Investigation on Microliter Free Jetting Using a Piezoelectric Micro Pump

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Abstract: By avoiding any interaction with surfaces, especially free jetting of liquid pharmaceuticals has numerous advantages such as no cross-contamination of substances, less waste in pipetting, or the possibility of needle-free subcutaneous injections. Dispensing a defined amount of a liquid into air without surface contact requires the formation of a free jet. Therefore, jet generation by a micro pump presents a solution which is versatile in jet volumes while providing minimal dimensions. Jetting is achieved by the quick actuation of the piezoelectric actuator on the pump, leading to the ejection of a volume package that is fast enough to overcome the surface tension at the outlet's interface, for example a dosing needle. Size and formation of the jet are controlled by matching the micro pump's driving parameters such as peak-to-peak voltage and signal waveform. The feasibility of this setup is tested as well as parameters optimized for free jetting aqueous solution. Furthermore, the influence of different actuation parameters on the dosing precision is evaluated. A rectangular signal with 250 Hz sine flanks is determined as suitable waveform for the jet generation with the evaluated 20 mm stainless steel micro pump. Simultaneously, varying and matching the amount of pump strokes and voltage amplitude results in adjustable dosing volumes.

1 INTRODUCTION

Delivery of small and accurate amounts of liquids is crucial in several applications. Many of those demand for a contactless way of applying the medium, for example to avoid cross-contamination while working with several liquids. This can be achieved by using a piezoelectric micro pump in a free jetting setup.

In pipetting, it is necessary to immerse the pipette's tip into the medium for aspiration and make sure that the liquid completely leaves the tip while dispensing. Therefore, tips are either immersed in a liquid which is already in the container or need to touch the container's wall to overcome capillary effects (Pushparaj, 2020). Consequently, tips must be disposed after every dosing cycle. Additionally, one tip can not be immersed in different containers or media as it leads to contamination. Instead, a metal piezoelectric micro pump with a nozzle or dosing needle generating a free jet can provide a contactless and waste free solution.

In this work, the feasibility of forming an aqueous free jet using a metal micro pump is shown. Therefore, parameters such as signal waveform and voltage are varied to optimize the reliable generation of a jet. The dosing precision of this technique is evaluated in a gravimetric setup by comparing the average dosed volume and the deviation from the median for five packages. Moreover, it is compared to the dosing precision of a comparable pump type in a non-free jetting evaluation from another publication (Thalhofer et al., 2021). The aim of the first and main part of this paper is to investigate the influence of different driving parameters on the free jetting performance of a piezoelectric stainless steel micro pump. The second step aims to find an optimal parameter set to deliver precise microliter jets evaluated with a precision scale.

2 MATERIALS AND METHODS

Key devices in the experimental evaluations of this work are the Fraunhofer EMFT's 20 mm diameter stainless steel micro pumps. Additionally, the param-

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eters for jet formation and the jet dosing precision are gravimetrically evaluated in one measurement setup.

2.1 Piezoelectric Micro Pumps

The steel micro diaphragm pump as shown in Figure 1 consists of a rigid bottom layer (5) with an inlet and outlet orifice (Wald et al., 2013). They are equipped with two valve foils (3+4) and one actuating diaphragm (2) that are laser welded to the bottom piece ensuring leak tightness. A round piezoelectric ceramic (1) is glued to the actuating diaphragm and pretensioned by applying a voltage during curing of the glue. Therefore, the pump chamber height benefits from the gluing of the piezoceramic in a curved state.

Actuation of the pump is established by the inverse piezoelectric effect. Alternating voltage signals lead to contractions and expansions of the piezoceramic thus causing an oscillating movement in the diaphragm. The check valves (a+c) in the valve foils passively open and close, resulting in a unidirectional pumping movement of the fluid. This working principle and the underlying voltages are depicted as zero position (green), suction (blue), and ejection (red) in Figure 1 (Bußmann et al., 2021).

The pump evaluated in this paper is of the type P320009, which describes a stainless steel micro pump with a 20 mm diameter.

