The Effect of Scale on Spray Nozzle Performance

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Graduate Program in Chemical and Biochemical Engineering  
A thesis submitted in partial fulfillment of the requirements for the degree in Master of Engineering Science  
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ABSTRACT

In the petroleum industry, Fluid Cokers™ are used to upgrade heavy oils into useful lighter products. When oil is injected in their fluidized bed of coke particles, it forms oil-coke agglomerates that slow down endothermic reactions and cause operating problems.

Spray nozzles of different sizes were used to show the effect of nozzle scale on spray characteristics. With open-air experiments, the stability, and angle of the spray during injection were analyzed. Fluidized bed experiments used Gum Arabic as injected liquid to simulate agglomerate formation and match the viscosity of the oil in coker nozzles.

Open air experiments were used to adjust the injection system so that all the studied nozzles provided a stable spray. The main impact of nozzle size on wet agglomerate formation in the fluidized bed was indirect: larger nozzles formed longer jet cavities, so that the liquid was deposited in regions that were more intensely fluidized.

Key Words: Fluid Coking™, Spray Nozzle, Fluidized Bed, Nozzle Scaling
ACKNOWLEDGMENTS

I would first like to thank my advisors, Dr. Cedric Briens and Dr. Franco Berruti, for their endless support and guidance as I progressed through this research program. Their guidance not only lead to the successful completion of my thesis, it supported my growth both academically and professionally. I am very grateful for the opportunity I was given to be involved in this industry related research.

Next, I would like to thank Syncrude Canada Ltd., ExxonMobil, and the National Science and Engineering Research Council of Canada for their financial support and for making my research possible.

I would also like to extend a special thanks to Dr. Jennifer McMillan for her role as an advisor and industrial expert through the development of my research.

ICFAR and the University of Western Ontario has provided me with a vast amount of resources for my Master’s. Francisco Sanchez has been a great postdoc and friend to me, as well as a remarkable person to learn from. The staff at ICFAR have been nothing but kind and helpful of which I would like to thank the efforts of Tom Johnston, Chantal Gloor, and Christine Ramsden. My experiments would never have happened without help of Cody Ruthman and Jake Vlemmix from the University Machine shop. Their talents supported the builds of the equipment used in my experiments. Beyond the support for my research, ICFAR and the University of Western Ontario provided me the opportunity to make many new friends, which I will we remember time and again. My dearest gratitude of those I’ve met goes to Amanda Kuhn for her love and encouragement.

Finally, I would like to thank my parents and all my family for all their love and support. My family has been there for me every step of my life and my masters was no exception. I know they will always be there whenever I need them, and I want to let them know how much I appreciate them.
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Nomenclature

$CV_{PRE}$  Coefficient of variation of Pre-mixer Pressure

$D_C$  Conduit Diameter (mm)

$D_{C/N}$  Conduit over Nozzle Diameter Ratio

$D_N$  Nozzle Diameter (mm)

$D_{P/C}$  Pre-Mixer over Conduit Diameter Ratio

$F_G$  Gas Flux ($g/(mm^2 \cdot s)$)

$F_L$  Liquid Flux ($g/(mm^2 \cdot s)$)

$Fps$  Frames per second

$GLR$  Gas Liquid Ratio (wt%)

$HID$  High Intensity Discharge

$I_{GS}$  Instability Index Gray Sum Method

$I_{PRE}$  Instability Index Pre-Mixer Pressure Method

$L_C$  Conduit Length (m)

$LED$  Light Emitting Diode

$LS$  Length to Max Spray Width (mm)

$M_i$  Mass Injected (g)

$P_{ATO}$  Atomization Pressure (psig)

$P_{BTK}$  Blow Tank Pressure (psig)

$P_{PRE}$  Pre-mixer Pressure (psig)

$Q_{G1}$  Gas Volumetric Flowrate Gas Distributer 1 (kg/s)

$Q_{G2}$  Gas Volumetric Flowrate Gas Distributer 2 (kg/s)
\( t \)  Time (s)

\( T_B \)  Average Bed Temperature (°C)

\( V_{GI} \)  Fluidization gas velocity for injection (m/s)

\( W_S \)  Max Spray Width (mm)

\( \theta \)  Spray Angle (°)

\( \rho_P \)  Bed particle density (kg/m\(^3\))

\( \Delta t_S \)  Spray Injection Time (s)
1. **INTRODUCTION**

The research work presented in this thesis addresses how changing injection nozzle sizes affect agglomerate formation inside a fluidized bed. The stability, Pre-Mixer pressure, and characteristics of the spray during injection were analyzed. To aid in this research, experiments inside a fluidized bed were developed using Gum Arabic, as an injection fluid and binding solution, to simulate agglomerate formation and match the viscosity and performance of heavy oils inside of a Fluidized Coker™ (Reyes & Andrea, 2015). The motivation for this research was to understand the correlation between the nozzle size and the quantity and quality of agglomerates formed. Agglomeration is a problem in Fluid Coker™ units as it reduces the yield of valuable products by increasing mass and heat transfer resistances and influencing the thermal cracking reactions (House, Briens, Berruti, & Chan, 2008). This chapter presents a short introduction to the world energy sector, explaining the movement towards heavy oil exploitation, bitumen, Fluid Coking™, agglomerates, and finally, an overview of the specific objectives of this research are outlined.

### 1.1 **WORLD ENERGY**

Worldwide there are primarily five types of energy consumed. These include, from highest to lowest usage: oil, natural gas, electricity, biofuels and waste, and coal. According to the International Energy Agency (IEA) total energy consumed in 1973 was 4,661 Million Tons of Oil Equivalent (Mtoe) compared to 9,555 Mtoe in 2016, which is more than double the usage over the 43-year span. When comparing the 2016 ratios with 1973, oil remained the top consumed fuel at ratios above 40% (International Energy Agency (IEA), 2018). Figure 1-1 shows the world fuel consumption comparison between 1973 and 2016.
As noted above, oil consumption remained the highest energy type consumed between the years 1973 and 2016. During this time, according to the IEA, (2018), oil consumption increased from 2,252 Mtoe to 3,908 Mtoe. This oil energy group is further tracked and analyzed by sector usage. During this period of time the sector “Road” (transportation) was the largest consuming group. In 1973 the “Road” sector accounted for 30.8% (694 Mtoe) of the consumption, jumping to 49.3% (1,926 Mtoe) of the total consumption in 2016. This large increase in the “Road” sector was offset my downturns in consumption within the “Industry” sector and the “Residential” sector. The “Industry” sector fell from 19.9% (448 Mtoe) to 7.8% (304 Mtoe) while the “Residential” sector fell from 13.4% (302 Mtoe) to 5.4%. (211 Mtoe). In both these sectors the consumption totals, Mtoe, decreased over the 43-year span (IEA, 2018). Figure 1-2 shows the world oil consumption comparison between 1973 and 2016.
As total consumption of oil continues to grow, conventional oil reserves become strained, due largely to the transportation sector using conventional oils, which there are no easy substitutes (Miller & Sorrell, 2014). This depletion of conventional oils is creating a need to improve the techniques and efficiency of oil development in other oil reserve categories known as unconventional oil reserves. Unconventional oil reserves include extra heavy oil, oil shale, tight oil, and oil sands (Miller & Sorrell, 2014). Oil sands, found in Alberta, are currently contributing to the global oil supply and, according to Miller and Sorrell, (2014), are forecasted to expand supply over the next 20 years. Oil sands are described by Miller and Sorrell, (2014), as a “near-surface mixture of sand, water, clay and bitumen”, where the bitumen can be diluted or upgraded to a synthetic crude for transport by pipeline. Although the upgrading of bitumen is not a preferred source, compared to the light oils, which are easier and less expensive to produce, it is a viable alternative.
1.2 BITUMEN UPGRADING

Unconventional oil has attracted attention as a sustainable alternative but with challenges. For example, due to its complex composition and high concentration of heteroatoms, such as nitrogen, sulfur, nickel, and vanadium, unconventional oil cannot be recovered by the well-established processes used for conventional oils (Santos, Loh, Bannwart, & Trevisan, 2014). Unconventional oil is characterized as oil with a gravity smaller than 20 API and a viscosity greater than 100 cP (Li, Yan, & Xiao, 2015). This gives the oil a flow resistant characteristic restricting it from being transported in the pipelines. Consequently, it must be upgraded, which typically involves reducing its viscosity to allow for shipment in the pipelines. Furthermore, processing then allows the unconventional oil to be processed into fuels at existing refineries, which would otherwise be incapable of dealing with the untreated unconventional oil. Upgrading adds processing costs to the unconventional oil but increases its value by creating a substitute for high quality and priced conventional oil, known as a synthetic crude oil. Once upgraded the synthetic crude oil can be transported and refined using the established processes.

