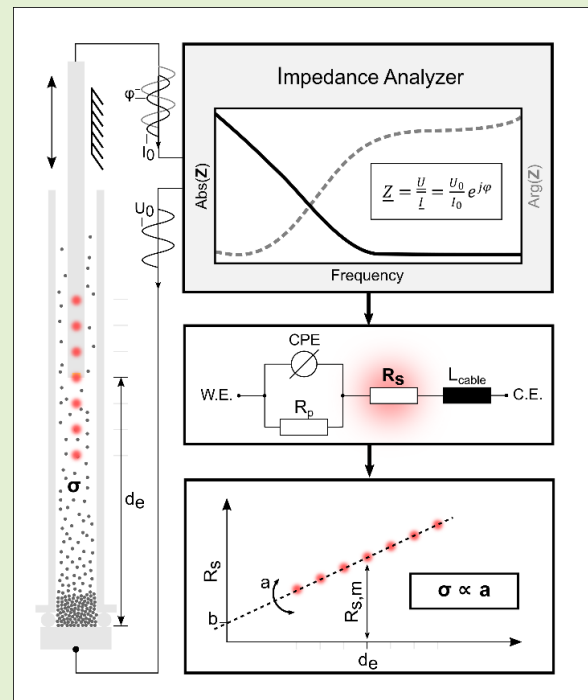


Using Moving Electrode Impedance Spectroscopy to Monitor Particle Sedimentation

Nikolaus Doppelhammer¹, Member, IEEE, Nick Pellens², Christine E. A. Kirschhock, Bernhard Jakoby¹, Senior Member, IEEE, and Erwin K. Reichel, Member, IEEE

Abstract—We have devised a method that employs moving electrode electrochemical impedance spectroscopy to monitor the sedimentation of particles in conductive suspensions. In contrast to standard electrochemical cells with a fixed geometry, our cell has a flexible design with a movable counter electrode that allows precise adjustment of the electrode distance. Measuring the electrical impedance at various electrode spacings and utilizing the linear dependence of this function on the electrode displacement enables probing of a small section of the sample. This has considerable advantages when heterogenous liquids (e.g., suspensions) are to be analyzed. We applied our moving electrode approach to various test cases and obtained the following results: (i) We demonstrated by experiment that the bulk conductivity can be measured correctly even if particle sediments cover the electrode surface. (ii) We studied monodisperse suspensions of various compositions and investigated the effect of particle concentrations and size on conductivity. (iii) We monitored the particle sedimentation process and, by combining experimental and theoretical results, identified a correlation between the growing mass of the sedimentation layer and the impedance measured. The intended application of our approach is to monitor crystallization processes in ionic liquids for use in zeolite synthesis.

Index Terms—Conductivity, electrochemical impedance spectroscopy, sedimentation, suspension, zeolite synthesis.



I. INTRODUCTION

THE conductivity of a liquid sample is usually measured by means of a cell with immovable electrodes. The simplest setup are two electrodes immersed in the test liquid. The specific conductivity can then be determined, for instance, by using electrochemical impedance spectroscopy (EIS) [1].

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For a simple parallel-plate electrode arrangement, the mathematical relation between the macroscopic solution resistance R_s and the microscopic conductivity σ is given by Pouillet’s law

$$R_s = \frac{d_e}{\sigma A_e},$$

where d_e is the electrode distance and A_e the effective cross section of the sample. In static cells, the term d_e/A_e is called *cell constant* and determined by calibration with a liquid of known conductivity.

A static cell design is suitable for measuring homogenous mixtures, where the conductivity is the same throughout the whole sample [2], [3]. For a heterogenous sample, such as a suspension which tends to phase-separate, a fixed cell design may provide results that differ from the average conductivity of a uniform sample, depending on the shape and spatial distribution of the suspended particles [4].