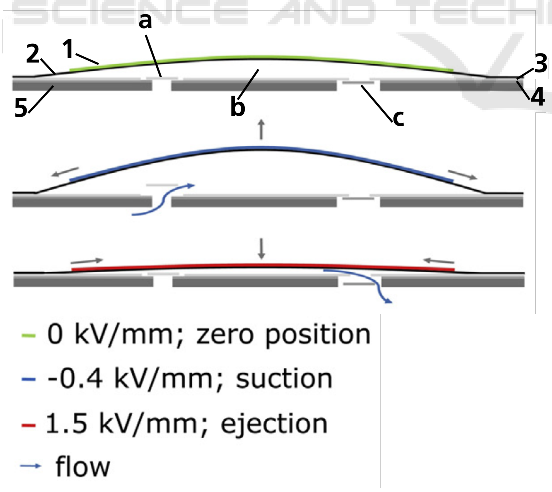

Figure 1: Schematic of the piezoelectric micro pump, consisting of a piezoelectric actuator (1) on an actuator diaphragm (2), inlet and outlet valve foil (3+4), and bottom piece (5). The working priniciple of the pump cycle is described by the colors while the fluid flows through the inlet valve (a), into the pump chamber (b), and out through the outlet valve (c). Adapted from Bußmann et al. (2021).

2.2 Gravimetric Measurements

All measurement data is obtained using a gravimetric setup as depicted in Figure 2. It consists of an inlet reservoir filled with distilled water and an outlet reservoir placed on a precision scale (Sartorius MC410, resolution 0.01 mg). A dosing needle (ID: 0.41 mm, L: 1.0", Vieweg GmbH) is installed in a 3D printed holder fitting into the scale's chamber, thus freely suspended above the outlet reservoir to enable free jetting. Inlet reservoir and dosing needle are fluidicly connected via a piezoelectric micro pump by silicone tubing. Measurement and driving equipment such as waveform generator, amplifier, and scale are controlled and logged by a connected computer.

The scale drift and evaporation effects are emended by additional measurement steps described in section 2.4.

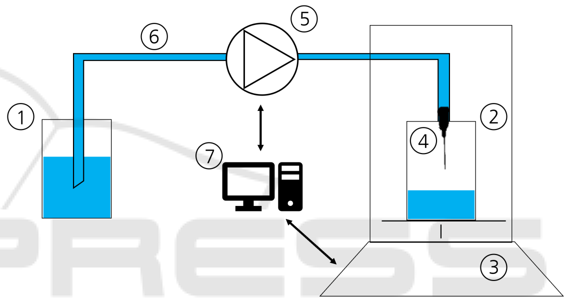

Figure 2: Schematic of the gravimetric setup: Inlet reservoir (1), outlet reservoir (2) on scale (3), dosing needle (4), micro pump (5), tubing (6), computer control and recording (7).

2.3 Jet Generation

Different types of jetting techniques and how they can be generated have already been described in another publication (Wackerle et al., 2002). Basically, a minimum flow rate must be achieved to overcome the surface tension at an orifice thus forming a jet. Achieving this using a micro pump, ideally each pump stroke results in a defined jet. This enables the optimal translation of the kinetic energy of the diaphragm movement into the liquids flow rate.

In this publication, the dosed water is distinguished in either a detaching droplet or the required free jet. The difference is quantified visually as shown in Figure 3 and by comparing the dosed weights of repeated volume packages. In Figure 3, the four steps on the left detail a formed droplet, how it tears off, and falls down from a dosing needle. In contrast, the right part of Figure 3 depicts the beginning of a free jet before its tear-off.

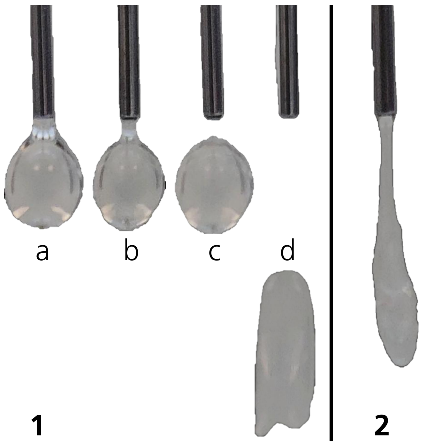

Figure 3: Comparison of droplet detachment (1) and free jet (2) from a dosing needle. Forming of droplet (a), tear off (b), droplet falling down (c), and deformation of droplet in the air (d).

