NEW DESIGN FOR THE MEASUREMENT OF LIQUIDS WITH NANOPARTICLES

Marian Klampar, Milan Spohner, Aneta Lontrasova

Doctoral Degree Programme (4), FEEC BUT

E-mail: xklamp00@stud.feec.vutbr.cz

Supervised by: Karel Liedermann

E-mail: liederm@feec.vutbr.cz

Abstract: Measuring dielectric properties of liquids is a very complex issue. Researchers have been trying hard to find the most precise way to evaluate the characteristics of liquids. This is why we deal with this issue in this paper. Measurements procedures for liquid samples have changed throughout decades. The carrying out measurements have changed their shape and size; the revolutionary feature is the ability to adjust the thickness and the amount of liquid because the thinner the samples, the larger capacity and better ability to measure material attributes. This improved design cannot eliminate errors of metering equipment, like values around 50 Hz where it leads to realignment of crystals, but aids to do away with many inaccuracies during measurements of dielectric attributes of liquids.

Keywords: dielectric relaxation spectroscopy, liquids with nanoparticles, Teflon, metal

1. INTRODUCTION

Several methods are used in measurement of liquid samples in dielectric relaxation spectroscopy. One of them is insertion of electrodes into a container with a liquid. This method was used around 1969 [4]. Its core advantage is simplicity, because the container itself was one electrode and the second electrode was submerged into the liquid to form a circuit [3]. This initial experiment was used to acquire only approximate results because it was difficult to determine piston area and liquid thickness; thus correct permittivity calculation was not possible. Over the time, research led to a more sophisticated way to evaluate liquids. The electrode system in fig. 1 contains the liquid studied, evenly distributed in order to properly fill space, the volume of which we know. It is easier to calculate particular dielectrical properties of the liquid using this value [5].

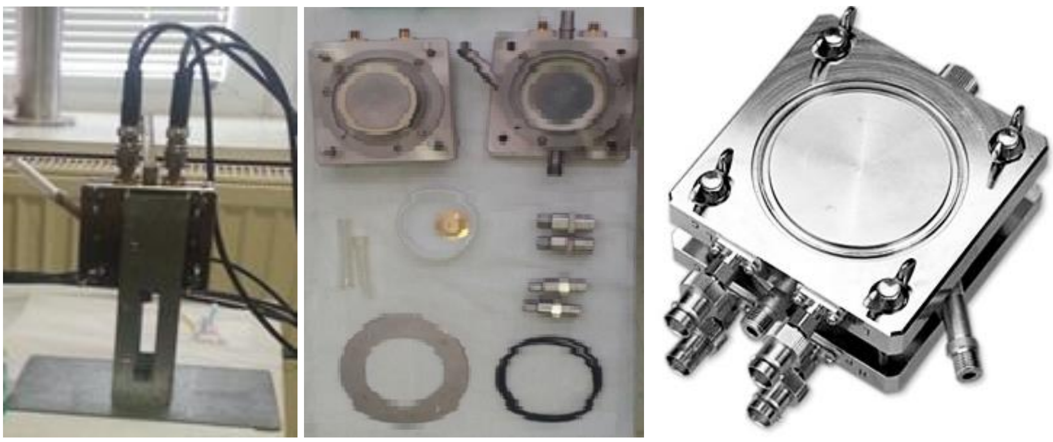

Figure 1. The current use of system

2. PRESENT STATE AND BACKGROUND

Dielectric relaxation spectroscopy (DRS) has been an established tool for the investigation of molecular dynamics of materials for a long time [1]. Developments in instrumentation and in measurement technique allow the study of dielectric properties in wide frequency and temperature ranges (10^{-6} Hz – 10^{15} Hz, 10 K – 1000 K) and for very different materials, ranging from materials for biomedical engineering up to insulations for HV rotating electric engines and power grids [2].

At the present time there are many devices and methods used for the measurement. In this research, the sample was inserted into an electrode system shown in Fig. 1. This system test fixture from Agilent was connected to the LCR bridge and the data were further processed by the computer. In spite of corrections that have been enabled on the device, the data was reflecting large errors in the samples. Therefore, the research had to focus to the test fixture design.

3. EXPERIMENT AND RESULTS

Principle of the electrode measurement system is very similar to syringe. We can always accurately determine the volume of its content, even when it's very small. Syringe can be used to put the liquid into the measurement system, but the system can be filled without it.

The system test fixture consists of: main section, upper electrode, lower electrode, pressure electrode and supplemental components and an adjustable screw with micrometric thread used to set sample thickness.

Subsequently a demonstration of a measurement is plotted into a graph, where results from older and newer electrode measurement system can be compared.

