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Characterization of applied fields for ion mobility separations in traveling wave based structures for lossless ion manipulations (SLIM)

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Abstract

Ion mobility (IM) is rapidly gaining attention for the separation and analysis of biomolecules due to the ability to distinguish the shapes of ions. However, conventional constant electric field drift tube IM separations have limited resolving power, constrained by practical limitations on the path length and maximum applied voltage. The implementation of traveling waves (TW) in IM removes the latter limitation, allowing higher resolution to be achieved using extended path lengths. Both of these can be readily obtained in structures for lossless ion manipulations (SLIM), which are fabricated from arrays of electrodes patterned on two parallel surfaces where potentials are applied to generate appropriate electric fields between the surfaces. Here we have investigated the relationship between the primary SLIM variables, such as electrode dimensions, inter-surface gap, and the applied TW voltages, that directly impact the fields experienced by ions. Ion trajectory simulations and theoretical calculations have been utilized to understand the dependence of SLIM geometry and effective electric fields on IM resolution. The variables explored impact both ion confinement and the observed IM resolution using SLIM modules.

Keywords

Ion mobility; Ion trajectory simulations; Structures for lossless ion manipulations (SLIM); Resolution; Electric field; Traveling waves

1. Introduction

Ion mobility in conjunction with mass spectrometry (IM-MS) is of broad analytical applicability, including biological applications in metabolomics, glycomics and proteomics [1–5]. The benefits and applicability of IM-MS increase with the achievable IM resolution. However, in conventional drift tube ion mobility (DT IM) [6–8] resolution increases with drift path length and requires increased voltages as the length of the DT increases. In contrast, in traveling wave ion mobility (TW IM) [9,10] a dynamic but relatively low voltage

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profile is repeated throughout the separation region regardless of its length, allowing extended path lengths without the requirement for increased voltages. Our laboratory has recently introduced TW IM based upon structures for lossless ion manipulations (SLIM) [11]. SLIM are fabricated from array of electrodes patterned on two parallel surfaces where the appropriate potentials are applied to generate the desired electric fields between the surfaces. In TW-based SLIM (TW SLIM) DC and RF potentials are generally applied to three different electrode types: the guard, TW, and RF electrodes. These electrodes are arranged on two mirror-image surfaces typically separated by a gap of ~ 3 mm for TW and TW electrode widths of ~ 0.5 mm. The applied DC voltage to the guard electrodes provides the required lateral ion confinement, while the applied potential to the RF electrodes prevents ion losses to the SLIM surfaces, and in combination confine ions to a selected path between the two surfaces. Thus, TW SLIM devices circumvent the voltage limitations associated with constant fields used in DT IM, providing a basis for extended path lengths, and thus achieving higher IM resolution in compact devices without requirement of excessive voltages [12–14]. SLIM also enables lossless ion transmission along with a wide range of ion manipulation and ancillary capabilities, such as the abilities to select, trap and accumulate selected species after separations [13,15–19].

In a TW SLIM device, the IM resolution mainly depends on the average applied electric field, length of the device, TW electric field, speed of the TW, temperature, ion-molecule collision reduced mass, and collision cross-section of the ions [20]. While drift tube IM resolving power is typically assessed using a single peak for measuring the separation quality, for traveling wave IM the resolving power is not a useful metric for separation quality (for example, under surfing conditions peak widths do not increase with path lengths but provide no separation). Hence the resolution of selected peaks is a more useful parameter and is used throughout this work. Increasing path length was proven to be an effective route of increasing the resolution by combining the attributes of TW and the flexibility of SLIM modules [12,15,21]. However, the applied field-related effects, and consequently the relationship between the SLIM variables such as the electrode size, inter-surface gap and the applied voltages on the obtained IM resolution have not been well studied to date.

Here we report on IM separations achievable using TW SLIM modules having various electrode sizes and inter-surface gaps using both theoretical and experimental studies. In addition, the ion trajectory simulations and theoretical calculations provide insights on the dependence of the IM resolution on SLIM design.

