Non-destructive Microstrip Resonator Technique for the measurement of moisture / permittivity in crude oil

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Abstract: An accurate, instantaneous and non-destructive Microstrip Resonator Technique has been set up for the measurement of permittivity and moisture of materials in solid, liquid and powder form. This paper reports the permittivity measurements in crude oil and water emulsions made in the laboratory. The resonant frequency of the microstrip resonator is measured in the presence of crude oil and the real part of the permittivity of the material under test is determined with the help of spectral domain analysis of multi-layer microstrips, which are embedded in dielectric cover. The relatively low values of permittivity of high oil percentage emulsions may be attributed to the in-pouch testing procedure.

Keywords: crude oil, emulsion, permittivity, watercut

1_Introduction:

Many multiphase flow and watercut meters used for fiscal and quality purposes are based on established electrical impedance technique. Knowledge of the relative permittivity of flow components is fundamental to such technologies. Calibration of multiphase flow meters requires permittivity data of crude oil and its components [1]. The other reason for importance of the permittivity measurements lies in remote sensing applications. Microwave radiometry of regions with oil spills needs oil and emulsion permittivity data to establish the thickness of oil films from the emissivity data [2].

In the petroleum refinery industry, there is increasing demand for on-line determination of product quality. Dielectric spectroscopy has previously been shown to be a relevant method for quality characterization of petrochemical products. Several parameters describing the quality of the oil can be found from the measured permittivity spectrum. Oil has low permittivity and loss, long relaxation times and a broad frequency dispersion region. Hence, the permittivity spectrum has to be measured with high sensitivity over a broad frequency range (typically 1 kHz to 10 GHz), which requires sensitive measurement cells and adequate models for calculating the permittivity.

At low frequencies (1 kHz to 10 MHz), the impedance measurements of coaxial cells give high-precision measurement of permittivity [1]. At radio and microwave frequencies, transmission and reflection measurements of coaxial cells using network analyzers are a suitable method for fast and broad-band permittivity determination of low-loss liquids [3]. The accuracy in the measurements is lower than that which is obtained with resonator techniques, but the resonator methods are time-consuming when applied in a broad range of frequencies [4]. In comparison, a coaxial cell can typically be used over 1-2 frequency decades.

When measuring the parameters of petroleum products, a representative sample of the flow must be taken. Considering the industrial applications of dielectric spectroscopy, an obvious drawback is that the cell is intrusive. Waveguide resonant cavity techniques have been widely used to perform non-destructive measurement of the moisture as well as complex permittivity of materials. Though considered accurate, the waveguide cavity technique is associated with difficulties like loading and unloading of samples. Out of the possible measurement principles, electromagnetic interaction is promising for cases where the fluid components have distinct dielectric properties.

ABB has published a report on a multiphase meter based on a special slow-wave resonator operating in the radio frequency range [4]. The dielectric properties of the fluid (permittivity, conductivity) determine the dispersion and, hence, the resonance spectrum of the measurement device. Microstrip resonator techniques, based on statistical or approximate closed form dependence of ε' and ε'', which are being used in the industry, do give sufficient accuracy for the crude oil industry.

To go a step ahead, we have implemented a novel technique for the measurement of complex permittivity for crude oil and water emulsions. The technique has used the microstrip resonator as a non-intrusive sensor, which can be used along with the flow meter itself without the need for sampling of the oil.

2_Experimental set-up:

The set up for measuring the rise in effective permittivity and the drop in the resonant frequency of the ring resonator with increase in the percentage of moisture in the crude oil sample were specifically designed for petroleum-drilling applications, keeping in mind that the occurrence of crude oil is always accompanied by water or moisture. It is this concurrent occurrence that could pose difficult challenges. Hence, by providing a non-destructive technique to measure the percentage of moisture in a sample of crude oil, these difficulties could be alleviated. In order to measure the percentage of moisture in the crude oil, firstly, the resonant frequency of the microstrip resonator is measured in the presence of crude oil while the real part of the permittivity of the material under test is determined with the help of spectral domain analysis of multi-layer microstrips embedded in dielectric cover.