In this work we present a method – hereafter referred to as moving electrode electrochemical impedance spectroscopy (MEEIS) – which overcomes this problem by using a flexible

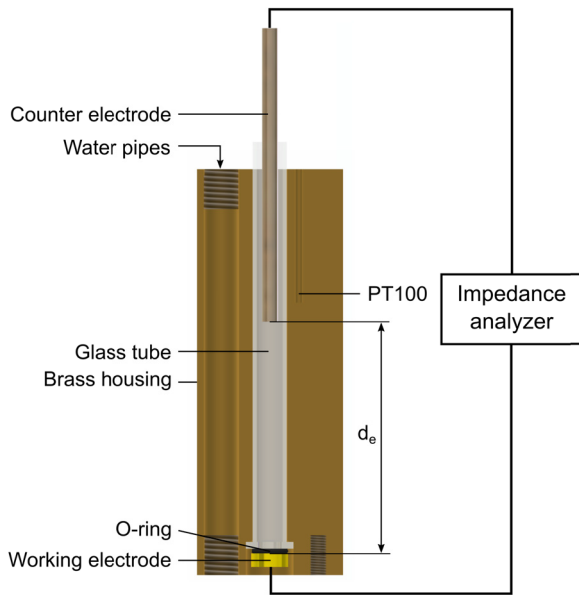


Fig. 1. Design of the MEEIS setup.

cell design where one of the two electrodes is movable. Thus, the inter-electrode distance can be adjusted, and the sample resistance can be probed at various points along the vertical axis. The information about how the impedance of the sample changes with the electrode spacing provides a focused view on the electrical bulk properties of a smaller proportion of the sample.

To test the applicability of our method, we performed several experiments in a specifically designed test setup. First, we discovered that the bulk conductivity measured by MEEIS is independent of layers of sedimented particles. Second, we analyzed the influence of particle concentration and size on the electrical parameters in monodisperse suspensions with uniform particle distribution. Finally, we demonstrate that our method is suitable for monitoring the particle sedimentation process, as it enables a direct conversion between change in impedance and the mass of sedimented particles.

We describe how this measurement technique can be applied to analyze complex crystallization processes, such as zeolite synthesis [5], [6]. MEEIS complements previously presented ways of measuring sedimentation, such as in-situ viscosity measurement [7] and acoustic streaming combined with an immersed microbalance [8].

Preliminary results from this work were presented at the IEEE Sensors 2019 in Montreal [9].

II. TEST SETUP

Fig. 1 depicts the experimental setup. The sample is contained in the elongated glass tube in the middle of the cell. The working electrode is located at the bottom of the setup and is squeezed against the flange of the glass tube using a silicone O-ring. The counter electrode is a commercial electrode (LAUE gold electrode, ALS, Japan), which is introduced from the top and mounted on a motorized linear stage. The distance between working and counter electrode is

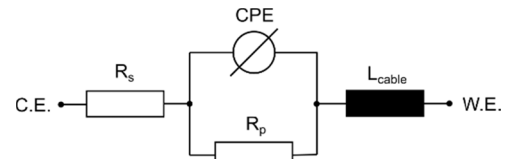


Fig. 2. Equivalent circuit model used to fit the EIS data.

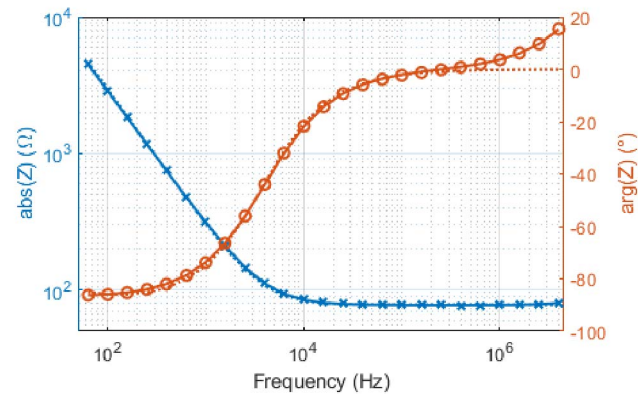


Fig. 3. Bode plot of measured and modelled impedance spectrum. 'x' and 'o' are the measurement values of impedance magnitude and phase, respectively. The dotted line is the fit using the standard SRC model and the solid line the fit using the modified SCR model.

