

Proceeding Paper

Electrodeless Studies of MXenes in Aqueous and Polar Non-Aqueous Aprotic Solvent [†]

Oksana Gutsul ^{*1} and Vsevolod Slobodyan ²

¹ Bukovinian State Medical University, Chernivtsi, Ukraine; gutsul@bsmu.edu.ua

² Yuriy Fedkovych National University, Chernivtsi, Ukraine; v.slobodyan@chnu.edu.ua

* Correspondence: gutsul@bsmu.edu.ua

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Abstract: MXenes attract a lot of attention due to their unique properties, in particular, high electrical conductivity. The physical processes occurring during the electrodeless studies of the specific electrical conductivity σ of MXenes in distillation water and in a polar non-aqueous solvent of N-Methyl-2-Pyrrolidone (NMP) at fixed resonant frequencies for five solenoids ($f_1 = 160$ kHz, $f_2 = 270$ kHz, $f_3 = 1.6$ MHz, $f_4 = 4.8$ MHz, $f_5 = 23$ MHz) are considered. The oscillating circuit was tuned to resonance by changing the capacitance of the BM-560 Q-factor meter. The Q factor of the oscillating circuit was measured in the range of 100–300 with a maximum relative error of $\pm 5\%$, and in the range of 30–100 with a maximum relative error of $\pm 3\%$. The cylinder with the liquid was placed in the middle of the measuring solenoid, in the area of a homogeneous magnetic field. The measurements were performed for four control volumes of the liquids under study (1 mL, 2 mL, 3 mL, 4 mL). The best measurement sensitivity was observed for the maximum volume of the liquid (4 mL). A difference between the experimental dependences of the introduced attenuation d of the oscillating circuit with a cylinder with MXenes in aqueous and non-aqueous polar solvent NMP was observed. The nonlinear dependence of the attenuation of the oscillatory circuit d on the volume of the studied liquids was analysed. The maximum value of the attenuation of the oscillating circuit for the solenoid at the resonant frequency of 160 kHz was observed for the NMP-MXenes measurement, in contrast to the study of MXenes in distillation water have the highest attenuation at a frequency of 1.6 MHz.

Keywords: MXenes; Q-factor; electroconductivity; aqueous and non-aqueous solvents

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1. Introduction

MXenes attract a lot of attention due to their unique properties, in particular, high electrical conductivity [1–5].

Electrodeless studies of the specific conductivity of liquids are relevant for many fields of science and production, including technological processes using both chemically clean and chemically aggressive liquids. The study of the properties of aqueous solutions of electrolytes for a wide range of concentrations is of great interest both for practice and for the development of theoretical concepts [6]. The current state of research on the electrophysical parameters of liquids allows obtaining information about the behavior and structures of various liquids [7,8]. Measurement of electrical conductivity in solids and liquids is usually carried out using, respectively, the contact method using electrodes. Therefore, the corresponding measurement methods are often called contact or electrode.

Currently, electrodeless and electrodeless methods are used to study the specific electrical conductivity of a liquid. Most of these methods were developed on the basis of the analysis of transient and stationary processes involving the object under study. The occurrence of these processes is caused by eddy currents formed in the object under study,

which depend on the object's structure and electrical conductivity. Measurement of weak eddy currents using inductive sensors leads to a significant increase in measurement error. An alternative to reduce the measurement error is the use of high-frequency resonance methods.

2. Materials and Methods

The solvent replacement method was used to prepare the solutions under study, as previously reported in [2]. Before the study, the prepared suspensions of Ti₃C₂-based MXenes in two solvents: in the polar aprotic solvent NMP and in distilled water were sonicated at 37 kHz during 30 min to prevent particle aggregation.

