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Sustainability vs suitability in granulation

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ABSTRACT

Little is known about the sustainability of competing granulation technologies, despite them being key and widespread industrial processes. This study has evaluated the sustainability of four prominent granulation technology pathways with respect to material wastage (Yield %), Specific Time (Hours/kg) and Specific Energy (kWh/kg). The Fluidised Bed Granulator proved to be the most material efficient while the Roller Compactor was the most time efficient. The energy efficiency of the granulators improved in the following order: Twin Screw Granulator, Fluidised Bed Granulator, High Shear Granulator and Roller Compactor. The wet granulation techniques proved to be the more energy inefficient technologies due to energy expensive sub stages such as drying. This was especially the case with the Twin Screw Granulator and High Shear Granulator where the actual granulating equipment only accounted for around 13% and 15% of the total energy consumed by the entire production pathway. Comparatively, the drying stage accounted for around 45% and 84% of the Twin Screw Granulation and High Shear Granulation production pathways. The different granulation technologies were shown to produce granules with significantly varying properties due to changing granule shape and porosities. The use of visual tools such as parallel co-ordinate graphs and radar plots alongside analytical methods such as priority scoring are suggested as important tools to help manufacturers choose the optimum process pathway based on these varying sustainability and granule suitability factors.

1. Introduction

Process sustainability, especially with regards to energy consumption is an aspect of food production that has experienced renewed interest, with significant reductions in the energy consumption of the food manufacturing sector needed to meet upcoming sustainability targets (Ladha-Sabur et al., 2019). This focus on energy consumption is driven by increased demand on manufacturers to produce cheaper products to accommodate financially pressured consumers, especially in developing markets. In developed markets meanwhile, manufacturers face a pressure both from the public and governmental bodies to adhere to their social responsibility of being sustainable and to reduce their carbon footprint.

Granulation is a well-established size enlargement mechanism which is commonly used in the powder processing industry to improve material handling properties, stability and aesthetics. Granulation can occur with the use of a liquid aid (wet granulation) or without (dry granulation). Wet granulation is commonly carried out in High Shear Granulators (HSG), Fluidised Bed Granulators (FBG) and Twin-Screw Granulators (TSG). Whereas Dry Granulation is mainly carried out in Roller Compactors (RC).

While the literature investigating the mechanisms of these individual processes are quite mature, studies looking at the energy consumption of them, especially in relation to each other are very much in the infancy. With regards to the HSG, the majority of the existing work focuses on using the power requirement of the process to predict the behaviour of the granulation mix, rather than as a basis for rating energy efficiency. For instance Pepin et al. (2001) evaluated the power consumption of a HSG process as a basis for identifying the overwetting point of the mix. Meanwhile, Betz et al. (2004) evaluated the ratio between temperature rise and power consumption in the HSG due to changing process and formulation parameters to evaluate whether it could be used as a tool to develop an artificial neutral network for in-process control. It is a similar story with the TSG, with studies investigating the use of torque as a process control aid to monitor granule size (Ryckaert et al., 2021), or attempting to correlate power consumption with granule shape (Zheng et al., 2022). The focus on energy efficiency is slightly more pronounced in the FBG with literature discussing how to make the technology more

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energy efficient through initiatives like temporarily separating the spraying and drying stages (Mielke et al., 2016). The lack of literature was worst with regards to the RC with no literature evaluating the energy consumption of the process to be found. No literature could be found which evaluates the energy consumption of the whole granulation technology pathways by accounting for the power consumption of additional units such as the binder pump or additional stages like drying.

Furthermore, while broad multi-granulator studies do exist, they only consider a limited number of parameters such as granule attributes (Arndt et al., 2018). No study evaluating the granulation technologies against each other based on energy efficiency could be identified. In order to make informed decisions about the optimum granulation pathway, it is essential to develop knowledge of the energy economy of different granulation pathways and key process parameters effecting it. The aim of this paper is to carry out a comprehensive study looking at the process energy consumption of the main granulation technologies currently used in industry, considering the entire production pathway as a whole. The process energy efficiency will be judged according to their specific energy (kWh per kilogram of in-specification granules produced) factor which not only accounts for the energy consumption of a process but also its granular output. Other metrics for evaluating process sustainability and affordability such as yield and specific time (Hours per kilogram of in-specification granules produced) were also considered. Key granule characteristics such as flowability, friability and dissolution time were also evaluated to add a quality element to differentiate the technologies by. Graphical and analytical tools to help manufacturers make judgements based on process sustainability (judged according to the material, time and energy facets) and process suitability (judged according to granule characteristics) together have then been proposed.

2. Materials and methods

2.1. Materials

Glucidex IT29 (Roquette, France) has been chosen as the model granulation material due to the commonplace use of Maltodextrins in both the food and pharmaceutical industries. In the food industry, Maltodextrins are added as a bulking and carrier agent due to their cheap nature and ability to significantly alter product texture and viscosity. Meanwhile in the pharmaceutical industry it is used as a stabilizer and thickner in cosmetic products and as a cheap filler in oral dosage forms. This popularity has seen Maltodextrins establish a large global market size of $3.15 billion, which is only projected to increase, with current projections putting it at a market size of $4.5 billion by 2029 (Maltodextrin Market Value). Maltodextrin also makes for a very good model material to represent amorphous food powders which makes the conclusions of this work not only applicable to Maltodextrin but to a wide variety of amorphous food powders. The powder size characteristics can be described by a d10 of 143 μm, d50 of 255 μm and d90 of 455 μm. Powder was conditioned at 20% Relative Humidity and 20℃ for 72 h in a Memmet IN110 Humidity Chamber (Memmet, Germany). This was done to remove moisture from the powder to reduce the powder's glass transition temperature and reduce the powder's tendency to cake. The chosen binder for the wet granulation processes was distilled water at 20℃.

2.2. Equipment and methods

Four different granulation equipment were tested and evaluated to produce granules. The granulation equipment includes the Fluidized Bed Granulator (FBG), the High Shear Granulator (HSG), the Roller Compactor (RC) and the Twin Screw Granulator (TSG). For each evaluated granulation condition, the experiment was repeated a multiple of three times and the average taken to check for reproducibility.

When comparing the energy efficiency of competing technologies, it is important that the process conditions used to represent each technology undergo a degree of optimization. This is done to ensure that the technology is being represented in its best light to allow for fair comparison. Therefore, the process parameters were optimized to maximize yield. This is because a higher yield results in less material wastage, which is another major consideration for food manufacturers in the drive to reduce costs and improve sustainability. For example, in the wet granulation processes this involved using the highest L/S possible without caking or over-wetting becoming a problem to maximize total granular yield. The remaining auxiliary parameters were then honed to favor the production of in-spec granules.

After the process had been optimized for yield, the process batch size/throughput was chosen as the primary condition of interest for this paper. This is because throughput, along with the yield, determines the mass of useful granules produced and therefore will play a significant role in determining the specific energy (kWh/kg) of a process. For the FBG and HSG processes, this meant batch sizes between 0.3 – 1 kg and 0.2 – 0.4 kg respectively were evaluated. In the RC, throughputs between 6.66 - 16.7 kg/hr was evaluated. This was done by activating the roll gap function and altering roll speed between 3 – 9 rpm, to change the throughput of the process. In the TSG, throughputs between 0.3 and 0.7 kg/hr was evaluated. The maximum and minimum values for batch size/throughput were chosen based on the limits at which the process could be run stably.

2.2.1. Fluidized bed granulator

The FBG technology was represented by a Glatt GPCG3 Fluidised Bed (Glatt, Germany) with a side entry spray nozzle. Batch Sizes between 0.3 and 1 kg were evaluated. The inlet air speed required to maintain bed fluidization varied with batch size as follows: 2 m/s (0.3 – 0.5 kg), 2.5 m/s (0.7 kg), 3.2 m/s (1 kg). A L/S of 0.18 was used because beyond this value there was no significant increase in granular yield. A spray rate of 4.5 g/min was used because it was the fastest the liquid could be added, which minimized run time, without caking occurring. Air temperature was limited to 30 °C because temperatures higher than this led to bulk adhesion in the powder bed. An atomization pressure of 1 bar was used as it generated a good droplet size and spray zone. The total granulation run time varied due to the changing binder spray times as follows: 12 mins (0.3 kg), 20 mins (0.5 kg), 28 mins (0.7 kg), 40 mins (1 kg).

