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DETERMINATION OF HUMIDITY IN OIL IMPREGNATED CELLULOSE INSULATION SYSTEMS

SUMMARY

The lifetime of oil impregnated insulation systems is strongly dependant to the mechanical strength of the cellulose molecules. Water accelerates the degradation of the paper insulation. For this reason the humidity in oil and oil impregnated cellulose insulation systems of power transformers is an essential factor for accelerated aging. Therefore the determination of the water content in the liquid and solid insulation is of high interest for lifetime assessment. In this paper the focus is laid on the different methods for the determination of humidity. Chemical as well as physical and dielectric methods were compared and the results of laboratory tests and tests at distribution transformers were discussed.

Key words: moisture determination, oil-paper insulation, diffusion, dielectric response

1. APPLIED TEST METHODS

For these investigations following diagnostic methods respectively calculation methods were carried out:

- Chemical determination of water in oil content with Karl-Fischer-Titration
- Dielectric response measurement in time domain with PDC and in frequency domain with FDS as well as combined method
- Physical determination of humidity in cellulose by weighing with a high accuracy balance
- Calculation of solid moisture by measurement with capacitive oil sensor

The focus on this paper is to investigate the different action-related dependences on the methods and to compare the results. Only with the knowledge of the sorption behavior of the cellulose the measured water content in oil can be converted to the water content in the oil impregnated cellulose. For this reason the sorption behavior of the cellulose is an important factor for the precision of the conversation from chemical to dielectric test results.

2. CHEMICAL DETERMINATION: KARL-FISCHER-TITRATION

Detecting the moisture content of solids or liquids by using the Karl-Fischer-Titration is a determination method that was introduced in 1935, so it is used for decades and therefore it is very well known [1].

The Karl Fischer coulometric titration determines the water content indirectly by electrochemically and chemically processes in absolute values. For this process there is also a chemical balance needed,

which accuracy must be at least 0,1µg, so that the accuracy of the complete measuring system is not degraded. The measured differential weight must be entered into the device and the relative moisture of the specimen is calculated digitally and shown by the Karl-Fischer-coulometer.

The range of the water content of mineral oil is within the range of 1 to 20 ppm (mg/kg) water. This value is not far away from the trace level, so the hole determination process with all handling must be done very carefully.

Basic functionality

Determining moisture by the coulometric Karl Fischer method is based on a two step chemically an electrochemically reaction that is specific to water. In the first stage sulfur dioxide forms with methanol an ester (alcyle sulphite) which is neutralized by a nitrogen base (RN):

$$CH_3OH + SO_2 + RN \rightarrow RNH_+ + CH_3OSO_{-2}$$

In the second step the alcyle sulphite is oxidated by iodine to alcyle sulphate and there is consumed water.

$$RNH_+ + CH_3OSO_{-2} + I_2 + H_2O + 2\ RN \rightarrow 3\ RNH_+ + CH_3OSO_{-3} + 2\ I_-$$

The electrochemically part of the process is the production of the iodine. It is produced at the generator electrode of the titration cell.

$$2I_- + 2e \rightarrow I_2$$

By Faradays law the amount of produced Iodine molecules is by determining the applied charge exactly specified.

In simple phrases, the Iodine created by the generator electrode from iodide combines with water and it is exactly determinable. And so the water content is by the molar weight easily calculable.

And if all water brought in the titration cell by the sample is combined with Iodine, there will be an overproduction of Iodine and this overproduction is noticed immediate by a sensing electrode with the amperometric measurement method.

The aim of the titration algorithm is to touch the of overproducing Iodine, because there is always a small amount of water entering the cell, what causes the permanent drift.

