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A Study of the Colors of Contamination in Used Oils

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The color of contaminants in used oils has not been investigated until recently. There are several good color standards. The CIExy coordinate, which is used in color engineering, is a popular color standard to locate a color of a product. The CIExy coordinate can be used for the colors of contaminants in used oil, but it is difficult to understand how the color is located in that position in the coordinate. The authors have developed a colorimetric patch analyzer (CPA) with the red-green-blue (RGB) color system to study the color of contaminants in used oils that were collected by membrane patches. The first CPA model examined the color of contaminants trapped on the surface of the membrane patches by reflected light similar to the currently available colorimetric analyzers. Some contaminants are soluble in working gas turbine oils at temperatures of 65°C or higher but become insoluble in oils when the oils are cooled to room temperature for 24 h. Therefore, the colors of contaminants differ according to oil temperature at the time of filtering through membrane filters. When the membrane patches through which hot turbine oils were filtered were examined with the light from the back, they showed light brown colors, although the color of the surfaces were white. This suggests that the membrane patch traps contaminants in the oil passages of the membrane patches when oil flows through the patches. The molecular weight of contaminants that are soluble in oil at high temperatures but not at room temperature was examined by gel permeation chromatography (GPC). The University of Fukui developed a state-of-the-art CPA that can examine the contaminants on the surface of membrane patches by reflected light and contaminants in the membrane patches by transmitted light.

KEY WORDS

Lubricant Decomposition; Gas Turbine Oils; Oil Oxidation Products; Colorimetric Analysis; Molecular Weight; Gel Permeation Chromatography; Maintenance

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INTRODUCTION

Contamination in oil is harmful to lubrication, but contamination is not usually paid much attention. When we check oil contamination, particle count is used because oil cleanliness standards such as NAS 1638 (1), ISO 4406 (2), and SAE AS4059 (3) are based on particle count. Sometimes, contaminated oil is filtered through a membrane filter disc to measure the weight of contamination, which is known as *gravimetric analysis*, consistent with oil cleanliness standards such as ASTM D4898 (4). When contaminated oils are filtered through a membrane filter, the membrane patches will be colored, but if a state-of-the-art oil cleaner like an electrostatic oil cleaner (Sasaki and Uchiyama (5)), which can remove almost all oil-insoluble contaminants, is used to control the oil contamination, the color of the membrane patches will be almost white. Therefore, color will be first noticed when we see a membrane patch, but we usually do not pay attention to the colors, because there are no standards to judge contamination of the oil by color. Robert Humphrey of Whirlpool was probably the first person who used a colorimetric method for analysis of hydraulic oil contamination, because the company had used the colorimetric technology for quality control of plastic products. He developed a 10-point color scale standard and implemented it for oil maintenance. As a result, twice-yearly oil changes were eliminated and downtime of injection molding machines was substantially reduced (Ogando (6)). Doug Robertson of Kleentek (USA) introduced the technology and improved it for application to other hydraulic oils with cooperation of one of the authors. The technology was used as one of the tools for oil maintenance at the Saturn plastic plant under the supervision of Mickey Jackson of Henkel. However, application of the technology was still limited to hydraulic oils. Therefore, one of the authors brought the technology to the University of Fukui in 1998 to apply it to lubricating oils. The results of the development were published in the Society of Tribologists and Lubrication Engineers' journal *Lubrication Engineering* (Yamaguchi, et al. (7)). The colorimetric method is suitable for examining the contaminants collected on membrane patches, but it is limited. When we see an object, we recognize its shape and color by reflected light. Therefore, commercially available colorimetric analyzers examine the color of membrane patches by reflected light. The first model of a colorimetric patch analyzer (CPA), which was

developed at the University of Fukui before 2009, recognized the color of membrane patches by reflecting light only. Therefore, it was suitable for examining the color of contaminants collected on the surface of membrane patches.

In the case of modern gas turbine oils, the working oil temperature is high and oil oxidation products with relatively low molecular weights (MWs) are soluble in the working oil. When this oil was filtered immediately after it was taken from the oil tank, the membrane patch was almost white, but when the same oil was filtered after it was kept at room temperature for a few days, the membrane patch was brown. This phenomenon was confirmed at other power plants (Sasaki and Galli (8)). The oil user's concern is to know whether the oil is (1) safe to use, (2) needs to be topped up, or (3) needs to be changed. Therefore, the University of Fukui is reexamining the behavior of contaminants and the filtering mechanism of membrane filters in order to develop a colorimetric analysis technology. This article discusses the behavior of the oil contaminants in gas turbines, the filtering mechanism of membrane filters, and the mechanism and performance of a state-of-the-art CPA.

BEHAVIOR OF OIL OXIDATION PRODUCTS

Oil Oxidation Products and Varnish

From the perspective of energy efficiency, a combined cycle power generation system with gas turbines and a steam turbine has become popular. The fuel efficiency of gas turbines also improves with higher inlet gas temperature. Therefore, the operating oil temperature of gas turbines has become higher and varnish

problems have become serious (Kalyanaraman (9); Day (10); Sasaki, et al. (11)). Sludge (ASTM D4378 (12)), which is an oil oxidation product, is called *varnish* when it coats metal surfaces. Therefore, this article will call the products formed by thermal decomposition of oils *sludge* and the deposits of the sludge or oil oxidation products on machine parts *varnish*; see Fig. 1.