Bitumen is an important part of nonconventional oil. It is found in the Alberta Oil Sands, and characterized as a black, low-grade crude oil made up of much heavier hydrocarbons. Most of the oil sands contain about 10% bitumen but some areas have up to 20% (Alboudwarej et al., 2006). The goal for upgrading Bitumen is to produce light oil suitable for further applications, which can be realized through various techniques, which have been developed over the years. These include techniques that are based on carbon rejection, hydrogen addition, and combinations of both routes (Castaneda, Munoz, & Ancheyta, 2013). Carbon rejection processes represent 56.6% of the total worldwide processing capacity mainly due to its relative low investment (Castaneda et al., 2013). Carbon rejection is one of the first types of conversion processes applied in the oil industry and has been used since 1913 for different fuels and heavy hydrocarbons heated under pressure (Castaneda et al., 2013). This group of technologies includes processes such as the elimination of the heteroatoms, the lowering of the viscosity, the increasing of the H/C ratio, and cracking and removal of macromolecules. (Li, Yan, & Xiao, 2015).
Carbon can be removed by thermally cracking the heavy oil. The main two types of thermal cracking are delayed and Fluid Coking™ (including Flexicoking™). Both methods produce high carbon content petroleum coke as byproducts. These are done at high temperatures (about 500°C) and “relatively low pressures (350 kPa)” (Oil Sands Magazine, 2018). Typically, coking results in the production of about 20 to 30 wt% of coke (for delayed coking) (Eser, 2013). The addition of hydrogen, known as hydroconversion, is done in the presence of a catalyst and at high pressures (14,000 – 21,000 kPa) (Oil Sands Magazine, 2018). Since hydrogen is added to the oil there is no waste carbon-rich products. This process is however more complex and has a higher capital cost (Oil Sands Magazine, 2018). One of the most common process for the upgrading of bitumen is Fluid Coking™.

1.3 Fluid Coking™

Fluid coking is the continuous process to convert heavy oils and bitumen into more valuable petroleum products. Figure 1.3 shows a schematic diagram of the Fluid Coking process (Eser, 2013). The feed is generally vacuum residue, the heaviest portion of the extracted petroleum. The feed is generally very viscous and even solid at room temperature. The feed must be heated up to around 350 °C before being pumpable and able to be injected. The feed is atomized with steam and sprayed through injection nozzles into a hot fluidized bed of coke particles. The liquid bitumen coats a portion of the hot coat particles thermally cracking the bitumen into lighter hydrocarbon vapors and leaving a carbon rich deposit on the coke. This bed is kept between 500-550 °C by burning the outer layers of the coke in a connected vessel (House, Saberian, Briens, Berruti, & Chan, 2004). The coke is cycled between the burner and the reactor introducing hot coke particles back into the reactor. The coke particles are regenerated by subsequent bitumen thermal cracking. Steam is injected into a stripper section at the bottom of the reactor and passes upwards through the coke particles in the stripper as they descend from the main part of the reactor above and promotes fluidization of the particles in the bed. The fluidization of the hot coke particles promotes good heat transfer. The light hydrocarbon vapors, produced through thermal cracking, mix with the rising steam and leave through the cyclones, which remove entrained particles (Wormsbecker, Wiens, Mcmillan, Mcknight, & Knapper, 2016).
A significant problem that occurs in a reactor is the formation of agglomerates. When the liquid feed is injected into the coke particles it can act as a binder, clumping the coke particles together into larger masses. These masses or agglomerates trap liquid inside, restricting heat transfer and preventing quick cracking of the injected hydrocarbons. These agglomerates can flow to the bottom of the reactor where the bitumen reacts causing fouling in the stripper section. If the fouling in the stripper section becomes too severe the strippers can be completely blocked causing the reactor to be prematurely shut down, requiring them to be cleared. If the agglomerates don’t react at the stripper section they can flow into the burner where the bitumen is burned instead of cracked. This causes loss of valuable product and an overall drop in efficiency and profitability of the Fluid Coker™.
As stated above, a drawback for the fluid coking process is the generation of particle agglomerations (granulation). This phenomenon occurs when the solid particles stick to one another or to a solid surface. The main type of granulation used in industry is wet granulation. Wet granulation is the fusing of small particles together using a liquid with or without a binding agent. One of the main types of wet granulation is Fluid Bed Spray Granulation. This is done by
putting a spray nozzle, usually tangential, into a fluidized bed. The spray nozzle sprays a liquid binder into a bed of small fluidized particles, which bind together into larger agglomerates.

Although agglomeration is useful for some industries, it is detrimental for Fluid Coking™. The liquid bitumen injected into the fluidized bed is meant to be thermally cracked not used as a binding solution. The ideal condition for Fluid Coking™ would be to have the coke particles coated in liquid bitumen, without the formation of agglomerates.

1.5 Thesis Objectives

This study is being done to determine the effect that nozzle scaling has on agglomeration formation. For the reduction of agglomeration and a promotion of the overall efficiency of the Fluid Coker™ this study investigates if it is better to have fewer large nozzles or more small nozzles. To determine the real impact of scaling, properly scaled nozzles are first needed. If we can trust small scale nozzles to represent commercial nozzles is the first step. This is proven by matching spray performance in open air experiments between different nozzle sizes. Once nozzles of different scale and equal spray performance are found they can be used inside a fluidized bed to test the impact scale has on agglomeration.

Chapter 2 investigates the previous attempts at discovering how the injection system influences agglomeration. These attempts include the study of the parameters of the injection system and how fluids flow through. The spray itself has been studied and how to measure the dispersion of droplets and thus predict how those droplets would dissipated through a fluidized bed. Lastly, it reviews the methods used to measure agglomeration and liquid dispersion inside a fluidized bed after injection.

Chapter 3 goes into detail about the equipment setup and the methods used in this research. The injection system that is used for both open-air experiments and experiments inside the fluidized bed and all its parameters are shown.
Chapter 4 utilizes sprays in open air to determine how the parameters of the injection system affect the Pre-Mixer pressure and characteristics of the spray. After understanding the impact of the injection system’s parameters, the conditions that provide consistent spray conditions between nozzles of varying sizes can be selected.

Chapter 5 brings the injection system into the fluidized bed. The impact of properly scaled nozzles on agglomerate formation is measured. The stability of the sprays is confirmed inside the bed. A new method to measure free liquid inside the fluidized bed is compared to the standard Gum Arabic Method. The results of in-bed experiments are analyzed and the impact of the nozzle size is presented.
2. PREVIOUS RESEARCH

A Fluid Coker™ is a complex unit of which its various components must be studied to increase its efficiency and improve Bitumen upgrading. Focusing on a main detrimental impact, agglomeration, past research has seen how different variables inside a fluidized bed, the injected feed liquid, and the injection system affect the formation of agglomerates. Changing the temperature or fluidization velocity of the fluidized bed alters the initial formation of the agglomerates. The variables of the feed, such as its viscosity or atomization gas to liquid ratio, affects the liquid distribution of the liquid bitumen on the coke particles. Last is the impact of the injection system. This is the focus of this research. Previous studies show that the geometry of the nozzle and the stability of its flow are critical to control in order to minimize agglomeration. Along with how the variables and parameters of the Fluid Coker™ impact agglomeration, it is important to look back at old methods used to capture images of nozzle sprays and characterize them. This helps identify differences in spray performance between different nozzles. Past methods of measuring the amount of liquid trapped inside agglomerates are crucial for determining liquid trapped inside agglomerates and developing new methods to measure liquid trapped.