2.2.2. High shear granulator

The HSG technology was represented by an Eirich EL1 High Shear Granulator (Eirich GmbH, Germany). Binder was fed in using a Graseby 2100 Syringe Pump (Burtons, UK). Batch sizes between 0.2 and 0.4 kg were evaluated. A bowl speed of 170 rpm and a relatively low impeller speed of 900 rpm was used to give a good degree of mixing while minimising heat generation due to friction. The machine was operated in a co-current mode which means the bowl and impeller were set to rotate in the same direction. The mixing pan was configured horizontally (inclination angle of 0 °). A L/S of 0.07 and a spray rate of 1.5 g/min were the optimum values for each parameter that could be used while preventing caking. Wet massing time: 5 mins. Total granulation run time varied with batch size due to the changing binder spray time: 15 mins (0.2 kg), 20 mins (0.3 kg), 25 mins (0.4 kg).

2.2.3. Roller compactor

The RC technology was represented by an Alexanderwerk WP120 Pharma Roller Compactor (Alexanderwerk AG, Germany). The roll speeds evaluated were between 3 - 9 rpm. This corresponds to material throughputs between 6.66 kg/hr and 16.7 kg/hr. The hydraulic pressure used was 70 bar along with a roll gap of 2 mm. A compaction pressure of 70 bar was chosen because the use of higher pressures did not result in any improvements to the yield faction and only increased the power consumption. A milling speed of 40 rpm and mill mesh sizes of 2.45 mm and 1.25 mm were chosen to produce granules in the desired size range.

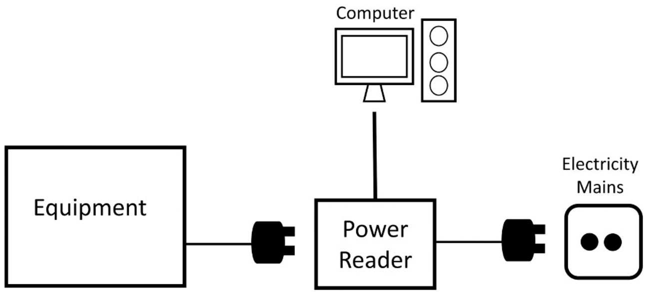

Fig. 1. Visual representation of the power measurement technique. The equipment to be measured was plugged into the power reader and the power reader into the mains. The power drawn by the machine as a whole could be recorded manually or automatically by connecting the power reader to a computer.

2.2.4. *Twin screw granulator*

The TSG technology was represented by a ThermoFisher Euro Lab 16 mm Twin Screw (ThermoFisher Scientific, UK). Incorporated into the TSG system is a loss in weight twin screw powder feeder (K-Tron Soder, Switzerland). An IC05C Water Chiller (ICS Cool Energy, UK) was used to maintain the cooling jacket on the TSG. Binder was fed in using a Graseby 2100 Syringe Pump (Burtons, UK). A screw speed of 30 rpm and L/S of 0.04 were the optimum values for yield before which jamming of the screws or caking over at the liquid inlet port became an issue. A barrel temperature of 10 °C was used to mitigate the heat generation within the unit. A conveying element only screw configuration was used as more aggressive mixing elements led to caking and screw jamming.

2.2.5. *Dryer*

Granules produced from the TSG and HSG were collected and drying was carried out at 50℃ in a Glatt GPCG3 Fluidised Bed (Glatt, Germany) with the target being to reduce the final granule moisture content to below 2.5%.

2.2.6. *Power measurement*

The power consumption of the equipment was measured using Socomec Countis E27 3 Phase Power Reader (NewFound Energy, UK) or DecDeal 1 Phase Power Reader (DecDeal, UK). The power measurement technique is visualized in Fig. 1.

2.2.7. *Granule size characterisation*

The desired granule size was classed as being between 0.5 mm and 2 mm. Sieving using a Retsch Sieve Shaker AS200 (Retsch, UK) at an amplitude of 0.45 mm for 3 mins was used to classify the granulation products. The sieve classes used were 0.5 mm, 1 mm, 2 mm and 4 mm. Granules less than 0.5 mm were classed as fines. Those between 0.5 mm and 2 mm were classified as within specification and to be the yield faction. Granules between 2 mm and 4 mm were classified as oversized and those greater than 4 mm were classed as lumps.

2.2.8. *Granule flowability characterisation*

A Shear Cell RST – XS.s (Dietmar-Schulze, Germany) was used to classify the flowability of granules between 1 mm and 2 mm. Three repeats were conducted for each experiment and the average taken. The normal load at preshear was 2000 Pa and the normal loads at shear to failure were 400 Pa, 1000 Pa, 1600 Pa and 400 Pa.

2.2.9. *Granule dissolution characterisation*

Dissolution testing was carried out using a Jenway 4520 Conductivity Meter (Jenway, UK). 4.5 g of granules between 1 mm and 2 mm was dissolved in 200 ml of distilled water at 25℃. Agitation was achieved using a magnetic stirrer at 700 rpm. The procedure was repeated a total of 5 times for each experiment and the average taken.

2.2.10. *Granule friability characterisation*

Friability or granule strength was assessed by evaluating the fine faction after vigorous shaking using a Retsch Sieve Shaker AS200 (Retsch, UK). 100 g of granules between 1 mm and 2 mm, was placed onto a 1 mm sieve and vibrated at a 1.5 mm amplitude for 2 mins. The friability was then calculated to be the reduced faction (mass less than 1 mm) of the product as a percentage. The experiment was repeated 3 times for each condition and an average taken.

2.2.11. *Granule sphericity characterisation*

A Camsizer (Retsch, UK) was used to evaluate the sphericity of granules between 1 - 2 mm. Random sampling was used to select granule samples of 25 g and three repeats were conducted for each condition. Sphericity is defined as $4\pi A/P^2$ (ISO 9276–6) where A is the measured particle projection area and P is the measured particle projection circumference. For an ideal sphere, the sphericity is expected to be 1.

2.2.12. *Granule porosity characterisation*

X-Ray Diffraction using a X-Ray Scanner μCT 35 (Scanco Medical, Switzerland) was used to obtain 2D imaged slices of granules between 1 mm – 2 mm. ImageJ was then used to evaluate the percentage of voids within the granular structure. For each condition, a total of 10 randomly

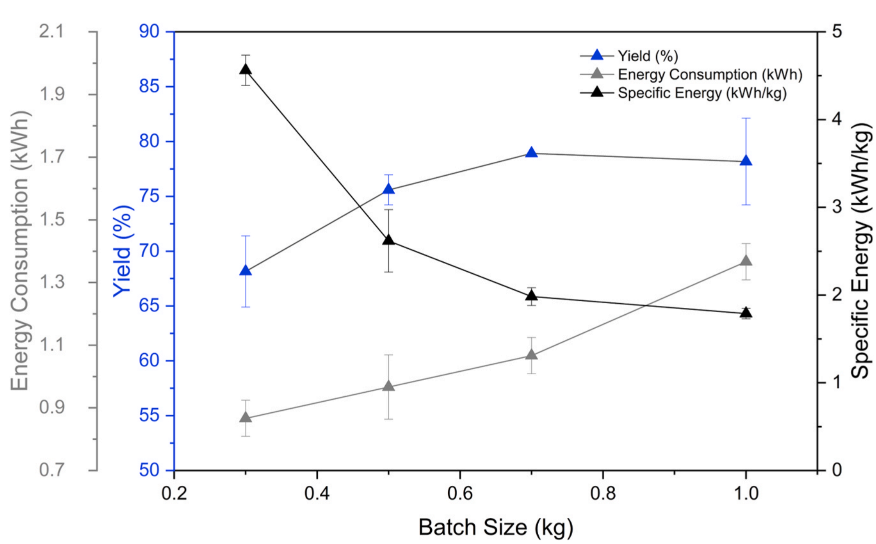

Fig. 2. Impact of Batch Size on Energy Consumption, Yield and Specific Energy in the FBG. The energy efficiency of the process was found to improve with batch size.

selected granules were evaluated.

3. Sustainability in granulation

The following section will present the energy efficiency results for each individual granulator, discussing the effect that batch size/ throughput had on the specific energy of the process. Then the Yield, Specific Energy (kWh/kg) and Specific Time (Hours/kg) of the production pathways as a whole will also be presented to allow the comparison between the technologies to determine the most sustainable.