2.4 Methods

A variation of parameters was evaluated in the gravimetric setup (see Table 1). Two main measurements are performed where one is underlying a variation of the waveform and the other a variation of the peak-to-peak voltage (Vpp). Both measurements consist of a sweep through ascending amounts of pump strokes from one to five.

Table 1: Driving parameter variations.

Actuation parameter	Variation
Waveform	Sine wave (SIN) Rectangular signal (RECT) Rectangular signal with 250 Hz sine flanks (SRS)
Voltage (Vpp)	Positive amplitude: +100 V to +300 V in 50 V steps
Amount of strokes	For each waveform & voltage: groups of 1, 2, 3, 4, and 5 strokes (5 volume packages per group)

For the first measurement, three different signal waveforms were evaluated at a fixed actuator frequency of 10 Hz and voltage amplitude of [+300 V / -80 V]. The chosen waveforms were pure sine wave (SIN) and pure rectangular (RECT) signal, as well as an artificial rectangular signal with 250 Hz sine flanks (SRS). Therefore, the first measurement holds three different waveforms (SIN, RECT, and SRS) with five groups (1, 2, 3, 4, and 5 strokes) each.

Comparably, the second measurement varies the positive amplitude of the voltage from +100 V to +300 V in 50 V steps while the negative amplitude is fixed to -80 V and frequency at 10 Hz, each variation with again five groups of stroke amounts. All groups of the second measurement are driven with a SRS signal. Every group of both measurements consists of five volume packages with the same driving parameters to evaluate their precision.

Each volume package is sampled by ten scale measurement points. Five measurement points are recorded before the dosing of the previously defined package. Five more points are recorded after a six second waiting period after the package was triggered.

The dosed weight per package is calculated by subtracting the average of the five points before the trigger from the average of the five points after the trigger. This allows to reflect the dosed weight without influences from a potential scale drift due to evaporation effects.

Calculating the volume per pump stroke or per package from the measured weight can be done by multiplying it with the density of water (0.998 kg*m^{-3} at 23 °C).

3 RESULTS

The driving parameters of the metal pump are varied as previously described to optimize their precision in microliter range free jetting. First, different waveforms are evaluated while the pump is driven in burst mode enabling the control of volume package groups of 1, 2, 3, 4, and 5 strokes per waveform, respectively. This burst mode allows the triggering of exactly one stroke in the period of the signal, starting with the negative amplitude and ending at the end of the positive amplitude. This guarantees a signal related resolution of the stroke instead of a time-resolved trigger leading to random starting points of a burst within the signal's period.

For the sinusoidal signal (see Figure 4), package volumes are at 3.34 mg ± 4.95 mg for one stroke, whereas for 2, 3, 4, and 5 strokes volume packages show 5.24 mg ± 2.41 mg, 9.82 mg ± 1.17 mg, 13.66 mg ± 1.07 mg, and 17.65 mg ± 1.08 mg, respectively. Thereby, the largest deviation from the median is visible for the 1-stroke packages where for three out of five packages no water is delivered onto the scale. The measured volume packages per waveform are also visible in the first column of Table 2.

Improvements are seen for the actuation with a rectangular signal in Figure 5. For this signal average deviations from the median are in the range of 1.85 mg. At the same time, the largest difference between the smallest and highest dosed volume package per

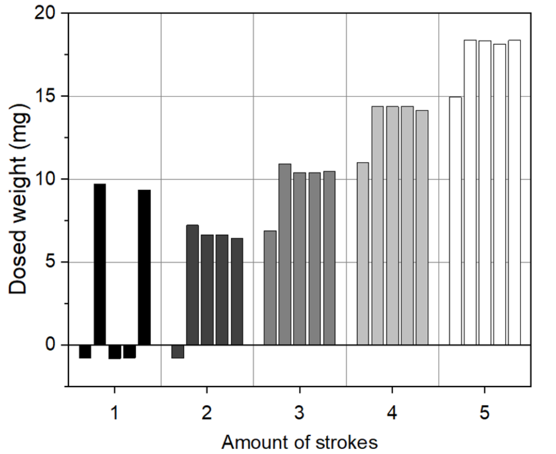

Figure 4: Dosed weight per package in groups of 1, 2, 3, 4, and 5 strokes for a sinusoidal signal. Five repeated packages per group.

group is 6.20 mg, as the first package in the 3-stroke group yields 7.60 mg and the other four packages are above 13.03 mg.

