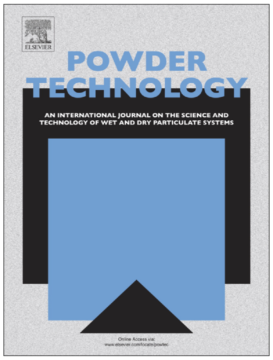

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Study of the feeding performance of mesoporous silica in a loss-in-weight feeder

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ABSTRACT

Accurate and reliable continuous feeding is essential for the continuous manufacturing of solid-dose pharmaceuticals to ensure the reproducible composition of the final product. Consistent feeding of cohesive powders is challenging and requires an understanding of the interplay between material properties and feeder configuration. This study presents the volumetric and gravimetric feeding behaviour of a cohesive pharmaceutical, excipient mesoporous silica, at low feed rates (< 0.6 kg/hr) using a twin-screw loss-in-weight feeder. The study investigates how the screw pitch, screen type and gravimetric setpoint impact the feed factor and feed rate variability. Additionally, the flow function and bulk density of the fed and unfed silica samples were determined and related to the feeding process parameters. Volumetric experiments highlight inconsistent feed due to the poor screw flight filling at high screw speeds and for configurations with the larger pitch, coarse concave screw. Poor screw flight filling also resulted in an inverse relationship between screw speed and feed factor in volumetric mode. In gravimetric mode, feed variability, expressed as %RSD, was greatest at the lower gravimetric setpoints with minimal impact due to tooling configuration. Discharge screen set-up was identified as the parameter which had a significant effect on the bulk density and flowability of the powder post-feeding.

1 INTRODUCTION

In recent years, continuous manufacturing (CM) has gained significant interest within the pharmaceutical sector [1]. In contrast to traditional batch processing, CM offers improved process control and product quality; easier scale-up, reduced waste, energy consumption and labour requirements [2].

Continuous manufacturing is defined as an integrated process of two or more unit operations, in which the input materials are continuously fed into the process and processed materials are continuously removed [3]. The initial feeding stage is a critical step for all powder-based CM processes. Accurate and reliable input of raw materials is required, as any inconsistencies of the powder feed stream input may pass compositional variability onto subsequent downstream processes, thereby negatively impacting the final drug product quality [4].

For pharmaceutical manufacturing, loss-in-weight (LIW) feeders are the preferred method for dispensing APIs/excipients in powder form [5]. The primary component of these feeders is a volumetric feeding device consisting of a moving element, usually a rotating cell, belt, vibratory channel, or screw. The latter being the most common for feeding pharmaceutical materials, however, the selection is highly influenced by the properties of the fed material [6]. The other two components of LIW feeders are a weighing platform and a control module. The function of the weighing platform is to provide a continuous reading of the net weight of material in the unit. This data is transferred to the control module which calculates the feed rate in real time [7].

Typically, LIW feeders are operated in either volumetric or gravimetric mode. In volumetric mode the screws in the feeding device rotate at a fixed speed defined by the operator. In gravimetric mode, the operator specifies a feed rate setpoint instead of a screw speed. During feeder operation in gravimetric mode, the control module compares the calculated feed rate with this setpoint. When deviation is detected between these values, a feedback signal is sent from the controller to the feeding device to adjust the screw speed, thereby minimizing the feed rate disparity [8]. The benefit of having this gravimetric system engaged is it allows the feeder to self-regulate the screw speed to compensate for material and process variables. This provides improved reliability and control over the feed rate.

Studies investigating the impact of pharmaceutical raw material properties on LIW feeding behaviour have demonstrated that there is no one feeding setup which is suitable for all materials [5,9–11]. This highlights the importance of a Quality-by-Design (QbD) approach when designing a LIW feeding process; where there is a good understanding of the relationship between critical material attributes (CMAs), critical process parameters (CPPs) and critical quality attributes (CQAs). Several recent studies have explored this relationship by using multivariate analysis techniques such as PCA (Principal Component Analysis) and PLS (Partial Least Squares) to correlate the input materials properties to the feeding performance [9,10,12–17].

Tahir et al. correlated 12 material properties (including flow function, tapped and bulk density, and particle size distribution) with the initial feed factor [14]. Li et al. generated a model which could estimate the initial feed factor for a new material based on the conditioned bulk density [18]. Conditioned bulk density refers to bulk density measurements performed using the Freeman FT4® powder rheometer following a conditioning step to remove the effects of powder handling and packing. Using this estimation, an operational feeding range was calculated where the feeding variability was minimised. Bostijn et al. characterised 15 materials and correlated the property descriptors with 4 feeding responses [10]. A notable difference in this study was that it examined relatively low gravimetric setpoints (0.1 and 0.55 kg/hr). Wang et al. established and validated a model to predict feeder performance based on 30 material properties [9].

While these previous studies outlined the impact that a range of material properties can have on continuous feeding behaviour; the focus of this study is to thoroughly investigate the feeding behaviour of a single, cohesive powder, mesoporous silica. As a pharmaceutical excipient, silica is often included in formulations in low proportions for several functional uses including moisture protectant, an anti-static agent, an aid in film-coating and glidant [19]. The mesoporous silica investigated in this study has the additional application as a carrier for active ingredients to enhance dissolution performance, which necessitates the use of greater quantities [20]. To manufacture such drug-silica systems in a continuous mode, it is an essential step to understand how such cohesive materials can be incorporated into the continuous manufacturing processes

Earlier studies have highlighted that cohesive powders can be particularly challenging to process [21]. Bostijn et al. found that higher feed rate variability was linked with material properties such as small particle size, low density, poor flow, and high compressibility [10]. While feeding a low-density material, Cartwright et al. encountered powder bridging which necessitated repetitive operator intervention to resolve [22]. Additionally, the material compacted within the barrel housing during operation which eventually led to the upper torque limit being reached, and the feeder shutting down. Engisch and Muzzio examined the feeding behaviour of colloidal silicon dioxide and reported that material adhered to the feeder outlet [21] which can contribute to feed rate fluctuations as powder aggregates may intermittently fall off.

As outlined above, studies to date have focused on examining the relationship between input material properties, process parameters and feeding performance. One aspect of continuous feeding not widely reported is the correlation between the feeder process parameters and output material properties following the feeding process. It is conceivable that the process of feeding could alter the physical properties of the fed material and this in turn could result in altered material performance downstream in subsequent processing steps. Engisch and Muzzio concluded that any impact from feeding was independent of the tooling used, although it was noted that this may not be representative for materials with different bulk properties [21]. Therefore, in this study it was considered important to not only investigate the impact of the feeder tooling configuration and feed rate on the feeding behaviour of the cohesive mesoporous silica material, but also to investigate the impact of the feeding process on mesoporous silica bulk density and flow behaviour.

2 MATERIALS AND METHODS

2.1 Materials

Syloid® 244 FP, a disordered mesoporous silica with a bulk density of 70 mg/cm^3 and an average particle size (D_{50}) specification of 2.5 to 3.7 µm, was supplied by Grace Davison GmbH & Co. KG (Germany)

2.2 Equipment setup

K-Tron MT12 micro feeder

The LIW feeder employed in this study was a K-Tron MT12 twin screw co-rotating LIW micro feeder (Coperion K-Tron) (Fig. 1). It was equipped with a 2 L hopper and within it was a rotating agitator to aid powder flow and screw flight filling. Four different screw types are available for this model: coarse/fine concave screws and coarse/fine auger screws. Coarse concave screws (CCS) and fine concave screws (FCS) were selected for this study as they are designed to have the screw flights closely interspersed in twin-screw configurations. This results in a self-cleaning ability by reducing powder build-up on the screws, which is advantageous when processing low density, cohesive powders [23]. Photographs and dimensions of the concave screws utilised in this study are included in supplementary material Fig. A1. Both the screws and agitator are powered via a gearbox which is connected to the motor. The equipment has a built-in gear reduction system within the feeder which reduces the screw speed in relation to the motor speed. The feeder in this study had a fixed gear reduction ratio of 1.392:1 from the motor to the screws, meaning at 100% motor capacity the screws would rotate at 108 rpm. Discharge screens can be equipped at the screw outlet to help break up aggregates and regulate flow. Three discharge screen options were investigated: a coarse square screen (CSqS), a fine square screen (FSqS), and no screen.