3.1. Fluidised Bed Granulator (FBG)

The yield of in-spec granules increases with batch size before reaching a plateauing point as shown by Fig. 2. This increase in yield can be explained by considering the increased particle-droplet and particle-particle interactions occurring at larger batch sizes. This increases binder droplet capture by the powder and the increased particle-particle interactions promotes coalescence and leads to better growth (Tan et al., 2006). Similar findings were presented by Geng et al. (2023) which showed that increasing the proportion of powder in the bed promoted powder to powder adhesion by increasing the collision probability. However, while increasing the collision probability also promotes growth, it also results in higher attrition rates of dried granules due to impact and higher breakage rates in wet granules if its yield strength is not sufficient to resist the collision force (Iveson et al., 2001). This higher attrition and breakage rates could detract from the improved growth factors at high batch sizes resulting in the observed plateauing effect. It is also possible that there is a critical particle concentration within the FBG required for good growth to occur which is achieved at a batch size of around 0.7 kg. Increasing particle concentration beyond this point might only result in marginal improvements in granular yield causing the plateauing effect. The smallest and largest batch sizes display the greatest variation in yield. This is due to greater instability in these processes with more caking seen at a batch size of 0.3 kg and fluidization problems observed at a batch size of 1 kg.

The energy consumption of the process meanwhile increases with batch size. As shown in Section 2, both the overall batch run time and the fluidising air velocity needed to maintain bed fluidisation increased with batch size. The higher energy requirement at larger batch sizes can therefore be linked to this additional run time, as well as the need to generate a larger heated air flow within the unit. Despite this increase in the energy consumption, the energy efficiency of the FBG was found to improve with batch sizes before reaching a plateauing value. This is reflected in Fig. 2 where the specific energy for the process decreased as batch size was increased. This decrease is due to the increased yield and material throughput of the process at higher batch sizes. This compensates for the increased energy consumption to result in a more energy economical process.

3.2. High Shear Granulator (HSG)

The energy consumed by the HSG is a very important parameter to be aware of when granulating amorphous food powders. This is because the energy consumed is almost completely converted to heat in the wet mass, which can then become problematic when granulating heat sensitive material (Kristensen and Schaefer, 1987). As shown in Fig. 3, yield of the in-spec granules first increased as batch size is increased from 0.2 kg to 0.3 kg and then decreased as batch size is further increased from 0.3 kg to 0.4 kg. This same trend is shown by Terashita et al. (2002) who showed that this peak where the maximum granule yield occurs is linked to the fill level that corresponds to the best particle circulation and greatest particle kinetic energy.The initial increase in yield with batch size can also be explained by considering the higher collision tendency at higher batch sizes promoting growth through coalescences. The higher collision tendency also increases the rate at which granules consolidate which forces liquid to the granule surface which will promote better growth (Iveson and Litster, 1998). However, it must be noted that this increase in yield is marginal, especially when the magnitude of the error bars is considered. The reduced yield at a batch size of 400 g meanwhile can be attributed to the high collision tendency promoting breakage over growth in this case. This increased breakage along with the less uniform binder distribution at high fill levels result in the lower yield seen here (Mangwandi et al., 2011). The Eirich granulator shares many similarities with planetary centrifugal granulators where the mixing motion is primarily induced by the rotation of the bowl. Studies in planetary centrifugal granulators have linked a decreased yield at higher fill levels to greater restriction of material movement occurring at high fill levels resulting in less agglomeration (Miyazaki et al., 2022). This reasoning is likely to apply to the Eirich granulator as well, indicating that there is an optimum fill level for mixing in the unit resulting in an optimum granulation condition.

Energy consumption increased with batch size. This is due to three factors, the first is the increased overall run time as a larger batch size required a longer binder addition time (since both L/S and binder addition rate were kept constant). The second is the greater load on the

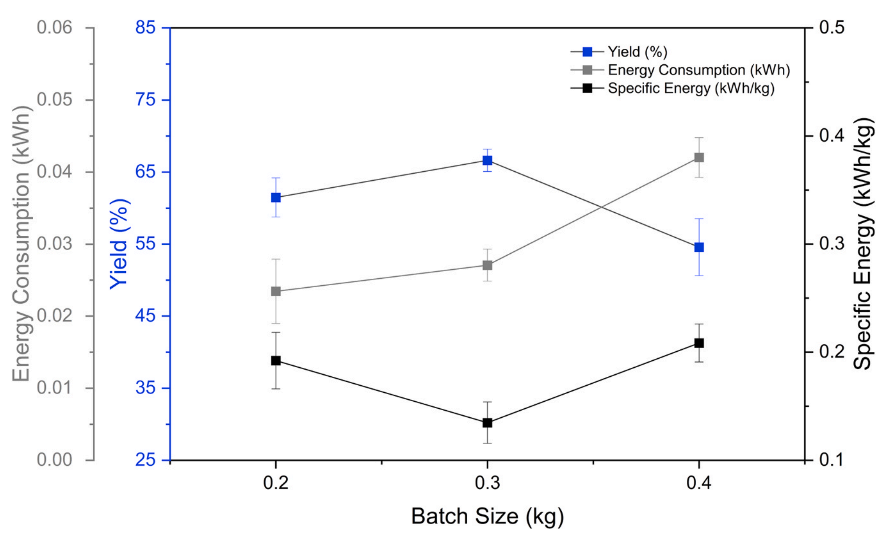

Fig. 3. Impact of Batch Size on Power Consumption, Yield and Specific Energy in the HSG. No specific trend between energy efficiency and batch size is observed here, although, an optimum point for yield and energy efficiency is observed at a batch size of 0.3 kg.

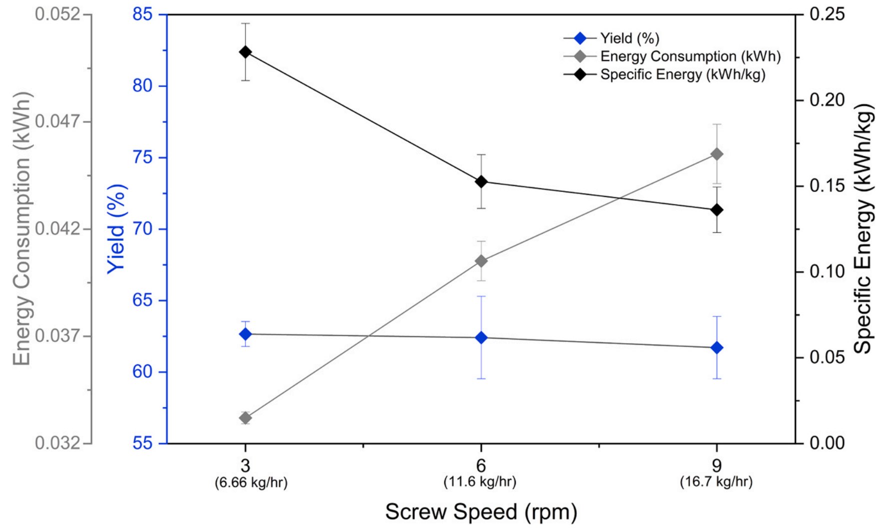

Fig. 4. Impact of Roll Speed on Power Consumption, Yield and Specific Energy in the RC. Energy efficiency improves with increasing roll speed due to the increased throughput at this condition.

