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THE USE OF ACTIVATED ALUMINA FOR RECLAIMING AND RECONDITIONING
TURBINE, TRANSFORMER AND CIRCUIT BREAKER OILS

by

Thomas O. English

A

T H E S I S

Submitted to the faculty of the
SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI
in partial fulfillment of the work required for the
D E G R E E O F
ELECTRICAL ENGINEER

Rolla, Mo.

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Approved by

F. H. Frame

Professor of Electrical Engineering.

STATEMENT

The results obtained and reported in this thesis were obtained at my direction, or under my supervision, during the period of Nov. 5, 1930 and Jan.30, 1935, while employed as Electrical Engineer by the Aluminum Ore Company; with the exception of those results reported as being done at Alcoa, Tennessee - which work was supervised by Mr.J.E.Housley, Electrical Superintendant.

T.O.ENGLISH.

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THE USE OF ACTIVATED ALUMINA FOR RECLAIMING AND RE-
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BREAKER OILS.

Advancements during the past few years have provided oil manufacturers with ample reason to claim that good oils no longer wear out because of use, but that deterioration by various destructive agencies such as moisture, air, heat, oxidation, emulsification and electro-chemical action may have the effect of rendering the oil totally unfit for use.

Turbine manufacturers and lubricating engineers have cooperated to provide the best of lubricating systems for turbine use. Many improvements have been made, but because of these certain destructive agencies the average life of turbine oil has been somewhat limited. One of the most important jobs left for the operating engineer is that of getting the maximum possible service from these oils which the refiners have improved for attaining that objective by the use of a material known as Activated Alumina.

TURBINE OIL

Oxygen is considered the most active and destructive agent in the deterioration of turbine oil. It is absorbed in proportion to the amount of oil exposed.

Because moisture continuously finds its way into turbine oil in spite of all precautions, either by the way of packing glands, small leaks at the oil cooler, or by condensation, petroleum acids are formed, in the presence of water, by the oxidation of the hydro-carbon compounds in the oil.

Heat ranks next to oxidation as a destructive agent of oil. The presence of heat and water and the resultant insoluble impurities accelerate the rate at which further oxidation, with production of additional acid, takes place in the oil. Because the trend of modern turbine design and operating practice is toward higher steam pressures and temperatures, more heat is consequently transmitted to the bearings contiguous to the steam end of the turbine. There is no relief, therefore, from high oil temperatures.

The most serious effects of these destructive agents are: Governor trouble due to sluggishness caused by sludge; increased temperature of oil and bearings; emulsions resulting usually from the entrainment of water and air; reduction of the lubricating quality of the oil; coated and clogged cooler-tubes; poor circulation of oil in system making necessary more frequent cleaning of oil system and replacements of oil;

more frequent shutdowns, with the resulting increase in cost of producing each kilowatt. Numerous other effects could be given, and all hamper ideal operation in one way or another, so that any positive method for eliminating or correcting any of these destructive agencies should be well received by everyone concerned.

An aluminous material of new and unusual form has recently been developed in the Aluminum Research Laboratories of the Aluminum Company of America. It is a granular form of alumina that possesses a very large surface area per unit of weight. It is sold under the trade name of "Activated Alumina". A typical chemical analysis is as follows:

Al ₂ O ₃ -----	91.23%
Loss on Ignition---	7.10%
Na ₂ O-----	.80%
SiO ₂ -----	.08%
Fe ₂ O ₃ -----	.03%
TiO ₂ -----	.01%

Soon after Activated Alumina became known as a most efficient and satisfactory adsorbent for gases, vapors, etc., a series of laboratory tests were performed to ascertain its effect on turbine oil. A sample of discarded, filtered turbine oil was heated to

120°F. - which is approximately the temperature of the oil in service - in the presence of a mass of 8 to 14 mesh Activated Alumina. The sample was agitated for varying periods with the following reduction in Neutralization Number or Acidities. (Neutralization Number or Acidity is expressed as the number of milligrams of potassium hydroxide required to neutralize one gram of oil):

	<u>N.N.in Mg.KOH per gm.of oil</u>
Original oil-----	.955
15 minutes-----	.740
30 minutes-----	.660
60 minutes-----	.430

This favorable reduction in acidity indicated that the oil was susceptible to improvement by treatment with Activated Alumina.