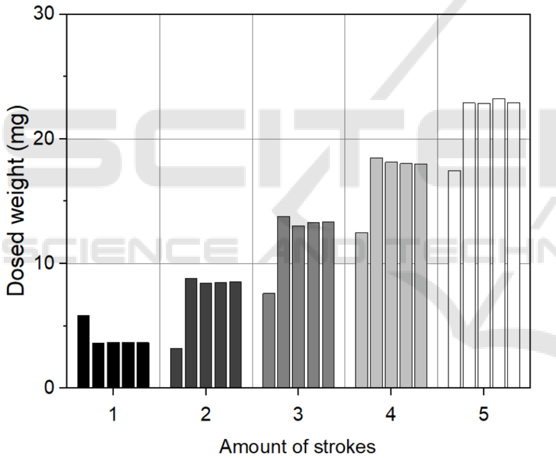

Figure 5: Dosed weight per package in groups of 1, 2, 3, 4, and 5 strokes for a rectangular signal. Five repeated packages per group.

The SRS signal in Figure 6 has only ±0.53 mg maximal deviation from the median for the five packages. The bar graphs for sine and rectangular signal show that the first dosed package of each group of the same number of strokes is different from the other four, thus mostly resembling the volume of the previous group. This indicates an incorrect signal trigger when switching the stroke settings from one group to the next which is verified by an oscilloscope measurement of the rectangular input signal. The recorded signal shows exemplary five times four triggered strokes. During the switch from four to five strokes, there is an aperiodic stroke with only half of the required voltage amplitude. Consequently, the following first package out of the group with five strokes, only shows four. This wrong trigger is observed for every switch between groups for the sine and rectangular signals.

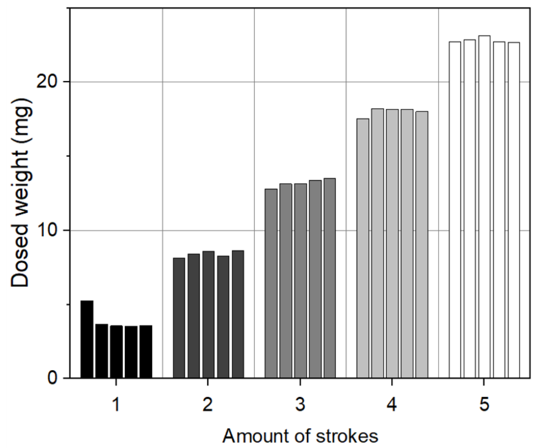

Figure 6: Dosed weight per package in groups of 1, 2, 3, 4, and 5 strokes for a SRS signal. Five repeated packages per group.

Therefore, the first package of each group is removed and the average deviation from the median is recalculated for the remaining four packages. This leads to an improvement of the median deviation in nearly all groups independent of the waveform. The corrected values are summarized in the second column of Table 2.

Table 2: Dosed weight before and after correction.

Waveform	Dosed weight (mg)	Dosed weight corrected (mg)
SIN	3.34 ± 4.95	4.37 ± 5.16
	5.24 ± 2.41	6.75 ± 0.24
	9.82 ± 1.17	10.55 ± 0.19
	13.66 ± 1.07	14.33 ± 0.08
	17.65 ± 1.08	18.32 ± 0.08
RECT	4.10 ± 0.70	3.67 ± 0.01
	7.48 ± 1.72	8.56 ± 0.13
	12.22 ± 1.85	13.37 ± 0.21
	17.04 ± 1.82	18.17 ± 0.16
	21.89 ± 1.76	22.99 ± 0.13
SRS	3.93 ± 0.53	3.59 ± 0.04
	8.40 ± 0.16	8.47 ± 0.13
	13.20 ± 0.20	13.31 ± 0.15
	18.03 ± 0.20	18.15 ± 0.06
	22.83 ± 0.14	22.85 ± 0.15

The second parameter that is examined is the voltage amplitude. Therefore, the negative voltage of -80 V is fixed while varying the positive voltage from +100 V to +300 V in 50 V steps. Figure 7 shows that the overall volumes per stroke are increasing for increasing voltages with mostly linear behavior between the ascending amounts of strokes.