The tests were performed using a 5 GHz ring resonator on substrate of dielectric of 4.7. We intended to quantify a relationship between the change in parameters of the resonator with change in its environment. The change in the composition of oil and water is effectively changed in the environment of the resonator circuit. We used this fact in our testing procedure.

3_Theoretical Background and Post-processing of Data:

Microwave resonator technique is a powerful tool for the measurement of moisture / dielectric constant of the material under test since, with the availability of modern network analyzers, frequency measurement is not subject to measurement device errors like drift and repeatability. The present paper reports an accurate and fast watercut measurement, using a microstrip resonator. The material under test is kept as an overlay on the microstrip probe. The resonant frequency and the quality factor of microstrip resonator are measured. The change in the effective permittivity of the microstrip probe is given by

$$f_o^2/f_s^2 = \varepsilon_{effs} / \varepsilon_{effo} \quad (1)$$

where suffixes “o” and “s” indicate resonant frequency (f) and effective permittivity (ε_{eff}) without and with the sample as an overlay on the resonator cavity. For low loss materials,

$$\Delta f_o = f(\varepsilon_{rs}') \quad (2)$$

$$\Delta Q = f(\varepsilon_{rs}', \varepsilon_{rs}'') \quad (3)$$

where ε_{rs} is the relative permittivity of the overlay sample and ε_{rs}' and ε_{rs}'' indicate real and imaginary parts of the complex permittivity. ε_{rs}' of the material under test is determined with the help of spectral domain analysis[3,4].

Specially-created and dedicated software EPSILONCALC™ has been used to determine the real of the permittivity of the oil-water emulsions in the plastic pouch. EPSILONCALC™ The software for material permittivity measurement is applicable to the materials with infinite as well as finite depth. Thickness and width of the samples need to be supplied as parameters to the software.

4_Results and Discussion:

Tables 1-4 give present and other published data on permittivity of crude oil, which seems to be around 2. The standard deviation is 5% in most cases. Figure 1 gives the experimental data on the variation of resonant frequency with water in the emulsion kept on ring resonators as overlay. The emulsions were kept in the zip-bag pouches for avoiding oil on the resonators. Figures 3-4 give simulated data using the software EpsilonCalc™, which takes only a fraction of a second for single frequency point. The software has been validated using other commercial suppliers and experiments. The resonator data as well as material height needs to be supplied to the interactive software. Figures 4-6 give coaxial probe permittivity data for crude oil. However, it was not possible to compare higher moisture data due to unavailability. Table 4 gives a comparative review of the various publications.

Table 1: Average value and standard deviation for the high-frequency permittivity, Є, of the 16 repeated measurements[5]

Fluid	Average permittivity	Standard deviation
p-xylene	2.327	0.005
Crude oil	2.034	0.113

Table 2: Estimated Cole–Cole parameters of three North Sea crude oils [3]

Crude oil	ϵ_S	ϵ_∞	τ (ns)	α	σ (nS m^{-1})
Oil 1	2.295	2.169	3.35	0.54	42.9
Oil 2	2.324	2.198	11.3	0.52	2.0
Oil 3	2.248	2.151	2.40	0.50	11.4

Table 3: Permittivity calculations from the present study using EPSILANCALC Open Resonant Frequency 5.3GHz, (Material FR4-Єr=4.7)

Oil %	Refractive index equation Є'$_{oil}$	Present tech Є'$_{oil}$	Resonant Frequency (GHz)	Effective permittivity Є$_{eff}$
100	2.0	1.56045	5.148	3.757227
90	4.37	1.865625	5.086	3.800496
80	7.66	4.159212	4.915	4.159212
50	15.85	20.741224	4.272	5.383967
20	58.97	39.11376	3.709	7.140352

