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An Experimental Study on Flow Behaviour in Suction Filling of Pharmaceutical Powders

Omar Ismail[1], Chao Zheng[1], Thomas Chamberlain[1], Anastasiya Zakhvatayeva[1],

Colin Hare[1,2], Edward Yost[3], Ariel R Muliadi[3], Chuan-Yu Wu [1*]

[1] School of Chemistry and Chemical Engineering, University of Surrey, Guildford, Surrey GU2 7XH, UK

[2]School of Chemical Engineering, Newcastle University, Newcastle upon Tyne, NE1 7RU, United Kingdom

[3]Synthetic Molecule Pharmaceutics, Genentech Inc., One DNA Way, South San Francisco, California 94080, USA

*Corresponding Author: Prof. Chuan-Yu Wu, Email: c.y.wu@surrey.ac.uk.

Suction filling is critical for industrial tablet production but poorly understood.

Critical velocity ratio is introduced to characterise the flow regimes.

Full die fill achieved when filling-to-suction velocity ratio is below the critical velocity ratio.

Insufficient fill resulted when velocity ratio exceeded critical velocity ratio.

Targeting the critical velocity ratio optimizes die fill quality.

Abstract

Die filling is a crucial step in the pharmaceutical tablet manufacturing process. For industrial-scale production using rotary presses, suction filling is typically employed due to its significant efficiency advantages over gravity filling. Despite its widespread use, our understanding of the suction filling process remains limited. Specifically, there is insufficient comprehension of how filling performance is influenced by factors such as suction velocity, filling velocity, and the properties of the powder materials. Building on our previous research, this study aims to further investigate the effects of powder properties and process parameters (e.g., filling velocity, suction velocity, fill depth) on suction filling behaviour. A systematic experimental investigation was conducted using a model suction filling system, considering both cohesive and free-flowing pharmaceutical powders. The effect of fill depth on the suction filling of these powders was examined at different filling and suction velocities. The results demonstrate that two distinctive flow regimes in suction filling can be identified: slow filling and fast filling. These regimes are delineated by a critical filling-to-suction velocity ratio. In the slow filling regime, the filling-to-suction velocity ratio is lower than the critical ratio, meaning that the filling phase is slower than the suction phase. Conversely, the fast filling regime occurs when the filling-to-suction velocity ratio exceeds the critical ratio, implying that the filling phase is faster than the suction phase. This study reveals, for the first time, that when the powder flow pattern during suction filling is dominated by plug flow, full die fill (i.e., the fill ratio equals unity) is achieved in the slow filling regime. However, in the fast filling regime, incomplete die fill is obtained. It is also found that when plug flow prevails during fast filling, the fill ratio has an inverse correlation with the filling-to-suction velocity ratio. This study further reveals that when the plug flow assumption is valid, the filling ratio at various fill-to-suction velocity ratios can be well predicted mathematically. Furthermore, it is also found that once the powder flow pattern differs from the ideal plug flow, which could be induced by the filling conditions and powder cohesion, the fill ratio can be overpredicted.

Keywords: Powder flow, Die filling, Suction filling, Tabletting, Pharmaceutical powder

1. Introduction

In the manufacturing of pharmaceutical tablets, die filling is a pivotal step (Cho et al. 2020, Goh et al. 2018a, van Snick et al. 2018, Xu et al. 2021, Yaginuma et al. 2007, Zhong et al. 2021, Zakhvatayeva et al. 2018, Tang et al. 2020, Schomberg et al., 2020 & 2023). Typically, a powder formulation is deposited into a die, followed by compression into a tablet. Achieving consistent and complete filling of the die is crucial for ensuring the quality of the manufactured tablets. Many important tablet attributes, such as weight variability and content uniformity, depend on the die filling step (Guo et al. 2-011a,2011b; Hilderbrandt et al. 2020, Schiano et al. 2018, Sinka et al. 2009, Zakhvatayeva et al, 2019a).

In practical industrial settings die filling typically takes place in continuously operating rotary tablet presses (Cho et al. 2020, Goh et al. 2018a). With these tablet presses, two die-filling methods can be employed: i) gravity filling, where a specified-volume cavity is initially created by setting the position of the lower punch before introducing the powder into the die under gravity, and ii) suction filling, wherein the die is initially fully occupied by the lower punch and covered with powder. The lower punch then moves downward, pulling the powder into the cavity as it is being formed by the descending punch (Wu and Guo, 2012; Mills and Sinka, 2013; Zakhvatayeva et al. 2019a & 2019b; Zhong et al., 2021). These tablet presses usually come with a forced feeding system, often referred to as the feed frame (Hilderbrandt et al. 2020). This system uses one or more rotating paddles to agitate and move powder from the hopper through the system, exiting through the feeder plate outlet and into the die (Le et al., 2010; Grymonpr´e et al. 2018; Goh et al. 2019; Tang *et al.,* 2020; Schomberg et al 2020). Over the last two decades, considerable efforts were made to understand powder flow behaviour during these types of die filling processes, either using lab-scale model die filling systems (Wu *et al.,* 2003; Schneider *et al*., 2007; Zakhvatayeva *et al.,* 2019a&b; Tang *et al.,* 2020; Mendez *et al.,* 2010; Jackson *et al.,* 2007), the industrial scale rotary presses (Peeters et al. 2015; Goh et al. 2019; Grymonpre et al. 2018; Schomberg et al. 2023a&b), or through computer modelling (Gopireddy et al. 216, Guo et al. 2011a, 2011b; Nwose et al. 2012, Ojha et al. 2018, Widartiningsih et al. 2020, Wu 2008, Wu and Cocks, 2006). Although die filling processes in rotary presses typically involved rotational motion of the die, it is generally assumed the centrifugal force is not too strong to significantly affect the die filling behaviour of pharmaceutical powders. Consequently, a linear die filling system could be used to explore powder flow behaviour and was hence employed by many researchers (Wu *et al.,* 2003; Schneider *et al*., 2007; Zakhvatayeva *et al.,* 2019a&b; Tang *et al.,* 2020; Mendez *et al.,* 2010; Jackson *et al.,* 2007).

In gravity filling, powder is deposited into an empty die under the influence of gravity. The performance of die filling is substantially affected not only by powder properties, such as particle size, shape and cohesion (Sinka et al. 2004, Wu et al. 2012), but also by the air initially present in the die, which can be particularly significant for light and fine particles (Guo et al. 2008). Powders with larger and more spherical particles are known to perform better during gravity filling (Zhao et al., 2018; Zhong et al. 2021). To quantitatively describe the powder flow behaviour during gravity filling, Wu *et al.* (2003) introduced the concept of the critical filling velocity, V_C, and related it to the fill ratio. The fill ratio is defined as the ratio of the mass of powder deposited into the die to the mass of powder in a completely filled die. They found that, with a linear die filling system, when the feeding shoe velocity, V_f, was lower than the critical velocity (V_C), the fill ratio (η) was 1, indicating complete filling of the die. A decrease in the fill ratio was observed once V_f exceeded V_C . Wu et al. (2003) introduced the following empirical equation to describe the fill ratio when $V_f > V_C$:

$$\eta = \left(\frac{V_C}{V_f}\right)^n \quad (1)$$

where *n* is a material dependant parameter. Both V_C and *n* are empirically derived parameters that apply only to a specific combination of the powder and the system geometry used. Further investigations using a linear die filling system, conducted by Wu and Cocks (2004), as well as Mills and Sinka (2013), revealed that Equation (1) could describe the variation of the fill ratio with the filling velocity when the filling velocity exceeded the critical velocity in the case of gravity filling.