The volumetric feeder was mounted on a weighing platform consisting of a high-resolution load cell. To further reduce outside influences, a protective acrylic draft shield surrounded the complete unit.

Catch scale

An Ohaus Pioneer precision balance was used as a catch scale to independently measure the mass of powder being dispensed from the feeder. The feed rate calculated by the MT12 feeder is subjected to pre-processing algorithms which make it difficult to compare data generated between different feeder models. Therefore, it was necessary to collect the raw mass readings with an independent load cell. This balance, with a 410 g max capacity, was situated directly under the feeder outlet with a beaker collecting the dispensed powder and connected to PC to record the mass readings every 1 second.

2.3 Feeding studies

Volumetric study

The first objective of the volumetric study was to generate feed factor profiles. Feed factor is defined as the mass of material dispensed from the feeder per screw revolution [10]. The profiles can highlight the consistency of screw flight filling, and it this case, can illustrate how screw filling varies as the hopper depletes. Two tooling configurations were tested: the coarse and fine concave screws with the coarse square discharge screen. For each configuration, the feed factor profiles were determined at 3 screw speeds defined as 30, 60 and 90% of the motor capability, which equates to 32.33, 64.66 and 96.98 rpm respectively when gear reduction is factored in.

Prior to each run, the feeder was dismantled and cleaned. After reassembly, the empty feeder and catch scale were tared; the hopper was filled with silica; the screws were primed; and additional material was added to achieve the 100% hopper fill level. This maximum fill was defined as the volume of silica required to occupy the complete hopper leaving a 2 cm gap to the upper hopper rim. The 100% hopper fill equated to a silica mass of approximately 178 g. The total net weight of material in the hopper was recorded for each experiment and used for subsequent calculations. The feeder was then operated until the hopper was fully depleted.

During feeder operation, the raw mass readings collected by the catch scale every 1 s were filtered by removing the periods of disturbance when the collection beaker became full and required to be replaced. An additional 5 s of mass readings were removed on either side of each disturbance to ensure the scales had adequate time to settle. The feed rate was calculated by determining the mass change between every consecutive 1 s mass reading (Eq. 1). Based on the screw speed used, the feed factor was then calculated from the feed rate (Eq. 2).

$$feed\ rate\ \left({}^{kg}/_{hr}\right) = \frac{\Delta\,Weight\ (Kg)}{\Delta\,Time\ (hr)} \qquad \text{(Eq. 1)}$$

$$feed\ factor\ \left({}^{g}/_{rev}\right) = \frac{feed\ rate\ (g/hr)}{screw\ speed\ (rev/hr)} \qquad \text{(Eq. 2)}$$

Feed factor profiles were generated by plotting the feed factor as a function of the % hopper fill level. To improve the interpretability, a 20 s moving average was applied to the calculated feed factors that derives the average feed factor over successive 20 s segments.

The second objective of the volumetric study was to determine the maximum volumetric capacity ($\dot{m}_{max}$) for each configuration. The $\dot{m}_{max}$ represents the feed rate (kg/hr) achievable at 100% screw speed. It is an important parameter to investigated as it has been shown that the volumetric capacity of screws is affected by the pitch size (distance between adjacent screw flights), and the magnitude of this effect is dependent on the properties of the material being fed [13]. Additionally, calculating the $\dot{m}_{max}$ would identify the feed rate range for each configuration which would be required in the subsequent gravimetric feeding study.

As for the previous volumetric method, the feeder was dismantled, cleaned, reassembled, and tared. The hopper was filled with silica, the screws were primed, and the hopper was refilled to achieve 100% fill level. For each experiment the feeder operated at maximum screw speed for a total of 6 min. The first 1 min of data was excluded to allow the feed rate to stabilise. The $\dot{m}_{max}$ was calculated as the mean feed rate of the following 5 min which was reasonably stable. All 6 possible tooling configurations (Table 1) were tested in triplicate.

Gravimetric study

The objective of the gravimetric study was to evaluate the feeding performance of silica using different tooling configurations and gravimetric setpoints (i.e. target feed rates) (Table 1).

Two approaches were considered to define the gravimetric setpoint. The first was to select fixed feed rate values to use across every configuration. The drawback with this option was that each configuration has a different feed rate range. For example, the coarse concave screw has a higher capacity versus the fine concave screw due to larger screw flights. Using fixed values to accommodate all configurations would only partially examine configurations with different capacities. Instead, an alternative new approach was used which defined the gravimetric setpoints as a percentage of the $\dot{m}_{max}$. Each configuration was investigated at 3 gravimetric setpoints, defined as 20, 55 and 90% of the $\dot{m}_{max}$ for that specific configuration. The key benefit of this method was that the full feed rate range of each configuration could be examined.

Prior to each run, the specific tooling was selected; the feeder was tared; and the screws were primed. The operating hopper fill level was maintained between 60-80% of the maximum capacity, which was defined as the mean of the net weights recorded at 100% hopper fill level from the volumetric study. When the lower threshold of 60% fill was reached the hopper was manually refilled with fresh material back to the 80% fill level. This refill procedure using small frequent additions was selected based on the findings of Engisch and Muzzio [24] and limited impact of hopper fill between 60 and 80% on feed factor determined during the volumetric study, Fig.2. More frequent hopper refilling was considered; however hopper refilling switches the feeder to volumetric mode which can result in deviations from the feed rate setpoint. More frequent refills increase the total time the feeder will operate in volumetric mode where it is essentially blind to changes in screw filling and powder density.

Feeders require time on start-up for the feed rate to reach a steady state. The initial period of greater deviation can be attributed to movement of unsettled powder in the hopper and incomplete screw flight filling. The duration required to reach a steady state is influenced by the powder properties and the target feed rate [25]. In this study, low feed rates are used which will increase the start-up period required as the powder will adjust more slowly. This movement will be further hindered due to the poor flow of the silica. Therefore, a relatively long start-up period (15 min) was selected to compensate for the above factors.

In total, the feeder ran for a minimum of 45 min for each experiment. As discussed, the data from the first 15 min was excluded to ensure a steady state was achieved. Feed rate analysis was based on the data from the subsequent 30 min. During the 30 min test period, mass readings were collected by the catch scale every 1 s. This data was then processed with a filtering method to remove interruptions caused by changing the collection container. Firstly, an initial mean feed rate and standard deviation (SD) (Eq. 3) were calculated. The SD was used to set upper and lower limits around the mean of ± 3 SD. Data outside the limits was excluded, along with 5 s on either side of each outlier point. This filtering method solely removes large deviations caused by collection container changeovers while keeping the in-process deviations that represent the feed rate variability. A new mean feed rate, termed as $\overline{Feed}$, and new SD was calculated using the filtered data. From this SD, the relative standard deviation (RSD) was calculated by expressing the SD as a percentage of the $\overline{Feed}$. No moving average was applied to this data as to obtain a more accurate representation of the feed rate variability.

$$Standard\ Deviation\ (SD)\left({}^{kg}/_{hr}\right) = \sqrt{\frac{\sum_1^n \left(feed\ rate\left({}^{kg}/_{hr}\right) - \overline{Feed}\left({}^{kg}/_{hr}\right)\right)^2}{n-1}} \qquad \text{(Eq. 3)}$$

n = number of time points

The material dispensed after the 15 min start-up period was collected for the original gravimetric experiments and stored in airtight containers. These samples were used for post-feeding characterisation.

It was previously reported that moisture sorption can impact the flow and feeding behaviour of powders [26]. To mitigate this additional factor, the relative humidity (RH) of the room was monitored, and volumetric and gravimetric feeding experiments were conducted at RH levels between 35-55% RH. These limits were derived from the silica moisture sorption experiments, see supplemental data (Fig. B1).