adjustable from 80 to 110 mm. All electrode surfaces are gold coated to avoid corrosion due to the highly alkaline test liquids. The glass tube is enclosed by a brass cylinder which keeps the sample at constant temperature. The temperature control is done with a thermostat bath (HE-4 heating circulator, Julabo, Germany) and an external PT100 temperature sensor mounted inside the brass cylinder. Working and counter electrodes are connected to a potentiostat (Reference 600+, Gamry, USA), with which the impedance measurements were carried out.

III. DATA ACQUISITION AND ANALYSIS

Impedance spectra were recorded in the frequency range from 10 Hz to 4 MHz. The measured data was analyzed by fitting an equivalent circuit (EC) model. A frequently used model and usually a good starting point for analysis is the simplified Randles circuit (SRC) [10]. In our case, we used a slightly modified version of the SRC, depicted in Fig. 2. In comparison to the SRC, the double layer capacitance was replaced with a constant phase element (CPE), which proved to better resemble the non-ideal capacitive behavior of the double layer at the electrode-liquid interface. Also, a serial cable inductance (L_{cable}) was added to capture the influence of the wires at elevated frequencies. The quality of fit of both models, the SRC and our modification of it, are compared in Fig. 3. We see that both models adequately resemble the experimental observation with a slightly better fit quality for the modified SRC model.

By recording impedance spectra at different electrode distances and subsequent fitting of our EC model, we obtained the solution resistance R_s as a discrete function of the electrode distance $R_s = f(d_e)$. The theoretically expected and experimentally observed linear behavior of this relationship

was approximated with a linear equation of the form

$$R_s = ad_e + b, \quad (1)$$

where a denotes the slope and b the y-intercept of the function. The former is related to the electrical conductivity

$$\sigma = \frac{d_e}{R_s A_e} = \frac{1}{\frac{\Delta R_s}{\Delta d_e} A_e} = \frac{1}{a A_e}. \quad (2)$$

Compared to static cells, where the conductivity is determined from the absolute value of the solution resistance, the conductivity in Eq. 2 is calculated from the slope a , which represents the change of the solution resistance in a small section of the sample, in our case in the range of the adjustable electrode distance from 80 mm to 110 mm.

To probe for the rest of the sample as well, especially for sedimentation effects near the electrode-liquid interface, the absolute value of the solution resistance was needed. In principle, any value of $R_s(d_e)$, determined from Equation 2, could be used, but the most reliable value was found at mid-electrode distance ($d_e = 95$ mm). This parameter is referred to as mid solution resistance $R_{s,m}$.

For homogenous mixtures, the conductivity can be determined from a single EIS measurement at a fixed electrode distance. In this case, the moving electrode functionality is not necessarily used, and the setup operates as a static conductivity cell. However, we will see in Section 4B that MEEIS has an advantage over conventional conductometry, even for homogenous liquids.

IV. MEASUREMENTS & RESULTS

To test our moving electrode approach, we performed several experiments. As test suspensions, we prepared monodisperse mixtures of aqueous sodium hydroxide solutions and commercially available PMMA spheres with 40 μm and 6 μm diameter (Spheromers CA 40 and CA 6, Microbeads, Norway). The sodium hydroxide solutions had molal concentrations of 5 mol kg^{-1} (solution A) and 0.5 mol kg^{-1} (solution B), respectively. Once the cell was loaded, a temperature equilibration time of 30 minutes was given. After that, the sample was stirred and impedance spectra at seven different electrode spacings ($d_e = 80, 85, 90, 95, 100, 105$ and 110 mm) were recorded. The value of the effective sample cross-section area $A_e = 0.799 \text{ cm}^2$ in Eq. 2 was determined by calibration with a commercially available conductivity standard (1 M KCl, Thermo Fisher Scientific, Chlemsford, USA).