3.1. MAIN SECTION (BODY)

The main body has cylindrical shape with height ranging from 10 mm to 40 mm; diameter is in range of 5 to 45 mm. This part is made of Teflon, see fig. 2.

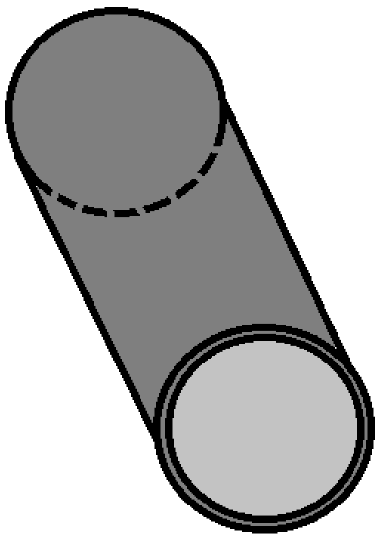

Figure 2. Main body made of Teflon.

3.2. UPPER ELECTRODE

This electrode serves also as a sealant for the upper part of the main body. It consists of two rings; the smaller one has the same outer diameter as the inner diameter of main body, so it seals the main body as a bottle cap. Another feature is a small hole, through which residual liquid and air can escape, which might otherwise lead to measurement errors. This part is usually made of solid and highly conductive material, like copper, see fig. 3

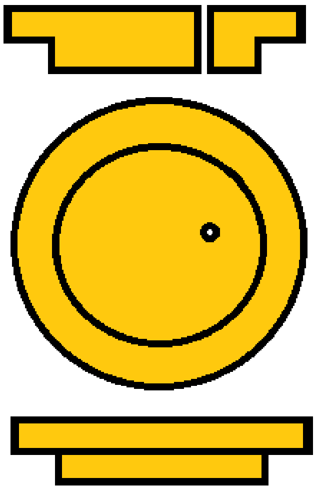

Figure 3. Upper electrode with a hole through which the air can escape

3.3. Lower Electrode With Pressure Adjustable Electrode

Lower electrode, as the name suggests, is located in the lower part of the measurement text fixture. It is similar to upper electrode, except that it has large threaded hole in the middle. This opening prevents air from taking in. It serves also as entry for pressure adjustable electrode. This electrode can be compared to a screw, which has flat head and precisely grips to Teflon sides of main body, so the liquid cannot flow along them. This screw is reversely screwed in from lower part, see fig. 4.

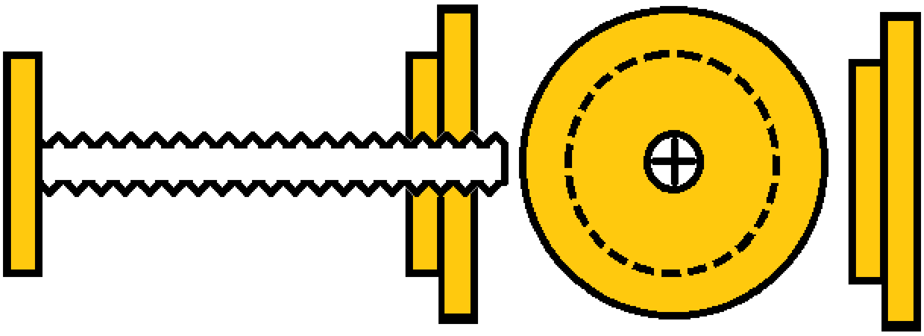

Figure 4. Lower electrode with pressure adjustable electrode body

3.4. Complete Electrode System With Configuration

As observable in fig. 5, after assembling all the parts, an adjustable electrode system is built, which is configured by adjustable micro-metric screwdriver, using which we can precisely set the thickness of the liquid sample with accuracy of micrometres. This leads to very accurate sample determination and more precise measurement.

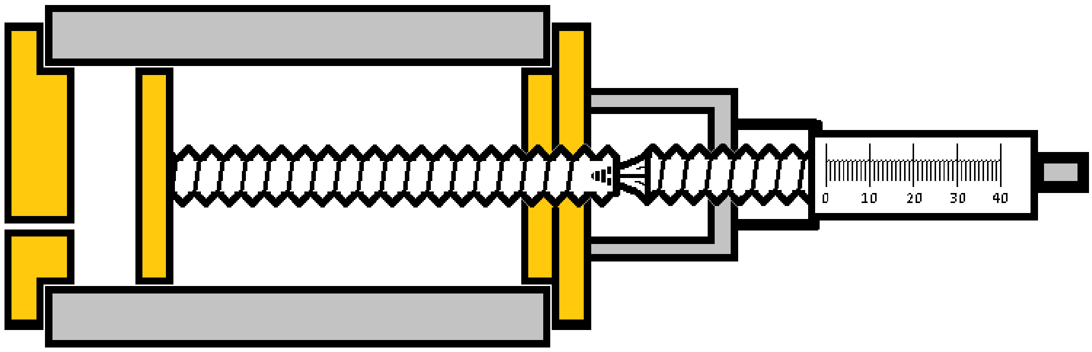

Figure 5. Complete assembly of the newly proposed test fixture with calliper.