2.4 Silica Characterization

Powder flow and bulk density analysis

The flow and bulk density of silica before and after feeding was measured using the Brookfield Powder Flow Tester (PFT) (Brookfield Engineering Laboratories, Inc.). Firstly, a standard 230 cc aluminium trough was gently filled with the sample. After using a shaping blade to remove excess powder, the mass of the remaining silica was determined. The trough was loaded into the tester and a 34 cc vane lid was equipped. The standard flow function test program was selected using 5 consolidation stresses (0.3, 0.6, 1.2, 2.4 and 4.8 kPa) and 3 over consolidation stresses. The tester calculated the major principal consolidation stress and the unconfined failure strength, which was plotted to determine the powder flow function (ff_c). Flow and bulk density characterisation were completed for the fed silica samples using a low or high gravimetric setpoint. Additionally, a control sample was analysed which was created by exposing silica to the environmental conditions (i.e. humidity) of the processing room, without feeding, prior to storage in an airtight container. All measurements were performed in triplicate.

Microscope imaging

Images of the silica samples were obtained using an Olympus BX43 light/fluorescence microscope (4×/0.10 magnification) and Olympus SC100 camera. A DAPI filter was equipped as it improved the clarity of the silica particles. The samples characterised included the control sample, and 2 fed samples to represent the extremes of the bulk density range. This qualitative assessment of the silica was completed in place of a quantitative measurement of the particle size distribution using laser diffraction due to challenges acquiring consistent reliable results due to powder electrostatics.

2.5 Data Analysis

The objective of the data analysis was to investigate correlation between the 3 process parameters (gravimetric setpoint, screw type and screen type) to the measured responses. Feed rate RSD was selected as the feeding behaviour response. The bulk density and flow function of the fed silica were selected as the responses to investigate if the feeding process physically altered the material. Using the ordinary least squares method, the data was fit to individual regression models which could determine the correlation. This analysis was completed using Minitab® 17 software. The setpoints tested in the gravimetric feeding study were based as a percentage of the $\dot{m}_{max}$ and as a result, each configuration consisted of a different feed rate range. To account for this, the gravimetric setpoints were coded as low, medium and high levels (Table 1) rather than using the setpoint in kg/hr. First order interactions were included in the models and a confidence interval of 95% was applied.

3 RESULTS

3.1 Investigating feed factor consistency during volumetric feeding

Feed factor profiles were generated by applying a 20 s moving average to the feed factor and plotting it against the relative hopper fill level as the silica was fed in volumetric mode (Fig. 2).

The first observation was the impact of screw type on feed factor. The coarse concave screw produced higher feed factors relative to the fine screw. This was expected given the larger pitch of the coarse screws which results in a bigger pocket to transport powder. Additionally, comparing the profiles of the 2 screws, the feed factors produced by the coarse screw had higher variability. Distinct peaks and troughs are seen across the range of the hopper fill level. The second observation was the impact of screw speed on the feed factor. In both configurations the 30% screw speed produced higher feed factors, and as the screw speed increased the feed factors decreased. This finding was clearer in the fine screw profiles as there was less variability. Feed factor behaviour in relation to screw speed suggests that there is reduced flight filling as the screws rotate faster. The third observation was the impact of hopper fill level on the feed factor. For the fine concave screw, it was evident that the feed factor gradually decreases as the hopper empties. A similar trend can also be seen with the coarse concave screw although due to the higher variability it was only evident at hopper fill levels below 20%.

The final observation was that "rat-holing" occurred in the hopper during feeding (Fig. 3). "Rat-holing" is a term used to describe extreme cases of funnel flow where only the central material over the hopper outlet is discharged and the material nearer the walls remains stationary [27]. A contributing factor to this unwanted flow pattern was the rotation of the vertical agitator rods which compacted the silica against the walls, aiding adhesion. No operator intervention occurred during the runs as the stationary zones eventually collapsed as the hopper depleted.

3.2 Impact of tooling configuration on the maximum volumetric capacity

A volumetric study was conducted to determine the maximum volumetric capacity ($\dot{m}_{max}$) for each of the screw/screen tooling configurations when operating the feeder at maximum screw speed. It was found that the tooling configuration significantly impacted the $\dot{m}_{max}$ (Fig. 4). As expected, the coarse concave screw produced a higher $\dot{m}_{max}$ in comparison with configurations using the fine concave screw and the same screen. The higher $\dot{m}_{max}$ of the coarse screws agrees with the findings of the previous volumetric experiment where the coarse screws produced higher feed factors. Evaluating the effect of the discharge screens, the no screen configuration control illustrates the

$\dot{m}_{max}$ achievable when there is no obstruction to flow at the feeder outlet. When a screen is equipped the $\dot{m}_{max}$ decreased, suggesting the screen was limiting powder flow. The fine square screen produced the largest $\dot{m}_{max}$ reduction as the narrow gratings resulted in greater resistance to flow. Another observation is that the coarse concave screw-no screen configuration had the greatest variability in $\dot{m}_{max}$ relative to all other configurations.

3.3 Impact of tooling configuration on feed rate variability in gravimetric mode

The erratic, variable nature of the silica feed factor during volumetric feeding (Fig. 2), emphasised the necessity of gravimetric feedback control. To evaluate the impact of tooling configuration on feed rate variability in gravimetric mode, relative standard deviation (RSD) was selected as the indicator of feeder performance control. The feed rate variability calculated in relation to the gravimetric setpoint for each screw/screen tooling configuration is shown in Table 1 and plotted in Fig. 5. There is a clear trend in which the RSD decreases as the feed rate increases. This inverse relationship is most notable at the lower end of the setpoint range, with the lowest feed rate of 0.049 kg/hr producing the highest RSD of 45.6%. The RSD significantly reduces from this point as the feed rate increases, until it levels out at approximately 0.35-0.4 kg/hr. The feed rate profiles for each run are displayed in the supplemental data (Fig. C1).

Comparing the impact of tooling configurations on RSD, no clear trend was observed to suggest an optimal tooling selection to reduce RSD across the feed rate range studied. To quantitatively assess the relationship between the process parameters and the RSD, the data was fit to a regression model (Table 2). From this model 2 parameters, the gravimetric setpoint and screw type, were shown to be significant. The feed rate RSD main effects plot (Fig. 6) indicates that the RSD decreased as the gravimetric setpoint increased. Additionally, lower RSDs were produced by the coarse square screen.

As observed in the volumetric study, “rat-holing” in the hopper occurred during the gravimetric runs, although the effect was reduced by the hopper refill process. The fresh incoming material of the small, frequent refills aided the collapse of the stagnant regions near the walls and as a result no operator intervention was required.

3.4 Characterization of Fed Silica

The flow and bulk density of silica was determined after feeding and compared to a control sample which was unfed. All fed silica samples were processed using a unique combination of screw type, screen type, and gravimetric setpoint (Table 1). As shown in Fig 7, the flow and bulk density varied between the fed samples and differed from the control, which would indicate that the process parameters studied did impact these silica properties. A simple linear regression model ($R^2 = 0.983$) was produced for the fed samples which highlighted that flow function (ff_c) inversely correlated with the bulk density.

To further explore the relationship between the process parameters and the continuous responses (ff_c and bulk density), individual regression models were generated with the significant outputs of both models displayed in Table 2 and the main effects of the parameters shown in Fig 7. For both responses, the most significant process parameter was the discharge screen. The fine square screen, which has the narrowest gratings, had the most pronounced effect that produced samples with a low ff_c and high bulk density. Reducing the obstruction to flow at the feeder outlet, by using the coarse screen with larger gratings or by removing the screen, resulted in an increase in ff_c and decrease in bulk density. Increasing the gravimetric setpoint produced better flowing, lower density silica, however it was only found to be significant for the bulk density response. Screw type was determined to be not significant for either material property response.