Short Review of Previous Studies on the Molecular Weight of Oil Oxidation Products

The mechanisms of oil oxidation have been studied by many scientists and researchers. During the period from the 1970s to the 1980s, many papers, which measured molecular weight of oil oxidation products, were published. Ali, et al. (13) and Cho and Klaus (14) showed that oxidation products are polymerized via intermediate oxidation products such as dimer, trimer, tetramer, although the MW of the majority of the base oil molecules remains unchanged. Ali, et al. (13) reported that polymerized oxidation products are molecules with an MW of 3,000–50,000 and that intermediate oxidation products are molecules with an MW of 580–860, although they did not provide a definition of



Fig. 1—Varnish on a valve piston (color figure available online). (Courtesy of Fortum UK.)

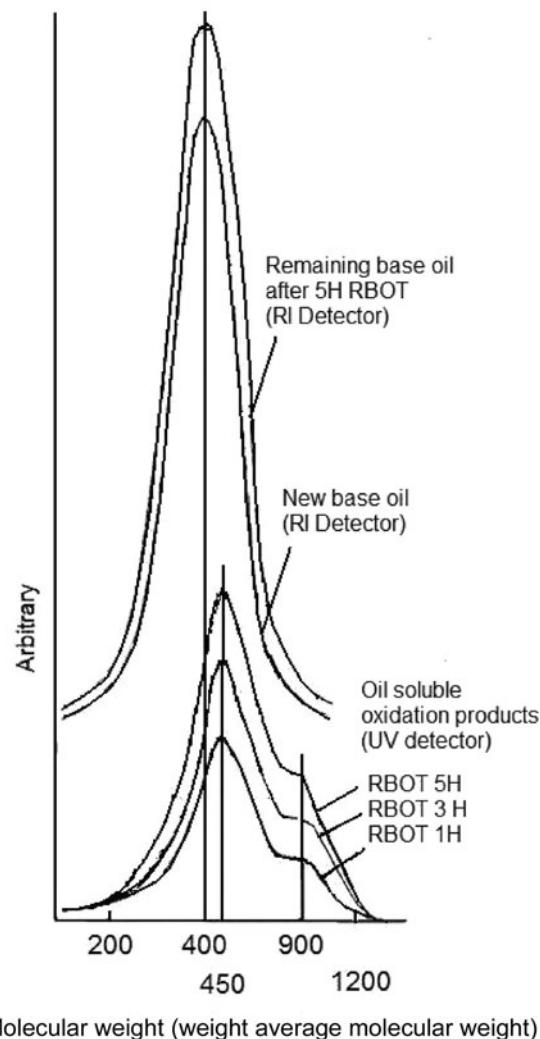


Fig. 2—Example of MW of group II base oil and oil-soluble oxidation products before and after RBOT tests.

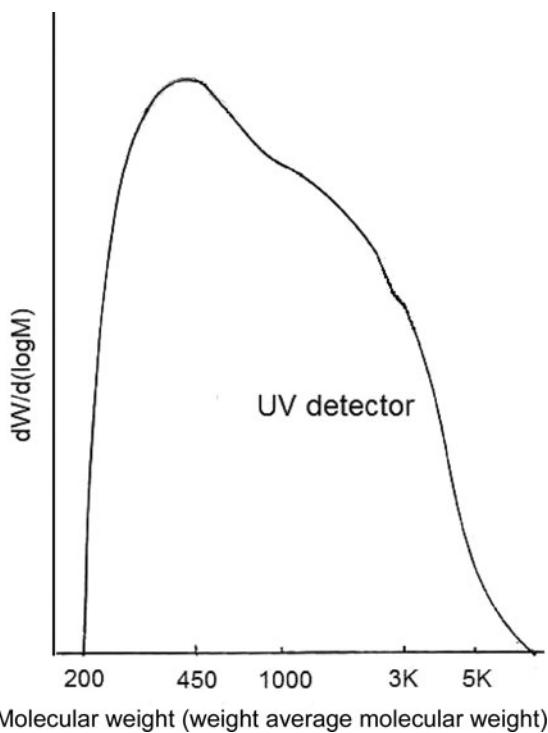


Fig. 3—Differential MW distribution of oil-insoluble fraction of 3 h RBOT base oil.

