Hodnocení vybraných olejů po zrychleném tepelném stárnutí pomocí FT-IR spektroskopie

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Evaluation of The Chosen Oils after Accelerated Thermal Ageing Using FT-IR Spectroscopy

Abstract – **This paper is focused on the analysis of liquid electrical insulation materials used in electrical devices. Three types of oil were tested by accelerated thermal ageing. The oils were aged in a thermal chamber at elevated temperature of 130 °C during 672 hours. FT-IR spectroscopy was used for the oils analyzing during the whole process of accelerated ageing. The change of the conversion substances of oils was observed. Antioxidants were reduced. The experiment results provided some interesting information about the change of the substances in oils and showed the way to determine the state of new or aged oils.**

Keywords – FT-IR spectroscopy; liquid electrical insulation; accelerated thermal ageing; change of antioxidants and antioxidants; oils

I. INTRODUCTION

There are a lot of methods to assess the state of liquid electrical insulations but each of them is based on testing. Some of the tests are performed in laboratories and others on site. The methods of assessment of the liquid electrical insulation state depend on the possibilities (i.e. on the equipment available) and how fast the results are required. Moreover, each method of assessment of the mentioned insulation materials brings advantages and disadvantages. In some cases it is better to prefer laboratory testing while on site testing may be better in other cases. The test for assessment of the state of liquid electrical insulation is described below. It is focused on laboratory testing, because of the necessary long time observation and special equipment, which are not available under service conditions. The first step is utilising of the thermal chamber for the accelerated thermal ageing and simulating of the service environment. In the second step, the FT-IR spectroscopy is used for analyses of three types of oils. After ageing at elevated temperature during the accelerated thermal test the change of conversion substances of the oils was observed

II. DESCRIPTION OF THE EXPERIMENT

The experiment was focused on seeking new knowledge in the area of electric insulation oils by accelerated thermal test. For gaining the new knowledge, three types of oils were used and measured by FT-IR spectroscopy, providing information about chemical changes inside the oils. Based on FT-IR spectroscopy, changes of antioxidants during ageing of oils were observed. In future, this information could be assigned to some electric parameters (e.g. breakdown voltage) with the aim to decide about residual lifetime of oils in service conditions.

A. Preparing of oil samples

Three types of electrical insulation oils were studied and their properties are chosen in table I. The transformer oil – Nynas Y3000 is a high-quality oil, because it is made from naphthenic raw material. The transformer oil - Mogul trafo CZ-A is inhibited. It is made from a high-quality paraffin oil. The transformer oil - Mogul trafo CZ-A is inhibited. It is made from a high-quality paraffin oil. [1] [2] [3]

TABLE I. TYPE OF OILS AND THEIR PROPERTIES

Typ of oil	Amout of antioxidants [% of weight]	Electric streght [kV] (before drying)	Oxidation stability [During 120 °C, 500 hours]
Nytro Lyra X	0,38	40-60	IEC 61125 B
Nynas Y3000	0,38	40-60	IEC 61125 B
Mogul trafo CZ-A	0,40	Is not available	Is not available

The oils are highly resistant against degradation. For preparing of the experiment it is necessary to divide each kind of oil into four special 50 ml testing glasses. Twelve testing glasses (3 x 4) were prepared, each containing 40 ml of oil. The oil had to be mixed up before filling the glasses. Then the glasses had to be marked by oil type and number. In the next step a strip of paper was added between glass neck and stopper. The strip helps drying of oil during the accelerated thermal ageing. Finally the glasses were placed into the thermal chamber. Their position had to allow their fast taking out from the chamber.

B. Accelerated thermal ageing

The accelerated thermal ageing is performed according to the international standard IEC 60216-1. The principle of the test is the samples of oils are exposed to elevated temperature for a certain period of time. The time of exposure is chosen based on the standard and performed experiments, as the standard does not provide enough information about testing time. The thermal chamber was used to ensure the elevated temperature. The elevated temperature chosen was 130 °C and started all the accelerated degradation processes, which take a very long time under normal service conditions. The thermal chamber fan speed was 50 % and ¼ of its back door was left open. A higher temperature (>140 °C) would lead to flash processes, according to datasheet. In this experiment, three types of electrical insulation oils were exposed each to four different time periods of thermal ageing. There were four groups of samples of each type of the tested oils. Every 168 hours one group of samples was taken out, the periods of ageing were therefore 168, 336, 504 and 672 hours. After finishing of each time period the taken out samples of oils were left at room temperature for 24 hours. In the next step, the FT-IR spectroscopy was used for analyzing of the aged samples. Each glass provided samples for 3 measurements. All infrared spectrums of the oils were recorded.