Based on the differences in flow function and bulk density of the fed silica, it was clear that that these properties after feeding were dependent on feeder tooling configuration and gravimetric setpoint. To provide a better insight into the physical changes, fed silica samples from either end of the material property spectrum were visually inspected with a microscope (Fig. 8). The images of a high-density silica sample Fig. 8(b) was composed of smaller powder particles compared to the low-density sample Fig. 8(c). Additionally, both fed samples displayed more angular powder aggregates, whereas the particle shape in the control unfed sample, Fig. 8 (a) was primarily spherical and uniform in size and shape.

4 DISCUSSION

The study presented outlines the relationship between the feeding performance of a cohesive pharmaceutical excipient, mesoporous silica, and the feeding process parameters employed. Findings also demonstrate how the density and flow of the fed silica was also dependent on process parameters employed. In an ideal feeding process material would uniformly flow through the hopper and consistently fill the screw flights, however this can prove challenging for cohesive materials such as, Syloid® 244 FP. In this study feeding uniformity was assessed by determining feeding consistency in the volumetric feeding study and the RSD of the feed rate in the gravimetric feeding study.

Variability was expected during volumetric feeding, although the distinct feed factor peaks and troughs, would suggest that transient powder bridging is occurring which is impeding the flow of material into the screws. Two screw types were investigated in this study, a coarse and fine concave screw, where the only difference was the size of the screw pitch. High variability in the feed factor profiles was noted, particularly evident when using the coarse concave screws (Fig. 2). As expected, the larger pitch of the coarse screws increased the capacity to transport material which was reflected by higher feed factor (Fig. 2) and $\dot{m}_{max}$ (Fig. 4) values. However, Fig. 2 highlighted that the coarse screw configuration produced greater feed factor variability in comparison to the fine screw which suggests that the prevalence of inconsistent flight filling may be linked to screw pitch size.

The regression model for the gravimetric feed rate RSD determined that screw type was a significant parameter (p-value = 0.042), however the main effects plot (Fig. 6) indicated that the RSD decreased when using the coarse screw. While this result appears to contradict the findings in the volumetric feeding study, it is important to consider that each tooling configuration (screw and screen combination) was examined over different feeding setpoint ranges, based on the calculated $\dot{m}_{max}$. As the coarse screw configurations have a higher capacity, the setpoints employed were higher relative to the fine screw configurations. This aspect of the study design becomes relevant as the model also found the gravimetric setpoint to be highly significant (p-value = 0.001) where the RSD decreases with an increase in setpoint. As shown in Fig. 5 feed rate RSD was shown to decrease with increase in gravimetric setpoint.

The observed "rat-holing" behaviour, (Fig. 3) which was attributed to an incompatibility between the silica properties, hopper design and the hopper agitator, can also contribute to feeding variability and raise concerns regarding material residence times. Feeding studies of materials with comparable flow functions have reported similar hopper flow complications [28]. Bekaert et al. observed ratholing issues when feeding materials with a high wall friction angle, which consequently resulted in an inability to get consistent screw filling [29]. Evaluating the overall gravimetric feeding performance of the silica as expected variability was high to the low setpoints investigated and the cohesive nature of the silica. Comparing the feed rate RSDs of 9.74–45.62% determined in this study to the variability previously reported by Bostijn et al., which fed materials at 0.1 and 0.55 kg/hr, the RSD ranges are similar [10].

The screen was not found to significantly impact the feed rate RSD of the silica in this study; however this may not be the case for other materials as previous studies have shown that the effect of discharge screens are material dependent [21]. The impact of powder accumulation due to triboelectric charging on feed rate RSD also had to be considered. Ramirez-Dorronsoro et al. had previously demonstrated that colloidal silicon dioxide was highly prone to acquiring a negative electrostatic charge [30]. Allenspach et al. investigated the electrostatic charging in feeding systems and similarly highlighted that the electrostatic powder build-up was impacted by the material properties, along with the powder feed rate [31]. Accumulation of powder clumps due to static could periodically be dislodged and result in mass loss spikes, thereby negatively impacting the LIW feeder control.

For both screw types, the volumetric studies revealed a strong correlation where the feed factor decreased as the screw speed increased. This inverse relationship between screw speed and feed factor may be attributed to a reduced forced screw filling time at higher screw speeds. A similar observation was made by Bekaert et al. during the volumetric feeding of Microcelac®100 [16]. In contrast, Bostijn et al. reported that the screw speed was only weakly anti-correlated with the maximum feed factor [10]. In relation to the feeder discharge screen, the primary finding was that greater flow obstruction at the outlet, achieved with narrower screen gratings, reduced the overall $\dot{m}_{max}$.

While designing a CM process using a QbD approach, it is important to measure the critical properties of output materials prior to each unit operation as process-induced variation in the material properties can lead to downstream process deviations [32,33]. Based on the post-feeding characterization data shown in Fig. 7, continuous feeding did alter the flow and density of silica. Moreover, this impact was not uniform across all runs, but rather correlated with the process set-up and parameters investigated. It was noted that the process of feeding increased silica's flow behaviour, irrespective of the bulk density change. This is visually shown in Fig. 7 as the control sample is an outlier to the trend displayed by the fed samples (i.e. with a bulk density of 71.5 g/L, the control sample has a lower ff_c in comparison to a predicted fed sample with the same density). One theory to explain this finding is that the general process of feeding, irrespective of the tooling used, is altering the packing of the primary silica particles. In Fig. 8 it is evident that the unfed control sample is composed of particles more spherical in nature, which may suggest more loosely bound aggregates. In contrast, both fed samples appear to contain a higher proportion of angular particles with defined edges which may indicate the aggregates are more densely packed. Regression models determined that screen type was the most significant parameter to influence both flow function (ff_c) and bulk density (Table 2). Supporting the theory that differences in flow and density were related to primary silica particles packing into agglomerates during feeding, microscope images (Fig. 8) of samples of high and low density fed silica samples highlighted that they varied in relation to the particle size distribution of the silica agglomerates.

Discharge screens are routinely used when feeding cohesive materials as they can help to break up powder clumps. The findings of this study (Fig. 6) validated this function as it was shown that using a finer screen decreased the ff_c and increased the bulk density, which can be attributed to the creation of finer powder aggregates which can fill voids between larger particles. In contrast, with no screen present the feeder produces coarser particles evident by the lower bulk density and improved flow. Agglomerated particles created within the screws can be sheared while passing through the screen resulting in more angular aggregates.

The gravimetric setpoint set was also shown to have a significant effect on bulk density (Table 2); higher feed rates created silica with a lower bulk density. One possible explanation for this effect may be related to the backpressure built up within the feeder barrel during operation. By examining each screw configuration individually in Fig. 4, it is evident that the screen reduces the $\dot{m}_{max}$ and thereby acts as the feed rate limiting step. The capacity of the screws is not changing between runs, which means the same volume of powder is conveyed, however by restricting powder flow, the screen generates pressure at the outlet. Using higher gravimetric setpoints would lead to greater pressure which may compact the silica into stronger aggregates that are not as easily broken apart, leading to larger particles in the fed material, resulting in a less packing and a lower bulk density. One finding which supports this reasoning is the minimal difference in bulk density found between the low and high gravimetric setpoint runs for configurations where no screen was employed. With no obstruction to flow, the higher feed rate did not produce greater pressure at the outlet, thus the silica was not compacted into stronger aggregates by back pressure as can occur when a screen was employed. A factor which must be noted for the backpressure theory is that finer screens would generate greater resistance to flow and pressure at the outlet, however from the characterization results (Fig. 6) the fine screen produced higher bulk density samples. This may indicate that although stronger aggregates are formed due to the increased backpressure produced by the fine screen, the narrower grating provide sufficient shear to break down the aggregates into a wider particle size distribution as shown in Fig. 8(C), which could increase bulk density due to increased packing. Mapping the motor torque over the duration of the feeding process could provide a useful insight into the generation of backpressure within the barrel. This was not covered within the scope of the current paper; however, it would be a valuable output response to monitor in future studies.