intermediate oil oxidation products. Gunsel, et al. (15) demonstrated that base stocks of mineral oil, ester, and poly-alpha-olefin followed similar oxidation processes. A group II base oil with a viscosity of 32 mm²/s, which is popular as a base oil for turbine oils, was oxidized at 150°C by a rotating bomb oxidation test (RBOT; now called a rotary pressure vessel oxidation test) apparatus without water and without catalysts for 1, 3, and 5 h. The oxidized oils were filtered through a 0.8-μm membrane filter after cooling to room temperature. Because the liquid part of the oils contains oil-soluble oxidation products, the oil-soluble oil oxidation products were extracted from the liquid part by silica gel chromatography. The MW of the new base oil, the remaining base oil after 5 h RBOT, and the extracted oil-soluble oxidation products (at 1, 3, and 5 h RBOT) were measured by gel permeation chromatography (GPC). The procedure for GPC analysis was the same as that described in a previous paper (Sasaki, et al. (16)). The results are collectively shown in Fig. 2. A refractive index (RI) detector showed a peak at a MW of 400 for the base oil. An ultraviolet detector showed peaks at MWs of 450 and 900 for the extracted oil-soluble oxidation products. The MWs of 450 and 900 may indicate monomer and dimer. This corresponds to the studies of Ali, et al. (13) and Cho and Klaus (14). The MW of the oil-insoluble fraction collected on membrane patches was also measured by GPC. The MW of the oil-insoluble fraction at 3 h RBOT is shown in Fig. 3; the membrane patches of the oils at 1 and 3 h RBOT were white but that of the oil at 5 h RBOT was light brown when it was filtered while hot. This suggests that the chromatograph has three peaks at MWs of 400–450, 900–1,200, and 3,000. Ali, et al. (13) reported that the oxidized oils had four groups of MW at 1,900–3,000, 580–860, unreacted part of the base

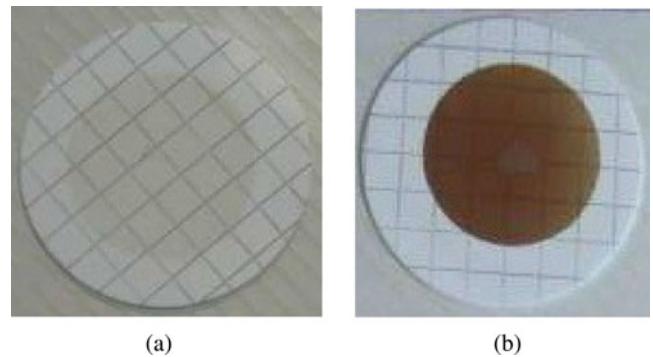


Fig. 4—Membrane patches of FR9FA gas turbine oil: (a) filtered immediately after sampling (at 51°C) and (b) filtered 24 h after sampling (room temperature) (color figure available online).

oil (about 400), and less than 220. Therefore, our GPC measurement corresponds to the data of Ali, et al. (13).

Examination of Used Gas Turbine Oil

A 25-mL oil sample was filtered through a 0.8-μm membrane filter with a 25 mm diameter immediately after being taken from GE FR9FA gas turbine oil tank in operation. The membrane patch is shown in Fig. 4a. The working oil temperature was about 65°C. The oil temperature was 51°C at the time of filtration. The same oil was kept at room temperature for 24 h and filtered using the same procedure. The membrane patch is shown in Fig. 4b. The oil was already cooled to room temperature at the time of filtration. The remaining oil samples were sent to us by courier service for further investigation. Four hundred milliliters of gas turbine oil, which was cooled to room temperature, was reheated in a glass beaker on a hot plate to raise the oil temperature to 65°C and then it was held at that temperature for 30 min. Thereafter, the oil was naturally cooled down to room temperature. The first 25 mL of sample oil (sample A) was filtered when the oil temperature was 65°C. Thereafter, 25 mL of samples B–G were filtered at 10, 20, 40, 60, 90, and 120 min after the first sample oil was filtered while the oil was naturally cooled. The cooling times and the filtered oil temperatures are shown in Table 1.

Twenty-five-millimeter-diameter, 0.8-μm membrane filters were used to filter samples A–G. The scanned pictures of the top surface and the bottom surfaces of the membrane filters and the oil temperatures at the time of filtration are shown in Fig. 5. The colors of the surfaces of all membrane patches were brown but the bottoms were white. This suggests that almost all of the contaminants were caught on the surface of the patches or in the oil passage.

TABLE 1—COOLING TIME OF THE HEATED OIL AND FILTERING TEMPERATURE

	Sample ID						
	A	B	C	D	E	F	G
Cooling time (min)	0	10	20	40	60	90	120
Filtering temperature (°C)	65.0	56.7	50.0	41.7	34.8	31.3	28.3

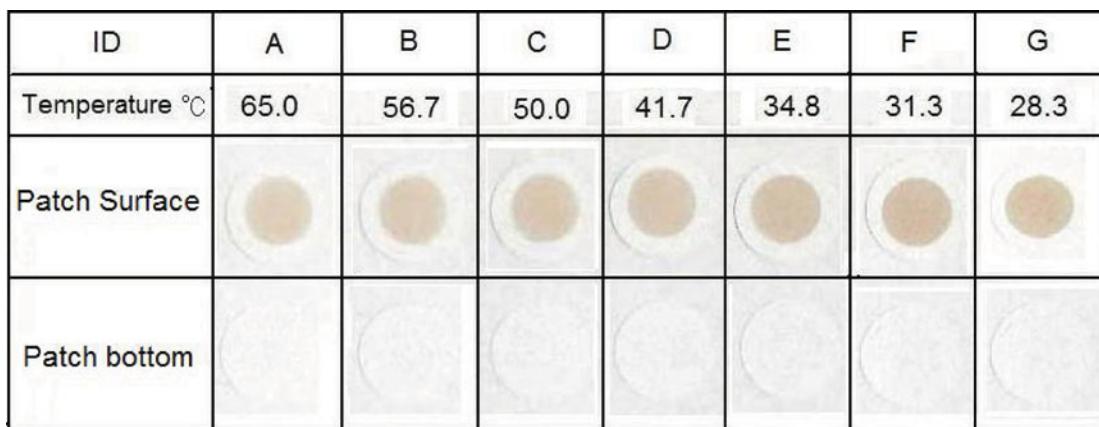


Fig. 5—Membrane patches filtered after heating oil sample (color figure available online).

