies are going on to contribute on this goal.