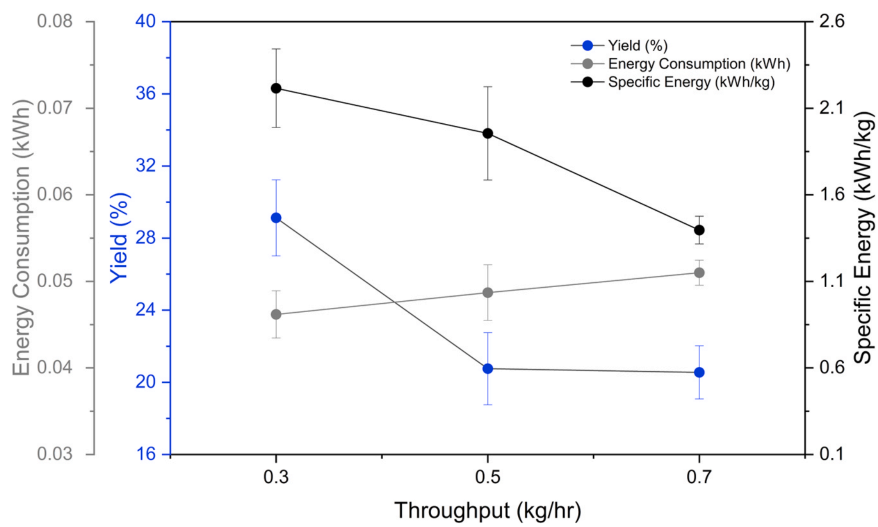

Fig. 5. Impact of Throughput on Power Consumption, Yield and Specific Energy in the TSG. Energy efficiency improves with increasing throughput as indicated by the decreasing specific energy.

bowl motor, as it needs to support a greater mass as it rotates, and the third is the increased load on the impeller motor which must maintain an impeller speed through a much larger bed mass. This is in agreement with the work of Betz et al. (2004) which showed that the energy consumption in the HSG increased with filling level in the granulator. Overall, it is hard to conclude what effect batch size has on the specific energy with there being no clear trend.

3.3. Roller Compactor (RC)

The Roller Compactor was run with the Roller Gap control function activated. This meant the unit would automatically alter the powder feeder screw speed to keep the roll gap constant. At faster roll speeds, more material is required to maintain the roll gap which means the process throughput is higher. This increase in throughput is considerable. For instance, the following average throughputs were recorded at the evaluated roll speeds: 6.66 kg/hr (3 rpm), 11.6 kg/hr (6 rpm) and 16.7 kg/hr (9 rpm).

There is a negligible change in yield with changing roller speed as shown in Fig. 4. This trend can be explained by considering the work of Osborne (2013) who granulated Maltodextrin IT21 under varying roll speeds in the same Roller Compactor as this study. Similarly, to this study, the roller gap control function was used to maintain a constant gap. Osborne (2013) showed that roll speed generally did not have an impact on the ribbon strength and therefore extent of bonding when roll gap is controlled to be constant. A minimal change in bonding with increasing roller speed therefore translates to the minimal change in granular yield seen here.

Energy consumption increased with roller speed. This is because as roller speed is increased, the demand on the roll motors increases as well. The power requirement of the powder screw feeder motor also increases as it needs to produce a greater throughput to maintain the 2 mm ribbon gap at higher roll speeds. This larger throughput along with the relatively stable yield means the overall mass of useful granules produced increases significantly as roll speed is increased. Therefore, despite the higher energy consumption, the specific energy decreases with increasing roll speed to result in a more energy efficient process.

3.4. Twin Screw Granulator (TSG)

The in-spec granule yield for the TSG process is low compared to the other granulation techniques with the highest yield being 29% at the 0.3 kg/hr condition as shown in Fig. 5. This reflects the idea that agglomeration in the granulator is not effective since the screw is only composed of conveying elements which are primarily used to transport material and not for mixing (Bandari et al., 2020). This resulted in a very high fine faction for all conditions (64%, 65% and 60% for 0.3 kg/hr, 0.5 kg/hr and 0.7 kg/hr respectively). The use of more aggressive mixing elements resulted in powder caking, which leads to screw jamming and therefore could not be utilized with this material.

The highest in-spec yield is recorded at the lowest throughput of 0.3 kg/hr. This is because at this throughput, the granules that are produced are predominantly within the desired size range (0.5 mm – 2 mm). At larger throughputs, a large portion of the granules produced are larger than the 2 mm upper limit. This means that they are classed as oversized which leads to a lower in-spec granular yield. This can be explained by considering two phenomena. The first is the increased residence time at low powder feed rates as the throughput force is low (Dhenge et al., 2011). This increased residence time facilitates better binder distribution and more incorporation of fine powder which leads to the preferred granule size distribution (Lute, 2018; Kumar et al., 2016). The second is the increased compaction forces at high throughputs leading to more interactions between granules and ungranulated material (Dhenge et al., 2011). This leads to the production of larger granules (which are out of spec) as throughput increases (Yu et al., 2014; Djuric et al., 2009).

The energy consumption of the TSG increases with throughput. At larger throughputs, the material offers more resistance to the motion of the screws meaning that more energy is required by the screw motor to overcome this resistive force. Meanwhile, the specific energy decreases with increasing throughput indicating a more energy favorable condition. This is despite energy consumption increasing and yield decreasing with increasing throughput. Similarly, to the RC, this is because the actual mass of useful granules produced is larger at higher throughputs despite the decrease in yield. This offsets the higher energy consumption to produce a more energy efficient process.

3.5. Comparing granulation pathways

When considering the affordability and sustainability of a manufacturing process there are multiple metrics they can be judged by. Examples of these metrics include Yield, Specific Energy and Specific Time.

3.5.1. Material Efficiency (Yield)

Yield efficiency indicates the amount of material wasted. A lower yield indicates more wastage. Linking this to the cost of the raw materials, a lower yield therefore correlates to a more expensive product in the case that the fines cannot be recycled. In the case that they can be recycled, this leads to a longer processing time which reduces the sustainability and viability of the process. As mentioned in Section 2, the technologies presented in this study were optimized based on yield to reflect its importance in deciding process economic viability. Fig. 6 shows the change in yield between the different processes. The process yield operating at optimized conditions was found to increase in the following order: Twin Screw Granulator, Roller Compactor, High Shear Granulator and Fluidised Bed Granulator. The yield of the TSG process is far below the rest which would result in a large amount of material wastage if it were adopted. The FBG meanwhile had the highest yield between the technologies at a value of 79% indicating a very material efficient process.

3.5.2. Energy Efficiency (Specific Energy)

Specific Energy is the second metric for evaluating process viability.

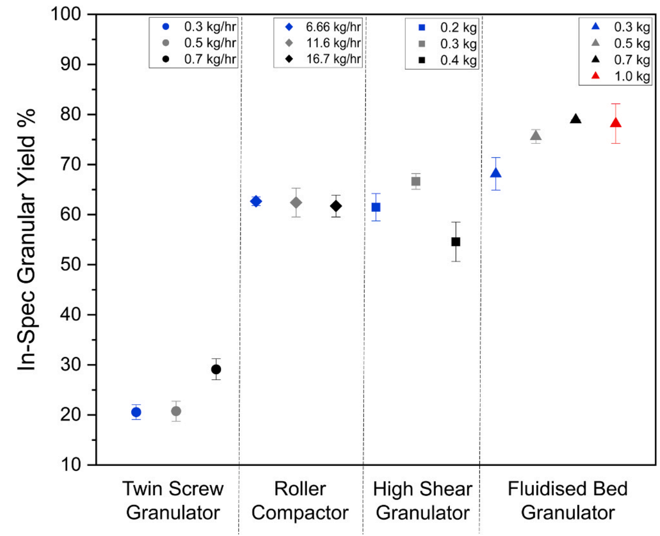

Fig. 6. Comparing yield of the different granulation pathways. The Twin Screw Granulator is represented by circles with the indicated throughput conditions. The Fluidised Bed Granulator is represented by triangles with the indicated batch size conditions. The High Shear Granulator is represented by the squares with the indicated batch size conditions. The Roller Compactor is represented by the diamonds with the indicated throughput conditions.