A. The Influence of Particle Sediments on the Bulk Conductivity and the Mid Solution Resistance

In a first experiment we investigated the influence of sedimented layers on the measured electrical properties. Suspensions with the 40 μm spheres at different concentrations in solution A and B were prepared and heated up to temperatures of 60°C. Two hours of time were given for the particles to settle on the bottom electrode. Then the MEEIS measurements were performed. The results are presented in Fig. 4.

For both solutions, A and B, we found that the resistance increases linearly with the electrode distance and rises monotonically with the particle concentration, in solution A about $0.5 \pm 0.14 \Omega$ and in solution B about $2.90 \pm 0.73 \Omega$

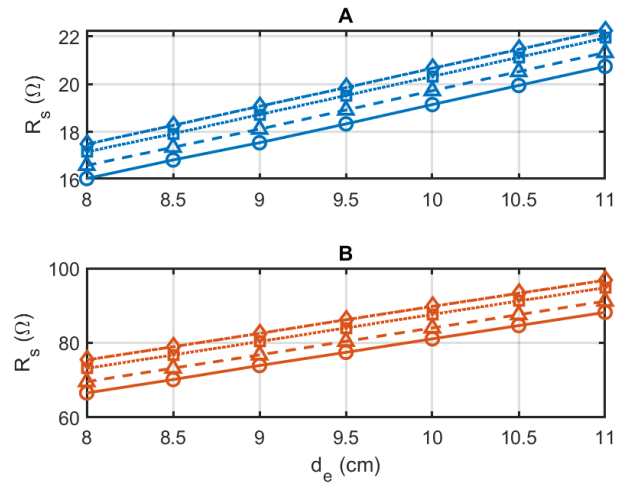


Fig. 4. Solution resistance vs electrode distance for fully phase separated suspensions. '○', '△', '□' and '◇' refer to particle concentrations of 0.00, 0.01, 0.02 and 0.03 g ml^{-1} in 5 m (A) and 0.5 m (B) NaOH solutions.

TABLE I
CONDUCTIVITY AND MID SOLUTION RESISTANCE
OF UNIFORM SUSPENSIONS

w_6 (%)	w_{40} (%)	σ (S m^{-1})	$\frac{\sigma}{\sigma_0}$ (%)	$R_{s,m}$ (Ω)	$\frac{R_{s,m}}{R_{s,m,0}}$ (%)
0.00	0.00	$40.79 \pm 0.00^*$	100.0	$34.29 \pm 0.00^*$	100.00
1.22	0.00	39.86 ± 0.11	97.72	35.00 ± 0.02	102.08
0.00	1.22	39.75 ± 0.15	97.46	35.03 ± 0.04	102.02
2.44	0.00	39.08 ± 0.22	95.81	35.66 ± 0.05	104.00
0.00	2.44	38.75 ± 0.29	95.00	35.82 ± 0.06	104.48

w_6 and w_{40} are the weight percent of 6 μm and 40 μm particles in concentrated sodium hydroxide solution (solution A). σ_0 and $R_{s,m,0}$ denote the conductivity and mid solution resistance of the test solution without suspended particles.

for every 0.01 g mL^{-1} of particles added. The conductivities of the sodium hydroxide solutions remained almost unaffected, with values of $79.06 \pm 0.59 \text{ S m}^{-1}$ for solution A and $17.41 \pm 0.08 \text{ S m}^{-1}$ for solution B. This is in reasonable agreement with data from [11], who reported values of 77.00 S m^{-1} and 13.63 S m^{-1} , respectively.

