

The effect of nebulizing gas pressure, volumetric flow rate and tip-to-surface distance on spot size in DESI-MS

Objective

Here, we investigate the effect of the nebulizing gas pressure and the volumetric flow rate on the spot size in the DESI experiment. Understanding the spot size of the DESI plume at the surface and how the various parameters affect its shape and size has implications for many applications such as chemical imaging. Here, we characterize the spot size and shape using water sensitive paper (Syngenta Corp.).

Introduction

The results obtained in DESI experiments are dependent on a number of operational parameters. These factors include geometric variables (e.g. incident angle (α), collection angle (β), and tip-to-surface distance (d_1)), physical variables (e.g. volumetric flow rate, nebulizing gas flow rate) and solvent/analyte, solvent/surface interactions. Typical operating conditions used in DESI are displayed in **Table 1**. Cooks and co-workers examined the effect of sprayer to surface distance and volumetric flow rate on signal intensities. Their results showed that the highest signal intensities were obtained when using a sprayer tip to surface distance of 1 mm and a solvent flow rate of 3 $\mu\text{L}/\text{min}$ [1]. These results are consistent with other work by Cooks and co-workers when phase Doppler anemometry was used to characterize the sizes and velocities of the impacting and ejected droplets while correlating (indirectly) these observations to signal intensities [2]. The results demonstrated that the highest droplet velocities are observed closest to the sprayer nozzle and thus the droplet impact force at that point is also greatest leading to more efficient desorption/ionization at the surface.

Table 1. Omni Spray Ion Source & Instrument Settings*

Parameter	Setting
ES voltage	5 kV
Solvent flow rate	2 $\mu\text{L}/\text{min}$
Gas pressure	150 psi
Distance from tip to surface	2 mm
MS inlet temperature	300 °C
Spray impact angle (α)	55 °
Solvent	70:30 Methanol/Water
Surface scan rate	200 $\mu\text{m}/\text{s}$

* Refer to *Understanding Omni Spray Ion Source Operating Parameters* for a description of the parameters listed.



Figure 1 - Omni Spray® ion sources

Experimental Description

Typical DESI experimental conditions are shown in Table 1. A 1D surface automated Omni Spray DESI Ion Source was used in these studies (**Figure 1**). In each experiment, the motion system was operated in point-to-point dwell mode allowing each spot, spaced in 2 mm increments, to be exposed to the DESI spray plume for approximately 3 seconds.

Discussion of Results

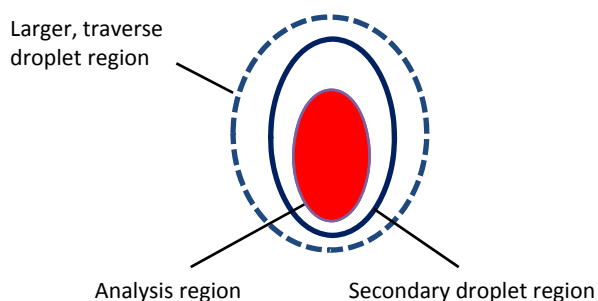
Figure 1 shows a digital photograph at 60x magnification of the DESI spray plume at the surface of a plain glass slide. As shown in the figure, the spray plume on the surface presents as an ellipse having three overlapping zones.

Figure 1. Photograph of DESI spray plume



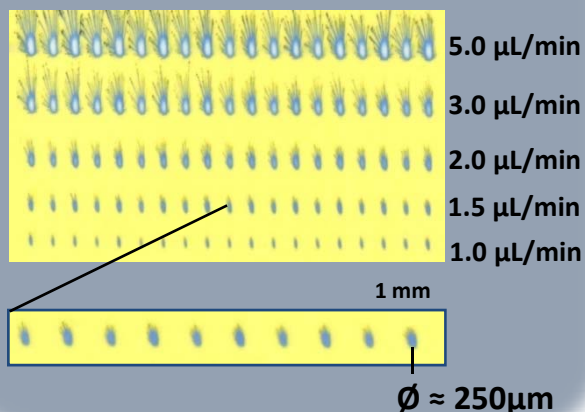
Figure 2 shows a representation of the droplet impact region on a typical surface. Within the plume exists a smaller elliptical region where desorption/ionization is most efficient –the analysis region. The second region is composed of secondary droplets which originate from the inner plume. The third region appears to be composed of larger, secondary, slow-moving droplets. Optimal signal levels were obtained when the analyte was sampled in the analytical region of the plume as opposed to being sampled in the second or third region of the impact plume where desorption/ionization is less efficient. **Figure 3** shows the effect of solvent flow rate on the spot size. As the aqueous/organic solvent droplets impact the yellow dye, the impact area turns blue to white. At high flow rates (i.e 5 μ L/min) the spot size is significantly larger in

Figure 2. Spray impact area representation



comparison to that which is obtained at lower flow rates. Higher flow rates lead to significant “splashing” having a dilutive effect through analyte re-distribution.

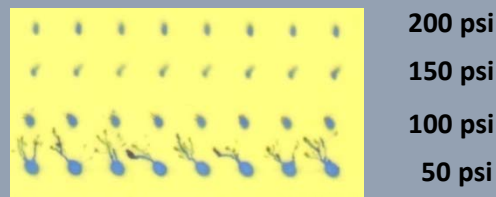
Figure 3. Effect of volumetric flow rate on spot size



At typical solvent flow rates, 1.5 -2 μ L/min, the spot sizes (in the direction orthogonal to the inlet of the MS) are between 250-400 μ m.

Other parameters influencing the spot size are the nebulizing gas pressure and the tip-to-surface distance. **Figure 4** shows the results when the nebulizing gas pressure was varied from 50 to 200 psi. As shown in the figure, at lower pressures the spot size is larger as compared to higher pressures, with the presence of solvent jets composed of large droplets.

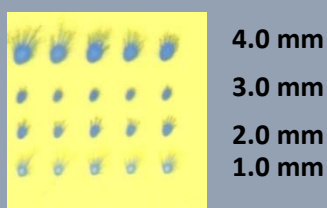
Figure 4. Effect of nebulizing gas pressure on spot size



At higher gas pressures the primary droplet beam is confined to a small spot, presumably composed of smaller droplets traveling at higher velocities.

Figure 5 shows the results when the tip-to-surface distance (d_1) was varied from 1 to 4 mm. As shown, the spot size depends on the

Figure 5. Effect of tip-to-surface distance on spot size



tip-to-surface distance. At closer tip-to-surface distances (1 mm) the desorption event is most efficient and produces a compact primary droplet beam. This is consistent with the analytical results reported in [1] and the fundamental studies presented in [2]. It is inferred from the above data that the desorption rate is also highest at smaller d_1 values, leading to higher sensitivity.

Conclusions

The Omni Spray Ion Source produces spot sizes that are dependent on the solvent flow rate, the nebulizing gas pressure, and the tip-to-surface distance. We have illustrated these dependences in each case using water sensitive paper. The effective spot size can be reduced to diameters of less than 250 μ m under certain conditions. This aspect of the method is especially important when small surface area sampling is required as in surface imaging.

References

- [1] Z. Takats, J. M. Wiseman, R. G. Cooks, *Journal of Mass Spectrometry* **2005**, *40*, 1261.
- [2] A. Venter, P. E. Sojka, R. G. Cooks, *Analytical Chemistry* **2006**, *78*, 8549.
- [3] S. P. Pasilis, V. Kertes, G. J. Van Berkel, *Analytical Chemistry* **2007**, *79*, 5956-5962.