Section 3 so far has only considered the energy consumption of producing granules, between 0.5 mm and 2 mm, within the granulators units themselves. However, to fairly compare different technologies, the energy consumption of the entire granulation pathway going from dry powder to dry granule must be considered. This is because not all the granulators produce the same end product, with the Roller Compactor and Fluidised Bed Granulator producing dry granules, while the High Shear Granulator and Twin Screw Granulator produce wet granules that require an additional drying stage. Fig. 7 shows the specific energies of the entire granulation pathway for the different technologies compared against each other. As evidenced by the decreasing specific energy, the

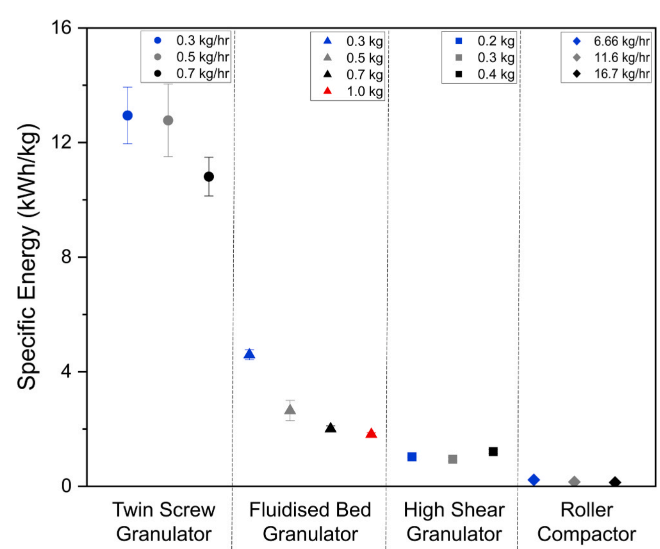

Fig. 7. Comparing energy consumption of the different granulation pathways. The Twin Screw Granulator is represented by circles with the indicated throughput conditions. The Fluidised Bed Granulator is represented by triangles with the indicated batch size conditions. The High Shear Granulator is represented by the squares with the indicated batch size conditions. The Roller Compactor is represented by the diamonds with the indicated throughput conditions.

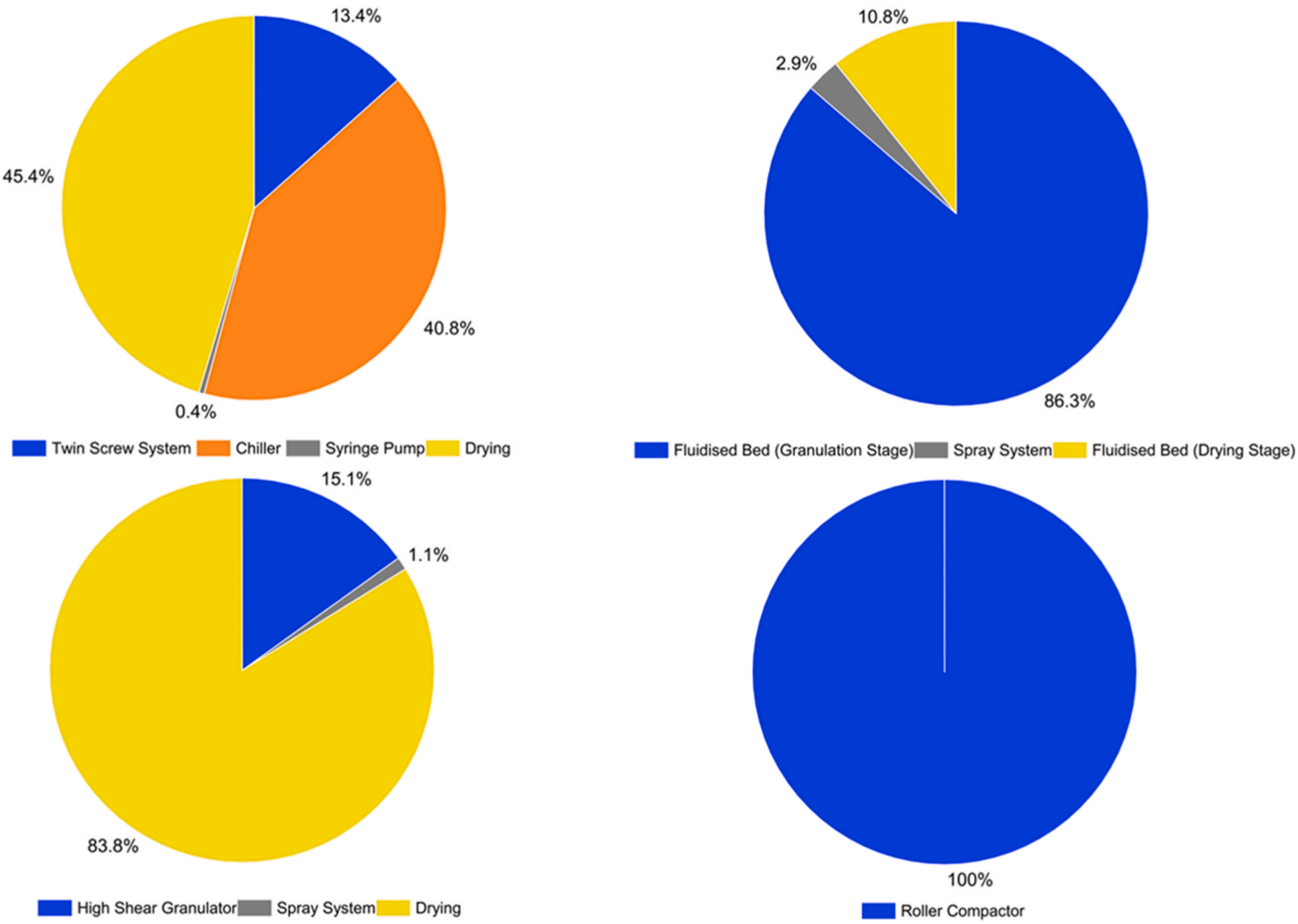

Fig. 8. Energy consumption distribution of the different units involved in each production pathway, represented by the most energy efficient condition for each technology. Top Left: Twin Screw Granulator operating at a throughput of 0.7 kg/hr. Top Right: Fluidised Bed Granulator operating at a 1 kg batch size condition. Bottom Left: High Shear Granulator operating at a 0.3 kg batch size condition. Bottom Right: Roller Compactor operating at a roll speed of 9 rpm (16.7 kg/hr)

energy economy of the pathway improves in the following order: Twin Screw Granulator, Fluidized Bed Granulator, High Shear Granulator and Roller Compactor.

The least efficient technology by a considerable margin is the TSG. This can be attributed to two things, the first is the low yield of the unit which meant that the mass of in-spec granules produced was very low. The second is the energy expensive sub stages that are involved in the Twin Screw Granulation process. Fig. 8 shows the energy consumption distribution for the most energy efficient condition for each technology. In the case of the TSG this is at a throughput of 0.7 kg/hr. It is evident that the actual granulation process requires a minimal amount of energy (13%) compared to the drying stage (45%) and the energy drawn by the chiller (41%). The chiller is necessary to counteract the buildup of frictional heat and keep the barrel temperature low. Failing to do so means that eventually the barrel temperature increases to the point where caking occurs leading to the jamming of the screws. The need to include such an energy expensive unit in the granulation process along with the low yield results in a far more uneconomical process pathway compared to the other technologies. The advantage of the energy distribution analysis shown in Fig. 8 is that the energy expensive stages can be easily identified so manufacturers are aware of what stages require more optimization when making the overall pathway competitive from an energy efficiency perspective. For the Twin Screw Granulator, it seems that the cooling process and the drying stage need the most attention when it comes to optimizing each to be as energy efficient as possible. Alternatively, a way must be found to improve the yield so that it can compensate for the energy intensive nature of the process.

The second most energy inefficient process was determined to be the Fluidised Bed Granulator. This is due to the large amounts of energy required to generate and maintain a heated air flow. Despite being very energy intensive, it produced the highest yield of all the evaluated technologies. This high yield compensated for its high energy usage and made it far more energy efficient than the TSG and meant its specific energy was more in line with the HSG and RC.

The HSG ranked third in terms of energy economy. Similarly, to the TSG, it was found that the energy consumption by the granulator is in the minority when the production pathway is looked at as a whole. This is because the drying stage encompasses the vast majority of the energy cost of the production pathway. For instance, at the 0.3 kg batch size condition the drying step accounted for 84% of the total energy consumed as shown in Fig. 8. It is this drying stage that manufacturers should focus on if the aim is to produce a more energy efficient HSG pathway.

Finally, Roller Compaction was found to be the most energy efficient production pathway. As displayed in Fig. 8, only one unit – the Roller Compactor, was involved in the transition from dry powder to dry granule. This makes it a rather simple pathway requiring less stages which translates to less energy. For instance, since it is a form of dry granulation it means that the energy cost associated with spraying the liquid and the subsequent high energy cost associated with drying the product wet granules are entirely avoided. This is a luxury that the wet granulation pathways do not have, making them far more energy expensive than the Roller Compactor.