B. Uniform Suspensions

In this experiment we analyzed monodisperse and uniformly mixed suspensions. The samples contained solution A and PMMA particles of different sizes and concentrations. At a temperature of 25°C, the density of solution A was almost identical to the density of PMMA ($\rho_{A,25^\circ\text{C}} = 1.18 \text{ g cm}^{-3}$, $\rho_{\text{PMMA}} = 1.2 \text{ g cm}^{-3}$). This resulted in a very slow sedimentation process with no significant particle sedimentation taking place during the measurements, which were conducted within 10 minutes after stirring. Each measurement of this series was performed 5 times to test for repeatability, only the measurements at a particle concentration of zero were performed just twice. The results are presented in Table I. We see that the conductivity is influenced mainly by the concentration of the particles. In average, the conductivity dropped about 1.7 % for the 6 μm particles and 2.0 % for the 40 μm particles, for every weight percent increase of the particle concentration.

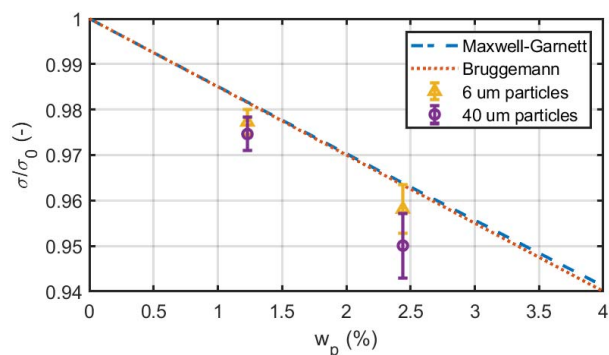


Fig. 5. Comparison of theoretical and experimental results of the influence of particle concentration on the electric conductivity.

A comparison to common effective medium approximation models from Bruggemann and Maxwell-Garnett [4], yielded that our measured conductivity drops are larger than the theoretically expected values. Furthermore, we found that conductivity is influenced by the particle size and not solely by the particle concentration, as assumed by the models. A comparison between theoretical and experimental results is illustrated in Fig. 5.

The study of uniform suspensions shows another advantage of MEEIS. As seen from Table I, the values of σ/σ_0 change to a greater extent than those of $R_{s,m}/R_{s,m,0}$, even though this variation should be equal for homogenous suspensions. The reason for this discrepancy lies in the contact and cable impedances which add to the actual impedance of the sample and distort the measurement result. In electrochemical cells with a fixed geometry so-called *load* or *short* calibrations are necessary to eliminate these errors. For conductivity measurements with MEEIS no such calibration is needed.

C. Monitoring the Sedimentation Process

In this third experiment, we employed our moving electrode approach to investigate the process of particle sedimentation in conductive solutions. Other than in section 3A, this time we studied the *dynamic case*, where, starting from a uniformly mixed suspension, particles slowly accumulated on the bottom and contributed to a steady buildup of the sedimentation layer.

1) *Experimental*: As test suspension we used solution A mixed with 40 μm PMMA spheres at a concentration of 2.44 w%. The sample was measured at temperatures of 25°C, 40°C and 60°C. Here we expected differences in the settling behavior, as the density of the concentrated NaOH solutions was markedly decreased upon heating, whereas the density of PMMA particles stayed approximately constant.

Measurements were carried out 5 minutes after stirring. A measurement prior to this point was not possible since stirring-induced temperature deviations needed to equilibrate first. As the bulk properties changed due to particle sedimentation, it was necessary to keep the data acquisition time short. Therefore, we recorded only the minimum of two EIS spectra at electrode distances $d_e = 8$ and 11 cm. This significantly reduced the measurement time to less than one minute.