2. Experimental setup

2.1. Mass spectrometer

The SLIM-QTOF MS platform developed for this work has been described in detail elsewhere [14,17]. Briefly, singly charged ions were produced by nanoelectrospray ionization of Agilent low concentration ESI tuning mix (Agilent, Santa Clara, CA) infused by a syringe pump (Chemyx, Stafford, TX) with a flow rate of 300 nL/min through a 20 μ m i.d. etched emitter. Ions were introduced into the first stage of vacuum through a 500- μ m i.d. stainless steel capillary heated to 140° C (Fig. 1A). After exiting the heated capillary, ions

were focused by a high pressure ion funnel (10 Torr) (RF: 960 kHz and 250 V_{p-p}) then stored for 10 ms in an ion funnel trap (IFT, RF: 1.3 MHz and ~160 V_{p-p}) at 3.95 Torr [22,23]. Ions were released from IFT by lowering the voltage applied to the exit gate for 486 $\frac{1}{4}$ s and then directed into the TW SLIM module maintained at 4 Torr with N₂. Ions exiting TW SLIM were collimated by an 18-cm long 'rear' ion funnel (RF: 830 kHz and ~350 V_{p-p}) and then guided to an Agilent 6538 QTOF mass spectrometer equipped with a 1.5-m flight tube (Agilent Technologies, Santa Clara, CA). Data was recorded with a U1084A 8-bit ADC digitizer (Acqiris SA, Switzerland) and processed using in-house developed control software written in C#.

2.2. TW SLIM modules

The two TW SLIM modules utilized electrode arrays on a pair of 30.5 cm $\times$ 7.6 cm surfaces that were fabricated using photolithography. The Fig. 1B shows a TW SLIM module arrangement of electrodes on one surface. The first module had IM paths that generated using 3 RF strips interleaved with 2 arrays of TW electrodes on each surface (i.e., a 3,2 configuration), while the second module has 6 RF strips interleaved with 5 arrays of TW electrodes (6,5 configuration) [13,14]. The TW electrodes are separated from adjacent RF electrodes by 0.13-mm gaps. The TW electrodes ranged in length from 0.5-mm to 4.0-mm. RF waveforms at 1 MHz (250 V_{p-p} and 180° out-of-phase for adjacent electrodes) were applied to the RF electrodes on each surface to create a confining pseudo-potential preventing loss of ions to the SLIM surfaces.

In this work, the TW was created by switching the DC on and off to individual electrodes of each eight electrode set at a selected frequency [13]. A symmetric TW sequence was utilized in the present experiments (HHHHLLLL), where L corresponds to 0 V and H to the application of the TW amplitude (e.g. 30 V_{p-p}) to each eight-electrode set, and which was stepped one electrode at a time in the direction of ion motion (i.e.: LHHHHLLL, LLHHHHLL, LLLHHHHL,...) in a repeating manner across all electrode sets to create the TW [13,14,17]. The gap between the two TW SLIM surfaces was varied between 2.8-mm and 4.8-mm by using aluminum spacers (McMaster-Carr, Los Angeles, CA).

2.3. Simulations

Electric potential, field calculations, and ion trajectory simulations using the TW SLIM electrode array designs were performed using SIMION 8.1 [24] (Scientific Instrument Services Inc., Ringoes, NJ, USA) [25]. The electrode geometries were generated in house using a geometry description language (GEM) integrated with SIMION. A finite difference method was used to estimate the potentials at different points inside a given volume, with the help of a relaxation method [24]. The fields due to the applied TW was extracted with the RF turned off. The potential array file (PA file) was converted into text file using the STL tools in SIMION. From the potential distribution, the electric fields were calculated by applying the gradient operator on the potential distribution using a Matlab code. An example of the instantaneous voltage and electric field for a 2-mm electrode length and 3-mm board spacing for a SLIM module is shown Fig. 1C. As the waveform is symmetric, one quarter of the waveform represents all other quarters. Hence, the average field over one quarter of the waveform, which is equivalent to two electrodes, represents the average electric field at any

instant. A Statistical Diffusion Simulation (SDS) [26] at 4.0 Torr was used to model ion motion. These simulations have been predictive of ion confinement, transport and separations [25], also consistent with ion current measurements and IM measurements [12,14].

3. Results and discussion

3.1. Theoretical field calculations

The average electric field at any instant at mid-plane of the SLIM electrode surfaces and along the axis of motion for a TW with an amplitude of $30 V_{p-p}$ was calculated for electrode sizes: 0.5-mm width, lengths varying from 0.25-mm to 5-mm, and for SLIM inter-surface gap from 2-mm to 10-mm, as shown in Fig. 2. The maximum field for a gap of 2.7 mm is obtained at 0.5-mm electrode size while the maximum field for a gap of 4.8-mm is observed for 1-mm electrode size. The average electric field is proportional to the average speed of the ions moving in the forward direction; thus, the greater this average field the greater the forward motion of an ion at a given TW speed for a given ‘rollover’ event. While direct correlation between this field and average ion motion is beyond the scope of this work, here we present empirical relationships for better understanding of the design and operational parameters for TW IM in SLIM under various electrode and field conditions.