TABLE 2—MOLECULAR WEIGHT OF SAMPLE A

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.024	22.667	23.176	23.776	24.866	25.291	25.833
Average MW	1,857	1,164	817	553	299	187	113

Molecular Weight of Contaminants by GPC

For GPC analysis, each membrane patch was washed with tetrahydrofuran (THF) in ultrasonic bath. The MW of the contaminants collected by the membrane patches was measured by GPC. The columns used were Sim-pack GPC-801, 802, and 803. Each 50- μ L sample was injected into the column, and THF was delivered as the mobile phase at a flow rate of 1.0 mL/min. The MW was detected by an RI detector. An MW calibration curve based on elution time (minutes) of polystyrene standard and an example of an elution curve of a sample oil are shown in Fig. 6. The elution peak time and average MW of each sample (A–G) are shown in Tables 2–8. Because there is some possibility that THF may dissolve cellulose acetate membrane filters, a blank membrane filter was also washed with THF and the MW of the solvent was measured. The result is shown in Table 9.

All of the data are collectively shown in Table 10. The MWs of all of the samples were almost similar at all elution time peaks. For easy comparison, the average MW of all of the membrane patches (A–G) at each elution time peak is shown as “Average” in the table. Both the MWs of all the membrane patches (A–G) and the average MW at peaks 1–7 were similar to those of the blank patch, which contained no oil oxidation products. This suggests that the MWs of the membrane filter materials were similar to those of oil-insoluble oxidation products.

TABLE 3—MOLECULAR WEIGHT OF SAMPLE B

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.059	22.658	23.191	23.800	24.886	25.317	25.800
Average MW	1,849	1,097	785	543	294	186	122

Because the same procedure was used on all membrane patches for MW measurement, the heights at the elution time peak were examined for all membrane patches. The results of the examination are shown in Table 11. The heights of all of the used membrane patches (A–G) were apparently greater at all of the peaks than those of the blank membrane filter. The facts indicate that there was a greater number of molecules for all used membrane patches (A–G) at each MW range or at each peak (1–7) than for the blank membrane patch.

Because the highest peak was found at peak no. 5, the highest MW was about 300, which was within the range of base oil molecules, but they were trapped in the membrane patches. As Ali, et al. (13) reported, it is possible to have oil oxidation products that have a lower MW than the mean MW of the base oil. The membrane filter was made of cellulose acetate. Based on the construction of molecules, there is a possibility that polar group

Molecular weight calibration curve based on elution time and an example of the elution time of sample oil "A"

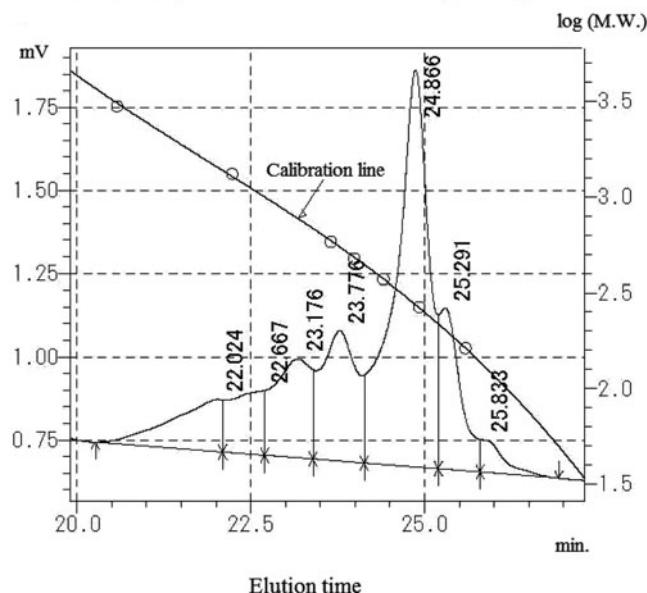


Fig. 6—Example of elution curve of sample A and MW calibration curve based on the elution time of standard polystyrenes.

TABLE 4—MOLECULAR WEIGHT OF SAMPLE C

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.133	22.630	23.168	23.783	24.883	25.318	25.767
Average MW	1,889	1,157	813	540	301	193	116

TABLE 5—MOLECULAR WEIGHT OF SAMPLE D

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.050	22.459	23.175	23.793	24.889	25.323	25.800
Average MW	1,987	1,130	794	547	302	193	97

TABLE 6—MOLECULAR WEIGHT OF SAMPLE E

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.030	22.601	23.148	23.772	24.863	25.290	25.733
Average MW	1,887	1,136	806	556	303	192	99

TABLE 7—MOLECULAR WEIGHT OF SAMPLE F

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.053	22.471	23.139	23.768	24.861	25.292	25.733
Average MW	1,905	1,148	815	553	303	193	121