In these experiments, qualitative tests showed that an opaque, black oil could be clarified to the pale straw color of new oil by varying the length of time of treatment, or the temperature of the oil. When the treatment was made under conditions that enabled the oil to come in contact with the Activated Alumina by convection or gentle flow, the acidity and color were both decreased and the product was free of turbid-

ity.

Since the laboratory tests had proved so successful, one on a much larger scale was started. The Aluminum Ore Company was in possession of 250 gallons of discarded turbine oil, which had been used continuously for a year. It was very dark, contained considerable sludge and the Neutralization Number was .9. This was obviously a splendid sample for such a test. One hundred pounds of Activated Alumina was submerged in a wire basket in the oil under which a coil supplied with boiler feed water at 212^oF. was placed to heat the oil and create some circulation of it thru the Activated Alumina. The complete analysis of this oil before the treatment and after two months of treatment is given in the following table:

	<u>BEFORE</u>	<u>AFTER</u>
Specific Gravity	.8720	.8710
Viscosity @ 104 ^o F.	150 Sec.	148 Sec.
Flash	408 ^o F.	410 ^o F.
Fire	458 ^o F.	464 ^o F.
Pour	14 ^o F.	16 ^o F.
B.S.& W.	.05 %	Trace
Neutralization No.	.9 <u>Mg.KOH</u> gm.oil	.047 <u>Mg.KOH</u> gm.oil

For purposes of comparison, the following analysis of new oil is given:

Specific Gravity	.8680
Viscosity @ 104 ^o F.	145 Sec.
Flash	406 ^o F.
Fire	460 ^o F.
Pour	8 ^o F.
B.S.& W.	None
Neutralization Number	.01

From these analyses it is readily observed that there is but little difference between the new oil and treated oil and that no objectionable change of properties took place in the oil during treatment. The color was restored to almost that of new oil and this batch of reclaimed oil is being used for make-up to the oil in turbine service.

From this test there was reason to believe that if such oil could be reclaimed so successfully, oil that had not become so badly contaminated could be brought up to and kept at high quality for an indefinite period by continuous treatment while in service. One year of continuous service had constituted the useful life of oil in the Company's 7500 K.W. Allis-Chalmers turbine, even though a Nugent by-pass

filter of standard design was in continuous service. At the end of one year the oil would have a Neutralization Number of about .9, be very dark in color, contain much sludge and was usually ready to be discarded.

A continuous by-pass treater was designed and installed for use on the turbine. The principal features of the treater are three trays filled with 4 to 8 mesh Activated Alumina and having baffles that cause the oil to take the longest route in running by gravity through the device. After leaving the treater, the oil flows through the Nugent filter unit as a precautionary measure to prevent accidental inclusion of particles of Activated Alumina in the treated oil. The treater contains 140 lbs. of Activated Alumina. The turbine oil reservoir and accessories contain 300 gallons of oil. The oil is by-passed through the treater at the rate of 35 gallons per hour. When the treater was first put in service, March 15, 1932, the oil had been in service since November 1, 1931. Its Neutralization Number was .05 and it was still considered good. Immediately after the treater was put in service, the Neutralization Number of the oil had begun to diminish and continued to do so as shown in the following table:

	<u>Date</u>	<u>Neutralization Number</u>
Start.....	3-15-32	.050
	3-16-32	.040
	3-17-32	.040
	3-18-32	.040
	3-21-32	.030
	3-23-32	.023
	3-30-32	.020
	4- 4-32	.013
	4-27-32	.011
	5-12-32	.009
	5-25-32	.006
	6 -1-32	.003

After the oil had reached a Neutralization Number of .003, the acidity began to increase very slowly until September 6, 1932, when the Neutralization Number was .022. This Neutralization Number was maintained until March 15, 1933, at which time the treater was discontinued. After discontinuing the treater, the acidity began to increase as shown in the following table:

	<u>Date</u>	<u>Neutralization Number</u>
	4 -5-33	.034
	4-26-33	.039

<u>Table</u> <u>Cont'd</u>	<u>Date</u>	<u>Neutralization Number</u>
	5-17-33	.045
	6 -7-33	.050
	6-22-33	.056
	7 -5-33	.056
	8 -2-33	.056
	8-30-33	.078
	9-21-33	.056
	11-1-33	.062
	12-27-33	.067
	1 -18-34	.073

On March 2, 1934 when the Neutralization Number was .073, the treater was again put in service, retaining the same Activated Alumina first used. By March 14th, the acidity was down to .039. This low point was of short duration, however. By March 21st, the acidity had increased to .045. This Neutralization Number continued until May 17th, when the turbine was taken down for a general inspection and the oil was placed in storage.