However, achieving consistent and uniform die fill with gravity filling is challenging, as it often leads to issues such as large fill weight variation, reduced die filling efficiency, and a high tendency for particle segregation. It is well recognized that the flow performance of powder during gravity filling heavily relies on powder properties and process conditions (Wu et al., 2003; Wu and Cocks, 2006; Sinka et al., 2004; Schneider et al., 2007). These studies identified three flow patterns for gravity filling: nose flow, bulk flow, and intermittent flow. Nose flow prevails when the flowing powder bed in the feed shoe forms a nose-shaped profile induced by inertia or powder discharge (Wu et al., 2003; Wu and Cocks, 2006). Bulk flow dominates when the filling process primarily involves the discharge of powders from the bottom of the powder bed (Wu et al., 2003; Wu and Cocks, 2006). Intermittent flow typically occurs when cohesive or air-sensitive powders are used, causing the powder to flow into the die either as chunks of agglomerates or in a discontinuous manner (Sinka et al., 2004). Among these flow patterns, nose flow can promote efficient die filling, but it is not generally achieved with continuously recharged feed frames in the process. Consequently, both bulk flow and intermittent flow prevail in actual production, but typically leading to poor die filling performance.

Hence, suction filling is frequently employed in tablet manufacturing. In suction filling, the downward movement of the lower punch initiates when the feeding shoe traverses over the die (or in the case of rotary die filling, when the die moves beneath the feeding plate). Consequently, the die opening is fully covered by the powder bed, mitigating such adverse effects as air presence, a reduced die filling rate and an increased tendency toward segregation.

In contrast to gravity filling, suction filling hence presents several advantages. Firstly, suction filling enables the complete filling of the die at much higher operating velocities compared to gravity filling in air, as demonstrated in studies by Zakhvatayeva et al. (2019a), Wu and Guo (2012), and Jackson et al. (2007). Suction filling typically results in a higher fill rate, as indicated by Wu and Guo (2012). This proves particularly advantageous in manufacturing processes designed for high throughput without compromising tablet quality. It has been well recognised (Wu & Cocks, 2004; Wu et al., 2003; Guo et al. 2008) that the presence of air significantly hampers die filling efficiency in gravity filling. In contrast, suction filling overcomes these challenges by allowing the powder to enter the die without overcoming a cushion of air inside, a common issue with gravity filling. Additionally, as the lower punch descends, it creates a partial vacuum inside the die, establishing a pressure gradient between the partial vacuum and the air above the powder. This further facilitates the smooth flow of the powder into the die (Wu & Guo, 2012). Schneider *et al.* (2007) also found that powders with small particle sizes, low bulk densities and low permeabilities were more affected by air presence, and for these powders suction filling provided a significant improvement to the fill ratio when compared to gravity filling. Secondly, suction filling not only facilitates the die filling process but also minimises segregation tendencies. The nature of powder being drawn into the die as a plug during suction filling creates limited space and time for particles with distinct properties to disperse. This attribute, emphasised by Wu and Guo (2012), plays a role in diminishing segregation occurrences throughout the filling process.

It Is worth noting that suction filling differs fundamentally from gravity filling, and the powder's permeability plays a crucial role in its performance in suction filling. Powders consisting of small and irregularly shaped particles often exhibit low permeabilities, as shown by Le et al. (2010). Baserinia and Sinka (2019) further demonstrated that low powder permeabilities are advantageous in suction filling. In contrast, powders containing large spherical particles are typically less affected by the presence of air, as demonstrated by Guo et al. (2008), and have high permeabilities, making them less likely to benefit from the effects of suction.

Suction filling typically encompasses the simultaneous operations of powder feeding and suction. Previous studies performed by Wu and Guo (2012) and Zakhvatayeva *et al* (2019a) demonstrated that, for a given suction filling system, there is a critical ratio of the feeding velocity to the suction velocity, denoted as the critical filling-to-suction velocity ratio (V_{CR}). The critical filling-to-suction velocity ratio is defined as a function of the system geometry (Zakhvatayeva *et al.,* 2019a), as follows

$$V_{CR} = \frac{V_{fc}}{V_{sc}} = \frac{(L_s - L_d)/t_{fc}}{H/t_{sc}} = \frac{L_s - L_d}{H} \quad (2)$$

where V_{fc} and V_{sc} are the filling and suction velocities when the effective filling duration t_{fc} and the suction duration t_{sc} are equal, i.e. $t_{fc} = t_{sc}$. L_S is the shoe length, L_d is the die opening length along the same axis as L_S and *H* is the suction depth (i.e., the fill height). Zakhvatayeva *et al* (2019a) showed that when suction filling operates at this critical velocity ratio, the lower punch attains its designated final position concurrently with the feeder just crossing the die opening. In other words, the tailing edge of the feeder reaches the position of the leading edge of die. The value of V_{CR} is considered the maximum value of the operational velocity ratio $V_R = V_f/V_s$ (where V_f is the filling /feeding velocity and V_s is the suction velocity) allowing for complete die fill. Specifically,

$$\eta = 1 \qquad (V_R \leq V_{CR}) \quad (3)$$

It is worth noting that Equation (3) is only valid for the powder exhibiting good flowability or poor permeability, as these powders can be drawn into the die at a rate matching that of the lower punch. In the case of powders with poor flowability and good permeability, the powder's entry into the die can be slow, resulting in the formation of significant voids within the die that may be filled with powder flow under gravity. This can lead to a fill ratio below unity. In such instances, it has been observed that, for a given feeding velocity, the fill ratio decreases with an increase in suction velocity (Zakhvatayeva *et al.* 2019a). The regime when $V_R \leq V_{CR}$ was referred to as slow filling, i.e., the filling phase is slower than the suction phase, in other words, The downward movement of the lower punch stops during the passage of the feed frame.

When the operational velocity ratio V_R is higher than V_{CR}, the process is referred to as fast filling, as the filling phase is faster than the suction phase (Zakhvatayeva *et al.* 2019a), and the lower punch still moves downwards when the feeder completely cross the die (in other words, the downward movement of the lower punch continues while the feed frame has completely passed the die opening.), indicating that no powder is above the die even though the lower punch has not attained its designated position. Therefore, only a fraction of the die volume is filled with the powder before the feeder crosses the die, consequently the fill ration is less than unity. Moreover, the fill ratio decreases as the filling velocity increases, for a given suction velocity, as the effective die filling time is reduced, causing a decrease in the fraction of die volume occupied by powder. Conversely, when the filling velocity is held constant, the effective filling time is determined, a decrease in the suction velocity leads to the decrease in fill ratio.

Zakhvatayeva *et al.* (2019a) experimentally investigated the suction filling of four pharmaceutical powders: lactose, two grades of microcrystalline cellulose (MCC) – Avicel PH102 and Celphere CP102, and acetysalicylic acid (ASA). The study varied operational velocity ratios achieved by increasing the filling velocity while keeping the suction velocity constant, and focused on only one suction depth, specifically set at 50 mm. They demonstrated that Equation (2) generally holds for all four powders tested, while Eq. (3) only gives a good prediction of fill ratio for free-flowing powders but tends to overpredict the fill ratio for cohesive powders; while for fast filling, the fill ratio is not sensitive to powder properties for the four powders considered.

However, the applicability of these flow regimes and their associated behaviours to suction filling under diverse process conditions remains uncertain, particularly when considering variations in velocity ratios. Moreover, it is still unclear whether Eq. (2), which defines the critical filling-to-suction velocity ratio solely based on system geometries, is applicable to other geometrical configurations, as our previous study (Zakhvatayeva et al. 2019a) only demonstrated its applicability for one suction depth. Furthermore, it is of scientific interest to explore whether the suction filling behaviour can be mathematically predicted, and whether the flow properties of feed powders and flow patterns during suction filling could affect the filling performance, subsequently the accuracy of the model prediction. Hence, this study conducts a systematic experimental investigation to elucidate the flow behaviour of both free-flowing and cohesive pharmaceutical powders during suction filling. Specifically, the research delves into the relationship between fill ratio and operational velocity ratio by altering both feeding and suction velocities, along with employing different suction depth values. The objective of this research is to further validate the flow regimes proposed by Zakhvatayeva *et al.* (2019a) and the critical velocity definition under a range of conditions. This study marks the first experimental investigation across different suction depths and velocity configurations, which aims to deepen our understanding of suction filling for pharmaceutical tabletting processes.