TABLE 8—MOLECULAR WEIGHT OF SAMPLE G

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.123	22.526	23.118	23.768	24.860	25.286	25.733
Average MW	1,874	1,147	816	548	303	193	112

TABLE 9—MOLECULAR WEIGHT OF THE DISSOLVED BLANK MEMBRANE PATCH IN THF

	Peak No.						
	1	2	3	4	5	6	7
Peak time (min)	22.078	22.500	23.190	23.792	24.889	25.327	25.931
Average MW	1,799	1,131	798	545	290	190	122

TABLE 10—SUMMARY OF THE MEASURED MOLECULAR WEIGHT

Sample ID	Peak No.						
	1	2	3	4	5	6	7
A	1,857	1,164	817	553	299	187	113
B	1,849	1,097	785	543	294	186	122
C	1,889	1,157	813	540	301	193	116
D	1,987	1,130	794	547	302	193	97
E	1,887	1,136	806	556	303	192	99
F	1,905	1,148	815	553	303	193	121
G	1,874	1,147	816	548	303	193	112
Average MW	1,893	1,140	806	549	301	191	111
Patch	1,799	1,131	798	545	290	190	122

TABLE 11—COMPARISON OF THE HEIGHTS AT ELUTION TIME PEAKS

	Sample ID							
	A	B	C	D	E	F	G	Blank
Peak 1	157	170	138	333	143	206	182	92
Peak 2	192	193	157	351	162	230	203	93
Peak 3	297	286	220	438	234	302	268	137
Peak 4	392	381	293	614	329	422	370	177
Peak 5	1,185	1,304	643	1,698	1,162	1,232	1,100	652
Peak 6	484	519	381	1,058	512	562	483	256
Peak 7	98	83	84	158	133	102	99	36

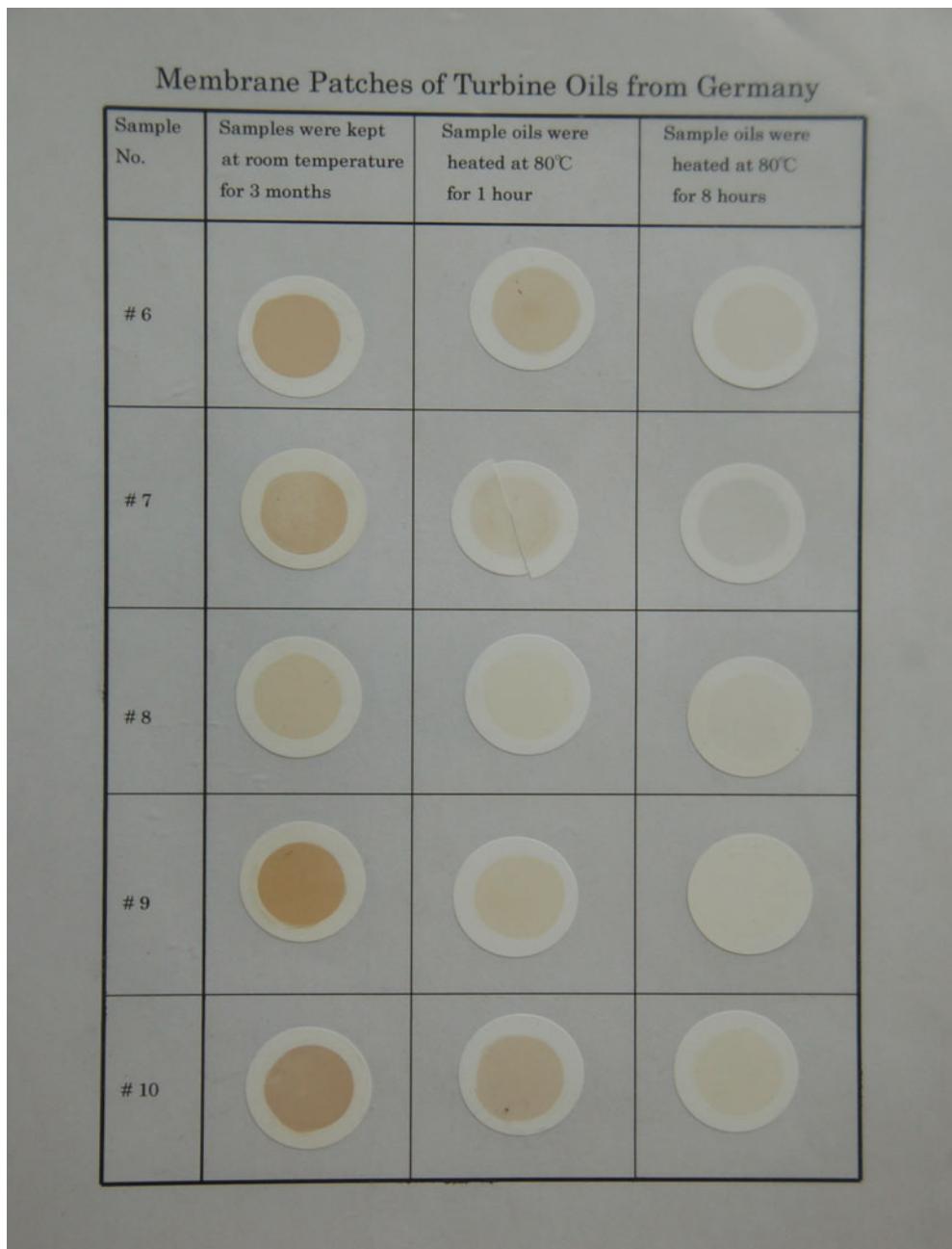


Fig. 7—Comparison of solubility of oxidation products in oil by heating at 80°C for 1 and 8 h (color figure available online).