3.2. Experimental IM resolution

IM resolution was experimentally measured as a function of the applied TW speed for 0.5-mm and 2.0 –mm TW electrode lengths. Performance was evaluated using the resolution of

m/z 622 and 922 ions $\left(R_{622-922} = \frac{2(t_{922} - t_{622})}{\Delta t_{922} + \Delta t_{622}} \right)$, where t_{922} and t_{622} represent the arrival

times of m/z 922 and 622 ions while t_{922} and t_{622} are the FWHM of the m/z 922 and 622 peaks, respectively. Fig. 3(A–C) show the effects of varying the TW speed on the measured IM resolution while varying the TW electrode length and inter-surface gap in the two TW SLIM modules (3,2 and 6,5). The observed trend of the TW speed on the measured resolution achieved by the SLIM TW IM modules are similar to previous experimental findings and theoretical predictions [13,14,20]. The resolution observed at low TW speeds is attributed to an additional drift IM separation component after the TW SLIM, primarily in the 18 cm-long rear ion funnel region where a drift field of 18 V/cm is applied. In TW SLIM, when the TW speed is sufficiently low or the ion’s mobility sufficiently high, ions will move with the waves in a “surfing” mode. Thus, ions are confined within traveling traps and no separation is obtained. Once the TW speed increases sufficiently, the lower mobility ions (m/z 922) are ‘rolled over’ by TW and increasingly separate from the higher mobility ion (m/z 622). Further increasing of the TW speed leads to a resolution plateau followed by a decrease of the observed resolution. With significantly increased TW speeds ions experience only slight axial displacement, reducing both their speed and the overall separation achieved [12,14,20]. Although the separation cut-off (i.e. transition to surfing mode) obtained using 0.5-mm length TW electrodes was similar to that observed using 2-mm electrodes, the maximum resolution achieved was significantly lower (Fig. 3A). For a smaller inter-surface gap of 2.7-mm, a similar trend of the resolution dependence on the TW speed was observed (Fig. 3B). Reducing the inter-surface gap resulted in a 67% increase in

the optimum IM resolution using 2-mm electrodes and 230% upon using 0.5-mm electrodes (Fig. 3A&B). These experimental observations mirror the trends observed in the present theoretical studies due to the higher fields obtained with the lower inter-surface gap. The trends for the computational and experimental measurements match, as it can be seen that in order to increase the achieved resolution using shorter electrodes, the inter-board spacing needs to be reduced. This is understandable since at smaller electrode lengths the effective TW penetration into the inter-board gap (and where ions are confined) decreases mandating a reduction in inter-board gap to increase performance (Fig. 2).

Also, the obtained IM resolution using 0.5-mm electrodes was similar to that obtained with 2-mm electrodes, as predicted above: the maximum electric field is achieved with shorter electrodes for narrower inter-surface gaps (Fig. 2). In consonance with calculation, it can be seen that for 2.7-mm inter-board gap, the resolution for 0.5 mm electrodes is indeed higher than for 2-mm electrodes, albeit at larger traveling wave speeds (Fig. 3B). Closer to the surfing transition, there some overlap in the performance values between 0.5-mm and 2-mm electrode lengths. This difference could arise due to the shape of the electric fields created in 0.5-mm and 2-mm electrodes and the specific details of the surfing to separation transition in both the cases. For example, with 0.5-mm electrodes, the wave moving at a given speed has to step forward to the next electrode at a faster rate as compared to the case with 2-mm electrodes. Such details (which are not analyzed in this manuscript) might influence some of the specific observations while the overall trends between experimental and theoretical analysis are in agreement.

Additional resolution measurements were made using a SLIM (6,5 electrode configuration) having 1-mm, 2-mm, and 4-mm TW electrode lengths and the inter-surface gap maintained at 2.8-mm. As illustrated in Fig. 3C, comparable resolution dependence on the applied TW speed was observed with 1-mm and 2-mm electrodes. The IM spectra corresponding to the TW speed at which the highest resolution obtained in each case (200, 100 and 50 m/s for 1, 2 and 4-mm electrode size respectively) is shown in Fig. 3D. In contrast, 4-mm TW electrodes showed a quite different resolution dependence on the TW speed with a transition between surfing and separation occurs at a very low TW speed. In addition, the optimum resolution obtained with significantly lower than that obtained using 1-mm and 2-mm electrodes. This can be attributed to having significantly lower field when 4-mm electrodes are utilized in combination with a narrow inter-surface gap (Fig. 2).