In the device for electrodeless measurement of the specific electrical conductivity of liquids, the eddy current in the cylinder with the liquid under study is inductively connected to the measuring oscillating circuit. Before examining the liquid in the cylinder, the Q-factor of the oscillating circuit is measured in the absence of liquid in the cylinder (Q_0), as well as the wave resistance ρ_0 :

$$\rho_0 = \omega L = \frac{1}{\omega C_1} = \sqrt{\frac{L}{C_1}}, \quad (1)$$

At the same time, the VM-560 Q-factor meter is tuned to the resonant frequency ($f_1 = 160$ kHz, $f_2 = 270$ kHz, $f_3 = 1.6$ MHz, $f_4 = 4.8$ MHz, $f_5 = 23$ MHz). Tuning for voltage resonance is carried out by changing the capacitance C_1 of the oscillating circuit.

When a cylinder with an inner radius $r = 10$ mm is filled with a liquid with a specific electrical conductivity σ , there is a decrease in the Q-factor of the equivalent oscillating circuit Q and a change in its wave resistance ρ , which can be measured experimentally.

The effect on the parameters of the oscillating circuit caused by the presence of liquid in the cylinder is described by the applied damping of the oscillating circuit d , which is calculated according to the formula [9]:

$$d = \frac{1}{Q} - \frac{1}{Q_0} = \frac{(\omega M)^2}{\omega L} \frac{r}{r^2 + x^2}, \quad (2)$$

where Q is Q-factor of the oscillating circuit with the tested liquid in the cylinder; Q_0 —Q-factor of the oscillating circuit in the absence of liquid in the cylinder; ω is the circular frequency of the oscillating circuit; M is the mutual induction coefficient; L is the inductance of the measuring solenoid; r is the active resistance to the eddy current in the liquid placed in the cylinder, x — the reactive resistance to the eddy current.

If the cylinder is not filled with liquid, then $\frac{1}{r} = 0$, hence $\frac{1}{Q} = \frac{1}{Q_0}$. The specific electrical conductivity of a liquid is inversely proportional to the resistance of the liquid $r = A/\sigma$. Therefore, the dependence of the introduced attenuation d on the specific electrical conductivity σ can be obtained according to the formula:

$$d = \frac{(\omega M)^2}{\omega L} \cdot \frac{A/\sigma}{(A/\sigma)^2 + x^2}, \quad (3)$$

where x is the reactive resistance to the eddy current, $A = l/S$ is a constant of the measuring complex, where l is the length of the eddy current in the cylinder, S is the cross section through which the eddy current in the cylinder passes.

The dependences of the introduced attenuation d on the specific electrical conductivity σ were obtained, which were used as calibration dependencies for the electrodeless measurement of the specific electrical conductivity σ of a fixed volume V of liquid in a cylinder of radius r based on the experimental measurement of the introduced damping d of the oscillating circuit.

The physical processes occurring during the electrodeless studies of the specific electrical conductivity σ of MXenes in distillation water and in a polar non-aqueous solvent of NMP at fixed resonant frequencies for five solenoids ($f_1 = 160$ kHz, $f_2 = 270$ kHz, $f_3 = 1.6$ MHz, $f_4 = 4.8$ MHz, $f_5 = 23$ MHz) are considered. The oscillating circuit was tuned to resonance by changing the capacitance of the Q-meter. On Figure 1 observe the electrical schem of the experimental setup for electrodeless measurement of liquids.