C. FT-IR spectroscopy

The samples were taken out of the chamber and left 24 hours at room temperature, approximately 3 ml of oil were taken from each of the glasses by means of a hypodermic syringe and put into the liquid cell. The liquid cell must be perfectly cleaned before measuring. The thickness of oil layer was 1mm. The second hypodermic syringe was used to prevent oil bubbles, which are not allowed inside the cell. In this way the cell was prepared and inserted into the FT-IR spectrometer.

Finally, the infrared spectrum was measured on FT-IR NICOLET 380 spectrometer which was connected to a PC and controlled by OMNIC software. Then the infrared spectrum of the oil was measured in the range from 400 to 4000 cm-1, depending on absorbance. Each of the glasses provided three samples, i.e. three spectrums, of which one average spectrum was made. Finally, the average infrared spectrums of each tested type of oil were put into one diagram and compared together. The results are interpreted below.

III. RESULTS

All the infrared spectrums of the oils measured consisted of three areas with higher value of absorbance situated between 300 and 3500 cm-1, which can be seen on an example of one oil spectrum in Figure 1. They were caused by the change of C-H bonds. The very high value of absorbance of the area between 800-400 cm-1 was caused by the stage of oil refining. The experiment itself was focused on data of the area from 3800 to 3400 cm-1, with top in 3650 cm-1. During the process of ageing decrease of absorbance was observed in the mentioned area depending on the increasing time of ageing. This decline was most pronounced in oil Mogul transformer CZ-A and it is typical for the process of antioxidants decrease, i.e. the decrease of antioxidants, which can be seen in Figure 2.

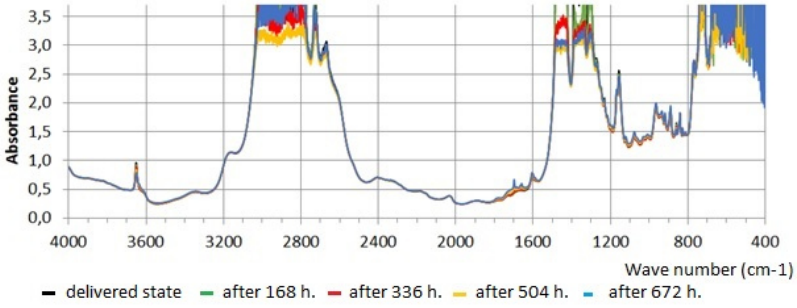

Figure I. **The infrared spectrum of Mogul trafo CZ-A oil in the whole measured range.**

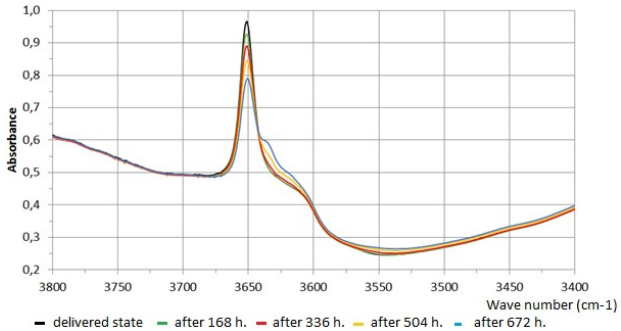

Figure II. **Infrared spectrum of Mogul trafo CZ-A oil in 3800-3400 cm-1.**

The increase of absorbance at 1610 cm-1 is typical for thermal oxidation which is called nitration. During nitration the organic compounds of nitrogen oxides (NO, NO2 ... N2O4) change at elevated temperature. This oxidation changes the quality of oil. The experiment results show the changes in antioxidants, which is shown in the Table II. It is possible to see that the changes of antioxidants are constant.

TABLE II. DIFFERENCE OF MAXIMUM VALUE OF ABSORBANCE

Type of Oils	Wave number	Change after 168 hours	Change after 336 hours	Change after 504 hours	Change after 672 hours
Mogul trafo CZ-A	3651,3	-0,04	-0,036	-0,046	-0,056
Nynas Y3000	3650,8	-0,016	-0,034	-0,045	-0,031
Nytro Lyra X	3650,8	-0,035	-0,045	-0,037	-0,043

IV. CONCLUSION

In the experiment a change of lineary thermal reducing of antioxidants was observed. For the evaluation of the actual state of oil, however, this experiment was insufficient.

It is necessary to carry out the same experiment again and follow also the dielectric strength of the oil during the ageing. It would be interesting to find a form between reducing of absorbation and reducing of electric streght in future. The values of the electric strength provide information about the actual state of oil. Thanks to this, it will be possible to decide about the actual state of oil only based on the values of the antioxidants decrease. [4] [5]

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