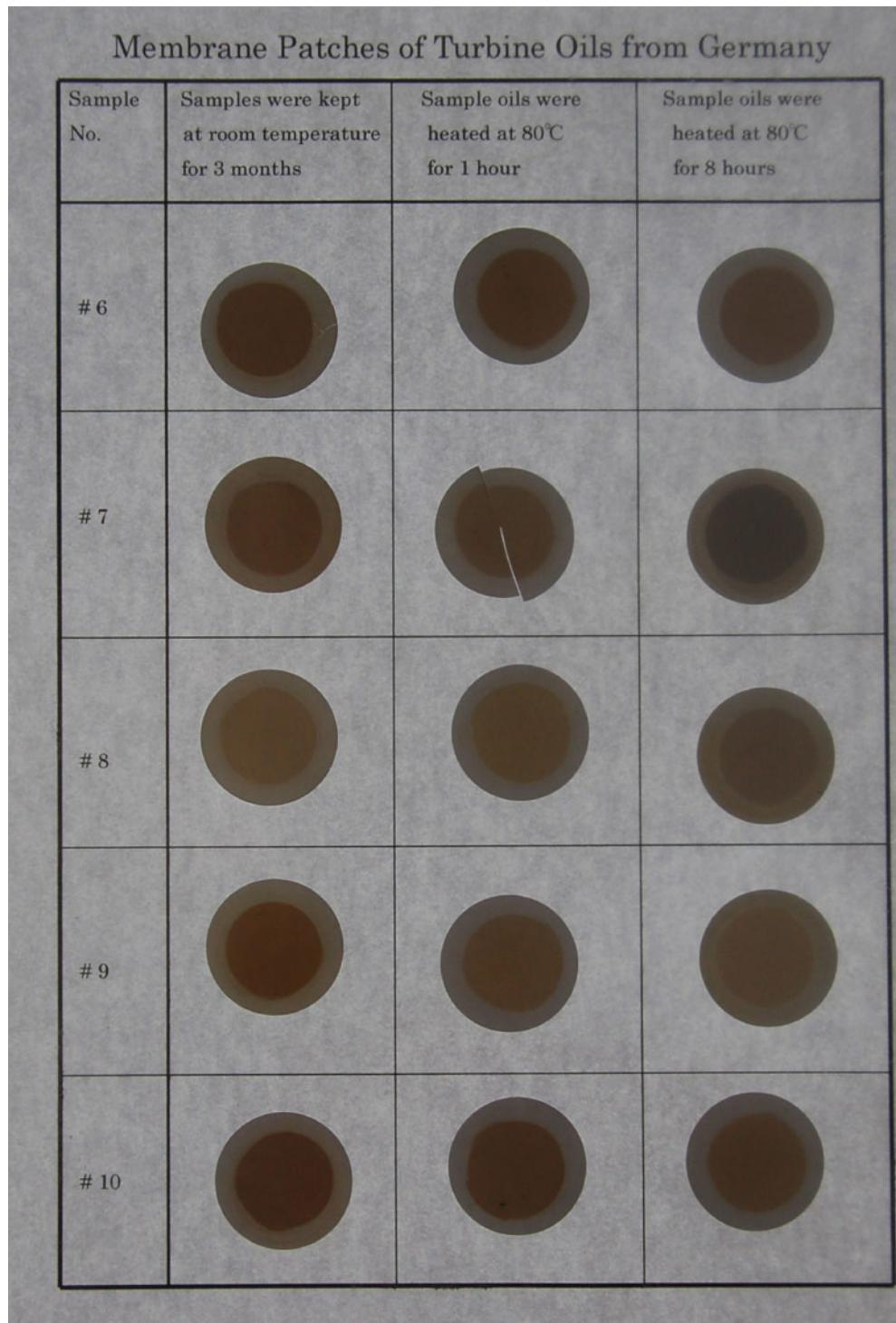


Fig. 8—Membrane patches seen with backlight (color figure available online).

molecules were trapped in the membrane filter material. After filtration, the membrane patches were rinsed with petroleum ether, which is nonpolar. Because polar and nonpolar molecules are not compatible, polar molecules will not be carried away by the nonpolar solvent.

The highest MW of oil-insoluble oxidation products was found in used gas turbine oil, which when cooled to room temperature was about 1,800–1,900. When the oil in question was taken from

an oil tank of a working gas turbine, the oil was clean, as shown in Fig. 4a. This means that the oxidation products of 1,800–1,900 in MW were soluble in oil at the working oil temperature (reported to be about 65°C) but that they became insoluble in oil at room temperature. This is why the membrane patch of the same oil that was filtered after cooling was brown. As shown in Fig. 4b, when the oil was heated up to 65°C for 30 min and filtered, the color of the membrane patch was still brown. This suggests that 30 min



Fig. 9—Schematic picture of a cross section of a membrane filter.

heating was not enough to dissolve the molecules with an MW of 1,800–1,900. Because the oil from the gas turbine was cleaned by an electrostatic oil cleaner, which can remove oil-insoluble contaminants, the oil was clean and the membrane patch is almost white when it is filtered through a membrane filter immediately after sampling. This means that molecules with an MW greater than 1,800–1,900 are insoluble at 80°C but molecules that are equivalent to or have an MW lower than 1,800–1,900 are soluble in oil at 80°C.