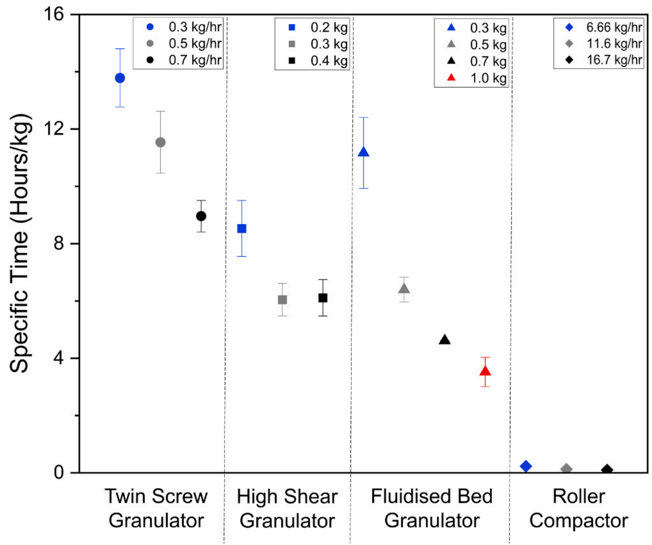

Fig. 9. Comparing production rates of the different granulation pathways in terms of specific time. The Twin Screw Granulator is represented by circles with the indicated throughput conditions. The Fluidised Bed Granulator is represented by triangles with the indicated batch size conditions. The High Shear Granulator is represented by the squares with the indicated batch size conditions. The Roller Compactor is represented by the diamonds with the indicated throughput conditions.

3.5.3. *Time Efficiency (Specific Time)*

The final metric that competing technologies can be judged by is the rate at which in-spec granules are produced. This has been classified as the specific time which is the hours required to produce a kilogram of in-spec granules. A lower specific time means facilities need to stay active for shorter durations of time which reduces energy and labour costs leading to a more affordable and sustainable product. The total run time of the granulation pathway to produce dry granules was accounted for in the total time. Also included with the batch technologies (HSG and FBG) was the time required to clean and reset the unit so that a new run could be begun. This was done to help highlight the differences between batch and continuous manufacturing technologies with regards to time. For the High Shear Granulator, the cleaning and reset time was 45 min. Whereas for the Fluidised Bed Granulator, this was 2 h. Cleaning time was not included for the continuous processes such as the TSG and RC. This is because these units are capable of running indefinitely as long as the process conditions chosen prevent significant material caking in the unit. Therefore, operating at optimum conditions, their cleaning time compared to their overall run time is negligible or many multitudes smaller than the batch technologies. For this reason, the cleaning time was not included when calculating the Specific Time for the TSG and RC. Once again, the Roller Compactor proved to be the most efficient technology based on Specific Time as shown by Fig. 9. This is due to its continuous nature, large throughput and elimination of the drying step leading to a reduction in overall operating time. Choosing the time-optimum condition, the wet granulation technologies can be ranked with the TSG being the least efficient, followed by the HSG and finally the FBG proving to be the most efficient. Whereas previously when considering Yield and Specific Energy, the TSG had been a major outlier, here it proved to be more competitive with the other technologies. This is due to the continuous nature of the process, meaning significant savings in the cleaning time, which compensates for the low throughput and low yield nature of the process.

4. Granule suitability

When choosing the optimum granulation technology, the efficiency of the process with regards to yield, energy and time are only some of the considerations that manufacturers have to make. Other considerations include the resultant granule characteristics and whether those characteristics fit with the ultimate product specification. This is an important consideration because the different technologies produce vastly different granules. These differences are highlighted by Fig. 10 which show X Ray scans of granules collected from the different technologies at the optimized conditions. The clearest difference is in granule porosity, with granule porosity varying significantly between the technologies. The High Shear Granulator and the Roller Compactor had the lowest porosities that were similar to each other. Porosity then increased going from the Twin Screw Granulator to the Fluidized Bed Granulation. These differences arise due to the varying extents of consolidation that the granules experience in the different granulators. Maltodextrin granules undergo the greatest densification and consolidation in the High Shear Granulator under the conditions evaluated in this paper. This is because the tightly packed granules are accelerated to high velocities in the HSG where they experience a high intensity of collisions. These high energy collisions squeeze liquid to the surface and consolidate the granules producing a product that has a very low porosity. This is especially true for the parameters chosen for this HSG process where the liquid was added rather slowly which resulted in an extended run time that provided ample time for consolidation to occur. Furthermore, the use of water binder also results in a granule with a lower porosity. This is because the water dissolves the Maltodextrin powder which then flows into the intergranular voids. During drying the water evaporates, leaving behind solidified Maltodextrin where the voids once were, which reduces the granule porosity. The low porosity nature of HSG granules is well documented in literature (Järvinen et al., 2015; Morin and Briens, 2014). The Roller Compactor also produced granules with a similar porosity to the HSG. This is because the basis for the bonding within the Roller Compactor involves minimizing the distance between particles as much as possible to increase the number of contact points and maximize the Van der Waals forces of attraction. TSG produced the next most porous granules with the granules undergoing densification as they are compressed between the narrow channel width between the screws and barrel. However, the granule porosity recorded by the TSG is much higher than that typically seen from the technology. This can be

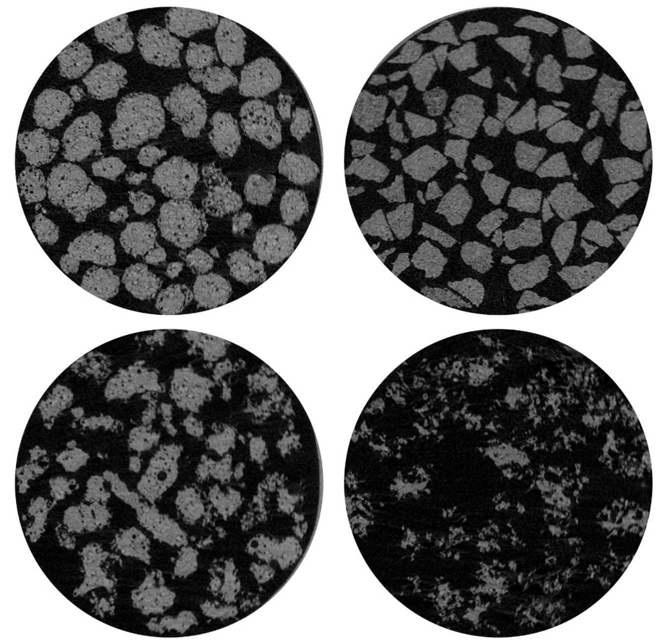

Fig. 10. X-Ray Scans of granules from the 1 - 2 mm size class shows significant variations in shape and porosity between the granulators. Conditions chosen represent the most energy efficient conditions. Top Left (HSG 0.3 kg), Top Right (RC 9 rpm), Bottom Left (TSG 0.7 kg/hr), Bottom Right (FBG 1 kg).

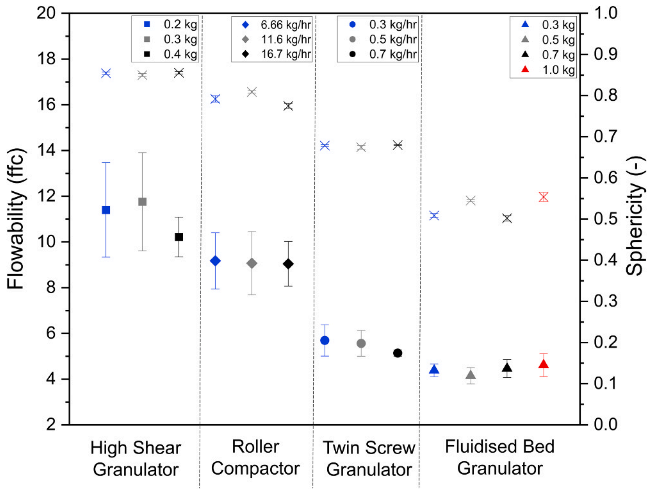

Fig. 11. Comparing the flowability of granules produced by the different technologies. Flowability is represented by the square (HSG), diamond (RC), circle (TSG) and triangle (FBG) symbols respectively. Granule sphericity is represented by the X signs. Flowability increased with increasing granule sphericity.