5 CONCLUSION

The study presented provides a comprehensive investigation into the feeding behaviour of a cohesive powder, mesoporous silica, through a twin screw loss-in-weight feeder with a range of screw and screen configurations. Volumetric experiments highlight feed variability due to inconsistent screw flight filling of the cohesive material which was more pronounced at high screw speeds and configurations with a larger pitched screw. Poor screw flight filling also resulted in an inverse relationship between screw speed and feed factor in volumetric mode. In gravimetric mode, feed variability, expressed as %RSD, was greatest at the lower gravimetric setpoints, with minimal impact by tooling configuration. A key finding of the study was the impact of the feeding process on fed silica bulk density and flow. The fine square screen had the most pronounced effect and produced silica with reduced flow and increased bulk density compared to set-ups with coarse square screen or no screen. The study reinforces the need for optimal feeding process design to accommodate the fed material properties and the importance of investigating the impact of feeder configuration and set point on the resultant fed material properties.

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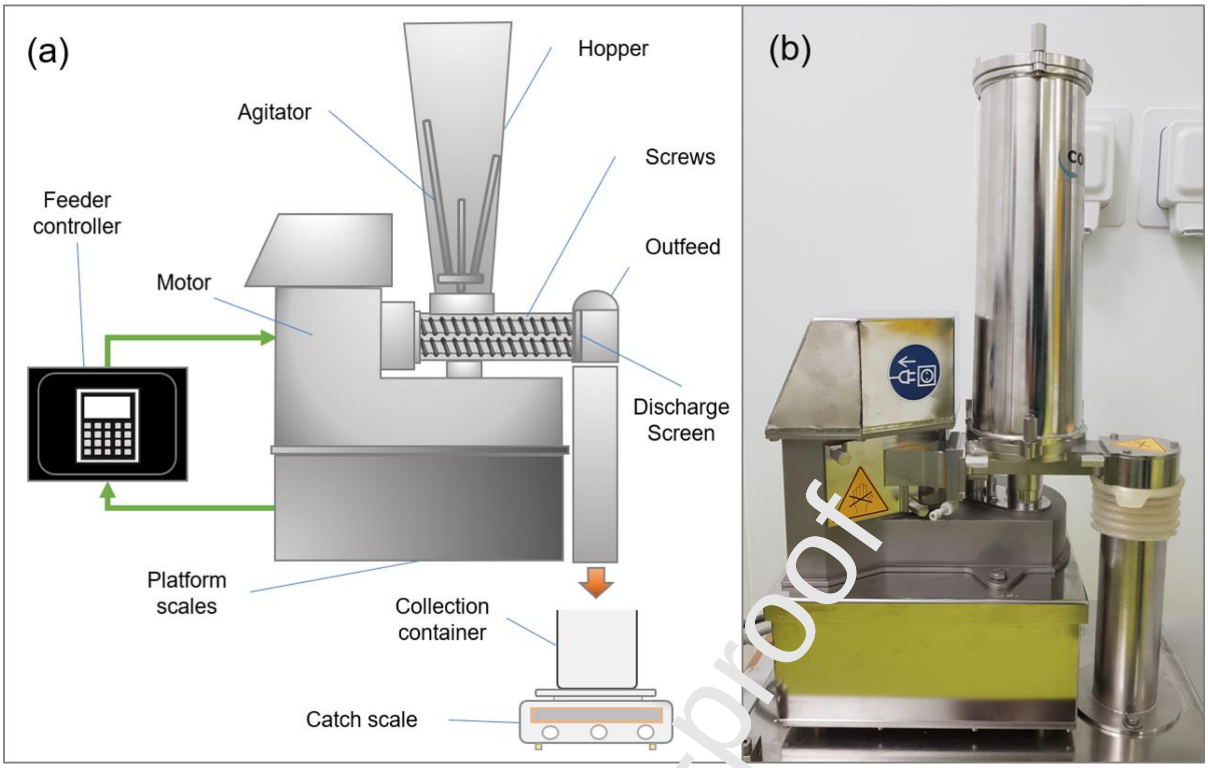

Fig. 1. (a) Diagram of the equipment setup including the feeder, control module, and catch scale. (b) Photograph of the K-Tron MT12 feeder.

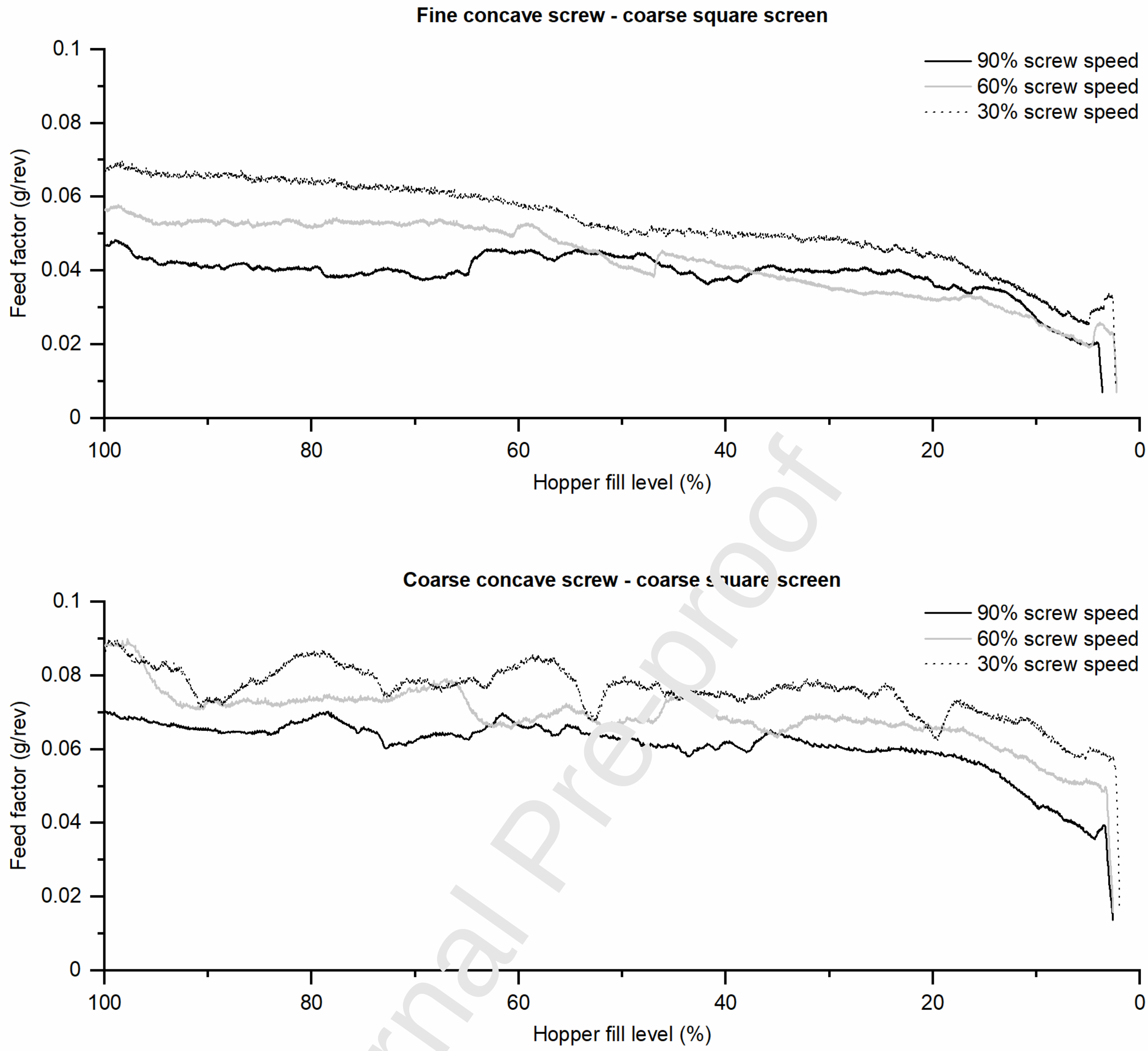

Fig. 2. Feed factor profiles illustrating the 2 examined tooling configurations at 3 screw speeds. The data shown is after the 20 s moving average was applied.