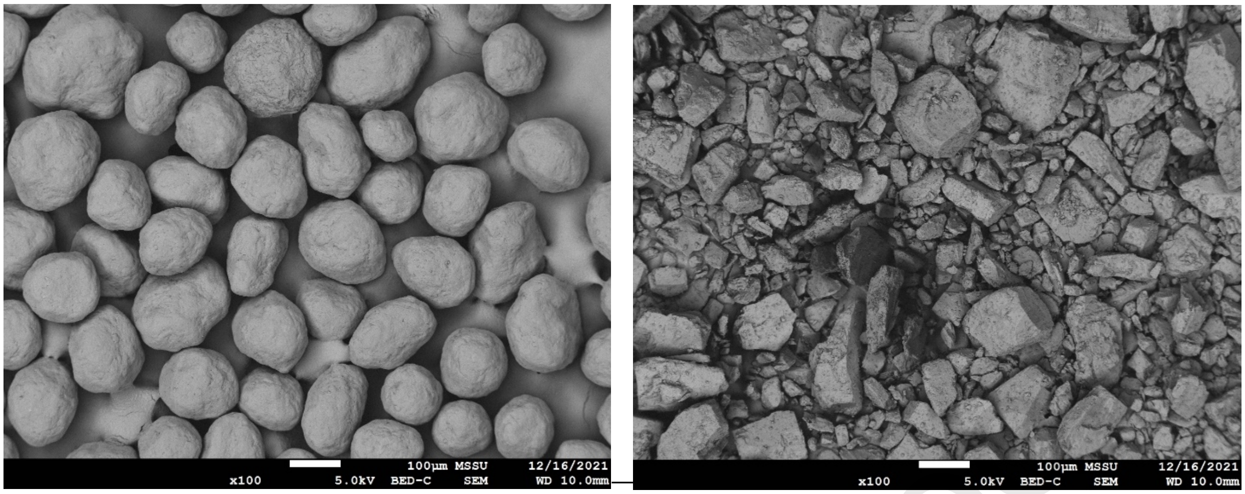
2. Materials and Methods

2.1 Materials

Two powders were considered: microcrystalline cellulose (MCC) of grade Celphere CP102 (Asahi Kasei Corporation, Tokyo, Japan) and lactose of grade Granulac 140 (Meggle Pharma, Wasserburg, Germany). These powders were chosen as examples of very free-flowing and cohesive powders, respectively, as reported in Zakhvatayeva *et al.* (2019a). MCC CP102 is a special celephrine grade of microcrystalline cellulose. Its particles are highly spherical in shape, which causes it to be highly free-flowing. Granulac 140 is a type of lactose monohydrate with fine, sharp-edged particles. It is marketed as being highly cohesive and compressible (Meggle, 2021), and is widely used as a tablet filler.

Figure 1 shows scanning electron microscope images of both powders used throughout this study. The particle size and sphericity distributions of both powders are shown in Table 1; these were measured using a QICPIC optical particle analyzer (Sympatec, Germany) equipped with a GRADIS air dispersion system. Sphericity of a particle is defined as the ratio of the diameter of a circle having the same projected area as the particle to the diameter of the smallest circumscribing circle that can encompass the particle. This method of quantifying particle sphericity is referred to as the equivalent projected circle (EQPC) method (Li & Iskander., 2021). In Table 1, D10, D50 and D90 are the 10th, 50th and 90th percentile of the cumulative size frequency distribution, respectively; S10, S50 and S90 are the 10th, 50th and 90th percentile of the cumulative sphericity frequency distribution, respectively. Three measurements were carried out for each value shown. Flowability of these powder swas measured using a Schulze RST-XS.s ring shear cell tester (Dr. Dietmar Schulze GmbH, Wolfenbüttel, Germany), and is quantified using the Jenkie flow function coefficient (ffc), which are also given in Table 1. These values indicates that MCC CP102 is a free flowing powder, while lactose Granulac 140 is cohesive and poorly flowing.

The permeabilities of these were determined from the observed pressure drops across compressed samples. These pressure drops were measured using an FT4 powder rheometer (Freeman Technology, Tewkesbury, UK). A 25 mm split vessel, along with the necessary accessories and a standard testing program, was utilised. The internal length of the split vessel was 20 mm. The testing program varied the applied normal stress from 1 kPa to 15 kPa. At each value of applied normal stress, air was passed through the powder bed at a velocity of 2 mm/s. Air entered through an aeration base attached to the bottom of the cylinder and escaped through a vented piston at the top of the powder bed. The pressure difference across the powder bed was measured for each applied normal stress value. The determined permeabilities were also given in Table 1. The data indicate that lactose Granulac 140 has a low level of permeability, whiel that MCC CP102 have a higher level of permeability.



a) **b)**

Figure 1: (A) MCC CP102 and (B) lactose Granulac 140 at 100x magnification.

Table 1: particle and flow proerties of the powders used in this study

Powder	Particle size distribution			Particle sphericity distribution			Flow function coefficient	Mean Permeability
	D_{10} (µm)	D_{50} (µm)	D_{90} (µm)	S_{10}	S_{50}	S_{90}	ffc	(m²)
MCC CP102	140.07 ±0.54	182.29 ±1.29	238.30 ±1.96	0.81	0.90	0.93	24.33±1.25	$2.54 x 10^{-11}$
Lactose Granulac 140	37.10 ± 2.11	100.27 ± 2.12	187.35 ±4.14	0.66	0.83	0.89	3.90±0.00	$4.07 x 10^{-12}$

2.2 Die filling experiments

The suction filling system previously used by Zakhvatayeva et al. (2019a, 2019b) was employed. This system is shown in Figure 2. The system consists of a pneumatically driven feeding shoe with inner dimensions of 150 mm in length, 30 mm in depth, and 55 mm in height. This feeding shoe translates across a stationary, transparent die that has a square cross section measuring 20 mm by 20 mm. The die contains a brass piston that can be moved downwards at controlled speeds by an electric motor in order to create suction. The velocities and positions of both the feeding shoe and the piston are controlled by a dedicated control unit.

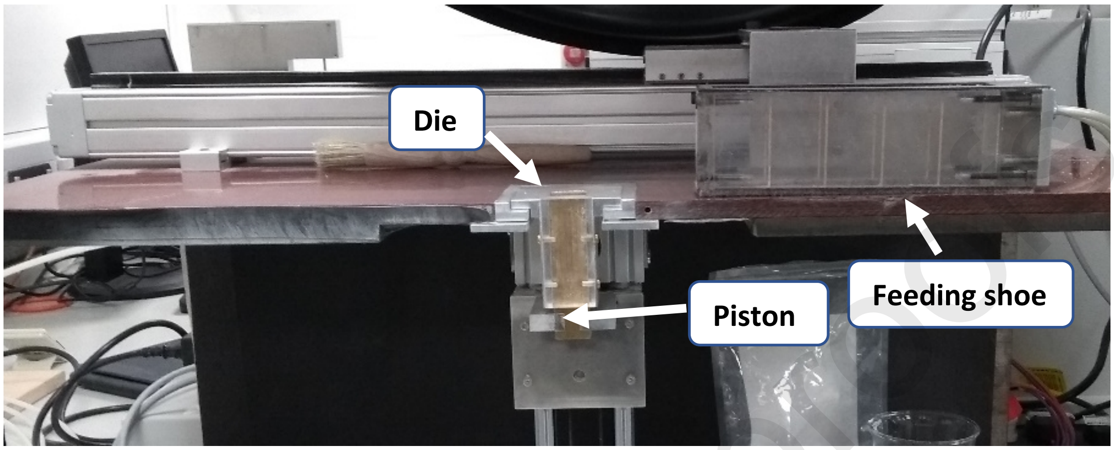


Figure 2: The linear suction die filling system used.