3.5. Graphical Method Comparison

Fig. 6 shows two measurements with identical liquid, but in different measurement systems. It can be seen that there are slight deviations in original system, whereas these deviations are negligible in the new electrode system. Permittivity deviation measured between old and new electrode system from permittivity supplied by Centro de Física de Materiales.

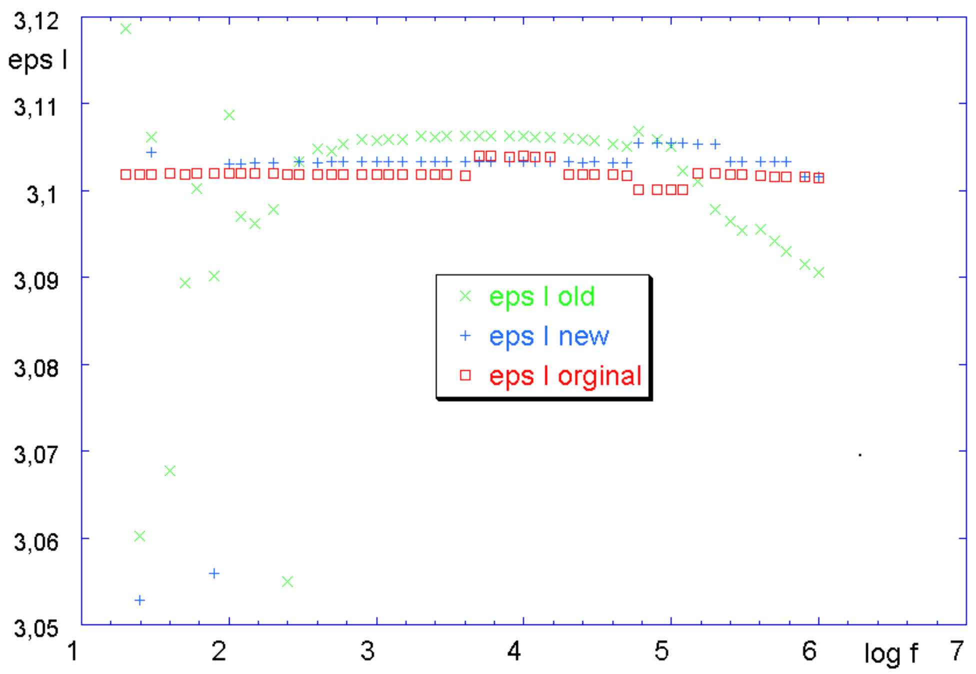

Figure 6. Graphical method comparison Sunflower oil (MINA) [5]

Graph in Figure 6 shows two our measurements. The first, tagged as "old", was measured on Agilent Test Fixture. The second, "new", was carried out on system we have designed. It can be seen that there are various differences between electrode systems, but our new system provides more accurate results.

4. CONCLUSION

To conclude, I would like to note that from the graph above it can be seen that this electrode system has a potential to replace the current measurement system, mostly in areas where the liquid studied is very rare or expensive, so they can be evaluated in small volumes.

Using this system it is possible to measure samples from maximum thickness 40 mm to 10 µm.

ACKNOWLEDGEMENT

Research described in the paper was financially supported by the European Centres of Excellence CEITEC CZ.1.05/1.1.00/02.0068 and by the Sensor, Information and Communication Systems (SIX) research centre. The SIX centre, CZ.1.05/2.1.00 /03.0072, was established by the operational program Research and Development for Innovation, which is a joint project of the Czech Ministry of Education and of the European Regional Development Fund

REFERENCES

[1] F. Kremer, A. Schönhals, “Broadband Dielectric Spectroscopy”, Springer, Heidelberg, Germany, 2002

[2] R. Pfaendner, “Nanocomposites: Industrial opportunity or challenge?” Polym. Degrad. Stabil., 95 (2010) 369 – 373

[3] K. Liedermann, M. Klampar, “Methods of production samples of dielectric spectroscopy” 2012, EEICT Brno

[4] 2nd LABORATORY COURSE ON DIELECTRIC SPECTROSCOPY 30 May -3 June 2011, San Sebastian – Spain

[5] M. SPOHNER, K. LIEDERMANN, M. KLAMPÁR, Electrical properties of natural oils and methyl esters of their constitutive fatty acids. In *IEEE Catalog Number CFP13EEI- USB.* 2013. s. 135-140. ISBN: 978-1-4673-4739- 6