In order to verify this hypothesis, six different turbine oils, which were kept at room temperature for 3 months, were heated to 80°C. The hot oils were filtered by membrane patches after 1 and 8 h of heating. The results of the tests are shown in Fig. 7. Although almost all membrane patches after 1 h of heating showed a light brown color or a yellow brown color, after 8 h of heating all of them were almost white. This suggests that intermediate oil oxidation products that were agglomerated and insoluble in the cooled oils became soluble in oil by heating at 80°C for 8 h, and that 1 h of heating was not enough to dissolve the oxidation products completely, even though the oil was heated at 80°C. There-

fore, it may be understood that oxidation products are insoluble in oil when the molecules of oxidation products are agglomerated and that they are soluble when the agglomerated molecules of oxidation products are disassembled from the agglomerated condition and dispersed in oil as individual molecules.

When the same membrane patches were seen with a backlight, a slightly brown color was seen on all of the membrane patches in the area where oil passed through the patch, as shown in Fig. 8. This suggests that the sizes of individual oil oxidation products with MWs of 1,800–1,900 or lower are smaller than 0.8 μm when they are soluble but are captured when passing through the membrane filter by electrostatic force, which is generated when oil passes through the membrane filter, as shown in Fig. 9. It is reported that the molecules of intermediate oil oxidation products, which are soluble in oil at working oil temperature but become insoluble in oil when cooled to room temperature for more than 5 h, do not cause varnish problems (Sasaki and Galli (8)). However, it is important to know that they will soon become polymers. Therefore, from the perspective of preventive and proactive oil management, it is imperative to detect molecules of intermediate oil oxidation products.

MECHANISM OF A PATCH ANALYZER DEVELOPED BY THE UNIVERSITY OF FUKUI

The University of Fukui has developed a CPA, which is shown in Fig. 10, that can determine the existence of intermediate oil oxidation products, even when the color of the membrane patches is white. It is different from a conventional colorimetric analyzer that uses reflected light only. The Fukui University CPA examines colors of membrane patches using both reflected light and transmitted light (Honda, et al. (17)). A schematic construction of the CPA is shown in Fig. 11. The measuring compartment was completely enclosed to shut off outside light. To measure the color on a membrane patch, a membrane patch is framed. The frame has openings at both the top and the bottom, as shown in Fig. 12. The framed membrane patch is placed in the measuring compartment. For measurement, white light is cast on the



Fig. 10—Photo of a prototype CPA (color figure available online).

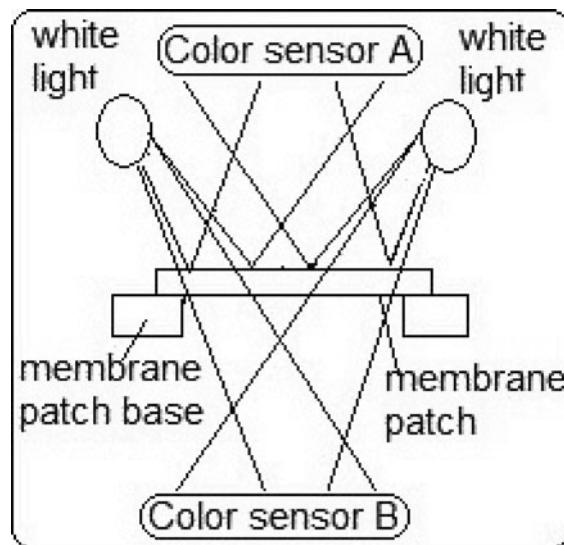


Fig. 11—Schematic arrangement of sensing parts of CPA.

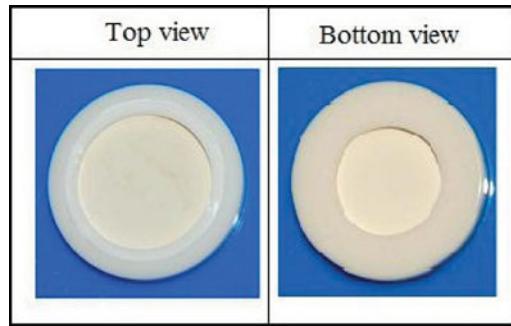


Fig. 12—Example of a framed membrane patch (color figure available online).

membrane patch from the direction of the surface. The light that is reflected from the surface is received by color sensor A, which is placed at the top, and the light that passes through the membrane patch is received by color sensor B, which is placed at the bottom of the framed patch.