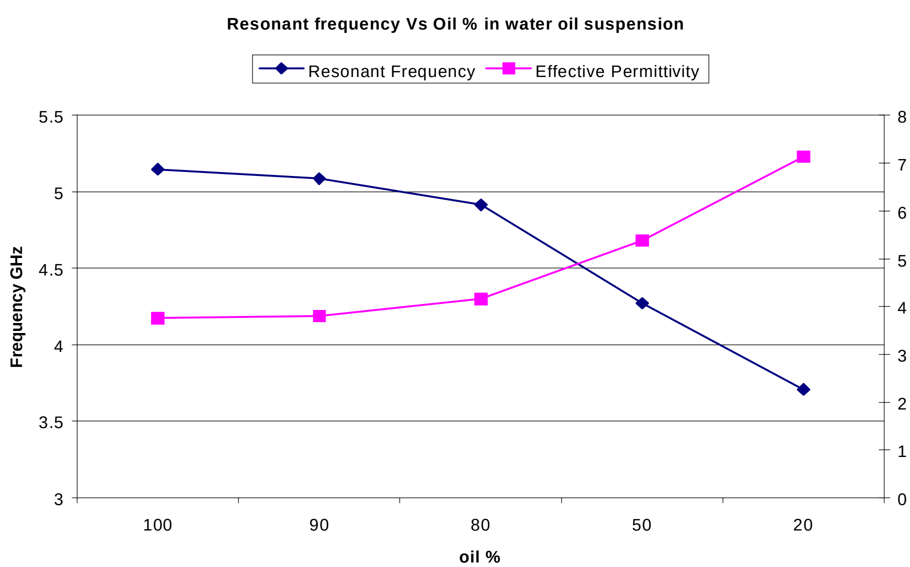

Figure 1: Resonant frequency variation with water percentage in crude oil.

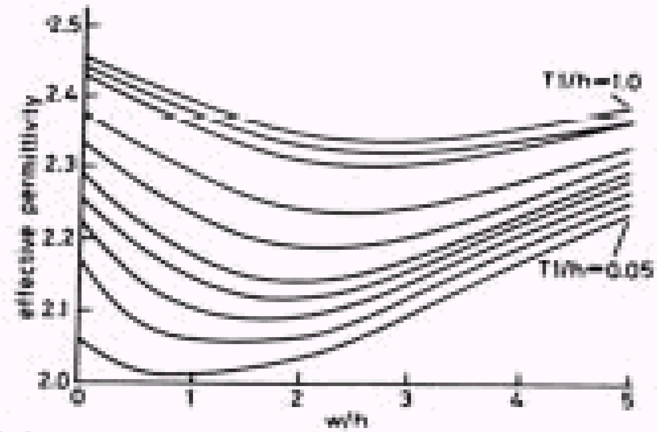

Figure 2: EpsilonCalc™ results for variation of effective permittivity with dielectric cover thickness and w/h ratio of the microstrip $\epsilon_{substrate}$ and ϵ_{cover} =2.53, h=1.27 mm

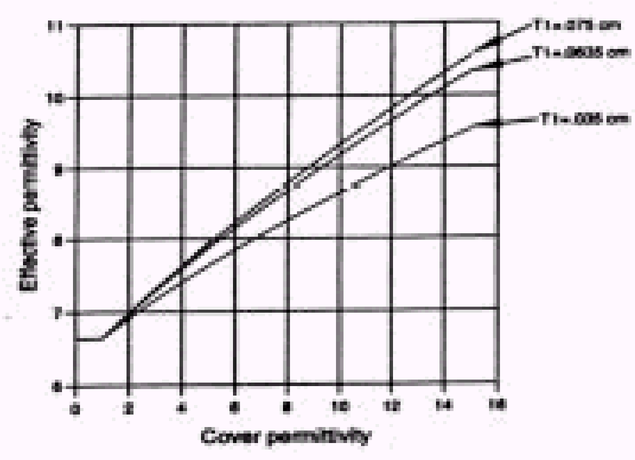

Figure 3: EpsilonCalc™ results for variation of effective permittivity of a microstrip with dielectric cover thickness and cover permittivity $\epsilon_{substrate}$ =9.99, h=0.635 mm