Determination Process:

- The titration cell has to be in a steady state with a constant drift
- The sample is brought into the cell and the process must be started
- The cell produces Iodine and combines it with water until the water is completely consumed and the titration value reaches the value of the drift prior the start of the titration
- The measuring electrode senses the unspent iodine and stops the process
- The current is totalized over the titration time and the absolute weight of the combined water is calculated
- After entering the weight of the specimen the relative water content is computed

Determining moisture in liquid specimen e.g. transformer oil

The determination of the water content in oil-samples is done by inserting a specimen of about one ml with a syringe with a canula through the diaphragm of the titration cell. After starting the titration process the Karl-Fischer coulometer produces enough iodine to absorb the whole moisture of the specimen and stops when the drift level from prior the titration-start is reached. Now the weight of the inserted specimen must be determined accurately by a differential weighing of the syringe and entered into the coulometer, so it can calculate the relative water content.

Determining moisture in solid specimen e.g. cellulose

Cellulose releases its moisture only slowly and at higher temperatures, so it is not suitable for the direct titration. It is necessary to extract the water by heating the specimen up until the water vaporizes. This is done with a Karl-Fischer titration oven. So the released moisture can be transported in the titration cell by flushing the oven with a flow controlled dry carrier gas e.g. nitrogen. The measuring procedure is the same as for liquid samples. Before inserting the specimen into the oven it has to be weighed for calculating the relative water content.

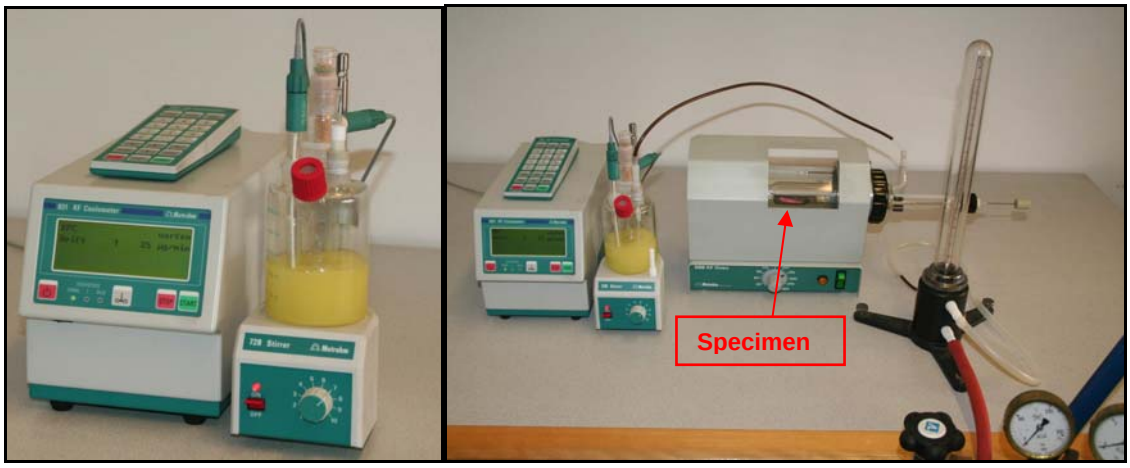

Figure 1 - Karl Fischer titration setup for moisture determination
LEFT: Setup for liquid specimen; RIGHT: Setup for solid specimen;

3. DIELECTRIC RESPONSE MEASUREMENTS: FDS AND PDC

The determination of paper moisture with dielectric measurement is sufficient known and many publications can be found. At this time only the results of a FDS and PDC measurement should be given. In following figure a FDS analysis was applied at a distribution transformer the humidity is determined automatically by the software of the measuring equipment. In the similar way the humidity of transformer board was determined by PDC measurements with the software of the PDC analyzer.