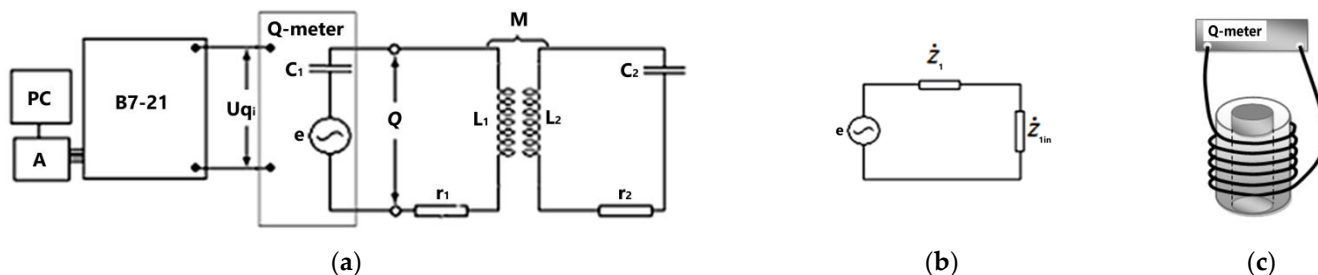
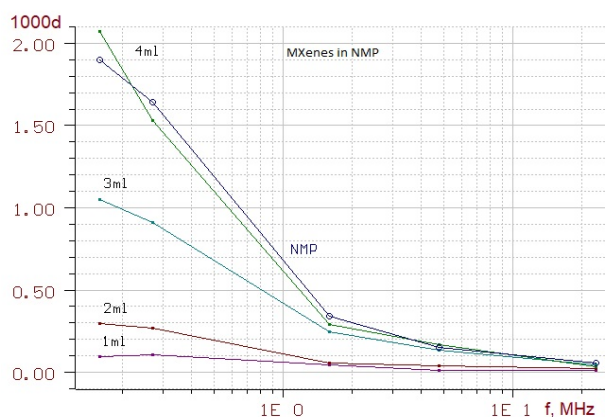


Figure 1. Schematic of the experimental setup for electrodeless measurement of liquids: (a) electrical schem of the measuring (1) and the test (2) parts of the setup; (b) equivalent diagram of two connected circuits; (c) schematic representation of the connection of the measuring solenoid to the Q-factor meter.

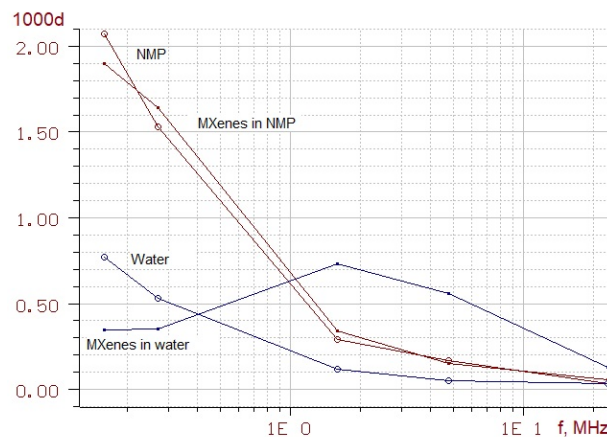
The Q-factor of the oscillating circuit was measured in the range of 100–300 with a maximum relative error of $\pm 5\%$, and in the range of 30–100 with a maximum relative error of $\pm 3\%$. The cylinder with the liquid was placed in the middle of the measuring solenoid, in the area of a homogeneous magnetic field. The measurements were performed for four control volumes of the liquids under study (1 mL, 2 mL, 3 mL, 4 mL).

3. Results and Discussion

The best measurement sensitivity was observed for the maximum volume of the liquid (4 mL). A difference between the experimental dependences of the introduced attenuation d of the oscillating circuit with a cylinder with MXenes in aqueous and non-aqueous polar solvent NMP was observed. The nonlinear dependence of the attenuation of the oscillatory circuit d on the volume of the studied liquids was analysed. The maximum value of the attenuation of the oscillating circuit for the solenoid at the resonant frequency of 160 kHz was observed for the NMP-MXenes measurement (Figure 2a), in contrast to the study of MXenes in distillation water have the highest attenuation at a frequency of 1.6 MHz (Figure 3).



(a)



(b)

Figure 2. Dependences of the introduced attenuation 1000 d on the resonant frequency f on the semi-logarithmic scale: (a) five different solenoids for four different volumes (1–4 mL) of MXenes dissolved in NMP and NMP solution separately; (b) comparison of 4 mL of four different tested solutions.