The complete analyses of the oil before the treater was placed in service, after it had been in service three weeks and after 18 months, was recorded as follows:

	<u>Before</u>	<u>Three Weeks</u>	<u>Eighteen Months</u>
Specific Gravity	.8700	.8700	.8720
Viscosity @ 104°F.	148 Sec.	145 Sec.	150 Sec.
Flash	413 °F.	410 °F.	404 °F.
Fire	462 °F.	463 °F.	458 °F.
Pour	10.5	11 °F.	10 °F.
B.S.& W.	.05	None	None
Neutralization No. $\frac{\text{Mg. KOH}}{\text{gm. oil}}$.05	.0135	.062

From the foregoing, it is evident that the lubricating quality of the oil was maintained and that the rate of breakdown as evidenced by acidity was kept very low. Perhaps the most significant fact shown is that the oil is essentially the same as new and is available for further use after two and one-half years of continuous service. Prior to these experiments, the average life of oil under the same conditions was one year.

TRANSFORMER OIL

The principal cause of deterioration of transformer oil like turbine oil is oxidation, which is accelerated by heat and moisture. When oxidation occurs in a transformer oil to the extent that sludge begins to precipitate, the windings and cores become coated with sludge. The resultant higher acidity of the oil

is injurious to insulation, and eventually breaks it down - a condition that should not be tolerated. For this reason, most users of transformers either filter or centrifuge the oil periodically to remove sludge. However, filtering or centrifuging does not remove acid in solution. Oil should be thrown away when the acidity has reached a Neutralization Number of .7, because it is then impossible to maintain satisfactory dielectric strength or freedom from excessive sludge. Oil having a Neutralization Number of .2 and .3 may be used satisfactorily, but it requires close observation and attention. Oil frequently increases in acidity from a Neutralization Number of about .2 to .7 in much less time than is required for the acidity to increase from the original low figure to .1.

In conjunction with the laboratory and field experiments on turbine oil with Activated Alumina conducted by the Aluminum Ore Company, similar experiments were made on transformer oil. Preliminary laboratory tests indicated that used transformer oil is also susceptible to treatment and that such oil, black in color, high in acid, and containing much sludge, can be restored to its original straw color and Neutralization Number. The experiments also showed that the dielect-

ric strength is increased from 15 KV. to 40 and sometimes to 50 KV.

Two methods were chosen for field application of Activated Alumina to transformer oil. The first method, which was used at the Aluminum Ore Company, was applied by placing bags of Activated Alumina in the transformer. The treatment of the oil in this application is dependent on convection currents. The second method was used at Alcoa, Tenn. The treatment of the oil in the second method is accomplished by the use of a by-pass treater similar to the one in use on the Aluminum Ore Company's turbine.

A bank of 3 - 200KVA, Type HE, 2300/460 - 230 volt, Moloney transformers was chosen for the first application because it was loaded to full capacity, contained oil of a Neutralization Number of approximately .8 and difficulty had been experienced in maintaining the oil at a proper dielectric strength. This bank will hereafter be referred to as transformers numbers 1,2 and 3. Before placing the Activated Alumina in transformer #1, a considerable portion of the oil was taken out for laboratory work. New oil was substituted for that removed, thereby reducing the Neutralization Number in #1 to .19. On May 13, 1932, one hundred

pounds of Activated Alumina were put into each of three Osnaburg cloth bags and one bag suspended in each transformer. A complete analysis of the oil before and during the treatment showed, as did the turbine oil analyses, that the changes in characteristics were almost nil, with the exception, however, of marked reduction in Neutralization Number and marked increase in dielectric strength. The following tabulation shows the improvement in Neutralization Number:

<u>Date of Sample</u>	<u>Neutralization Numbers</u>			
	<u>Transformers</u>	<u>#1</u>	<u>#2</u>	<u>#3</u>
5-13-32 (Activated Alumina was applied.)		.190	.890	.710
5-19-32		.080	.490	.330
6 -6-32		.017	.191	.135
6-17-32		.022	.106	.101
7 -6-32		.011	.063	.056
8 -1-32		.017	.039	.039
1 -4-33		.020	.025	.034
11-1-33		.022	.039	.036
6 -7-34		.022	.034	.036
12-1-34		.020	.039	.026