For all experiments, the velocity ratio V_R was varied from 1 to 12. This was achieved by keeping either V_s (the suction velocity) or V_f (the filling velocity) constant while varying the other velocity. For experiments with the variable V_f configuration, the suction velocity V_s was kept constant at 50 mm/s while the filling velocity V_f was varied from 50 mm/s to 600 mm/s. This configuration was previously used by Zakhvatayeva *et al* (2019a). For experiments with the constant V_f configuration, the filling velocity was kept constant at 300 mm/s while the suction velocity V_s was varied from 300 mm/s to 25 mm/s. The velocity ratio ranges used in the experiments were similar to those utilised by other researchers in previous studies (e.g., Jackson et al., 2007, Zakhvatayeva et al., 2019a, Wu et al., 2003).

For both configurations, the feeding shoe acceleration was set to 39.60 m/s^2, which is the upper acceleration limit of the system, in order to ensure that the required filling velocity was reached before the leading edge of the feeding shoe passed over the die. With the feeding shoe in the starting position, the distance between the external leading edge of the feeding shoe and the first edge of the die was 62 mm. The time interval between the movement of the piston and the feeding shoe was controlled so that the piston began to move at the moment that the powder bed fully covered the die. Table 2 listed all experimental conditions employed in this study.

Table 2: Experimental conditions employed in this study.

Powder	Suction depth (mm)	Filling velocity	Suction velocity
Lactose Granulac 140	20, 40, 50	50 - 600 mm/s	Constant at 50 mm/s
Lactose Granulac 140	20, 40, 50	Constant at 300 mm/s	25 - 300 mm/s
MCC CP102	20, 40, 50	50 - 600 mm/s	Constant at 50 mm/s
MCC CP102	20, 40, 50	Constant at 300 mm/s	25 - 300 mm/s

The mass of powder in a fully filled die was determined by manually filling an empty die using a 5 ml metal spoon, without tapping or compression. The filled die was weighed using a balance (PCB 1000-2, Kern Pharma, Terrassa, Spain). This manual filling and weighing process was repeated three times. The mass of powder in a full die was defined as the mean value from the three weight measurements.

For each experiment, three suction depths of 20 mm, 40 mm and 50 mm were considered. The feeding shoe was completely filled with powder and levelled, before a magnetic mesh lid was used to cover the top surface, allowing air to pass through while reducing powder spillage. Suction filling was performed at the specified V_f and V_s values. The amount of powder deposited into the die was measured using the balance. The system was then cleaned and reset for the next test. For each value of V_R, three test runs were carried out and, for each test run, the fill ratio was calculated as the ratio of the deposited powder mass to the mass of powder in a fully filled die, as determined previously. The mean and standard deviation of the three fill ratios were then determined.

3. Results and Discussion

Utilising the methodology outlined in Section 2, a systematic experimental investigation was conducted to delve into the fill ratios achieved under various suction filling process conditions. The results were presented in this section, aiming to elucidate the intricate relationship between filling ratio and two crucial process parameters - suction depth and velocity configuration. The presentation of results in this section facilitates insightful analysis, enabling a deeper understanding of the fill ratio's dependence on these critical independent variables of suction depth and velocity configuration.

In this study, the suction filling system employed was adjusted to accommodate three distinct suction depths: 20 mm, 40 mm, and 50 mm, each corresponding to specific critical velocity ratios V_{CR} of 6.5, 3.25, and 2.6, respectively, as determined by Eq. (2). The primary focus of the investigation was to scrutinise the variations in fill ratio in response to changes in the velocity ratio, considering different powder types, under the specified suction depths. This approach allowed for a targeted examination of how the interplay between suction depth and velocity ratio influences the fill ratio across various powders during suction filling.

3.1 Variable V_f configurations

Adopting the identical velocity configuration as utilised by Zakhvatayeva et al. (2019a), the velocity ratio in our study was manipulated by maintaining a constant suction velocity (V_s) at 50 mm/s, while systematically altering the filling velocity (V_f) within the range of 50 mm/s to 600 mm/s. Subsequently, the fill ratio was determined for each powder and suction depth under consideration. The resulting variations in fill ratio corresponding to different velocity ratios are depicted in Fig. 3. Furthermore, experimental data obtained from suction filling with a suction depth of 50 mm and an identical velocity configuration (i.e., maintaining a constant suction velocity of 50 mm/s), as reported by Zakhvatayeva et al. (2019a), were also superimposed in Fig. 3c.

It is evident that, across both powders and various suction depths under examination, two discernible regimes can be delineated. when the velocity ratio is low, the fill ratio remains relatively constant; however, at the higher velocity ratios, the fill ratio decreases with the increasing velocity ratio. This is consistent with the observation of Zakhvatayeva et al. (2019a) who categorised these two regimes as the slow filling and fast filling regimes, respectively. Notably, for both powders considered, the transition points from the slow filling to the fast filling regime are identical, aligning precisely with the critical velocity ratio defined by Eq. (2).

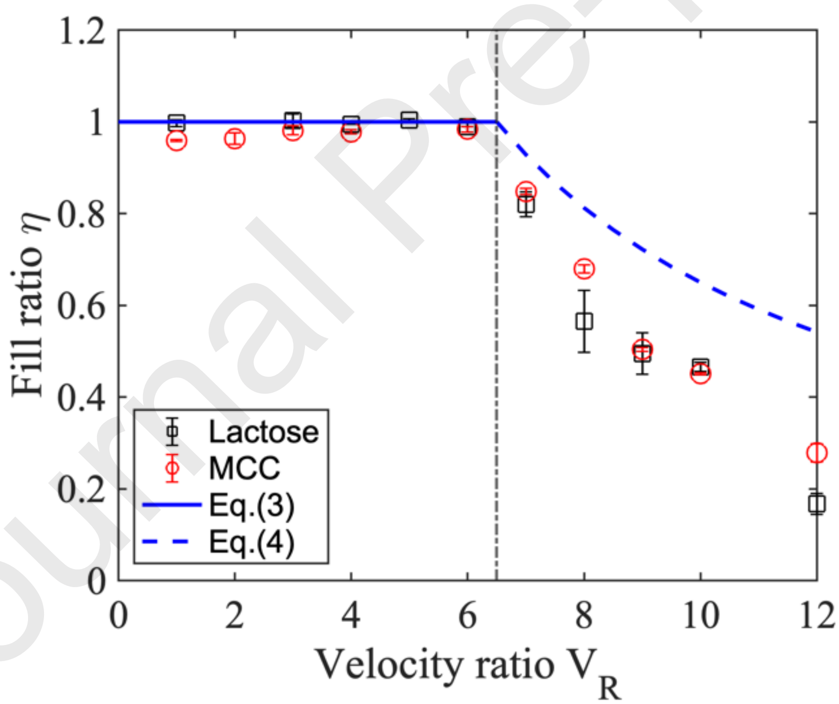
Furthermore, it is also clear that within the slow filling regime (i.e. V_R< V_{CR}), the fill ratio remains consistently close to unity across all cases considered in this study. This is in excellent agreement with the predictions derived from Eq. (3), implying that Eq.(3) provides an accurate estimate of the fill ratio for the powders and suction depths considered in this study. This is because plug flow prevails for these cases considered in this study. A significant deviation from the prediction from Eq. (3) was observed by Zakhvatayeva et al. (2019a) where the flow process is not dominated by plug flow. In contrast, in the fast filling regime, the fill ratio is generally less than unity. This observation underscores that the critical velocity ratio V_{CR} as defined by Eq. (2) accurately represent the upper limit of the velocity ratio at which a fully filled die (i.e. $\eta = 1$) can be achieved. Additionally, Figure 3 illustrates that the critical velocity ratio is independent of the powder used, emphasising its principal reliance on the system's geometry rather than the specific characteristics of the powder.