2.1 FLUIDIZED BED

As shown in Chapter 1, the Fluid Coker™ is kept between 500-550 °C and has steam injected from the bottom to fluidize the bed, and to strip hydrocarbon vapors from the cold coke leaving the reactor. High velocity steam is also injected in the lower section through attrition nozzles, which help attrition of agglomerates. The temperature of the reactor bed controls the reaction rate of the conversion of heavy hydrocarbons into more valuable products. The bed temperature was also seen to influence the initial formation of the agglomerates in laboratory experiments. A higher average bed temperature caused the agglomerates to dry quicker and form larger agglomerates. The binding solution solidified and bound the particles together before they were broken up (Reyes & Andrea, 2015). The fluidization of the bed had a large impact of the initial formation of agglomerates and their further breakup. Although it was shown to have
little impact at the nozzle tip, the fluidization velocity at the tip of the injected jet had a strong impact on initial agglomeration formation. Having higher fluidization at the end of the jet decreased the liquid trapped inside the agglomerates (Bhatti, 2017). This was due to the findings that most of the agglomerates were formed at the tip of the jet (Ariyapadi, 2004). The fluidization velocity was also shown to impact the further breakup of agglomerates. A high fluidization velocity promoted more breakup of agglomerates after their initial formation (Li, 2016).

2.2 Injected Feed

Heavy hydrocarbons, such as bitumen, are so viscous that they need to be heated up to 350 °C just to allow them to be pumped and injected into the Fluid Coker™. Along with this the feed is atomized with steam to promote dispersion of the heavy fluid. It has been studied that by decreasing the viscosity of a liquid injected into a fluidized bed the fluid will coat the particles more evenly. This is due to less forces keeping the liquid together and allowing it to form smaller droplets in the spray (Reyes & Andrea, 2015). It is studied that the range of viscosity and surface tension of liquid bitumen in commercial coking operating temperature and water at ambient temperature to have no appreciable change on Sauter mean diameter of the spray droplets (Ejim, Rahman, Amirfazli, & Fleck, 2010). Without the steam to atomize the feed there would be very little dispersion of liquid (Portoghese, 2007). By increasing the gas to liquid ratio of the injected spray we can improve the dispersion of liquid on the solid particles inside the fluidized bed (Portoghese, 2007).

2.3 Injection System

The injection system is a predominant variable in how the liquid disperses into the fluidized bed and coats the solid particles. Two-fluid injection nozzle systems are designed to atomize liquid into small droplets and evenly distribute them through a jet out of the nozzle tip. Small droplet sizes and even distribution of the droplets allows for a more surface area of the droplets to react with gas such as combustion or wet more solid particles for better heat transfer in a Fluid Coker™ (Portoghese, Ferrante, Berruti, Briens, & Chan, 2010). With inferior wetting and heat transfer of liquid bitumen on coke particles, agglomeration occurs.
With small, lab-scaled spray nozzles, different pre-mixer geometries can be used. Studies by Portoghese, (2007) showed that having a pre-mixer with the gas flow flowing straight into the nozzle conduit and the liquid entering at a 90° angle produced a more stable spray than a 30° pre-mixer. Movies taken with a transparent conduit showed the better pre-mixers produced a liquid flow with better dispersed gas in the conduit (Portoghese, 2007). Having a stable spray of feed into the reactor is beneficial as it improves the liquid distribution on the solid particles (Portoghese, 2007). It has been seen that all the best conditions for liquid dispersion had stable sprays. Stable sprays produced continuous streams of fine particles, while pulsing sprays alter in fine droplets and geometrically irregular liquid chunks (Ariyapadi, 2004). The pulsations in flow are caused by unstable flow upstream of the nozzle and can be identified from fluctuations in pre-mixer pressure (Ariyapadi, 2004).

The pressure fluctuations can detect what type of flow is in the injection system upstream of the conduit. If the pressure fluctuations are high in amplitude but low in frequency, then it is likely that there is slug flow in the system. If the pressure fluctuations are low in amplitude but high in frequency then the flow is more likely bubble flow (House, 2007). The pre-mixer is an important part of the injection system to create stable gas-liquid mixture upstream of the nozzle.

Having the pre-mixer as close as possible to the nozzle tip would reduce the time that the liquid has to form fully developed two-fluid flow. A conduit placed between the pre-mixer and the nozzle tip is required (Chan, Knapper, Mueller, McMillan Tyler, Kiel, Davuluri, 2015). The conduit must be long enough for the nozzle spray to reach well fluidized zones of the reactor without being blocked. This means the conduit needs to extend past the insulation, reactor wall, and any coke buildup on the interior wall of the reactor (Briens, Book, Albion, Briens, & Berruti, 2011). Adding static mixers or a helical insert to the conduit can reduce the formation of slugging flow but also affects the conduits rodability, the ability to clear the conduit of solid deposits with a rod (Maldonado, Fleck, Heidrick, Amirfazli, Chan, & Knapper, 2008) (Keon, 1992).
The nozzle used in Fluid Coking™ is based off Terence E. Base patent (Base, Chan, Kennett, Emberley, Jonasson, McCracken, & Bennett, 1997). It details separate inlets for both the steam and liquid bitumen. The bitumen inlet is downstream of the steam inlet and there is a restriction orifice on the steam line to stabilize the steam flow. The nozzle has a specific geometry to promote breakup of large liquid ligaments and droplets into fine droplets. It consists of an initial contraction, which accelerates the fluids and reduce the droplet sizes, an expansion that decelerates the liquid and may create a shockwave, and a final contraction, which accelerates the fluids to supersonic velocities. The diagram of the nozzle from the patent is displayed below in figure 2.1.

![Figure 2.1 TEB Nozzle Diagram (Base et al., 1997)](image)

The Fluid Coker™ is designed to run continuously. The TEB nozzles minimization of internal parts increases its corrosion resistance. Unlike other atomization nozzles that have the gas and liquid meet at the nozzle tip of the nozzle, the TEB nozzle is “rodable”, so that it may be unplugged by forcing a rod through to remove deposits. The TEB nozzle has been seen to reduce the size of liquid droplets from 12,000 µm to 300 µm (Base et al., 1997).
2.4 Spray Characterization Measurements

Obtaining the characteristics of a nozzle spray are important in order to understand how altering the injection system geometry and the fluids used in them impact nozzle performance. Multiple studies have been completed on diesel engine injection nozzles. These studies include research to improve fuel injector efficiency inside engines without the negative effects of going to extremely small scale (Tang, Feng, Zhan, Ma, & Zuohua, 2017), how to best scale diesel nozzles with varying engine and vehicle size (Zhou, Li, Lai, & Wang, 2018), and the impact switching from diesel to biodiesel has on the spray characteristics (Wang, Huang, Kuti, Zhang, & Nishid, 2010). Other areas of study involve the scaling of two-fluid nozzles for pharmaceutical granulation while keeping proper shape and size of products (Poozesh, Grib, Renfro, & Marsac, 2018), as well as previous studies of Fluid Coking™ spray characteristics by Li (2016). In all these studies, the identification of the spray characteristics was performed with a camera and illumination of the spray. In the studies performed by Tang et al. (2017), Poozesh et al. (2018), and Zhou et al. (2018), a shadowgraph method was used to create contrast between the spray and the background. In the shadowgraph method, the spray was between the light source and the camera. Having the spray blocking the light to the camera, the spray in the image appeared dark while the background very light. The studies by Li (2016) and Wang et al. (2010) illuminated the spray from the same side as the camera and Wang et al. (2010) study utilized focused mirrors and a light scattering technique. The equipment used to capture the images of the sprays ranged from regular cameras to extremely high-speed cameras. Utilizing a regular camera at 1 frame per 30 ms, Li (2016) captured the overall trend of the spray during continuous operation. High-speed cameras used by Poozesh et al. (2018) and Tang et al. (2017) at 49 kHz (49,000 fps) and 20,000 fps were used to observe the breakup of sprays into droplets and the evolution of the spray respectively. Tang et al. (2017) took it even further by also using a single pulse laser with a florescence diffuser in combination with a CCD camera with a telephoto microscope attached to obtain images of droplets in the spray. Ariyapadi, Balachandar, & Berruti, (2001) use Phase Doppler Anemometry to characterize the stability of
downward, vertical sprays, and Ejim et al. (2010) use changes in droplet size and liquid flux within horizontal sprays.

To measure the characteristics of the spray in the captured images, the studies developed thresholds to distinguish the spray from the background. Using MATLAB or another program most of these studies used the grayscale or intensity of the spray and set a cutoff that distinguish spray from background in the images (Tang et al., 2017), (Poozesh et al., 2018), (Zhou et al., 2018). After omitting the background, the studies used various methods to measure the droplet size, spray angles, spray area, and spray jet lengths. The high detail observation of initial evolution of the spray is important for diesel injection since a diesel engine works in quick bursts while a Fluid Coker’s™ injection is continuous. For this reason, Portoghese (2007) and Li (2016) looked at the steady state operation of the spray and its stability, using videos of the spray in open air. Portoghese (2007) characterized the spray stability with the coefficient of variation of the spray angle while Li (2016) used the total count of pixels that were spray and how they fluctuated with time. After obtaining the characteristics of the spray, predictions could be made in regard to the combustion efficiency of the injected fuel or the wetting of liquids onto solid particles.