Dielectric tests made at irregular intervals on the oil of these transformers showed that it quickly came up to and maintained a dielectric strength of 25 KV or better thereafter. No filtering has been done nor

has the oil received any attention except periodic sampling. The color and all other characteristics are now the equivalent of new oil. The Neutralization Numbers have remained almost constant for the past two and one-half years despite the fact that the original Activated Alumina is still in use. Obviously, if a poor transformer oil, high in acid and sludge and low in dielectric strength, can be so successfully restored to a good condition then a relatively small amount of Activated Alumina should remove acid, water and sludge and maintain a high dielectric strength in good oil for an indefinite period of time. The Neutralization Number and dielectric strength are the two governing characteristics of good transformer oil. Since they can be held or preserved at a favorable value by a continuous contact with Activated Alumina, the necessity for periodic filtering, centrifuging or replacing transformer oil is apparently eliminated by the use of this new material.

On the results of the tests herein above described, the Aluminum Ore Company applied Activated Alumina in all its power transformers, consisting of 22 banks, ranging from 75 to 3000 KVA capacity. The quantity of Activated Alumina used was calculated to

remove all existing acidity from the oil by the formula:

$$\text{Lbs. Activated Alumina} = .412 \times \text{Neutralization Number} \times \text{Gallons of oil.}$$

The second method of treatment was used at Alcoa, Tenn. where the Aluminum Company of America uses up to 300,000 K.W. and, of course, employs many large transformers. A portable treater was constructed similar to the stationary one employed on the turbine of the Aluminum Ore Company. A part of the transformer oil is by-passed by pumping through the bed of Activated Alumina until the desired dielectric strength and Neutralization Number are obtained. The following data were obtained from a typical curve, showing acidity correction against time. This test was made on a 14,000 KVA, 89,000 volt transformer.

<u>No. of Passes Thru Treater</u>	<u>Neutralization Number</u>	<u>Dielectric Strength-KV</u>	<u>Time in Minutes</u>
0	.94	24.0	0
1	.68	26.5	10
2	.58	28.0	20
3	.53	30.0	30
4	.49	31.5	40
5	.47	32.5	50
6	.45	33.5	60

<u>No. of Passes Thru Treater</u>	<u>Neutralization Number</u>	<u>Dielectric Strength-KV</u>	<u>Time in Minutes</u>
7	.44	34.0	70
8	.43	35.0	80
9	.42	35.5	90
10	.42	36.0	100 x
11	.33	36.0	110
12	.28	36.0	120
13	.26	36.0	130
14	.24	36.0	140
15	.23	36.0	150
16	.22	36.0	160
17	.22	36.0	170
18	.22	36.0	180 xx
19	.14	37.0	190
20	.13	36.0	200
21	.13	36.0	210
22	.12	36.0	220

Notes: x Treater was recharged at 10th pass.
xx Treater was recharged at 18th pass.

The total weight of oil in this transformer was 29,500 lbs. The treater contained 700 lbs. of Activated Alumina. The ratio by weight was thus 42 to 1. Neither the life of the oil nor the final results of this by-pass method of treating transformer

oil will be known until more time has elapsed. Enough has been learned, however, to prove beyond doubt that it is a quick, cheap, easy and effective method of reducing acidity and raising the dielectric strength of transformer oil, and from present indications, the life of this oil may be considered as indefinitely prolonged.

RECLAMATION OF MISCELLANEOUS CIRCUIT
BREAKER, COMPENSATOR AND SWITCH OIL.

In most any averaged size industrial plant, several hundred gallons of oil are replaced annually in compensators, motor starters, circuit breakers and small transformers. The quantity is somewhat governed by operating conditions and by maintenance practice but in most places it is surprisingly large. Oil taken from such electrical equipment is usually so saturated with water, acid, sludge and carbon that satisfactory reclamation is seldom possible and then rarely profitable by the usual methods of filtering or centrifuging.