Fig. 3. Example of the silica “rat-holing” in the hopper during a volumetric run.

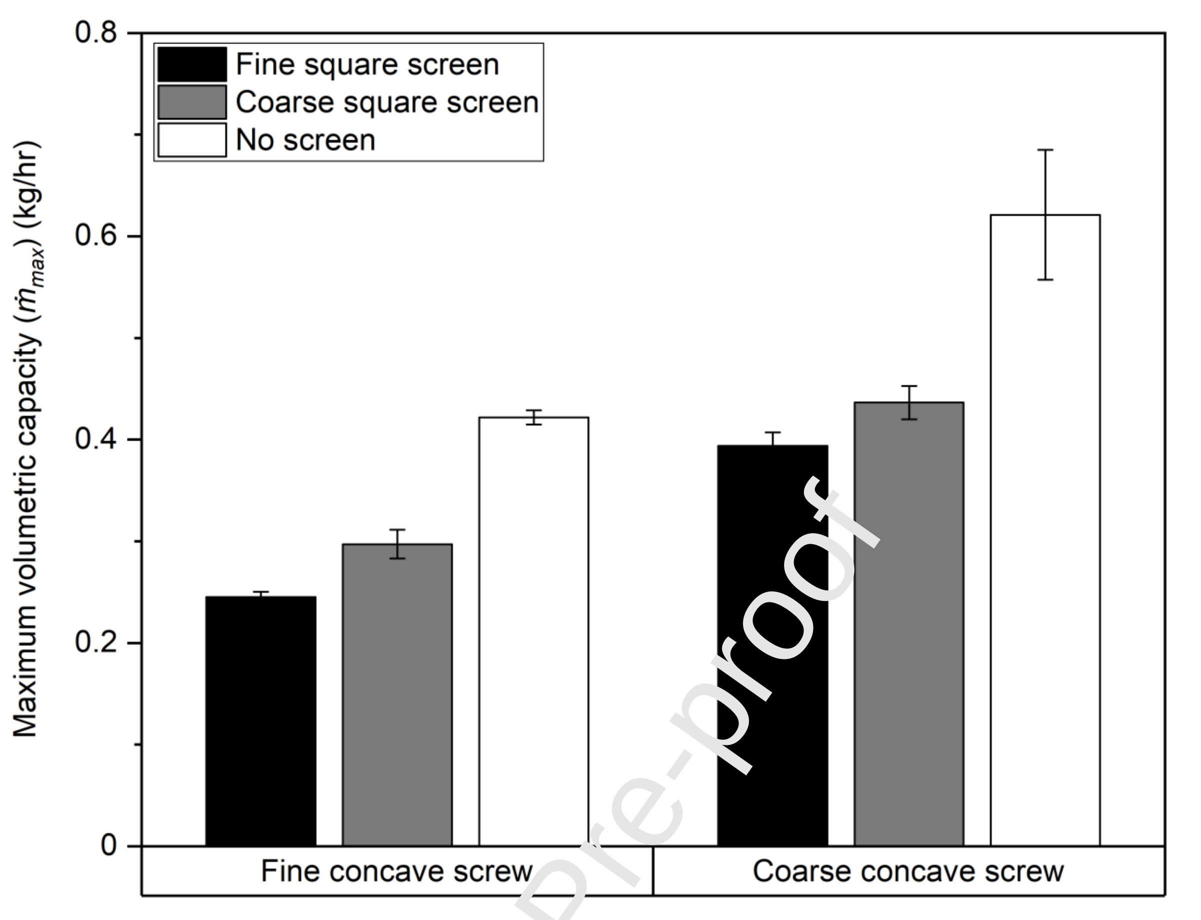

Fig. 4. The $\dot{m}_{max}$ for the feeder tooling configurations measured in volumetric mode. The error bars represent the standard deviation (n=3).

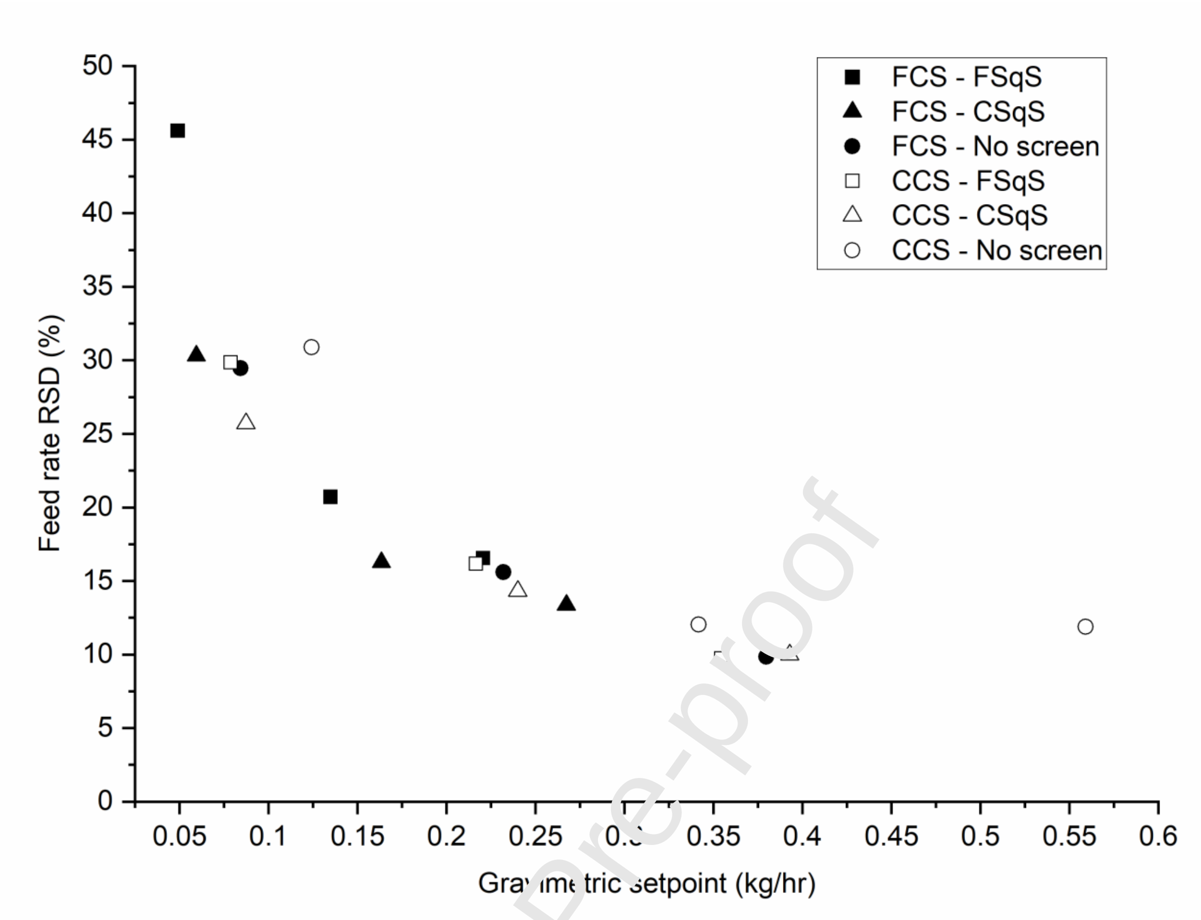

Fig. 5. Gravimetric feeding data plotting the relative feed rate variability versus the gravimetric setpoint. Coarse concave screw (CCS), fine concave screw (FCS), coarse square screen (CSqS) and fine square screen (FSqS).