Other studies have characterized the stability of open-air sprays from the vibrations of the nozzle assembly (Briens et al., 2011) or the sound made by the spray in open air (Ariyapadi, Berruti, Briens, Knapper, Skwarok, & Chan, 2005) (Sun et al., 2015). Ariyapadi et al. (2005) used cycle analysis of a microphone signal to quantify the spray stability and found that the stability could be characterized from either the microphone signal or the fluctuations of the pressure at the premixer of the liquid with the atomization gas, upstream of the nozzle. Other researchers have also used the fluctuations of the premixer pressure to study the stability of open-air sprays (Maldonado et al., 2008)

Characterizing the stability of sprays in fluidized beds is more challenging. The premixer pressure fluctuations can be used (Ariyapadi et al., 2005) as well as the signal form a triboprobe located within the cavity formed by the spray within the fluidized bed (Ariyapadi et al., 2005).
Since both methods agree reasonably well (Ariyapadi et al., 2005), using the premixer pressure fluctuations is more convenient.

2.5 Agglomeration/Free Liquid Measurements

Many previous methods have been developed to model the free liquid and agglomeration inside a fluidized bed. These models have utilized passive and simulation methods to model agglomeration. Some of the previous passive measurements include capacitance, conductance, and sound monitoring of the fluidized bed (Mohagheghi, Hamidi, Briens, Berruti, & McMillan, 2014) (Farkhondehkavaki, Soleimani, Latifi, Berruti, Briens, & McMillan, 2014) (Book, Albion, Briens, Briens, & Berruti, 2011).

Mohagheghi Dar Ranji (2014) injected Varsol into a fluidized bed of coke particles and used the relationship between the free moisture inside the bed, i.e. the liquid that is not trapped within agglomerates and the capacitance. By measuring the capacitance in the bed, the liquid escaping the agglomerates could be determined (Mohagheghi Dar Ranji, 2014). Another similar method measured changes in bed conductance, when injecting water into a fluidized bed of sand particles (Zirgachian, Soleimani, Briens, & Berruti, 2013).

One model that does well to simulate the formation of agglomerates is the Gum Arabic (GA) method developed by Reyes & Andrea (2015). This method utilized an Arabic Gum solution to simulate the binding effects of liquid bitumen on coke particles. “Gum Arabic is non-toxic, completely soluble in water, and stable up to about 200 °C (Imeson, 1997). This allowed for easy use in laboratory activities without the need for additional protection. Having a safer method has many benefits and the Gum Arabic solution used in a fluidized bed of sand is a good choice to simulate bitumen and coke in a Fluid Coker™. The wettability of the particles by the injected liquid greatly affects agglomeration formation. The Gum Arabic solution is mainly water and wets the silica sand well, as bitumen wets coke well in commercial Fluid Coker™ reactors (Mohagheghi Dar Ranji (2014). The viscosity of the solution was modified to match the viscosity of preheated
bitumen by adjusting its pH with HCl. It also allows for a measurement of the liquid trapped in the agglomerates if a dye is added to the solution that persists after the solution is evaporated.
3. EQUIPMENT, MATERIALS, AND METHODS

The objective of this chapter is to describe the equipment, materials, and methods used in the experiments of this thesis. The equipment, including the Spray Injection System, the High-Speed Video, and the Fluidized Bed have been used to simulate the operation and performance of an industrial Fluid Coker™ spray nozzle. It consists of three sections as follows:

1) The equipment, which was developed to supply and control pressurized gas and liquid flow in the experiments.

2) The camera used and the setup method developed to capture a video of the spray.

3) The methods and equipment used to evaluate the performance of the spray inside a fluidized bed.

3.1 Spray Injection System

A spray injection system was developed to disperse atomized liquid into a fluidized bed. It could also be used for open air experiments. The schematic diagram of the Injection System is illustrated in Figure 3.1. The injection system consisted of three components:

1. a gas supply system
2. a liquid supply system
3. a spray nozzle system
The gas supply system consisted of a bank of high-pressure nitrogen cylinders. Nitrogen was chosen as the injected gas due to its inert properties, its low cost and its ability to be stored as a gas in high pressure cylinders. A pressure regulator regulated the supply pressure $P_{ATO}$, which was measured with a pressure transducer connected to a data acquisition system. The gas flowed through a restriction orifice ("sonic nozzle") accelerating the gas. Table 3.1 shows the dimensions of the various sonic nozzles that were used in the experiments. Preliminary calibration experiments determined the relationship between the mass flowrate of atomization gas and the measured pressure $P_{ATO}$.

The liquid system consisted of a pressurized blow tank. Using a regulator, the blow tank was pressurized with nitrogen to a pressure $P_{BTK}$ that was measured with a transducer connected to a data acquisition system. Liquid flowed from the blow tank through a restriction (a solenoid valve and manual valve) that helped stabilize the liquid flow. The pressure $P_{BTK}$ was adjusted to provide the required liquid flowrate. Before each run the tank was refilled with the required mass.
of liquid and was completely emptied with each injection. The spray nozzle system consisted of a pre-mixer, where liquid and atomization gas were mixed, a conduit, and a spray nozzle. A pressure transducer was placed just upstream of the pre-mixer in the atomization line. It was placed in the closest location to the pre-mixer that would not impact the atomization or flow downstream of the pre-mixer. The spray nozzles were scaled-down versions of commercial TEB spray nozzles (Base et al. 1997). The TEB nozzle and the function of its geometry is explained in Section 2.3. Figure 3.2 shows the TEB nozzle and Table 3.2 shows the nozzle tip diameters that were used in this study. Table 3.2 also shows the corresponding conduit characteristics. Table 3.3 provides the pre-mixer characteristics. The pressure at the pre-mixer was measured with a transducer connected to a data acquisition system.

Three nozzle sizes were used, each having twice the liquid flowrate as the previous nozzle, providing information on the overall impact of nozzle scale. Various conduit diameters (Dc) were also used to evaluate the effect of conduit diameter on P_{PRE} and to match P_{PRE} for different nozzle sizes (DN). The conduit lengths were cut into controlled segments to determine the relationship between P_{PRE} and conduit length (LC).

A commercial scale nozzle was tested to scale up the effects of changing the conduits on the large scale. The commercial size nozzles used a 60° Pre-Mixer instead of the 90° used for the small-scale nozzles.

Figure 3.2: TEB Nozzle Diagram (Base et al. 1997)
Table 3.1 Sonic Nozzle Sizes used in Experiments

<table>
<thead>
<tr>
<th>Sonic Nozzle Inner Diameter (μm) [thou]</th>
</tr>
</thead>
<tbody>
<tr>
<td>254 [10]</td>
</tr>
<tr>
<td>305 [12]</td>
</tr>
<tr>
<td>356 [14]</td>
</tr>
<tr>
<td>406 [16]</td>
</tr>
<tr>
<td>457 [18]</td>
</tr>
<tr>
<td>508 [20]</td>
</tr>
</tbody>
</table>

Table 3.2 Nozzle and Conduit Dimensions used for Injection System, * for Standard Commercial Size

<table>
<thead>
<tr>
<th>Nozzle Diameter, (mm) [in]</th>
<th>Conduit Diameter, (mm) [in]</th>
<th>Conduit Length, (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Commercial Sizes</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13.00 [0.512]*</td>
<td>12.52 [0.493]</td>
<td>0.61</td>
</tr>
<tr>
<td>15.80 [0.622]</td>
<td></td>
<td>0.76</td>
</tr>
<tr>
<td>24.31 [0.957]*</td>
<td></td>
<td>1.00*</td>
</tr>
<tr>
<td><strong>Pilot Plant Sizes</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.00 [0.039]</td>
<td>1.55 [0.061]</td>
<td>0.40</td>
</tr>
<tr>
<td>1.41 [0.056]</td>
<td>2.16 [0.085]</td>
<td>0.45</td>
</tr>
<tr>
<td>2.00 [0.079]</td>
<td>2.67 [0.105]</td>
<td>0.50</td>
</tr>
<tr>
<td>2.92 [0.115]</td>
<td></td>
<td>0.55</td>
</tr>
<tr>
<td>3.18 [0.125]</td>
<td></td>
<td>0.60</td>
</tr>
<tr>
<td>3.76 [0.148]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4.01 [0.158]</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4.27 [0.168]</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The liquid flowrate through the nozzle was obtained from the total liquid mass introduced in the blow tank before each run and the start and end times of the spray. The start and end times of the spray were determined from the recorded pre-mixer pressure ($P_{\text{PRE}}$). A large rise of $P_{\text{PRE}}$ was seen when the blow tank was opened to the Pre-Mixer marking the start of the injection. The $P_{\text{PRE}}$ spikes then dropped as the spray went from liquid and gas to just gas, which marked the end of the spray. The determination of the start, end, and duration of the spray is shown below in Figure 3.3.