Because of the splendid results obtained in all trials of reclaiming and preserving turbine and transformer oils by use of Activated Alumina, a small treating unit was recently installed for reclaiming used switch and compensator oil which had formerly

been thrown away or burned. Because oil that has been subjected to such uses always contains much dirt and carbon, in addition to water and acid, a small filter press is used in conjunction with this treating unit to remove those impurities susceptible to removal by filtering before the oil is subjected to the Activated Alumina treatment. The remainder of the unit consists of three 110 gallon capacity oil tanks. One is reserved for dirty oil and the other two receive the oil after it has been filtered. Each is equipped with thermostatically controlled electric heaters for maintaining the oil at approximately 130°F. while the treatment with Activated Alumina is in process. The filtered oil is pumped upward through a bed containing 90 lbs. of 4 to 8 mesh Activated Alumina and returns by gravity into the same tank. The unit is compact, requiring a floor space of only 2½' x 7', and has a capacity for treating approximately 50 gallons of oil per day.

The results obtained so far are surprisingly good. Circuit breaker oil of doubtful dielectric strength and a Neutralization Number of .8, and very black from suspended carbon and sludge is restored to a condition as good as new, with the exception of

color, in approximately 24 hours. When the treatment is continued from 48 to 96 hours, this very black oil can be restored to its original pale straw color. Oil that is not excessively dark will usually be restored to a satisfactory color within 24 or 36 hours.

REACTIVATION OF USED ACTIVATED ALUMINA

When Activated Alumina is used for treating oil, the pores of the material gradually become clogged with acid, sludge, water, etc., to the extent that the Activated Alumina is no longer active. However, the clogging or inactivity of the material does not mean that it is no longer fit for further use, because Activated Alumina can be reactivated and restored to its original state. The above statement is based on various experiments both in the laboratory and on a large scale. The following data were taken during a typical laboratory reactivation of Activated Alumina test:

Reactivation Cycles	Activated Alumina Wt. Grams	Trans- former Oil Vol- ume c.c.	Time Agi- tated	Temp. °F.	N.N. Val- ues	Col- or (Imp.)	Reactivation Temp. °C.	Time in Hrs.
Blank		1000	1 hr.	100	.190	320		
Original	150	1000	1 hr.	100	.067	240		
1st	138.3	922	1 hr.	100	.056	200	400	2
2nd	137.6	917	1 hr.	100	.078	240	400	2

Reactivation Cycles	Activated Alumina Wt. Grams	Transformer Oil Volume c.c.	Time Agitated	Temp. °F.	N.N. Values	Color (Imp)	Reactivation Temp. °C.	Reactivation Time in Hrs.
3rd	139.5	930	1 hr.	100	.056	240	400	2
4th	137.5	917	1 "	100	.062	280	400	2
5th	136.2	908	1 "	100	.067	280	400	2
6th	136.2	908	1 "	100	.067	280	400	2
7th	135.5	903	1 "	100	.067	280	400	2
8th	136.2	908	1 "	100	.067	280	400	2
9th	136.3	909	1 "	100	.089	280	400	2
10th	136.3	909	1 "	100	.078	280	400	2

From the above data, one will observe that 150 grams of Activated Alumina is placed in 1000 c.c. of transformer oil that has a Neutralization Number of .19. The oil is heated to 100° C. before Activated Alumina is added, after which the oil and Activated Alumina are agitated for one hour at 100°C. The Activated Alumina is reactivated by placing the material in an oven for 2 hours at 400°C. The cycle was repeated through ten treatments, using the original Activated Alumina and another sample of old oil after each reactivation. The Neutralization Number of the oil after each treatment is almost constant throughout the test, as is the color.

These reactivation cycles, as performed in the laboratory, could have been continued indefinitely. In practical applications, however, such as the portable transformer oil treater used at Alcoa, Tenn. or the unit installed at the Aluminum Ore Company for reclaiming miscellaneous compensator, circuit breaker and switch oils, the Activated Alumina is usually subjected to rougher handling, which would ultimately disintegrate the material somewhere during the cycles of reactivation. However, if the material is handled with reasonable care during the course of handling and reactivation, it can be profitably reactivated many times.

It is obviously difficult to quote accurate costs per gallon of reclaimed oil at this time for all the applications described in this paper. But it can be stated that the cost of reclaiming the miscellaneous circuit breaker and compensator oils, by use of the above described treater at the Aluminum Ore Company, is less than \$.06 per gallon of oil. This figure includes the cost of all labor, electric power and 7 to 10 reactivations of the Activated Alumina. It should be stated that these oils were extremely degraded before treatment and less degraded oils

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could without doubt be reconditioned at a more favorable cost.

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