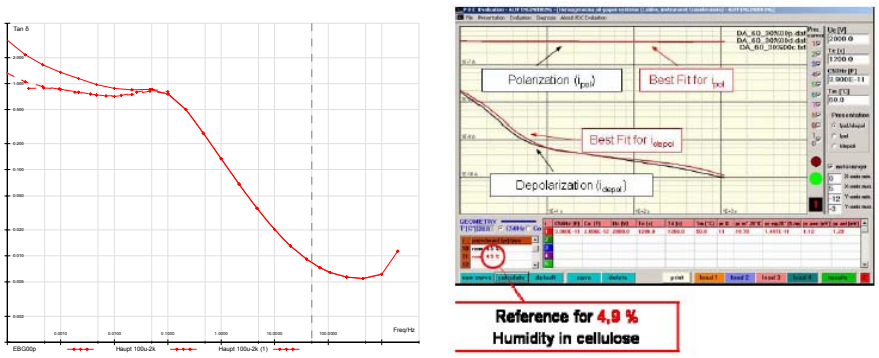

Figure 2 - Results of FDS and PDC Analysis

4. PHYSICAL DETERMINATION: WEIGHING

One of the most reliable methods to determine the moisture is to determine the absolute amount of humidity in the insulation material by weighing with a chemical balance. The best accuracy can be achieved if the test objects were first dried in a vacuum chamber (e.g. at 70°C for 2 days). The test objects should have a minimum of residual moisture as reference point.

To determine the sorption behavior the paper samples have to be stored in a climate chamber with defined environmental conditions. The temperature and air humidity should be measured with a

reference system. It takes some time until a equilibrium is reached, for this reason the paper samples should be placed in the climate chamber for a minimum of one hour. For the weighing process the samples should be done into a sealed vessel (small glass bulb). The content of moisture in % in cellulose is the difference of the measured weight of a sample at an assigned moisture value related to the dry weight.

At equilibrium, the relationship between water content and equilibrium humidity of a material can be displayed graphically by a curve, the so-called sorption isotherm. For each humidity value, a sorption isotherm indicates the corresponding water content value at a given, constant temperature. If the composition or quality of the material changes, then its sorption behavior also changes. Because of the complexity of sorption processes, the isotherms cannot be determined by calculation, but must be recorded experimentally for each product. Sorption isotherms (T=constant) often show a difference between adsorption and desorption. The hysteresis is determined by the hygroscopic nature of the wood fiber [2].

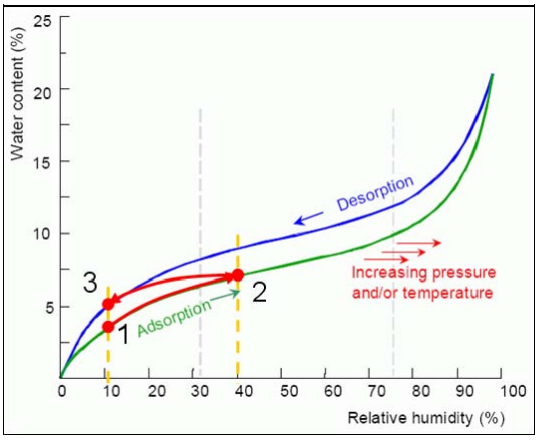

Figure 3 - Adsorption and desorption isotherm for cellulose [2]

The relative humidity can be calculated according to equation (1).

Table 1 – Moisture in insulation systems

p	[hPa]	Moisture vapour pressure in atmosphere
p_0	[hPa]	Moisture vapour pressure of water
T	[°C]	Temperature of moisture vapour in atmosphere
rh	[%]	Relative humidity

$$\text{relative humidity rh in \%} = 100 \cdot \frac{p}{p_0(T)} \qquad (1)$$

In following table the values of the vapour pressure of pure water and the saturation values of cellulose are shown in the temperature range from 0 to 100 °C.

Table 2 – saturatiuon values

temperature T in °C	***vapour pressure of pure water p_0 in Pa***	***saturation value of moisture in cellulose S_c in % (hypothetic)***
0	610	20,7
20	2300	19,5
40	7400	18,2
60	19900	17,0
80	47300	15,8
100	101300	14,6

$$\frac{W_{oil}}{S_{oil}} = \frac{p_{oil}}{p_0} \qquad (2)$$

The temperature characteristics of the solubility values of oil and cellulose are totally different. The solubility of moisture in oil increases with temperature, but in cellulose it decreases. Therefore moisture condenses in oil at dropping temperatures while in cellulose saturation occurs at increasing temperatures. Using the relative concentration W/S plotted versus the relative vapour pressure p/p_0 the influence of the temperature is minimized. In case of mineral oil a simple relation is given by equation (2). This equation appears as a straight line in Figure 4. The dotted line indicates that a non linear behaviour

could be found close to saturation. For fibrous cellulose the behaviour is more complicated. John D. Piper published sorption curves based on literature of moisture vapour pressure measurements of cotton and spruce wood. All measurements were performed in wet air without oil. The content of moisture in cellulose seems to be unaffected by the impregnation of oil. Oil merely retards the moisture migration process [3, 4, 5].