Fig. 6 illustrates the evolution of the bulk conductivity and the mid solution resistance. We see that both, locally measured

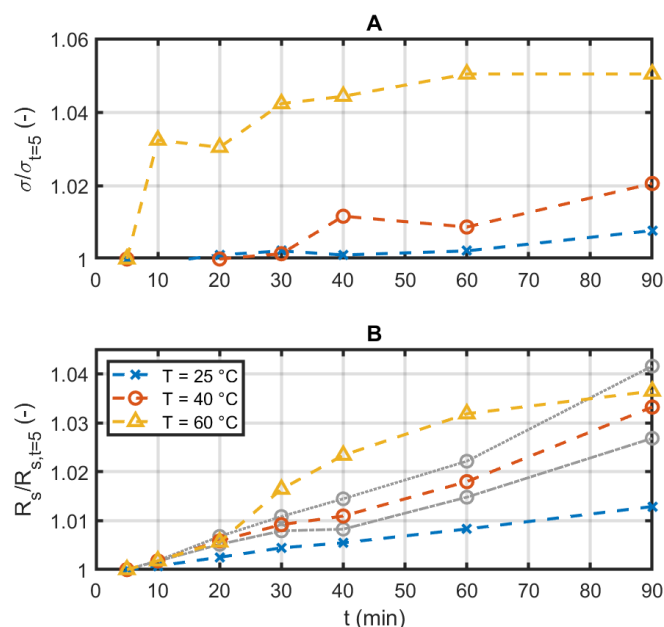


Fig. 6. Evolution of conductivity (A) and mid solution resistance (B) during particle sedimentation. The values are normalized to their respective values at $t = 5$ min for reasons explained in the text. For the 40°C case, solution resistances at electrode distances $d_e = 8$ cm and 11 cm (dotted and dash-dotted grey lines) are shown as examples. Lines are a guide to the eye.

conductivity and the solution resistances, increase over time. As conductivity and resistivity are in a reciprocal relationship, this behavior might appear counterintuitive at first, but makes sense when we consider that the conductivity is probed 80 to 110 mm above the working electrode. Due to sedimentation, the volume fraction of (non-conducting) particles in this part of the sample decreases, hence, the locally measured conductivity rises. In the lower part, the particles form a densely packed layer at the bottom electrode. This layer poses a current barrier, which is much larger than that of uniformly distributed particles. Therefore, the overall mid solution resistance rises.

The influence of the temperature on the sedimentation process is also clearly visible in both plots. At lower temperatures, a slow and linear increase is observed. At 60°C, the values increase much faster and tend to approach a plateau. At elevated temperatures the density difference between the liquid and solid phase is higher, whereby particles settle faster. The plateau in the 60°C case indicates the end of the sedimentation process, with all particles from the bulk having settled on the bottom electrode surface.

The absolute conductivity values for 25°C and 40°C after letting particles settle for 90 minutes are 41.53 S m^{-1} and 55.13 S m^{-1} . Even though particles were still present in the bulk in these cases (no plateauing behavior of the conductivity curve), the values are in good agreement with those reported for a homogenous NaOH solution (40.69 S m^{-1} and 55.82 S m^{-1}) [11]. Unexpectedly, the value at 60°C (72.77 S m^{-1}) is significantly smaller than previously determined (79.06 S m^{-1}) and as reported in the literature (77.00 S m^{-1}). Since all experiments in this series were performed with the same sample, and measurement and heating times in-between were long, this most probably was

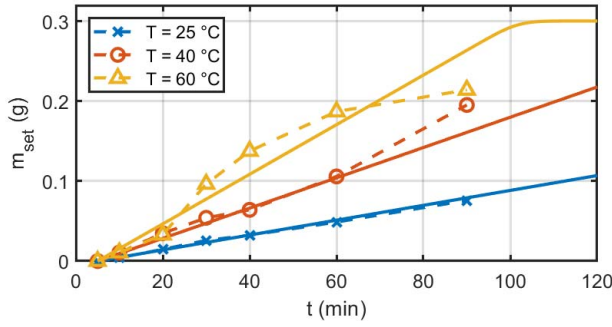


Fig. 7. Mass increase over time. Solid lines are the theoretical results and markers are the experimental observation. The dashed lines are a guide to the eye.

because of contamination effects. Caustic NaOH is known for its high hygroscopicity and CO₂ uptake tendency from atmospheric air [12]. Furthermore, the hydroxide ions in alkaline solutions attack the surface of the glass, forming soluble silicates [13]. These interactions of the sample with its environment are known to have a negative impact on the electrolytic conductivity.