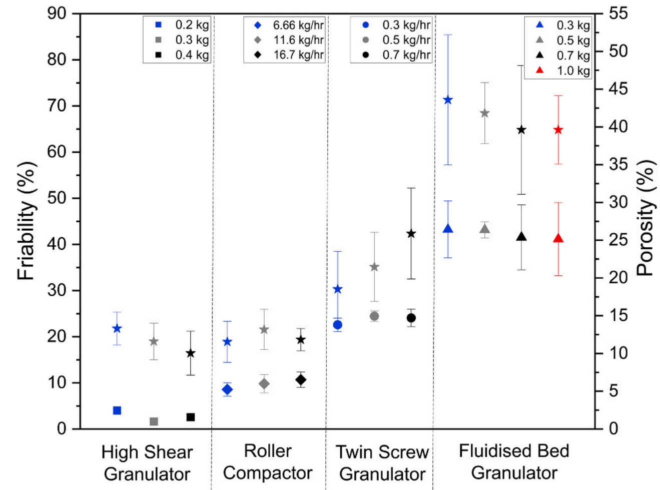

Fig. 12. Comparing the friability of granules produced by the different technologies. Flowability is represented by the square (HSG), diamond (RC), circle (TSG) and triangle (FBG) symbols respectively. Granule porosity is represented by the star signs. Friability was found to generally increase inline with granule porosity

attributed to the lack of kneading elements used in this work which is where densification predominantly occurs in the unit. Finally, the FBG produced the most porous granules by far which is a common finding in literature (Järvinen et al., 2015; Morin and Briens, 2014). This is due to the lack of consolidation and densification that occurs within the unit as there are little to no compaction forces acting within the granulator.

These differences in granule shape and porosity will significantly effect the product granule attributes. In order to quantify the differences between the granules produced via the different manufacturing routes; Granule Flowability, Granulate Friability and Dissolution Time were all evaluated. These are key attributes which will critically affect further manufacturing steps and influence the perceived quality of the product by the customer. Please note that granule attributes can vary significantly within each technology depending on the operating conditions used to produce them. For example, the use of a higher compaction pressure has been shown to significantly reduce product porosity during a roller compaction process (Osborne et al., 2013; Al-Asady et al., 2015). The values presented here are purely for the optimised operating conditions presented in Section 2.2 and are presented to highlight the variety in granule properties that arise depending on manufacturing route taken and process conditions used.

4.1. *Granule flowability*

The flowability of the granules developed by the granulation technologies follows the sphericity of the granules as represented by Fig. 11. The HSG produced the most spherical and best flowing granules which are classed as free flowing as they have a ffc above 10. This is followed by the RC, then the TSG and finally the FBG. All three technologies produced granules which are classed as easy flowing which is classified as granules with an ffc between 4 and 10. Whereas the RC and TSG are at the upper limit of this range, the FBG is at the lower limit of this range and is borderline cohesive. The superior flowability and sphericity of HSG granules compared to other technologies is well documented in literature (Ji et al., 2017; Beer et al., 2014; Megarry et al., 2020; Lee et al., 2013).

The flowability of the bulk IT29 powder was measured at a ffc of 7.6. It is interesting therefore, that despite particle size increasing in all cases, only the HSG and RC led to an increase in flowability. The reason the TSG and FBG led to a worsened particle flowability can be attributed to its aspherical shape. This means that during motion, granules tend to interlock together to resist motion. This interlocking phenomenon is

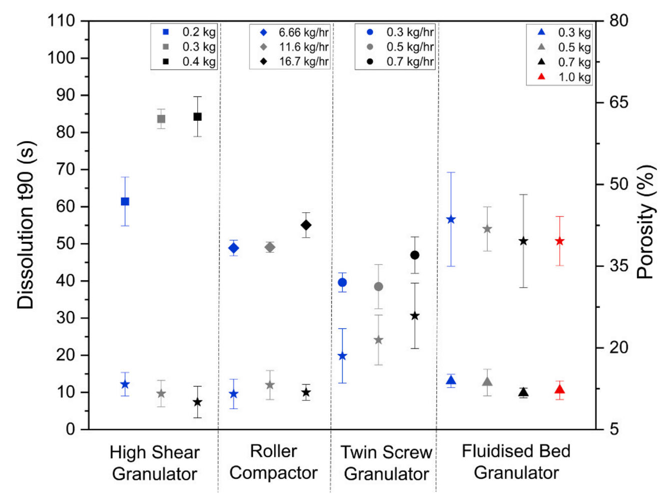

Fig. 13. Comparing the dissolution time of granules produced by the different technologies. Dissolution time is represented by the square (HSG), diamond (RC), circle (TSG) and triangle (FBG) symbols respectively. Granule porosity is represented by the star signs. Dissolution time was found to be inversely related to granule porosity.

part of the reason tablets produced from TSG granules have a relatively strong tensile strength compared to other technologies (Miyazaki et al., 2020). The FBG granules are also much weaker than those produced by the HSG and RC. This means that when pressure is applied to them during the operation of the shear cell, they are more likely to break. This produces granule fragments which further increases the likely hood of interlocking occurring in the sample, leading to a lower flowability.

4.2. *Granule friability*

The friability of the granules produced by the different technologies, which are presented in Fig. 12, seem to be inline with the granule porosity observations made when looking at the X Ray scans. This is because there is a direct link between granule porosity and granule strength. The lower the granule porosity, the closer the particles are to each other and the more contact points there are between them. This increases the attractive Van der Waals forces in the granules.