![Figure 3.3 Liquid Flux Calculation $P_{\text{PRE}}$ (2 (mm) $D_n$, $F_L$ 21.3 (g/(mm$^2$·s)), GLR 2.04 (wt%))](image)

The spray’s liquid flux ($F_L$) is calculated from the injected mass over the injection time. This is shown in equation 3.1.
The following steps were followed for each controlled experiment:

1. The correct sonic nozzle, conduit, and spray nozzle are installed into the system.
2. All valves in the injection system are checked to be shut.
3. The port on the blow tank is opened and the liquid is poured into the blow tank.
4. The port valve is shut.
5. The atomization gas line is opened and the pressure is set for the desired gas flowrate using the first regulator, then shut.
6. The gas line to the blow tank is opened.
7. The second regulator pressure is set for the desired liquid flowrate.
8. Any recording devices (DAQ/camera) are started.
9. The atomization line is opened.
10. The blow tank valve or solenoid valve are opened and the injection is started.
11. After the injection ends the blow tank valve/solenoid is shut.
12. The atomization line valve is shut.
13. The gas line into the blow tank is shut and the blow tank is depressurized.

### 3.2 High Speed Video

The use of a camera is essential to study the characteristics of a spray. Which camera to use depends on the characteristics you desire to obtain from the spray. The extremely high-speed cameras used by Tang, Feng, Zhan, & Zuohua (2017) obtain very high level detail of the spray but the collection and measuring of the images would be excessive past studying a small portion of time of the spray. This study observes the continuous operation of the nozzle injection therefore
the camera used was modeled closer to Li (2016) setup. A balance of detail and practicality was chosen by using a model EXILIM EX-ZR1700 high speed camera at 480 fps. 480 fps allowed for a better visualization of how the spray characteristics change in smaller amounts of time while still having enough resolution in the images to clearly identify the spray characteristics. The camera was positioned on a stand parallel to the spray to capture the length of the spray.

Initially problems were encountered transitioning from 30 fps to 480 fps due to the feedback from the frequencies of the HID lighting in the laboratory. The feedback caused flickering in the videos due to the camera’s framerate being faster than the laboratory’s lighting frequency. The feedback was solved by using a dark room and direct current LED lighting as seen in Figure 3.4.

Figure 3.4 Diagram of the Dark Room Used for Video Recording
The darkroom also provided a black background for greater contrast between the spray and background. This allowed for omission of the background during MATLAB calculations. Any gray value lower than 30 was considered background during analysis. The camera captured videos of 160 x 224 pixels, which represented a 480 x 343 mm area. For the video analysis, the camera was set in place with the spray nozzle just outside the view of the camera. The camera started recording and then the injection was started. The camera recording was terminated after the spray was finished. A frame of one of the videos taken can be seen in Figure 3.5.

![Figure 3.5 Spray Image; F<sub>L</sub> = 23 (g/(mm<sup>2</sup>.s)), 1.41 (mm) Nozzle, 2 (wt%) GLR, 4.01 (mm) Conduit Diameter](image)

The video analysis was done by importing the video files from the camera into MATLAB. Using MATLAB, the pixel values of each frame of the video could be read and analyzed. The RGB values were converted into gray scale values in MATLAB. Since the light was being reflected off the spray, the gray value was related to the density of the spray, resulting in a higher concentration of water with a higher gray value. For video analysis, the experiment utilized the steady state portion of the spray, between the spray start and spray end (see Figure 3.6). The start and end of the spray were found from the change of the sum of the gray value intensity for the whole picture between frames.
A significant increase in the sum of the gray values would occur when the video transitioned from only black background to the start of the spray. The sum of the gray values had a steep drop at the end of the spray as the spray went from a combination of liquid and gas to just gas. An example is shown below in Figure 3.6. Section 4.1 provides a detailed review and discussion of spray analysis methods.

![Figure 3.6: Spray Duration Determination Sum of Gray Values (2 (mm) D_n, F_L 21.3(g/(mm²·s)), GLR 2.04 (wt%)](image)

Utilizing the sum of the gray values, the spray start, end, and duration can be determined. From this calculation, we take 10% of the spray duration off the start and end of the spray to obtain the middle steady state portion of the spray. This calculation of the portion of the spray was used in all the video analyses of the spray.
3.3 Fluidized Bed

A rectangular fluidized bed was used in this study to represent a portion of the region in a Fluid Coker where a feed nozzle is injected. A diagram can be seen below in Figure 3.7.

**Figure 3.7 Fluidized Bed Diagram**
The fluidized bed equipment for the experiments was 1.2 (m) long, 2.3 (m) tall, and 0.15 (m) wide. The bed was filled with 150 (kg) resulting in a fluidized bed height of 60 (cm). The injection nozzle was inserted 0.32 (m) above the grid plate. For dual injections a second nozzle was inserted at the same height as the first on the opposite side of the bed. Before each injection, the bed was preheated to 130 °C with hot fluidization gas, using a 7.5 (kW) electric heater on each fluidization gas line. During injection the superficial velocity of the fluidization gas into the bed was 0.55 (m/s). The bed had one internal and one external cyclone. The bed was emptied out the bottom at the far end.

To control the fluidization velocity of the bed, a series of three sonic nozzles were used in the fluidization system for both halves of the bed. These can be seen in Figure 3.8 and are the same setup used previously by Bhatti (2017).

Figure 3.8 Sonic nozzle banks upstream of gas distributors which provide the fluidization air (Bhatti, 2017)
Using sonic nozzles and controlling the upstream pressure with a pressure regulator ensured that the mass flowrate of fluidization gas remained constant during each experiment, even when liquid injection and vaporization resulted in changes in the pressure drop through the fluidized bed equipment. A pressure transducer was placed upstream of the nozzles for each gas supply line. The sonic nozzles were calibrated to provide the mass flowrate of gas from the measured pressure. This calibration can be found in the appendix.

Fifteen thermocouples were placed into the bed to record temperature. Two were in the fluidization line, one after each heater. The other 13 thermocouples were placed in a matrix in the bed to obtain an accurate average bed temperature. Figure 3.9 shows the thermocouple locations.

![Figure 3.9: Thermocouple Locations](image)
For an experiment using the fluidized bed, the following steps were followed:

1. Fill bed with 150 (kg) of sand.
2. Steps 1:7 of the injection system procedure (see section 3.1).
3. Partially open atomization line value to have some gas flowing through the spray nozzle and prevent backflow.
4. Set the fluidization gas to optimum flowrate for heating.
5. Heat the bed up to 130 °C.
6. Fully open the atomization gas line.
7. Increase fluidization velocity to injection fluidization velocity (0.55 (m/s)).
8. Start recording devices (DAQ Pressure/Temperature), time equals zero.
9. Open blow tank valve or solenoid valve at 30 (s); injection starts.
10. Drop fluidization velocity to just above minimum fluidization velocity at time equals 60(s).
11. Shut blow tank valve or solenoid valve.
12. Shut atomization valve.
13. Shut the gas inlet to the blow tank and depressurize the blow tank.
14. Dry the bed at just above minimum fluidization until 660 (s).

3.4 MATERIALS

Several materials were used for the experiments. An overview of all the materials used in the research are summarized below.

3.4.1 Solids

Sand with a density of 2650 (kg/m³) and a Sauter mean diameter of 190 (µm) was used inside the fluidized bed. This was the same grade of sand used in previous studies and the size distribution is shown in Figure 3.10. The sand belongs to Geldart’s group B.