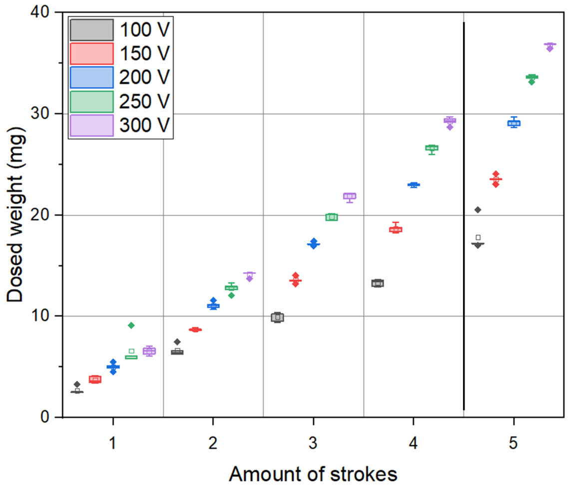

Figure 7: Dosed weight of 1, 2, 3, 4, and 5 strokes. Every group with ascending 100 V, 150 V, 200 V, 250 V, and 300 V positive driving voltage amplitude. Negative voltage fixed to -80 V.

The average volume for one stroke at +100 V was 2.67 µl ± 0.25 µl (first box of group 1 in Figure 7) and 6.57 µl ± 0.31 µl for +300 V actuation voltage (fifth box in group 1), thus 2.5 times more.

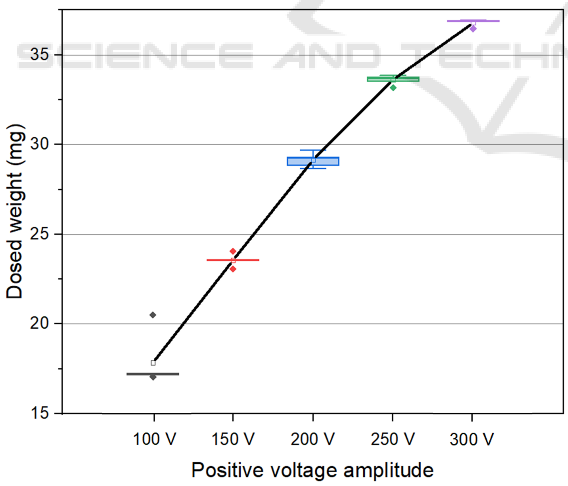

Figure 8: Detailed view of the dosed weight of 5 bursts for 100 V, 150 V, 200 V, 250 V, and 300 V.

Accordingly, the relationship between the voltage amplitude and stroke volumes is also almost linear, but slightly decreasing toward 250 V and 300 V as visualized in Figure 8. It shows group 5 from Figure 7 in detail. In this group, 5 bursts are dosed with positive voltage amplitudes of 100 V, 150 V, 200 V, 250 V, and 300 V, respectively. The mean of each box is connected for better visualization. It emphasizes the linear correlation of the first three voltage packages and the decline of the last two. Moreover, the deviation and outliers from the mean are decreasing toward higher voltage values. Similar behavior is observed in all groups, independent of the underlying amount of bursts.

4 DISCUSSION

The formation of a free jet using a piezoelectric micro pump is achieved and the dosing of different volumes is accomplished by combinations of voltage amplitudes and amount of strokes. In addition, the waveform of the driving signal has an influence on jet generation.

As already described in the publication of Thalhofer et al. (2021), the flanks of the signal must be sufficiently steep to provide defined volume packages. The aim of Thalhofer's publication was to evaluate the dependency of the dosed volume on signal shape, amplitude, and frequency but in a non-free jetting setup (Thalhofer et al., 2021). Consequently, the dependency of the described flank steepness is even more crucial in our paper, concentrating on a free jet creation. In addition to the delivery of defined volume portions per stroke in both setups, droplet detachment in the free jetting setup is also influenced by the kinetic energy of the liquids during a pump stroke which is based on the steepness of the signal flanks (Wackerle et al., 2002).