Nevertheless, the influence of sample contamination throughout the timespan of one experiment is arguably small, as the changes in conductivity are positive and can clearly be ascribed to particle sedimentation.

2) *Theoretical*: To describe the sedimentation process not only qualitatively but also quantitatively, we performed a simple 2D computer simulation using MATLAB (MATLAB R2019b, The MathWorks Inc., Natick, Massachusetts). As a basis, we generated a uniform distribution of particles in a sample volume, like in our test setup and assigned each particle a settling velocity according to Stokes law

$$v_s = \frac{2}{9} r^2 g \frac{\rho_p - \rho_f}{\eta},$$

where r denotes the particle radius, g the standard gravity, ρ_p and ρ_f the particle density and the density of the fluid, respectively, and η the dynamic viscosity. Knowing the position and velocity of each particle, we calculated the settling time. A particle was considered *settled* when it approached the bottom electrode. With this information, the total mass of settled particles m_{set} was calculated. Plotting this parameter as a function of time, a similar picture as outlined in Fig. 6(B), was obtained.

This provides a reason to assume that the change of the mid solution resistance qualifies as a measure for the total mass of sedimented particles. To associate these two parameters, we introduced a constant of proportionality α . Mathematically this relation writes

$$m_{set} = \alpha \frac{\Delta R_{m,s}}{R_{m,s,0}},$$

where m_{set} is the increase in mass.

A good fit for all measurement data was achieved for $\alpha = 5.910^{-3} \text{ kg } \Omega^{-1}$. The results are illustrated in Fig. 7.

We see that the fit is especially good for experimental results at 25°C and 40°C. At 60°C, the experimental observation shows a less linear behavior and trends to plateau earlier than in the model.

V. CONCLUSION AND OUTLOOK

In this work, we designed an electrochemical cell with a movable counter electrode and precisely adjustable electrode distance. This allowed us to perform impedance measurements at various positions in the sample and thus to probe different parts of the sample independently. This advantage of moving electrode impedance spectroscopy over conventional electrochemical cells is useful when studying heterogenous liquids, such as conductive suspensions.

In a series of experiments, we tested the applicability of MEEIS. In the *static case* we saw that the conductivity of the liquid phase remains unchanged even if sedimented particles cover the electrode surface. The mid solution resistance, though, which probes the sample between counter and working electrodes, increases significantly upon addition of particle layers.

Investigation of uniform suspensions revealed the influence of non-conductive particles on the bulk conductivity and the mid solution resistance. Conductivity decreases significantly with particle concentration and slightly with particle size. The former is supported by common effective medium theories, whereas the latter is not.

MEEIS can also be used to monitor the particle sedimentation process. Starting from a uniformly mixed suspension, the evolution of the mid solution resistance provides a qualitative measure of the sedimentation rate. Combining this with theoretical considerations allows a relation to the mass of settled particle to be determined.

The main application of MEEIS we envisioned is in monitoring crystal formation, for instance, the formation of zeolites from ionic solutions. Recent progress in this field has shown that zeolite formation from highly conductive hydrated silica ionic liquids has a qualified potential for tailored synthesis of various zeolite materials [14]. The initially clear synthesis liquid forms steadily growing crystalline particles upon hydrothermal treatment. These particles also tend to phase-separate once they have reached a specific size. The presented setup is intended for studying this process in detail.

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the field of fundamental zeolite science with focus on experimental techniques and material characterization.

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