Gum Arabic (acacia gum) powder was used for the Gum Arabic solution.
3.4.2 Liquids

De-ionized water was used for pure water injections and for the Gum Arabic solution. The Gum Arabic solution was developed by Reyes & Andrea, (2015), who showed that when sprayed in bed of sand particles at 130 °C, it provided a good representation of the agglomerate formation when bitumen was sprayed into a bed of coke particles in the small-scale reactor (Reyes & Andrea, 2015).

Ejim et al. (2010) studied the impact of liquid viscosity and surface tension of droplet size in the spray of atomization nozzles. It was found that no appreciable changes in Sauter mean diameter of the spray droplets was found in the range of bitumen at 300°C and water at ambient conditions. The viscosity and surface tension of bitumen being 2 mPa s and 14 mN/m, respectively, and 1 mPa s and 70 mN/m for water. With the adjustment of viscosity of the Gum Arabic solution, closer liquid conditions are obtained tough their impact is already negligible.
Methylene blue dye was added to the Gum Arabic solution. It facilitated the analysis of the recovered agglomerates. The Gum Arabic solution was made with 92 (wt%) deionized water, 6 (wt%) Gum Arabic, and 2 (wt%) blue dye. Its pH was adjusted with HCl until it reached a pH of 3.

3.4.3 Gases

Nitrogen gas was used as the injection gas and for the blow tank. Dry compressed air was used to fluidize and preheat the bed.

3.5 **Analysis Equipment**

Accurate scales were used to weigh the liquids and solids used in the experiments and analyses. A photo spectrometer was used to find the absorbance of the blue dye trapped in the agglomerates, using the method developed by Reyes & Andrea, (2015).
4. **Impact of Spray Injection System Dimensions on Pre-mixer Pressure and Open-Air Spray Characteristics**

In order to properly scale the nozzle diameters ($D_N$), of the flux ($F_L$), atomization gas-to-liquid ratio (GLR), pre-mixer pressure ($P_{PRE}$), and spray characteristics needed to be kept constant between different nozzle sizes. To achieve the proper scale, this research determined how the dimensions of the injection system attached to the spray nozzle affected each one of these variables. Chapter 4 uses open-air spray experiments to determine the relationships of the dimensions and variables of the injection system and shows how they influence the conditions chosen for in-bed experiments.

**4.1 Development of Methods**

During injection, some parameters such as the stability and geometry of the spray cannot be merely measured with a transducer or a caliper. These parameters required the development of methods for their quantitative evaluation. These methods utilized the values obtained from pressure transducers and the high-speed video recordings from open-air experiments to calculate the parameters of spray stability and spray geometry.

**4.1.1 Geometry Method**

To find the geometry of the spray the border of the spray had to be identified. The spray characteristics included the angle, the relative maximum width, and relative length to the maximum width. The relative maximum width was the maximum width of the spray over the diameter of the nozzle while the relative length was the length to the maximum width over the diameter of the nozzle.

After defining the background and gray values seen in Chapter 3.2, the average border of the spray could be determined. Pixels that were designated as background were given a value of 0 and pixels identified as spray a value of 1. The actual gray values were ignored, since for the border, it is only important to find the transition between the spray and background. Since the spray boundary fluctuated with time, this transition zone corresponded to the zone of largest
variability in gray value from frame to frame. A MATLAB code went frame by frame taking the difference of each local pixel value from its previous frame. This code can be found in the appendix. The sum of these differences was taken for each local pixel and a figure of pixel sums was formed. A colour-coded image of this can be seen in Figure 4.1. The MATLAB code further developed the border of the spray by selecting a single lined border around the spray from the pixels with the highest sums. This is seen in Figure 4.2.

![Image of local pixel fluctuation sum](image)

Figure 4.1 Local Pixel Fluctuation Sum (Flux: 21.5 (g/(mm²·s)), GLR: 2 (wt%), Dₙ: 2 (mm), Dₜ: 4.01 (mm))
Once the spray border was determined, the spray could be analyzed. MATLAB was used to find the maximum width of the spray ($W_S$ in Figure 4.3) and the distance to the maximum width ($L_S$ in Figure 4.3). The width and length of the spray were used to calculate the angle of the spray ($\theta$ in Figure 4.3).
4.1.2 Stability Methods

The first step to developing a system of stability estimation for the sprays was to develop a visual categorization of the spray. Human interpretation was used to compare fluctuations in area and density of the spray to differentiate between stable and unstable sprays. In this study, a ranking of 1 through 5 was used, as detailed in Table 4.1. An example of a stable and an unstable spray can be seen in Figures 4.4 and 4.5 respectively.
Table 4.1: Eye Ranking System for Spray Stability, 1 for most stable to 5 for least

<table>
<thead>
<tr>
<th>Stability Rank</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Stable spray cone with no visible fluctuations</td>
</tr>
<tr>
<td>2</td>
<td>Stable spray cone with only minor visible fluctuations</td>
</tr>
<tr>
<td>3</td>
<td>Periods of fluctuations and stable cone spray</td>
</tr>
<tr>
<td>4</td>
<td>Rapid Fluctuations while remaining a cone</td>
</tr>
<tr>
<td>5</td>
<td>Rapid fluctuation between cone and dense liquid stream</td>
</tr>
</tbody>
</table>

The next step was to develop a MATLAB calculation to compare numerical data obtained from the videos to the visual ranking system. The initial approach was to use the fluctuations in the spray area, as determined from video recordings, and the fluctuation in pre-mixer pressure ($P_{PRE}$). It was determined that the visual fluctuation of the spray should relate closely to the ranking system since both methods were based on the visual observation of the spray. The fluctuations in $P_{PRE}$ allowed for the observation of a slugging flow regime in the conduit and nozzle as well as having the benefit of being utilized for the in-bed experiments.
Three methods were chosen that utilized video analysis to measure spray stability. These methods included the Gray Bin Method, the Gray Diff Method, and the Gray Sum Method.

The Gray Bin Method utilized the background cutoff previously stated in Chapter 3.2 (Gray Value < 30). The method takes each individual pixel in the frame and counts how many times it fluctuates from spray to background or the reverse. The average of the moving standard deviation of Pixel Changes was taken over the average of the Pixel Changes for the steady state duration of the spray. The equations used for this method can be found in Equation 4.1.

\[
\begin{align*}
if \ (Gray\ Value \ < 30) \Rightarrow Bin &= 0 \\
if \ (Gray\ Value \ \geq \ 30) \Rightarrow Bin &= 0
\end{align*}
\]

\[
Pixel\ Changes = \sum_{p(pixel)=1}^{n} abs(diff(Bin_{f+1} - Bin_f))_p
\]

\[
I_{GB} = \frac{AVG(MOVSTD(Pixel\ Changes))}{AVG(Pixel\ Changes)}
\]

Equation 4.1 Instability Index Gray Bin Stability Method

The Gray Diff Method looks at the amount each pixel of the spray changes throughout the steady state duration of the video. Each individual pixel was taken and the absolute difference between its gray value and the gray value of the next frame was taken. The sum of these differences was taken for the steady state duration of the spray. The sum of all the pixel
sums were taken over the average gray value of one frame for the steady state duration of the spray. The equations used for this method can be found in Equation 4.2.

\[
\text{Gray Diff} = \sum_{f(frame)=1}^{n} \text{abs}(\text{diff}(\text{Gray Value}_{f+1} - \text{Gray Value}_{f}))
\]

\[
\text{Sum Gray Diff} = \sum_{p(pixel)=1}^{n} \text{Gray Diff}_p
\]

\[
I_{GD} = \frac{\text{Sum Gray Diff}}{\text{AVG(Gray Sum)}}
\]

Equation 4.2 Instability Index Gray Diff Stability Method

This Grey Sum Method was developed by calculating the average of the moving standard deviation for every 50 ms of the gray intensity of the spray divided by the average sum of gray intensity of the spray. The purpose of calculating the standard deviation over short, 50 ms intervals was to focus on the high frequency fluctuations in the sum of gray intensity without being influenced by any slow evolution of the grey sum, which would not impact the performance in a fluidized bed. This can be seen in Equation 4.3 and a stable and unstable example are shown in Figure 4.6 and Figure 4.7.
The stability index for Pre-Mixer pressure was deployed as an additional method since it was the only one that could be used for in-bed experiments. This method was not influenced by any changes in video capture procedure. The final version of this method provided the coefficient

\[ I_{GS} = \frac{AVG(MOVSTD(Gray\ Sum))}{AVG(Gray\ Sum)} \]