Within the fast filling regime, where V_R exceeds V_{CR}, the fill ratio decreases with increasing velocity ratio for both powders and all suction depths examined. Considering that the suction filling is generally dominated by plug flow, i.e. the powder mass is drawn into the die as a continuous column of materials, as illustrated by Wu and Guo (2012) and Zakhvatayeva *et al*

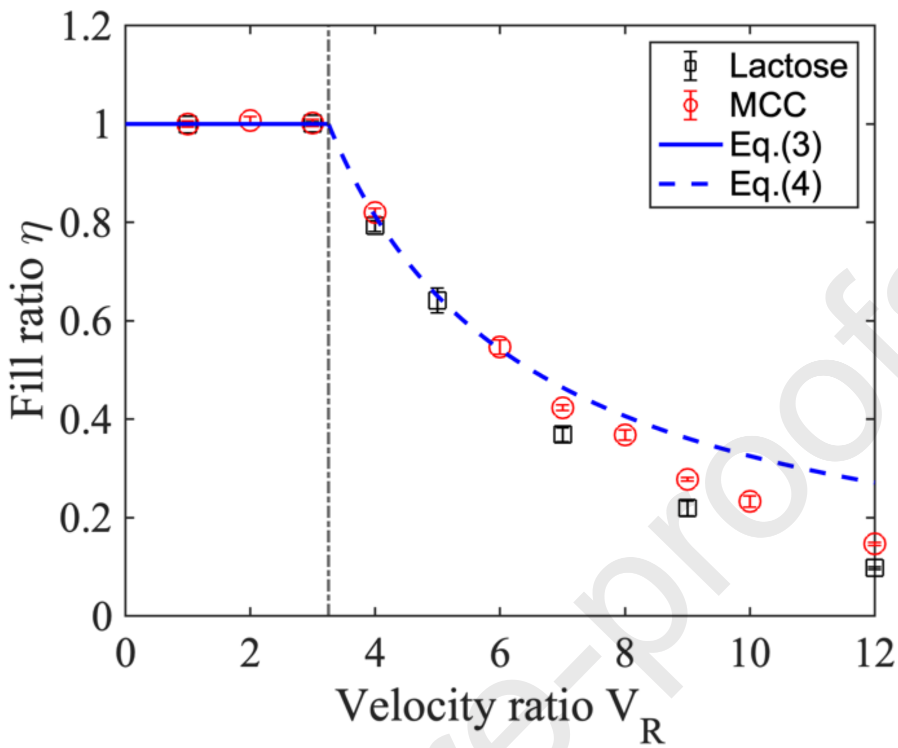
(2019a), and assuming that the packing density of the powder mass does not change during suction filling and a prefect plug flow occur during suction, the fill ratio can be approximated as the ratio of the effective filling time $t_f = \frac{(L_s - L_d)}{V_f}$ to the total travel time of the lower punch (i.e. the suction time) $t_s = \frac{\mathrm{H}}{V_s}$, i.e.

$$\eta = \frac{t_f}{t_s} = \frac{(L_s - L_d)V_s}{H\,V_f} = \frac{V_{cR}}{V_R} \qquad (V_R \geq V_{CR}) \qquad (4)$$

It is evident in Eq. (4) that the fill ratio $\eta = 1$ when $t_f = t_s$, i.e. V_R = V_{CR}. It is worth emphasising that Eq. (4) is applicable for powders that can achieve a perfect plug flow. The prediction of Eq.(4) is also superimposed in Fig.3.

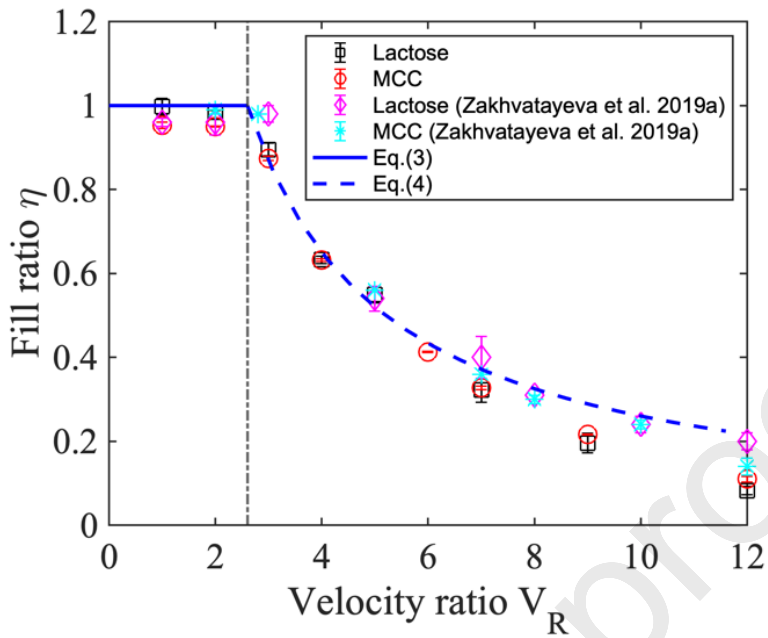


a) H=20 mm



1.2
1
0.8
0.6
0.4
0.2
0
0
2
4
6
8
10
12
Fill ratio η
Velocity ratio V_R
Lactose
MCC
Eq.(3)
Eq.(4)

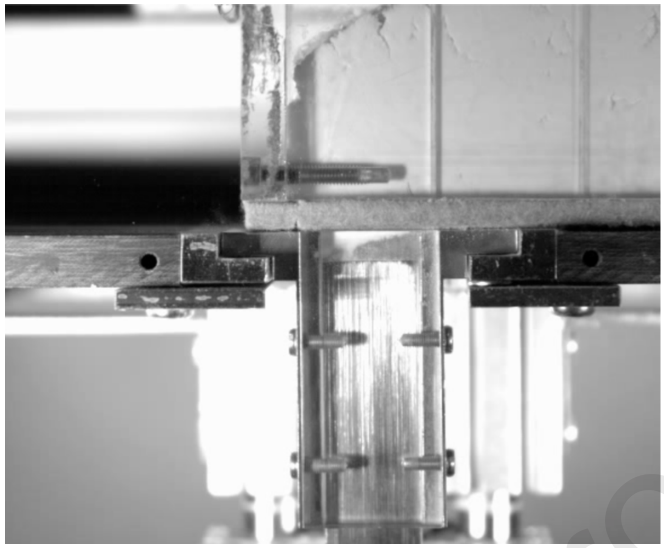
b) H=40 mm



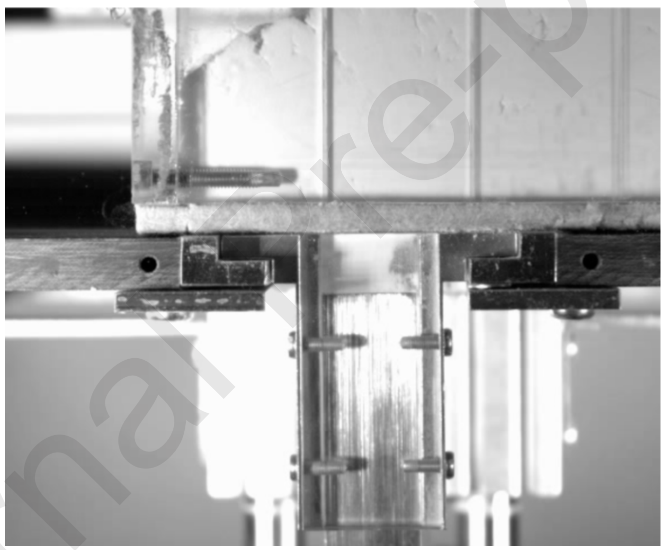
c) H=50 mm

Figure 3 The relationship between the fill ratio and the operational velocity ratio during suction filling with lactose and MCC and various suction depths (the vertical chain lines indicates the critical velocity ratio determiend using Eq.(2). The error bars denote the standard deviation of three measurements.