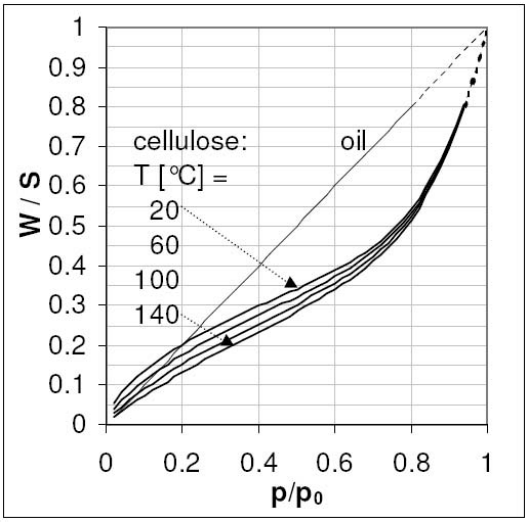

Figure 4 - Relative moisture in cellulose and oil [2]

5. DETERMINATION OF PAPER MOISTURE BY MEASURING THE OIL MOISTURE AND CALULATION WITH DIFFUSION EQUATIONS

With the knowledge of the moisture content of oil and the equilibrium curves of paper and oil the diffusion equation can be applied to calculate the moisture in board. The diffusion equations were applicable under steady-state conditions. Measuring the moisture content of oil with a capacitive sensor and knowing the temperature of the system the humidity content of paper can be calculated. In following figure 5 an example of calculation and measurement (Karl-Fischer-Titration) is shown.

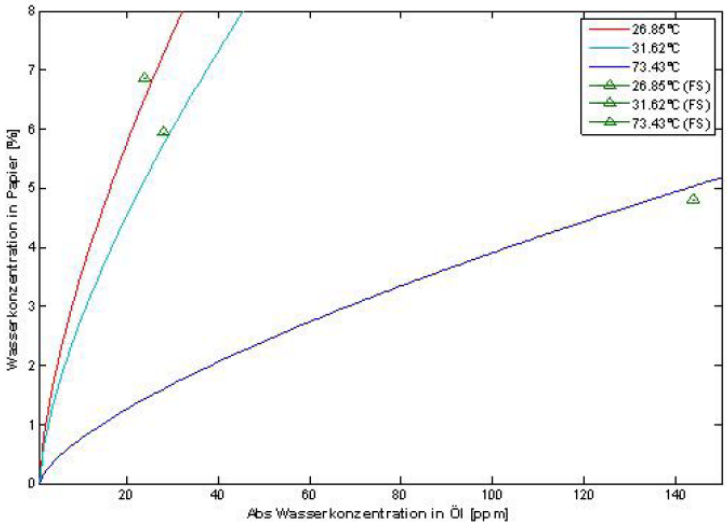

Figure 5 - Relative moisture in cellulose and oil calculated and measured with Karl-Fischer-Titration [6]

6. CONCLUSIONS

In these investigations different possibilities to determine the moisture content in paper were done. At first the chemical method by Karl-Fischer-Titration was applied. Then dielectric measurements were carried out at different oil board arrangements and power transformers. In the next step the moisture content was measured by weighing at defined climatic conditions. And finally the oil moisture was measured with a capacitive sensor following a calculation of the paper moisture by the diffusion equations.

Comparing the methods following results were obtained: The capacitive sensor and the weighing as well as the Titration showed a good accuracy. The results of the dielectric measurements can not be compared directly because different geometric arrangements and varying paper qualities were used [7].

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