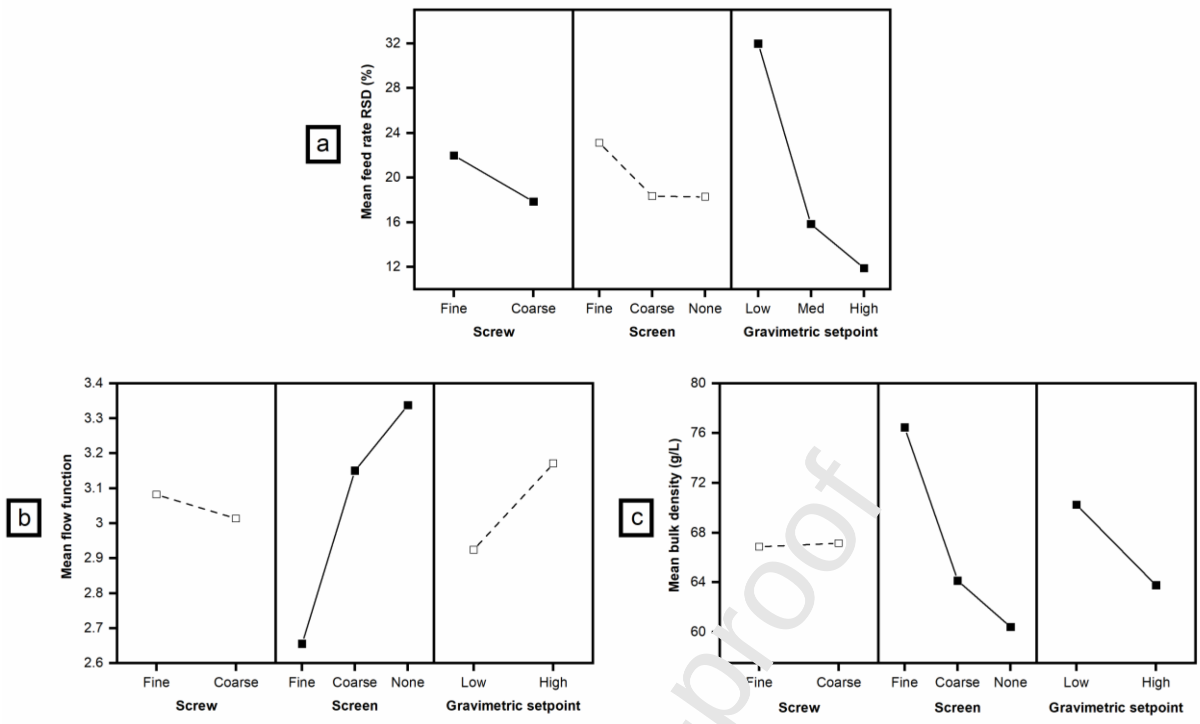

Fig. 6. The main effects plots for (a) feed rate RSD, (b) flow function, and (c) bulk density. Model parameters which have the solid symbol and line represent the significant parameters.

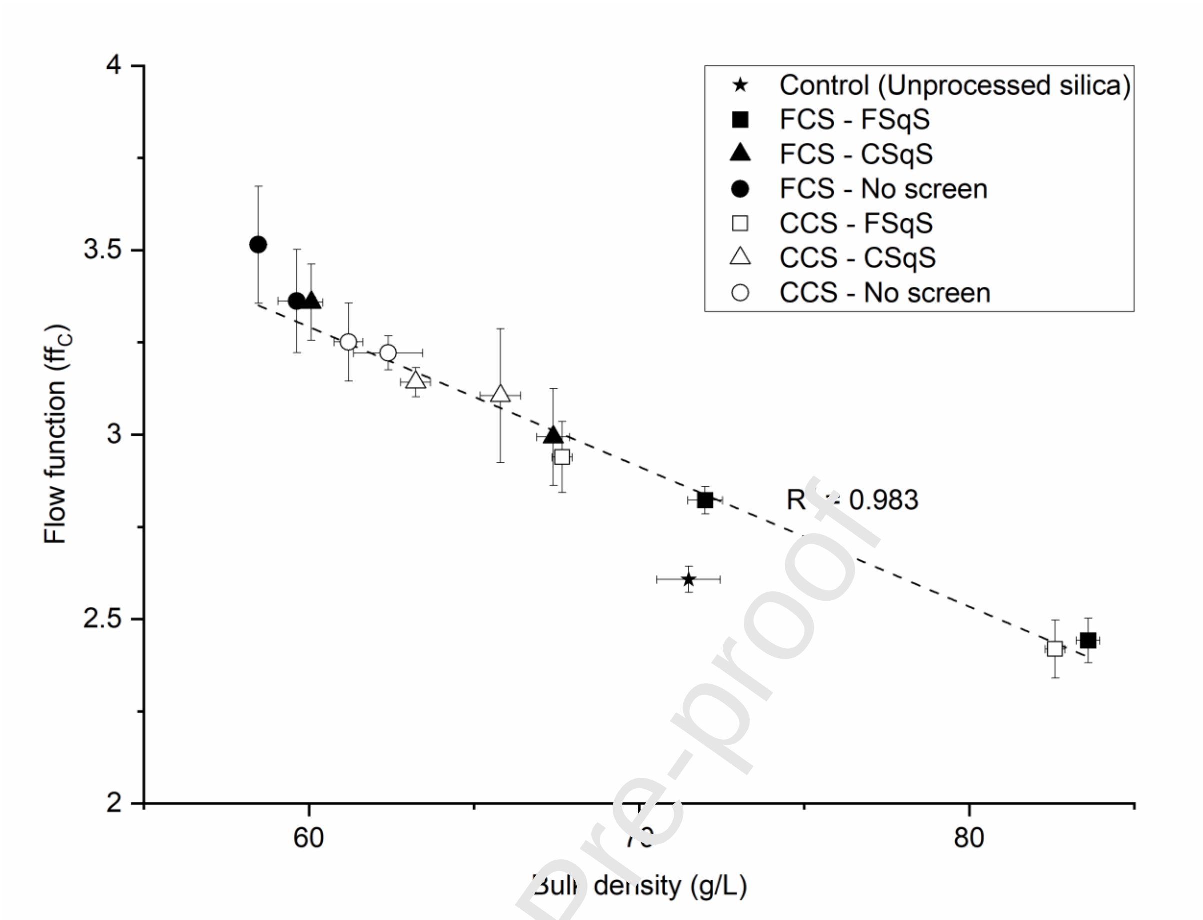

Fig. 7. Plot showing the relationship between the flow function (ff_c) and bulk density of the control and processed samples. A trendline was produced via linear regression using all the fed samples. The error bars represent the standard deviation (n=3). Coarse concave screw (CCS), fine concave screw (FCS), coarse square screen (CSqS) and fine square screen (FSqS).