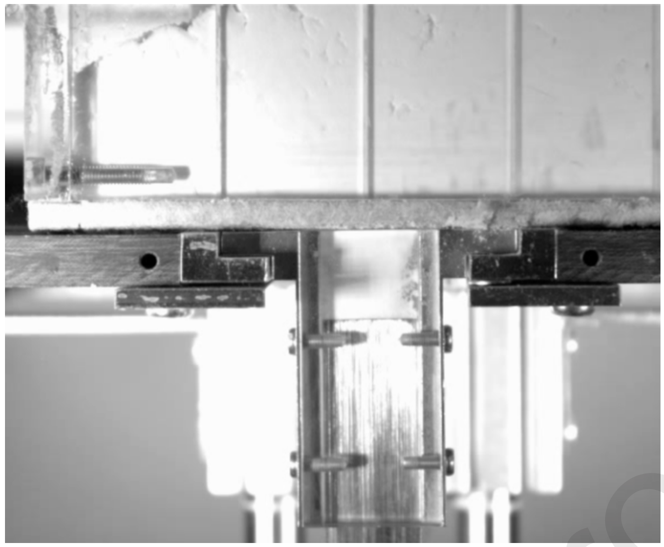
Figure 3 clearly shows that the trend of the variation of fill ratio with the velocity ratio qualitatively aligns with the expectations set by the prediction from Eq. (4). However, Eq. (4) tends to overestimate the fill ratio when the suction depth is set at 20 mm (see. Fig. 3a). Notably, this deviation diminishes as the suction depth increases, as illustrated in Figs. 3b and 3c. A close examination of all these graphs reveals that this discrepancy becomes more apparent when the velocity ratio is sufficiently high, typically when $V_R > 6$ for all cases considered in this study. Specifically, under the current variable V_f configurations, this occurs when the filling velocity surpasses 300 mm/s. The observed deviation is linked to the predominance of nose flow-dominated filling processes at extremely high filling velocities (as illustrated in Fig.4). In such instances, the flowing powder stream travels at a substantial horizontal velocity, often colliding with the leading edge of the die and rebounding backward. This observation aligns with the results presented by Zakhvatayeva et al. (2019a), as also highlighted in Figure 11 of their paper. Notably, a significant void is identified on the trailing side of the die, diverging from the assumption of perfect plug flow made in the derivation of Eq. (4). For suction filling processes predominantly governed by plug flow, Eq. (4) indeed offers an accurate prediction of the fill ratio, as evidenced by its excellent alignment with experimental data within the velocity ratio range $V_{CR} \leq V_R \leq 6$, as shown in Figs 3b and 3c.



(a)



(b)



(c)

Fig.4 A nose-flow dominated suction filling process (Lactose: V_f=700 mm/s; V_s=200 mm/s; H=50 mm).

Furthermore, an observation from Fig. 3 indicates that, within the fast filling regime, the rate of fill ratio decrease is less pronounced for MCC CP102 compared to lactose. Lactose, being a finer and cohesive powder with lower flowability, exhibits a more substantial decline in fill ratio under high velocity ratios. In this scenario, at higher velocity ratios, the rapid filling velocity is more prone to triggering initial densification. This occurs when the feed shoe initiates motion from a stationary position, causing the powder bed in the feeder to be propelled toward its tailing end (see also Fi.g 4a). This phenomenon is primarily driven by the substantial inertia effect experienced during the feeder's acceleration. These densification effects likely become more pronounced for free-flowing powders compared to cohesive powders, and can result in reduced permeability, facilitating suction filling and consequently yielding a slightly higher fill ratio.

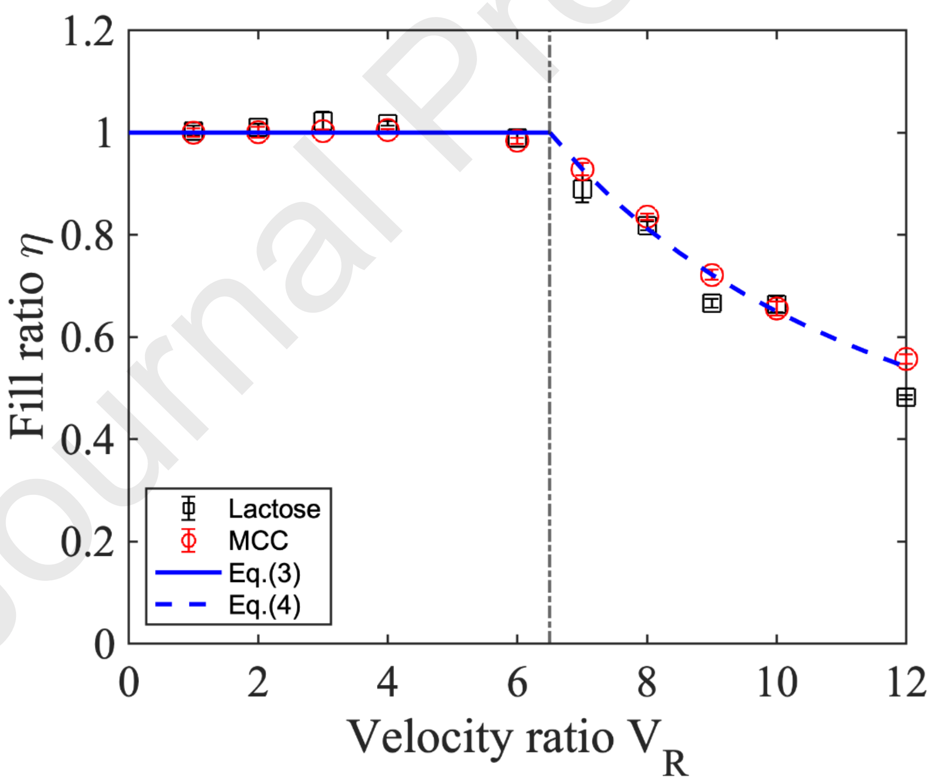
3.2 Constant V_f configurations

The preceding section illustrates that the suction filling behaviour becomes more intricate when the filling velocity exceeds 300 mm/s. It is logical to examine whether analogous die filling behaviour can be observed at the same velocity ratio by maintaining a constant filling velocity V_f while altering the suction velocity. To explore this, a second set of experiments was conducted holding the filling velocity V_f constant at 300 mm/s while varying the suction velocity Vs from 25 to 300 mm/s. This achieved the same velocity ratios of 1 to 12 previously tested. With this velocity configuration, the fill ratios during suction filling of lactose and MCC at various suction depths are depicted in Figure 5.