Now, separations quality is benefitted by both increased resolution and sensitivity. Sensitivity can also be increased by increasing the charge capacity of the device which increases with the increasing scale of the SLIM device. The previous discussion focused on how the geometry of the SLIM affects the traveling wave field and the corresponding performance of IMS separations. In SLIM, the out of phase RF voltage applied to the alternating electrodes (Fig. 1D) generates pseudopotential fields, which effectively confines the ions between the surfaces. The effective confinement potential the SLIM is dependent on the rf field [9]; and any change in the dimensions of the device will also alter the confinement RF field. The Fig. 4 show the SIMION simulations for the RF confinement when the geometry is scaled by a factor of 2 and 4. The dimensions of the scaled device change equivalently in all the three dimensions. That is, scaling two times means the gap and

electrode thickness increases from 3-mm and 0.5-mm to 6-mm and 1-mm respectively. Also the width of the electrode scales similarly. When scaled by a factor of 2 (Fig. 4B), the RF amplitude has to be raised from 150 V to 250 V peak to peak to have an effective confinement. Similarly, when scaled by a factor of 4 (Fig. 4C), the RF amplitude further has to be increased to 420 V peak to peak. Keeping the rf amplitude same in a scaled situation leads to confinement minimum moving closer to electrodes and eventually loss of confinement. With increasing electrode thickness and gap, the rf amplitude needs to be increased to create similar confinement. Traveling wave parameters (amplitude and speed) were kept the same in each case, which has no role in confinement but only on separation and the resolution. In the scaled geometries the length of the electrodes also were scaled, and the traveling wave performance in scaled situations can be optimized as was done in this manuscript.

4. Conclusions

Traveling waves can be applied either in the conventional stacked ring ions guides (e.g. Waters Synapt platforms) [9] or on SLIM which utilize electrodes patterned on parallel planar surfaces. We have investigated, using experimental and theoretical approaches, the impact of key TW SLIM parameters expected to directly affect the achieved electric fields and IM separations. The planar nature of SLIM provide the flexibility for constructing unique capabilities and manipulations, such as ion turning, switching, etc. In addition, the SLIM implementation described allows decoupling of rf confinement and TW voltages which enables facile fabrication of devices having different electrode size. Simulations have been performed for different electrode sizes and different inter-surface gaps, and the instantaneous electric field has been calculated. The simulations showed the electrode size at which the maximum effective field occurs shifts towards longer electrode lengths as the inter-surface gap increases, in agreement with the experimental observation that similar resolution can be attained for longer electrodes by increasing the inter-surface gap. This study provides a foundation for greater understanding of the variables that impact the observed resolution in TW SLIM modules, and for obtaining higher IM resolution with a broader range of SLIM geometries.

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Fig. 1.

(A) Schematic diagram of the experimental setup. (B) Portion of a TW SLIM module surface using a 3,2 electrode arrangement of three RF electrodes interspersed by two segmented electrode arrays (used to create the TW) (C) Potential (black) and the field (red) due to TW only along x-axis in middle plane between the two boards for 2 mm electrode size and 2 mm inter-surface gap. Region between points #1 to #2 represents one quarter of the wave which is equivalent to 2 electrodes distance, hence the average over this range represents the average field. (D) The plot of the equipotential surface generated by the RF pseudopotential for 622 m/z for a 2 mm inter-surface gap. The two surfaces are on the left and right.

Fig. 2.

The average electric field for a TW SLIM with a 30 V_{p-p} amplitude calculated for different board spacing as a function of varying electrode size.

Fig. 3.

IM resolution observed for m/z 622 and 922 ions as a function of TW speed for a SLIM module of 3,2 configuration using a symmetric TW sequence (HHHHLLLL), TW amplitude of 30 V, guard bias of 15 V, and RF amplitude of 300 V_{p-p} for TW electrode size 0.5-mm (red circles) and 2-mm (black squares) for SLIM surface gaps of (A) 4.8-mm and (B) 2.7-mm. (C) IM resolution for m/z 622 and 922 as a function of TW speed for a SLIM module of 6,5 configuration using the same conditions for 1-mm (black squares), 2-mm (red circles) and 4-mm (blue triangles) long TW electrodes with an inter-surface gap of 2.7-mm. (D) Ion mobility spectra corresponding to the TW speed at which the highest resolution obtained for the 6,5 configuration with electrode sizes 1-mm (black), 2-mm (red) and 4-mm (blue) and an inter-surface gap of 2.7-mm.

Fig. 4. The SIMION simulations showing the RF confinement for different geometry scaling. (A) RF voltage 150 V peak to peak, frequency 1 MHz, TW 30 V, TW speed of 45 m/s. (B) Scaled by a factor of 2, RF voltage 250 V peak to peak, frequency 1 MHz, TW 30 V, TW speed of 45 m/s. (C) Scaled by a factor of 4, RF voltage 420 V peak to peak, frequency 1 MHz, TW 30 V, TW speed of 45 m/s.