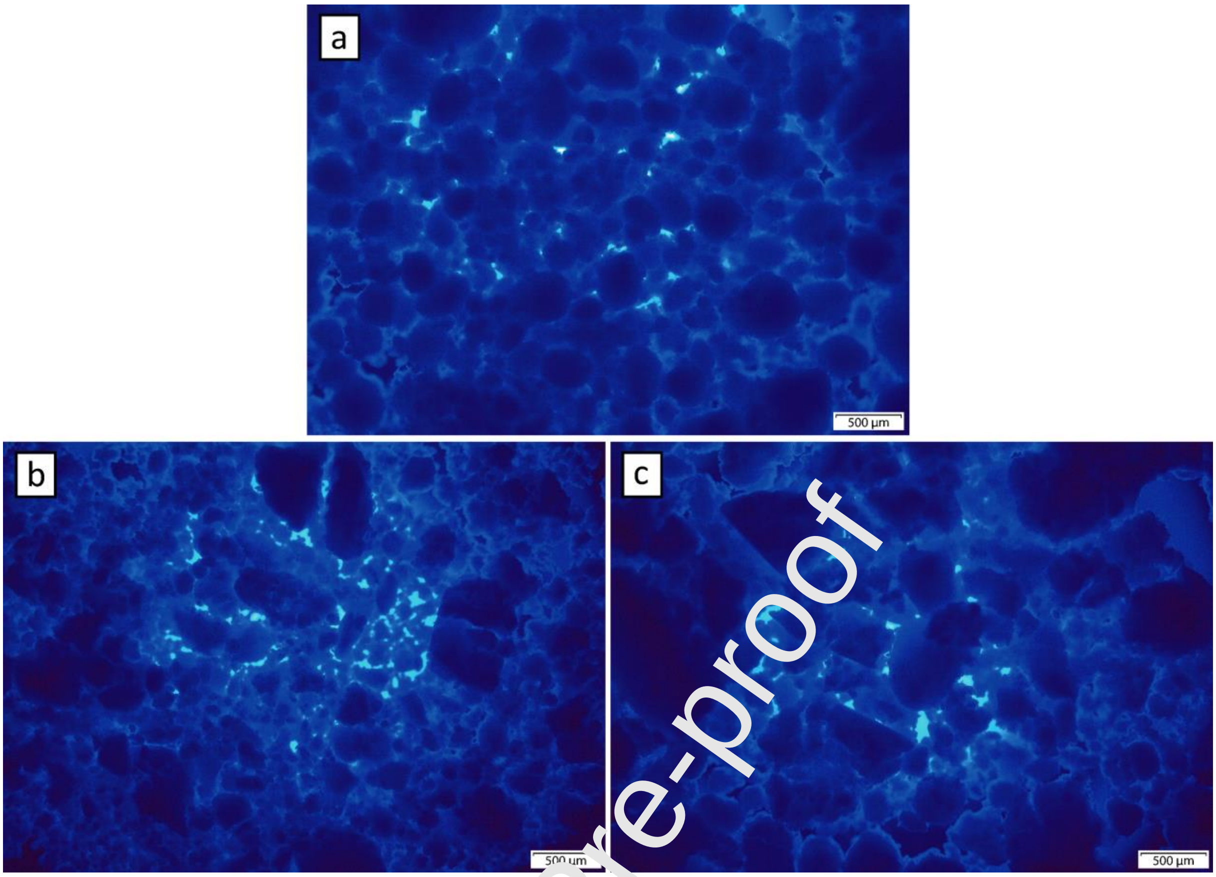

Fig. 8. Microscope images of (a) unprocessed silica control sample (ff_c = 2.61, bulk density = 71.5 g/L), (b) sample fed using FCS - FSqS at the low gravimetric setpoint (ff_c = 2.44, bulk density = 83.6 g/L), and (c) sample fed using FCS - no screen at the high gravimetric setpoint (ff_c = 3.51, bulk density = 58.5 g/L).

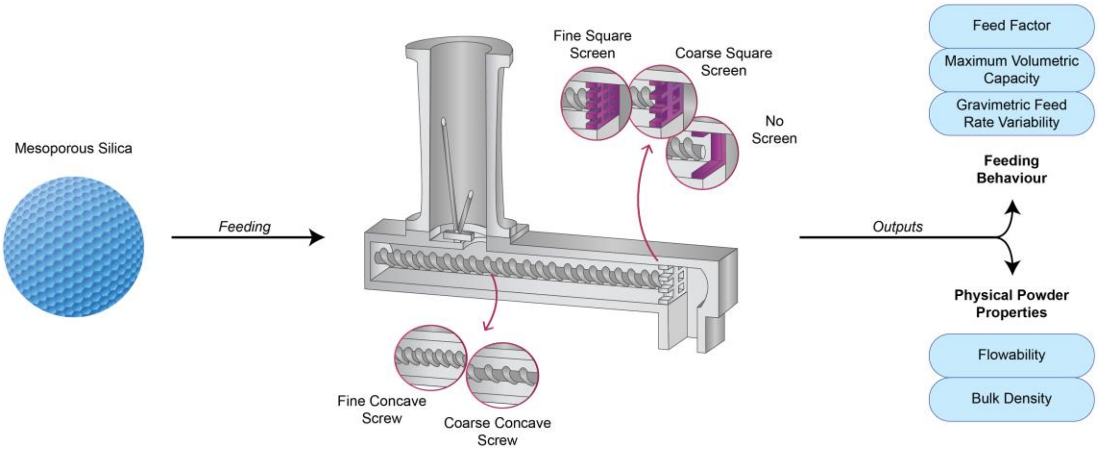
Mesoporous Silica
Feeding
Fine Square Screen
Coarse Square Screen
No Screen
Fine Concave Screw
Coarse Concave Screw
Outputs
Feed Factor
Maximum Volumetric Capacity
Gravimetric Feed Rate Variability
Feeding Behaviour
Physical Powder Properties
Flowability
Bulk Density

Table 1. Feeder tooling configurations and gravimetric setpoints used in the gravimetric study, feed rate relative standard deviation (RSD), and sample bulk density and flow function (average +/- standard deviation, n=3). Coarse concave screw (CCS), fine concave screw (FCS), coarse square screen (CSqS) and fine square screen (FSqS).

Screw	Screen	$\dot{m}_{max}$ (kg/hr)	Gravimetric setpoint			Feed rate RSD (%)	Bulk density (g/L)	Flow function
			Level	% of $\dot{m}_{max}$	kg/hr			
FCS	FSqS	0.2450	Low	20	0.05	45.62	83.6 ± 0.4	2.44 ± 0.06
			Med	55	0.14	20.72	-	-
			High	90	0.22	16.56	72.0 ± 0.5	2.82 ± 0.04
	CSqS	0.2971	Low	20	0.06	30.32	67.4 ± 0.5	2.99 ± 0.13
			Med	55	0.16	16.26	-	-
			High	90	0.27	13.36	60.1 ± 0.4	3.36 ± 0.10
	None	0.4217	Low	20	0.08	29.45	59.6 ± 0.6	3.36 ± 0.14
			Med	55	0.23	15.61	-	-
			High	90	0.38	9.85	58.5 ± 0.2	3.51 ± 0.16
CCS	FSqS	0.3938	Low	20	0.08	29.86	82.6 ± 0.3	2.42 ± 0.08
			Med	55	0.22	16.17	-	-
			High	90	0.36	9.74	67.7 ± 0.3	2.94 ± 0.10
	CSqS	0.4365	Low	20	0.09	25.71	65.8 ± 0.6	3.11 ± 0.18
			Med	55	0.24	14.31	-	-
			High	90	0.39	9.99	63.2 ± 0.5	3.14 ± 0.04
	None	0.6211	Low	20	0.12	30.90	62.4 ± 1.0	3.22 ± 0.05
			Med	55	0.34	12.04	-	-
			High	90	0.56	11.88	61.2 ± 0.4	3.25 ± 0.11
			Silica control sample (unprocessed)				71.5 ± 1.0	2.61 ± 0.04

Table 2. The p-values of the parameters determined from the regression models of the 3 responses. † denotes the significant parameters (α=0.05).

Model parameters	***p-values***		
	Feed rate RSD (R^2 = 0.979)	**Flow function (ff_c) (R^2 = 0.979)**	**Bulk density (R^2 = 0.990)**
Screw	0.042†	0.415	0.829
Screen	0.077	0.027†	0.014†
Gravimetric setpoint	0.001†	0.068	0.032†
*Screw * Screen*	0.131	0.468	0.356
*Screw * Gravimetric setpoint*	0.582	0.524	0.861
*Screen * Gravimetric setpoint*	0.458	0.289	0.098

CRediT author statement

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Marcus O'Mahony: Writing- Reviewing and Editing,

Abina Crean: Conceptualization, Methodology, Supervision, Funding acquisition, Writing- Reviewing and Editing

Declaration of interests

☒The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

ARTICLE HIGHLIGHTS

Study of the feeding performance of mesoporous silica in a loss-in-weight feeder

1. Poor screw flight filling causes inconsistency in feeding in volumetric mode.
2. Poor screw flight filling is more pronounced for screws with greater pitch.
3. Discharge screen setup altered silica density and flow function of post-feeding.
4. In gravimetric mode, screw or screen setup had minimal effect on feed consistency.