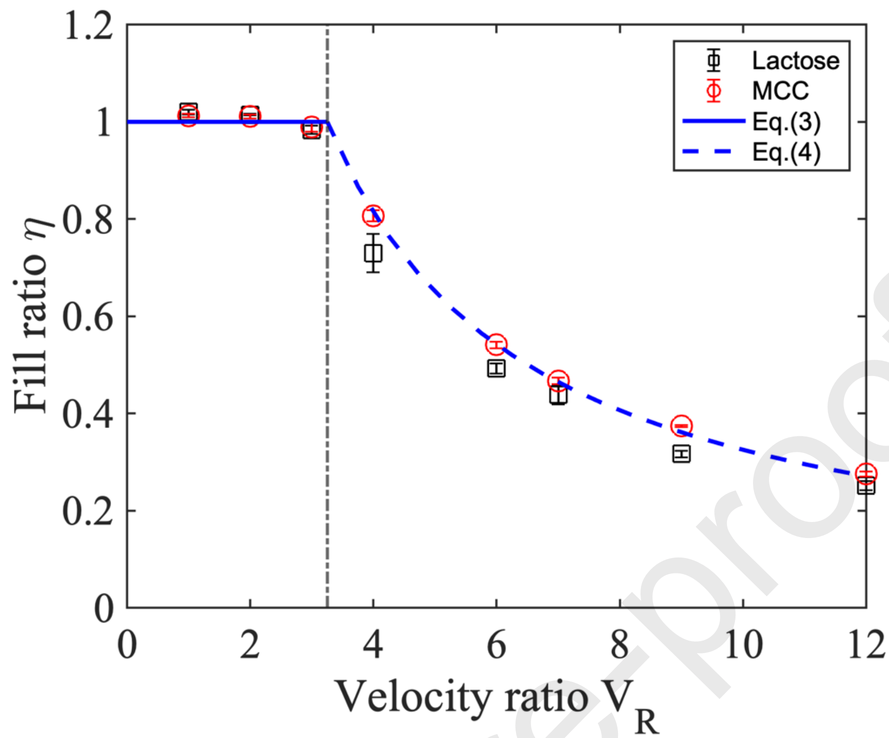
The results depicted in Fig. 5 clearly illustrate a consistent die-filling behaviour across all three suction depths considered. It is evident that die filling can be categorised into two distinct regimes, delineated by the critical velocity ratio V_{CR}. The first regime, referred to as slow filling, occurs when the velocity ratio V_R is below the critical velocity ratio V_{CR}. In this regime, the fill ratio η remains essentially equal to unity. The second regime, referred to as fast filling, is observed when the velocity ratio V_R exceeds the critical value V_{CR}. In this scenario, the fill ratio η decreases with the increasing velocity ratio.

An intriguing observation is that the fill ratios in these two regimes η can be accurately predicted by Eqs (3) and (4), respectively. The agreement between these predictions and experimental measurements is generally superior to that shown in Fig. 3. This suggests that when the impact of nose flow (predominant at higher filling velocities) diminishes—indicating suction filling dominated by plug flow, as illustrated in Fig.6, —Eqs (3) and (4) reliably predict the fill ratio η.

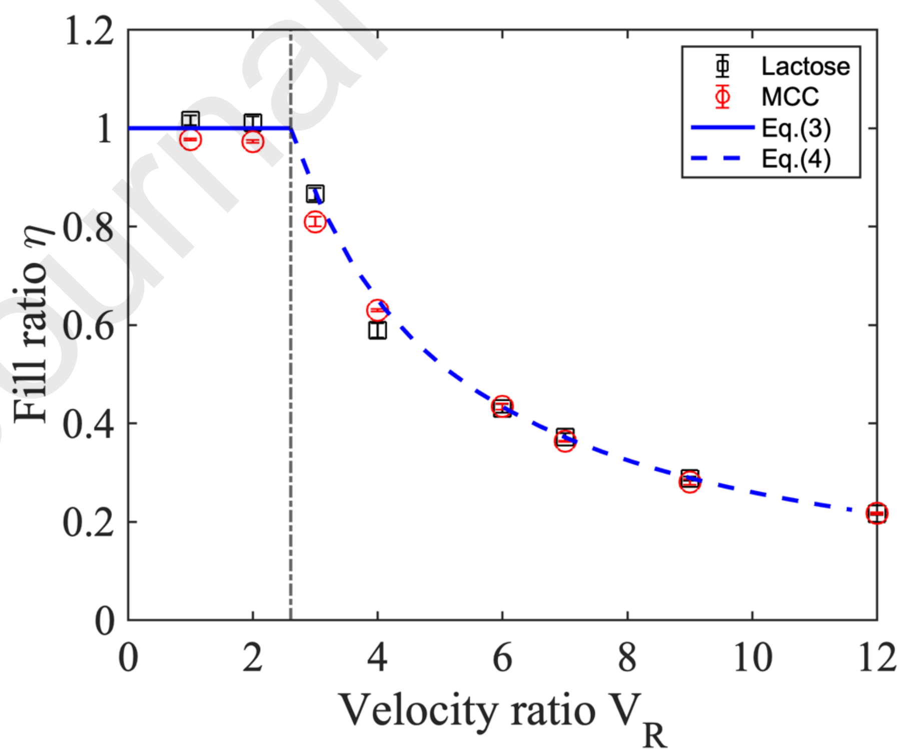
A detailed examination of Fig. 5 also highlights that the fill ratio for the free-flowing powder MCC is marginally higher than that for the cohesive powder lactose, particularly at suction depths of 20 mm and 40 mm. This is attributed to the slight densification caused by inertia when powders are accelerated in the feed shoe from a stationary position. This observation aligns with expectations and broadly corroborates findings by Zakhvatayeva et al. (2019a).



a) H=20 mm

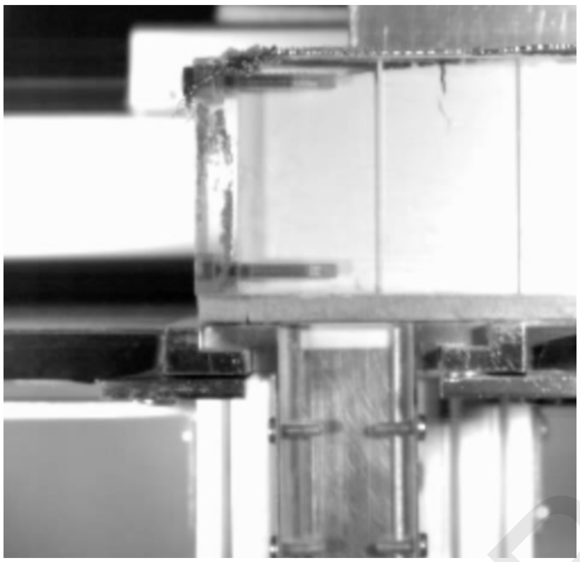

1.2
1
0.8
0.6
0.4
0.2
0
Fill ratio η
0
2
4
6
8
10
12
Velocity ratio V_R
Lactose
MCC
Eq.(3)
Eq.(4)

b) H=40 mm

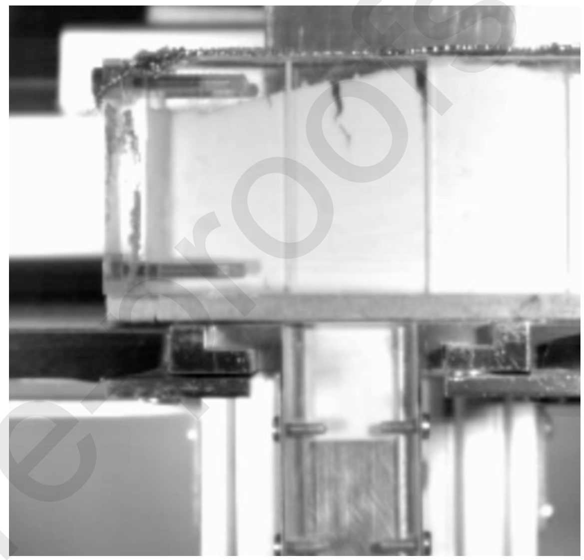

1.2
1
0.8
0.6
0.4
0.2
0
Fill ratio η
0
2
4
6
8
10
12
Velocity ratio V_R
Lactose
MCC
Eq.(3)
Eq.(4)

c) H=50 mm

Figure 5: The relationship between the fill ratio and the operational velocity ratio during suction filling of lactose and MCC with constant Vf velocity configuration and various suction depths. The vertical chain line indicates the critical velocity ratio given by Eq.(2). The error bars denote the standard deviation of three measurements.