For the sinusoidal signal, specifically the recorded weights for one stroke, only two out of five packages are measured on the scale indicating that a droplet at the needle's tip is formed without falling down. This droplet formation was observed during the operation of the measurements. This indicates that the flanks are not sufficiently steep in the sine signal, resulting in a gradually growing droplet. Only reaching a specific size and weight and the impulse of another pump stroke cause it to detach and fall. The inconsistency in the measured packages of group 1 in Figure 4, supports this correlation.

Instead, the required free jetting behavior for a predictable volume per package is achieved by providing a certain velocity of the liquid for every stroke. This leads to the formation of a defined jet instead of droplets accumulating at the dosing needle's tip. Therefore, combinations of different voltage amplitudes and signal waveforms provide steeper flanks, thus higher liquid velocity and more precise jets. As sharp signals can have an effect on the pump's mechanical properties and lifetime, the rectangular signal with 250 Hz sine flanks is preferred over the pure rectangular signal.

In another publication, a similar micro pump design was introduced to enable the delivery of high viscosity liquids (Surendran et al., 2024). It was equipped with a piezoelectric stack actuator, thus allowing high pressure actuation while limiting the voltage to 200 V. Surendran et al. (2024) also observed degradation of pump functionality due to actuation with a rectangular signal, although the overall voltage amplitude was smaller.

The non-free jetting single-stroke volumes from Thalhofer et al. (2021) yield 11.1 µl ± 0.1 µl for a voltage amplitude of [+300 V / -80 V], SRS signal, and pump stroke frequency below 20 Hz. In the free jetting setup of our publication, single-stroke volumes with the same parameters, and fixed frequency of 10 Hz, are at 3.59 µl ± 0.04 µl and 6.57 µl ± 0.32 µl for the first and second measurement, respectively. Smaller volumes in the free jetting results, compared to greater values in Thalhofer et al. (2021), can be explained by fluid that remained in the tubes or at the needle's tip. Moreover, even though the exact same pump type is used in the compared publications, each pump within one pump type can be different due to production. However, the results for the first and second measurement of this paper are different for the same parameters and the same pump evaluated. Therefore, small differences in the setup can highly influence the dosed volume. In this publication, the deviation of the average dosed weight is used as an indicator for the precision of dosing with the micro pump. A positive finding is that the deviation for dosed volumes in jetting and non-jetting setups is overall below 5% for the previously described parameters.

For both publications, it was observed that the dosed portions for the sinusoidal signal always show less volume than the SRS packages (Thalhofer et al., 2021).

5 CONCLUSION

Jet dosing with stainless steel micro diaphragm pumps is realized and yields reliable minimal jet volumes of 3.59 µl and 3.66 µl for the actuation with a [+300 V / -80 V] SRS and rectangular signal, respectively. Therefore, they can be implemented in products requiring defined volumes like pipettes.

As the previous comparison of jetting and non-jetting precision shows very different results due to comparing different pumps, future research will focus on the differences of jetting and non-jetting by comparing free jet and standard dosing results of the same pump.

Additionally, it is important to find a method to characterize and calibrate the presented micro pumps, as stroke sizes are varying from pump to pump. Moreover, the feasibility of microliter jetting of higher viscose mediums will be investigated to prepare the usage of micro pumps in dosing of complex pharmaceuticals.

The results presented in this paper, can enable the development of a new pipetting technique. The micro pump provides a jet with a defined volume that can be delivered contactless into a container, making the use of disposable pipetting tips obsolete. For this application, the robustness of free jet microliter dosing using a micro pump must be improved. Replacing the current single-layered piezoelectric actuator wit a stack actuator as described by Surendran et al. (2024), can be beneficial for precision and robustness of free jetting. Moreover, energy consumption and dosing volumes can be reduced while achieving liquid jets with higher velocity, pressure, and viscosity. This can enable the application of micro pumps in needle-free subcutaneous injections.

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