When used contaminated oil is filtered through a membrane filter, contaminants that are larger than the pores of the membrane filter are caught on the surface of the membrane patch, but small contaminants pass through the membrane patch. However, static electricity is generated on the surface of the oil passages of

the membrane filter when oil passes through the membrane filter. Therefore, small contaminants are trapped in the oil passage by the drawing force of static electricity. Figure 9 shows schematically how contaminants are trapped in the oil passages of a membrane filter. The surface color of the membrane patch is detected by color sensor A as the reflected color. Parts of the white light pass through the membrane patch and are detected by color sensor B as the transmitted color because some contaminants are trapped in the membrane patch by electrostatic force, and the transmitted light with the color of the contaminants is detected by color sensor B, even when the underside of the membrane patch is white. When the surface contains contaminants, the reflected light examines the color of contaminants on the surface, and the color of contaminants trapped in the oil passage will be measured with the transmitting light by the RGB method. The results of the measurement of the membrane patches (no. 9) shown in Figs. 7 and 8 are shown in Fig. 13. The three patches in the row “With Reflecting Light” are a photo extracted from membrane patches no. 9 in Fig. 7. The red-green-blue (RGB) readings of the patches as measured by the CPA were (240, 195, 139), (249, 231, 199), and (255, 246, 237). The RGB colors corresponding to the RGB readings measured by CPA were copied from the standard RGB colors and shown in the third line of the table in Fig. 13 for comparison. The colors of the patches were similar to those of the standard RGB colors. The photo of three patches in

With Reflecting Light	Patches				
	RGB Reading by CPA	240, 195, 139	249, 231, 199	255, 246, 237	
	RGB Color Related to the RGB Readings				
With Transmitting Light	Patches				
	RGB Reading by CPA	209, 132, 031	215, 156, 072	219, 173, 096	
	RGB Color Related to the RGB Readings				

Fig. 13—Patch no. 9 of Figs. 7 and 8 was extracted and shown with RGB values by CPA (color figure available online).

TABLE 12—EXAMPLE OF THE READINGS OF RGB, HLS, AND L*a*b* BY THE CPA

Color Mode	Readings
RGB with reflected light	240, 195, 139
HLS with reflected light	33°, 42%, 94%
L*a*b* with reflected light	82, 11, 35
RGB with transmitted light	209, 132, 31
HLS with transmitted light	34°, 85%, 82%
L*a*b* with transmitted light	62, 25, 62

the row “With Transmitting Light” was extracted from the photo of membrane patch no. 9 shown in Fig. 8. The RGB readings of the patches measured by the CPA were (209, 132, 031), (215, 156, 072), and (219, 173, 096). For measurement by transmitted light, the light passed through a membrane patch; therefore, the photons were generally dark but the color of the contaminants can be seen in the center of each membrane patch. The CPA can measure the color of contaminants in the closed chamber with strong light-emitting diode white light, although the light is weakened when it passes through a patch. The RGB colors corresponding to the RGB readings measured by CPA were copied from the standard RGB colors and shown in the third line of the table in Fig. 13 for comparison. The color of the patch that filtered 25 mL used oil after heating at 80°C for 8 h was almost white as observed by the naked eye when seen by reflected light, but the CPA could distinguish it from standard white. When the patch that filtered 25 mL of used oil after heating at 80°C for 8 h was checked with backlight, a yellow-brown color was observed. It was similar to the standard RGB color related to the RGB readings by CPA. It is known that molecules of oil oxidation products will become polymers following the processes of forming dimers, trimers, tetramers, pentamers, etc. (Gunsel, et al. (15)). When oil oxidation products are in the early stage of intermediate oxidation products, they cannot be captured by the surface of a membrane patch but are caught inside the oil passage instead. When they become polymers, they are captured by the surface of a membrane patch; therefore, the CPA is suitable for examining the color of the surface by reflected light when the surface contains contaminants and is also suitable for examining the color of contaminants inside the oil passage by transmitted light when the surface and the bottom are white. This means that the CPA can detect the formation of oil oxidation products at an early stage because the CPA detects intermediate oil oxidation products that are formed before polymerization.

There are several methods to define colors, including Munsell, Oswald, RGB, HLS, CIE, and L*a*b* methods. These methods were not originally developed to define the color of contamination of oils. When several methods coexist, it is inconvenient to translate the readings of one method to another; therefore, an advantage of the CPA is that it can measure readings of (1) RGB with reflected light, (2) RGB with transmitted light, (3) HLS with reflected light, (4) HLS with transmitted light, (5) L*a*b* with reflected light, and (6) L*a*b* with transmitted light. An example of the readings of RGB, HLS, and L*a*b* measured by CPA is shown in Table 12.

CONCLUSION

1. The color of the membrane patch through which gas turbine oil was filtered immediately after sampling was almost white, whereas the color of the membrane patch through which gas turbine oil was filtered after cooling at room temperature for 24 h was brown. The contaminants were soluble in the working oil at a temperature of approximately 65°C, and they were insoluble when the oil was cooled to room temperature for 24 h.
2. The MWs of the contaminants were examined by GPC and were lower than about 2,000. It was confirmed that they were similar to the intermediate oil oxidation products that were previously studied (Ali, et al. (13); Cho and Klaus (14); Gunsel, et al. (15)).
3. The CPA, newly developed by the University of Fukui, could detect contaminants on the surface of the membrane patch by reflected light as well as those trapped in the membrane patch via transmitted light, even if the surface of the patch was white by reflected light.

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