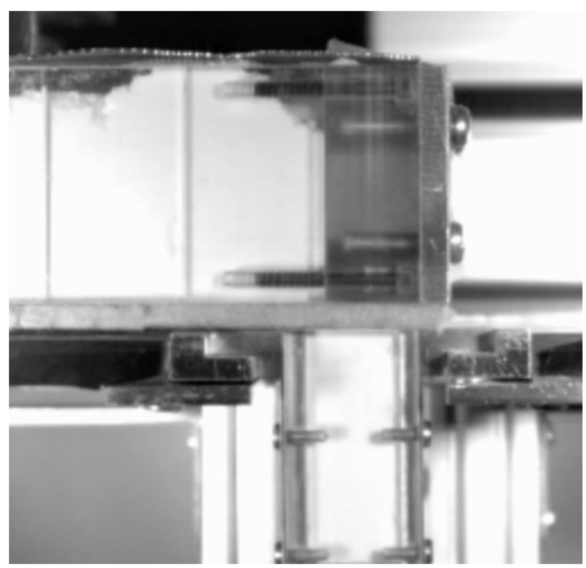


(a)



(b)

(c)



(d)

Fig.6 a plug-flow dominated suction filling process (Lactose: V_f=50 mm/s; V_s=50 mm/s; H=50 mm).

4 Conclusions

The suction filling of MCC and lactose powders into a die with varying fill depths further demonstrated the existence of two distinct regimes: slow filling and fast filling. The demarcation between these regimes is defined by the critical filling-to-suction velocity ratio. In the slow filling regime, complete die fill is attainable, resulting in a fill ratio reaching unity. Conversely, in the fast filling regime, the fill ratio diminishes with increasing velocity ratio. Moreover, for situations where plug flow dominates the filling process, the fill ratio in the fast filling regime can be approximated as the ratio of the critical velocity ratio to the velocity ratio. However, at sufficiently high filling velocities, nose-flow becomes dominant, leading to a significantly lower fill ratio. These observations hold true for both free-flowing MCC and cohesive powder lactose across various suction depths considered. It is plausible to hypothesise that these findings could extend to a broad spectrum of pharmaceutical powder blends. Nevertheless, the validity of this hypothesis requires further confirmation through experimentation with other powders and different suction filling systems.

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Declaration of competing interest

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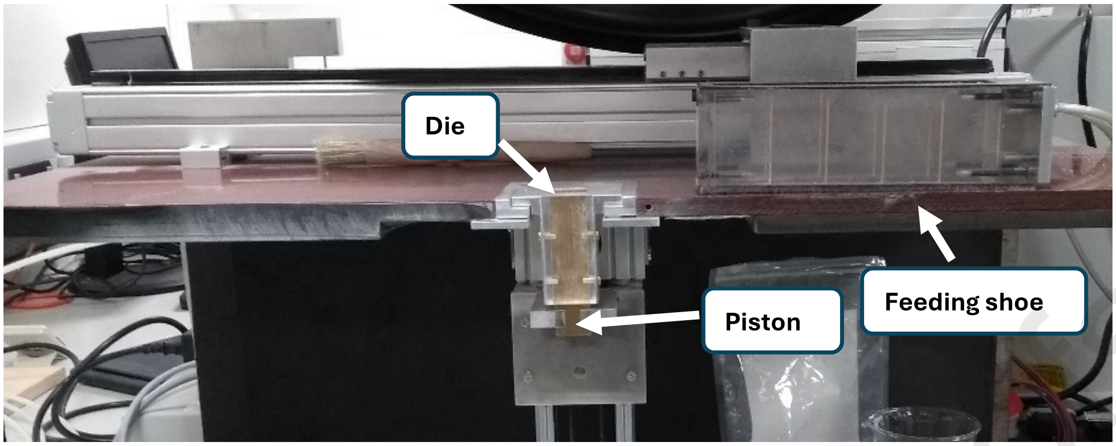
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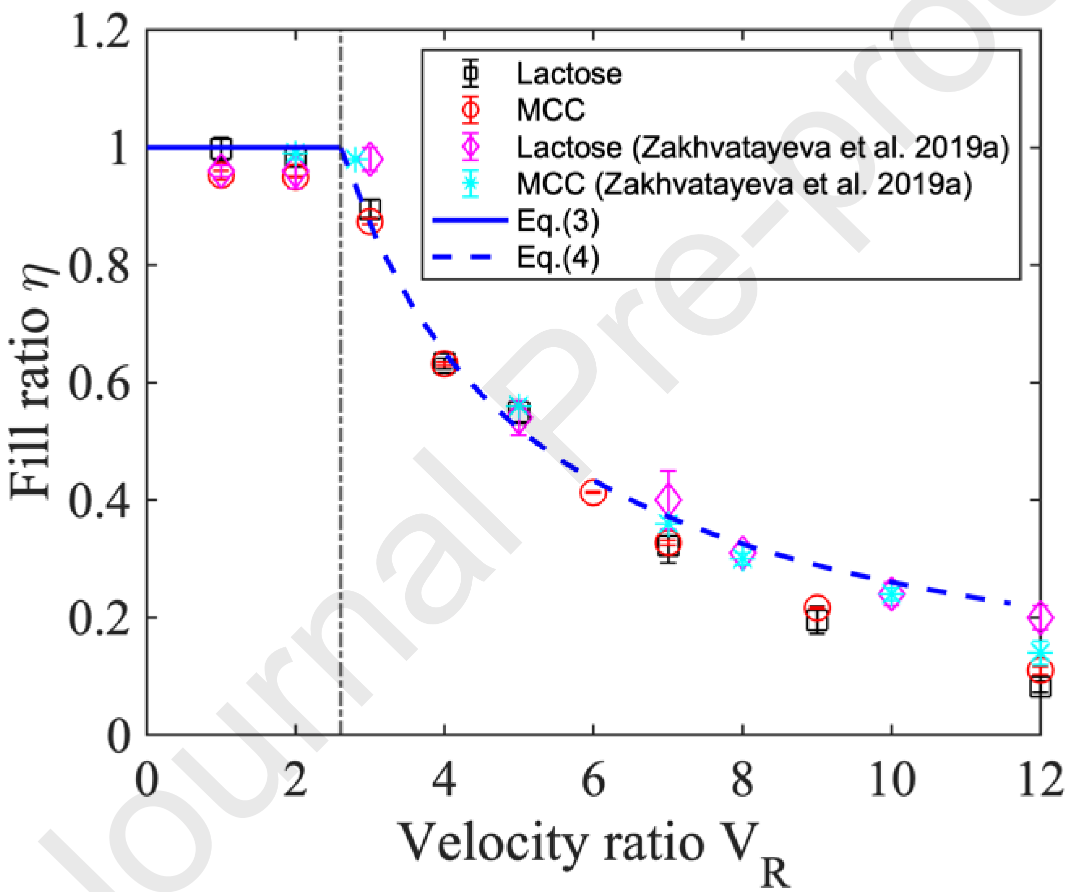
Piston
Feeding shoe


Lactose
MCC
Lactose (Zakhvatayeva et al. 2019a)
MCC (Zakhvatayeva et al. 2019a)
Eq.(3)
Eq.(4)
Fill ratio η
Velocity ratio V_R
0
0.2
0.4
0.6
0.8
1
1.2
2
4
6
8
10
12