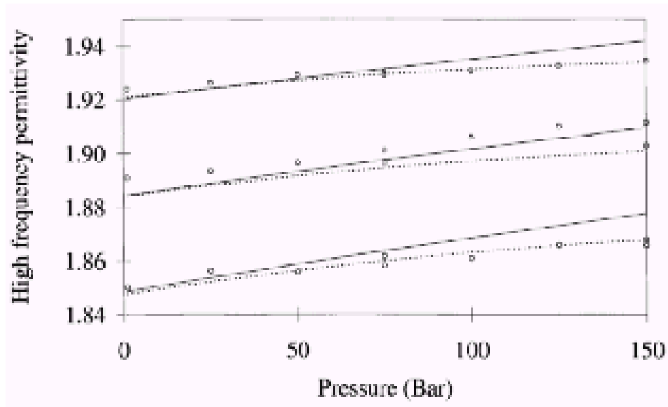

Figure 4: Measured permittivity (O) of n-heptane as a function of pressure and temperature at 20, 40 and 70^0C (from top to bottom respectively) using an open-ended coaxial probe[5]

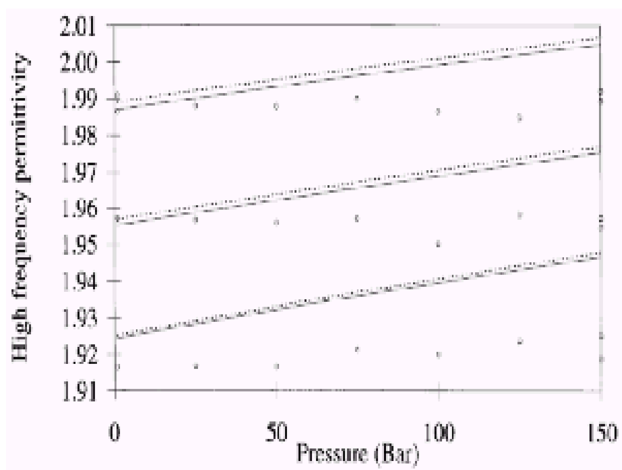

Figure 5: Measured permittivity (O) of n-decane as a function of pressure and temptarature at 20, 45 and 70^0C (from top to bottom respectively) using an open-ended coaxial probe[5]

Table 4: High frequency permittivity values from the literature

High Frequency permittivity				
Liquid	Simple calibration	Bi-linear calibration	Literature values	Reference
n-pentane	1.80	1.84	1.844 1.841	6 7
n-decane	2.02	1.99	1.987 1.991 1.989	8 6 7
p-xylene	2.45	2.27	2.270	6

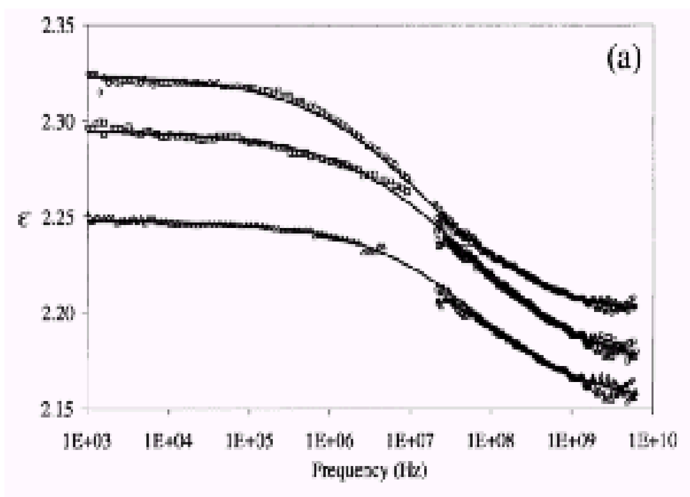

Figure 6: Measured permittivity (O) of North Sea crude oil as a function of frequency using transmision type coaxial cell[9]

5_Conclusion:

The results from the present work compared with the published data indicates that the novel numerical analysis technique can be used for crude oil watercut measurements. The technique is very fast in the numerical postprocessing and can give on-line measurement device with the measurement time in milliseconds. The possible applications would be well-logging, refinery flow meters, quality control and mixed fuel sensor. The lower values of permittivity are attributed to the in–pouch testing procedure. The measurements will be repeated for the direct contact and non-contact measurements to avoid contamination of the sensor surface.

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