Equation 4.3 Instability Index Gray Sum Stability Method

![Figure 4.6 Stable Spray Gray Sum Stability Method](image)

![Figure 4.7 Unstable Spray Gray Sum Stability Method](image)

The stability index for Pre-Mixer pressure was deployed as an additional method since it was the only one that could be used for in-bed experiments. This method was not influenced by any changes in video capture procedure. The final version of this method provided the coefficient

F_L 32.0 (g/(mm^2·s))  
D_N 1.41 (mm)  
GLR 1.36 (wt%)  
D_C 4.01 (mm)

F_L 15.9 (g/(mm^2·s))  
D_N 1.41 (mm)  
GLR 2.80 (wt%)  
D_C 4.01 (mm)
of variation of the pre-mixer pressure. This is presented in equation 4.4 and a stable and unstable example are shown in Figure 4.8 and Figure 4.9.

\[ I_{PRE} = CV_{PRE} \]

Equation 4.4 Instability Index \( P_{PRE} \) Stability Method

Figure 4.8 Stable Spray Pre-Mixer Pressure Stability Method

- \( F_L \) 32.0 (g/(mm\(^2\)·s))
- \( D_N \) 1.41 (mm)
- GLR 1.36 (wt%)
- \( D_C \) 4.01 (mm)

Figure 4.9 Unstable Spray Pre-Mixer Pressure Stability Method

- \( F_L \) 15.9 (g/(mm\(^2\)·s))
- \( D_N \) 1.41 (mm)
- GLR 2.80 (wt%)
- \( D_C \) 4.01 (mm)
4.2 Results

4.2.1 Pre-Mixer Pressure

The proper scaling of the different conduit sizes was accomplished in part by keeping the pre-mixer pressure of the injection system constant. Setting the pre-mixer pressure controlled the amount of energy injected into the system.

The \( P_{\text{PRE}} \) of the system was dependent on the \( F_L \), the GLR, and the geometry of the conduit and nozzle. In this study the \( F_G \) was kept constant whereas the \( P_{\text{PRE}} \) varied with \( F_L \), conduit length (\( L_C \)), and the conduit and nozzle diameter. The conduit and nozzle diameters affect the \( P_{\text{PRE}} \) due to sudden size restrictions from the pre-mixer to conduit and conduit to nozzle.

4.2.2 Effect of Conduit Diameter and Length

To determine the impact conduit diameter and length had on \( P_{\text{PRE}} \), multiple runs were conducted around commercial conditions (22.3 (g/mm\(^2\)·s), 2% GLR). A linear relationship was taken of these runs and the commercial condition was found through interpolation. Each set of runs for each conduit condition had an \( r \)-squared value for their linear regression of 0.95 or greater. This shows a small amount of error in obtaining the pre-mixer pressures for each conduit condition. An example is shown in Figure 4.10.
The conduit diameter sizes were plotted against the \( P_{\text{PRE}} \) required to achieve the same liquid and gas fluxes as in the commercial Fluid Coker™ nozzles (22.3 g/(mm\(^2\)·s), and 2% GLR), as seen in Figure 4.11.

\[
y = 0.095x - 3.0241 \\
R^2 = 0.9645
\]
Figure 4.11 shows that the pre-mixer pressure increased as the conduit diameter decreased. In all experiments, the pre-mixer size did not change. When the conduit diameter was reduced there was a higher contraction pressure drop from the pre-mixer to the conduit.

Figure 4.11 also shows that the pre-mixer pressure was higher for smaller nozzle diameters, when using the same conduit diameter. This was due to the diameter ratio between the conduit and the nozzle tip. When this ratio is higher the contraction pressure drop from conduit to nozzle was higher.

The overall conduit length was determined to have an effect on the pre-mixer injections performed at commercial conditions (22.3 g/(mm²·s), and 2% GLR), as shown in Figure 4.12. A decrease in conduit length showed an increase in pre-mixer pressure, which was the opposite of what was expected. Changing the conduit length by 1/3, from 60 (cm) to 40 (cm), resulted in less than an 8% change in pre-mixer pressure for all nozzle sizes. This was a negligible impact and therefore it was concluded that the effect of conduit length would be disregarded.
By understanding the impact of the conduit diameter and length, a consistent Pre-mixer pressure between the three nozzle sizes can be determined. The conduit diameters that resulted in the closest pre-mixer pressure between the different nozzle sizes were chosen for future study. This is shown in Figure 4.13.
Due to the restriction of using nominal tube sizes it was not possible to have perfectly matching pre-mixer pressures for the three nozzle sizes. The conduits that gave the closest $P_{\text{PRE}}$ along all the three nozzle sizes were chosen as the best representation for a constant $P_{\text{PRE}}$ between varying nozzle sizes.

The conduit conditions chosen for the in-bed experiments are shown below in Table 4.2.

**Table 4.2: Selected Conduit Dimensions**

<table>
<thead>
<tr>
<th>Nozzle Diameter (mm)</th>
<th>Conduit Diameter (mm)</th>
<th>Conduit Length (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.55</td>
<td>0.6</td>
</tr>
<tr>
<td>1.41</td>
<td>2.16</td>
<td>0.6</td>
</tr>
<tr>
<td>2</td>
<td>2.92</td>
<td>0.6</td>
</tr>
</tbody>
</table>
A conduit length of 0.6 (m) was chosen since it was determined that the conduit length had only a slight impact on $P_{PRE}$. The experiments varying conduit diameter were done with conduit lengths of 0.6 (m) in length, as this length was consistent with the previous experiments varying conduit diameter.

To see the effect of conduit diameter while scaling up nozzle sizes, the pre-mixer pressure was plotted against the dimensionless ratio of Conduit Diameter over Nozzle Diameter. The commercial scale nozzle of 13 (mm) in diameter, and the 3 conduit diameters used with it, was added to the graph as well, to compare commercial sized nozzle with the small-scale nozzles as shown in Figure 4.14. It should be noted that the commercial nozzle had been used for several years and was slightly eroded, so that its openings were slightly larger than would have been expected from its 13 mm nominal diameter.

Figure 4.14: Pre-Mixer Pressure vs. Dimensionless Conduit Diameter for the Three Nozzle Sizes and Commercial Size (Flux: 22.3 (g/(mm²·s)), GLR 2 (wt%))
The three small scale nozzles show comparable trends to each other, as can be seen in Figure 4-11. They do differ slightly compared to the commercial scale nozzle. The commercial scale nozzle has a shift to the left showing a lower pre-mixer pressure for the same diameter ratio as the small-scale nozzles, which may be due to a difference in pre-mixer size.

4.2.2.2 Spray Geometry

From the analysis of the spray border the spray angle, maximum width, and length to maximum width ratio for the chosen nozzles and their conduits were obtained. A table 4.3 presents the conditions of the runs used for the spray geometry analysis. Figures 4.15 – 4.17 show the three geometric variables measured for the sprays. The graphs show multiple runs for each nozzle size at commercial conditions.

Table 4.3 Spray Conditions used for Spray Geometry Analysis

<table>
<thead>
<tr>
<th>( P_{\text{pre}} ) (psig)</th>
<th>Liquid Flux (g/(mm(^2)-s))</th>
<th>GLR (wt%)</th>
<th>Nozzle Tip (mm)</th>
<th>Conduit (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>319</td>
<td>21.09</td>
<td>2.12</td>
<td>1.00</td>
<td>1.55</td>
</tr>
<tr>
<td>321</td>
<td>21.24</td>
<td>2.11</td>
<td>1.00</td>
<td>1.55</td>
</tr>
<tr>
<td>324</td>
<td>21.58</td>
<td>2.07</td>
<td>1.00</td>
<td>1.55</td>
</tr>
<tr>
<td>326</td>
<td>22.58</td>
<td>1.97</td>
<td>1.00</td>
<td>1.55</td>
</tr>
<tr>
<td>314</td>
<td>20.67</td>
<td>2.11</td>
<td>1.46</td>
<td>2.16</td>
</tr>
<tr>
<td>316</td>
<td>20.94</td>
<td>2.08</td>
<td>1.46</td>
<td>2.16</td>
</tr>
<tr>
<td>318</td>
<td>21.00</td>
<td>2.08</td>
<td>1.46</td>
<td>2.16</td>
</tr>
<tr>
<td>321</td>
<td>21.16</td>
<td>2.06</td>
<td>1.46</td>
<td>2.16</td>
</tr>
<tr>
<td>293</td>
<td>20.87</td>
<td>2.10</td>
<td>2.00</td>
<td>2.92</td>
</tr>
<tr>
<td>328</td>
<td>23.65</td>
<td>1.85</td>
<td>2.00</td>
<td>2.92</td>
</tr>
</tbody>
</table>
This table shows the closest conditions to the selected condition with analyzed spray geometries.