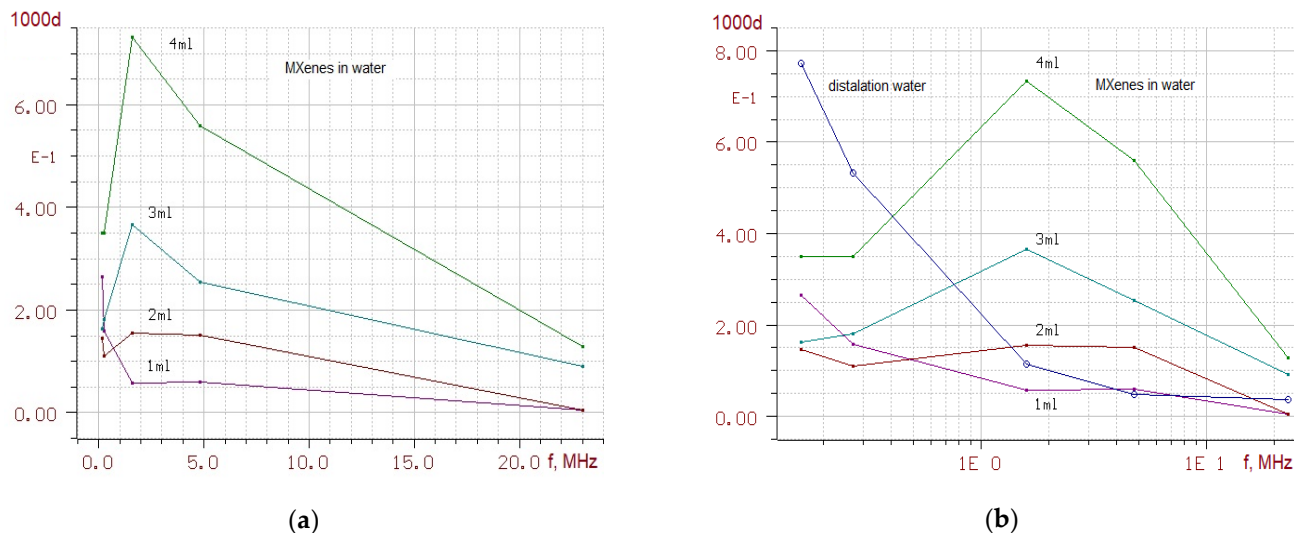


Figure 3. Dependences of the introduced attenuation 1000 d on the resonant frequency f of five different solenoids for four different volumes (1–4 mL) of MXenes dissolved in distilled water: (a) in simple scale; (b) in semi-logarithmic scale.

Figure 2b shows a comparison of the introduced attenuation at resonant frequencies for the same volume (4 mL) of the four different solutions studied: MXenes in NMP, NMP solution, MXenes in distilled water, and pure distillation water.

The experimental dependences of the introduced attenuation for aqueous suspensions of maxenes showed the greatest sensitivity at the resonant frequency of 1.6 kHz. With an increase in the volume of the suspension under study, an increase in the value of the introduced attenuation d was observed. For comparison, the frequency dependence of the introduced attenuation d for 4 mL of distilled water was also plotted. The graphical dependences of the introduced attenuation on the resonant frequency f on a semilogarithmic scale for distilled water (4 mL) and maxenes dissolved in distilled water for different studied volumes (1–4 mL) are shown in Figure 3b.

4. Conclusions

Experimental electroless studies of eddy currents depend significantly on the choice of solenoid and its resonant frequency. The sensitivity of the electroless method increases with the volume of the liquid under study. It has been experimentally established that it is advisable to conduct electroless studies of eddy currents in MXenes dissolved in the organic solvent N-methyl-2pyrrolidone in solenoids with resonant frequencies of 160 kHz and 270 kHz, and for MXenes dissolved in distilled water in a solenoid with a resonant frequency of 1.6 MHz. In general, this electrodeless method of investigation is appropriate for analysing the stability of MXenes in various solvents.

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