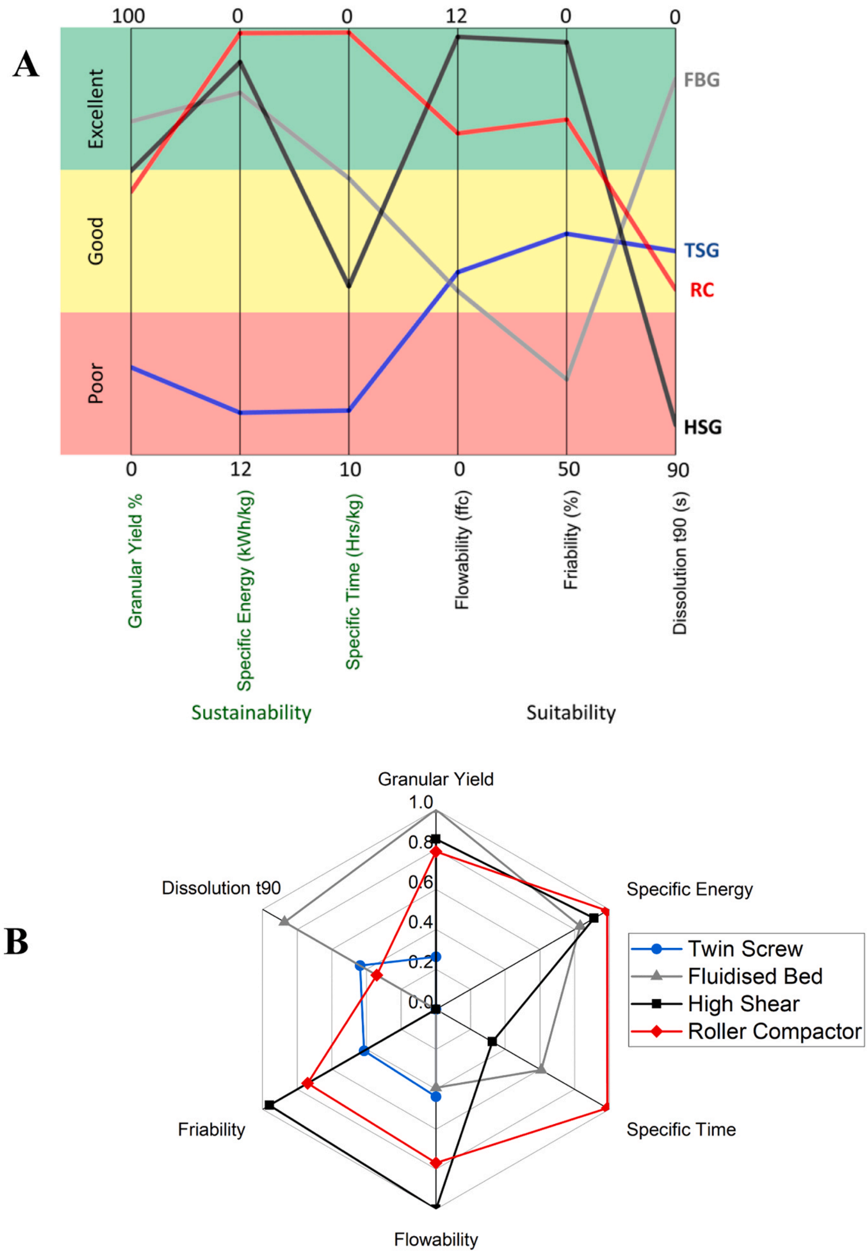

Fig. 14. Ways to visually represent process sustainability and suitability. The most energy efficient condition for each granulator was chosen to represent each technology as follows: Fluidised Bed Granulator (1 kg), High Shear Granulator (0.3 kg), Roller Compactor (16.7 kg/hr) and Twin Screw Granulator (0.7 kg/hr). **14A:** Parallel co-ordinate graph which categorises the performance of each considered aspect of the technology into poor, good or excellent. For the production of a strong, quickly dissolving granular product, a high friability and long dissolution time would be classed as undesirable traits leading to a poor performance. Values for the sustainability of the chosen conditions presented in Figs. 6,7 and 9 are paired with values for granule attributes shown in Figs. 11–13. The axis for each parameter is decided by whether a high value in that attribute is considered positive or negative. For instance, the higher the granule yield, the more attractive the process. Therefore, a yield of 0% indicates a poor process performance while a yield of 100% indicates an excellent process performance. On the other hand, when considering specific energy, a higher specific energy indicates a less attractive process. Therefore, a Specific Energy close to 0 kWh/kg indicates an excellent process while a high specific energy indicated by 12 kWh/kg indicates a poor process. This same principle was applied to all the attributes evaluated in this paper. The values for each parameter for the different technologies was then plotted on the graph. **14B:** Radar chart which presents the performance of each granulator in the different evaluated attributes ranked from 0 to 1. The higher the value, the more desirable the performance of the technology in that trait. A value of 1 would indicate the most desirable and a value of 0, the least desirable. The ranking was carried out by dividing the experimental data values for each parameter by the highest value of that attribute. Looking at Yield for example, the Yield of each chosen granulator condition is as follows: FBG (1 kg) – 78%, HSG (0.3 kg) – 66%, RC (16.7 kg/hr) – 62% and TSG (0.7 kg/hr) – 29%. Dividing each value by the highest yield (78%), gives a rating of 1 for the FBG, 0.85 for the HSG, 0.79 for the RC and 0.37 for the TSG which is shown on Fig. 14b. This procedure is repeated for every attribute in a similar manner. In certain cases, however, a high value is undesirable, for instance when looking at Specific Energy and Specific Time. Once again considering the development of a strong and quickly dissolving granular product, a high friability and long dissolution time would also be undesirable. In these cases, the produced ranking for these attributes are subtracted from a value of 1. For example, the TSG had the highest Specific Time of 9 hrs/kg while the RC had the lowest at 0.1 hrs/kg. Dividing these values by the highest Specific time then results in the TSG have a ranking of 1 while the RC has a ranking of 0.01 which does not reflect the idea that a ranking closer to 1 is more desirable. It is for this reason that these values are then subtracted from 1 to account for this. Subtracting by 1 gives a ranking for the TSG of 0 while giving a ranking for the RC of 0.99 as shown in Fig. 14b. In this manner the performance of each technology in a given aspect can be easily compared with each other. Overall technology performance can also be evaluated by looking at the area encompassed by the plot. The bigger the area, the better the performance.

Furthermore, it also leads to more mechanical strength due to more mechanical interlocking within the granule and the increased likelihood of solid bridges between the Maltodextrin particles. This means granule friability increases in the following order: High Shear Granulator, Roller Compaction, Twin Screw and Fluidized Bed Granulation, which is in line with that presented in literature (Arndt et al., 2018) (Fig. 12).

4.3. Granule dissolution

The dissolution time of the granules produced by the different technologies is inversely related to the porosity of their respective granules as shown by Fig. 13 (Le et al., 2011; Ansari and Stepanek, 2008). This is because the denser the granules, the slower the penetration time of the liquid into the granules and the longer the disintegration time. Dissolution time decreases in the following order: High Shear Granulator, Roller Compaction, Twin Screw and Fluidized Bed Granulation.

5. Choosing the optimum granulation pathway

As discussed in this study, there are a lot of considerations that should be accounted for when deciding the optimum process route. These include those considerations looking at the efficiency and sustainability of the process with regards to material, energy and time. As important are how suitable the product granule is to its purpose which depends on the granule characteristics such as flowability, friability and dissolution time. Methods to help account for all of these parameters together in a logical and sensible way are critical to the development of a coherent decision making system. One possible approach is the use of evaluating the technologies graphically as shown in Fig. 14 through the use of parallel co-ordinate graphs or radar graphs (Leane et al., 2015). For example, the parallel co-ordinate graph can be used to quickly and easily eliminate unsuitable processes from the decision making process.

Table 1
Priority based score ratings for each technology. In this method, the user assigns weightings for each parameter based on their priority. The higher the priority, the higher the weighting given to it. For instance, in the below example, the Specific Energy was the users highest concern so it was assigned the highest priority weighting of 0.3. Yield followed as the second highest concern for the user and was assigned the second highest weighting of 0.25. On the other end of the scale, the Specific Time was not a significant consideration for the user and so was assigned the lowest weighting of 0.05. To summarise, the priority weightings are essentially how important the user views those attributes as being based on their needs. These priority weightings are then multiplied by the technology coefficients for each parameter (Parameter Rating = Priority Weighting × Technology Coefficient). Coefficients can be produced by normalising the experimental data gathered for each parameter which are presented in Figs. 6,7,9 and 11–13. Negative signs are used when higher technology coefficient values are undesirable. For a strong and quickly dissolving product; a high Specific Energy, Specific Time, Friability and Dissolution Time are undesirable and therefore have been assigned negative signs. Please note, the example coefficient's given are for illustrative purposes and do not relate to any particular technology. Summing the parameter ratings then gives a total rating for that process. The most suitable process for a set of priority weightings can then be determined by comparing the total process ratings between different processes and seeing which has the most positive result.

Parameter	Priority Weighting	Technology Coefficient	Parameter Rating
Material Efficiency (Yield)	0.25	0.780	0.195
Specific Energy	0.3	-0.629	-0.189
Specific Time	0.05	-0.558	-0.028
Flowability	0.2	0.879	0.176
Friability	0.1	-0.486	-0.049
Dissolution	0.1	-0.470	-0.047
Process Rating Total			0.059

For instance , it is evident that the Twin Screw is predominantly in the poor region across the board and is therefore highly inappropriate as a general process option in this case. Another example scenario could involve the development of a process where very strong granules are required. Looking at the parallel coordinate graph, it's clear that the FBG is the only one present within the poor region for granule friability and can therefore be discounted from further evaluation. An alternative approach is to calculate a score for each process based on the priority the manufacturer assigns to each parameter based on their needs. An example and method outline is presented in Table 1. This quantification of desirability is a methodical and rational way of differencing between processes based on specific requirements.

6. Conclusion

While multi-granulator studies do exist they are often limited in scale, only looking at a small number of granulators, and narrow in scope, often choosing to focus primarily on granule attributes. This study has evaluated the predominant granulation technologies in a comprehensive and thorough manner. The sustainability of the process has been scrutinized with regards to material, energy and time. Also looked at was the suitability of the process to produce granules that are fit for purpose. Results showed that the granulator performance varied between the measured attributes with no granulator appearing the clear winner in all cases. The Roller Compactor was found to be the most efficient with regards to energy and time. The Fluidised Bed meanwhile was found to be the most efficient with regards to material usage and produced the best dissolving granules. The High Shear Granulator produced the best flowing and strongest granules. This highlights the difficulty faced by manufacturers when deciding the optimum production pathway in cases where there is no clear winner. It is crucial therefore that manufacturers take a 'big picture' approach to the decision making process and look at all the parameters discussed by this paper as a bare minimum. Both visual and analytical methods have been proposed by this study to help in this. Utilizing them to match sustainable manufacturing technologies to products they are suited for is key in the drive to make the food industry more sustainable and affordable.

List of Symbols

FBG	Fluidised Bed Granulator [-]
HSG	High Shear Granulator [-]
RC	Roller Compactor[-].
TSG	Twin Screw Granulator [-]

Declaration of Competing Interest

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.

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