Figure 4.15: The relative lengths to maximum spray width for the three nozzles and their chosen conduits. ($F_L = 22.3 \text{ (g/(mm}^2\cdot\text{s})$, GLR 2 (wt%), $P_{\text{PRE}} \sim 320 \text{ (psig)}$)

The lengths of the sprays increase relative to the size of the nozzle they are injected from.
Figure 4.16: The relative maximum width of the sprays for the three nozzles and their chosen conduits. ($F_L = 22.3 \text{ (g/(mm}^2\text{s)})$, GLR 2 (wt%), $P_{PRE} \sim 320 \text{ (psig)}$)

The relative maximum width of the sprays decreased as the nozzle diameter was increased.
The angle of the sprays for the three nozzles and their chosen conduits. ($F_L = 22.3 \text{ (g/(mm}^2\cdot\text{s})$, GLR 2 (wt%), $P_{PRE} \sim 320 \text{ (psig)}$)

The angle of the 1 (mm) and 1.41 mm sprays seem to be about equal, however for the angle of the 2 (mm) nozzle is lower. The spray angle continues to decrease to the 13 (mm) commercial sized nozzle but gradually compared to the drop between 1.41 (mm) and 2 (mm) nozzles.

**4.2.3 Stability**

To properly scale the nozzle diameter, the sprays for each nozzle size must be stable for commercial conditions. The Gray Sum Method for determining stability was the best method to determine a stable versus an unstable spray. The graph of over 100 Gray Sum Instability indexes vs. Eye Stability Ranking is shown below in Figure 4.18.
Figure 4.18: Stability Index of Gray Sum vs. Eye Stability Ranking

Figure 4.18 showed a good trend between the stability index for gray sum and the eye stability ranking. There was a gap between the eye ranking of 2 and 4 for the stability index for gray sum. This makes a good cutoff criterion for stable and unstable sprays. The cutoff was scaled to a value of 1 and the original values are shown in the Appendix.

The PPRE Stability Method was compared to the eye stability ranking since it will be the only method utilized for in-bed experiments. The pre-mixer pressure stability against eye stability is shown in Figure 4.19.
The stability index for pre-mixer pressure provided a trend with eye stability rank but not as reliable as the Gray Sum stability index. The stability index for pre-mixer pressure showed no clear gap between the eye stability ranks of 2 and 4. In a conservative approach the lower end of rank 4 was used as the cutoff point. The cutoff value for pre-mixer pressure was scaled independently of Gray Sum with a cutoff value of 1 also.
Since the index for gray sum was better for determining the stability but the index for pre-mixer pressure could be used for in-bed experiments, a relationship between the two were desired. This provides a relationship between the index for pre-mixer and the index for gray sum for in bed experiments. To verify that the index for gray sum was the best method to correlate with the index of pre-mixer pressure two other video analysis methods were tested against the index of pre-mixer pressure first.

The relationship between the Gray Bin Stability showed a poor relationship with the $P_{\text{PRE}}$ Stability Method. The Gray Bin Stability Method was not chosen to use with the $P_{\text{PRE}}$ Stability Method. The correlation is shown in Figure 4.20.

![Figure 4.20: Correlation Between Gray Bin and $P_{\text{PRE}}$ Stability Indexes](image)

$y = 0.2392x + 0.5725$

$R^2 = 0.582$

The relationship between the Gray Diff Stability Method also showed a weak relationship to $P_{\text{PRE}}$ Stability Method. This also verified that Gray Diff Stability Method was not suitable for determining stability from the $P_{\text{PRE}}$ Stability Method. This correlation is found in Figure 4.21.
The index for gray sum had a good correlation with the index for pre-mixer pressure as seen in Figure 4.22. This allowed for the use of the index of pre-mixer pressure to be used for in-bed experiments and to be compared to open-air experiments. The relationship was also best fit as a linear relation. A linear relation allows the use of just the pre-mixer pressure to compare in-bed or open-air experiments without a complex conversion.
The correlation has no districted deviation between nozzle sizes. This demonstrates how both these methods can be used for varying nozzle sizes without adjustment.

For the use of the instability index for pre-mixer pressure to replace the instability index for gray sum, the main criteria is whether the spray is stable or not. From all the stability runs conducted, 87.5% that were found to be stable for the index for pre-mixer pressure were also stable for the index for gray sum. For those that were not stable the greatest deviation was an index of 1.42 for gray sum. Although there was not a perfect linear relationship between the two indices, the probability of getting a false positive for a stable spray using the index for pre-mixer pressure was low.
4.2.4 Commercial Scale Nozzles

To determine how the stability method scaled up to commercial conditions, trial runs were completed with the commercial sized nozzle (nominal $D_N = 13$ (mm)) and 3 large scale conduit diameters. Videos of their sprays were analyzed using the Gray Sum Stability Method. Results are shown below in Figure 4.23.

![Figure 4.23: Commercial Sized Nozzles Flux vs. Stability Index Sum, Gas Flux 0.45 (g/(mm$^2$·s))](image)

These trials determined that the spray provided results within the stable regime of the Gray Sum Stability Index, for the commercial sized nozzle using the 3 large scale conduit diameters.

The conditions chosen for the small-scale nozzles for use in the fluidized bed are as follows in Table 4.4.
### 4.3 Conclusion

Methods were developed to measure the complex parameters of the spray geometry and stability. These methods utilized the high-speed camera, pressure transducers, and MATLAB code to analyze the gray scale values.

The results of the open-air experiments show how the injection system’s variables of liquid flux, conduit length, and conduit diameter affect the $P_{PRE}$. The Liquid Flux of the spray has the greatest impact on the $P_{PRE}$. While keeping the Liquid Flux of the system constant for comparison between nozzle sizes, the $P_{PRE}$ can be altered by changing the conduit diameter. The length of the conduit has little impact on the change of Pre-Mixer Pressure.

The geometry of the sprays were altered by changing the nozzle diameter. Using the three nozzles at commercial conditions and equal pre-mixer pressures it was found that the relative length to nozzle diameter to be relatively constant as nozzle size was increased. The relative widths of the spray to nozzle diameter decreased as the nozzle size increased. This showed that the spray jets were becoming narrower with increased nozzle diameters. The angle of the sprays remained relatively constant for the 1 and 1.41 (mm) nozzles, but the 2 (mm) nozzle had a smaller spray angle.

To measure the stability of the spray the best method used the fluctuations in Gray Sum. This method had a good relationship with visual tests and was set to a threshold value of 1 for

<table>
<thead>
<tr>
<th>$D_N$ (mm)</th>
<th>$D_C$ (mm)</th>
<th>$L_C$ (m)</th>
<th>$F_L$ (g/(mm²·s))</th>
<th>GLR (wt%)</th>
<th>$P_{PRE}$ (psig)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.55</td>
<td>0.6</td>
<td>22.3</td>
<td>2</td>
<td>317</td>
</tr>
<tr>
<td>1.41</td>
<td>2.16</td>
<td>0.6</td>
<td>22.3</td>
<td>2</td>
<td>320</td>
</tr>
<tr>
<td>2</td>
<td>2.92</td>
<td>0.6</td>
<td>22.3</td>
<td>2</td>
<td>304</td>
</tr>
</tbody>
</table>
the stability regime. A value greater than 1 corresponded to an unstable spray and less than 1 a stable spray.

For a liquid flux of 22.3 \(g/(mm^2\cdot s)\), GLR of 2 (wt%), and the selected nozzle conduits for each nozzle diameter, it was shown that stable sprays were achieved for all three small scale nozzles using the Gray Scale Sum Stability Method. This method also found stable spray conditions for the commercial scale nozzles at similar conditions. This proves that this method is valid for nozzles of different scales.

Open spray studies ensured that liquid injection experiments in the fluidized will be conducted with nozzles of different scales that, for the same liquid and atomization gas fluxes, have the same pre-mixer pressure and provide a stable spray.
5. **Impact of Spray System Dimensions on Jet Penetration and Liquid Distribution Inside a Fluidized Bed**

Previous chapters have presented the effects of altering the injection system, in particular, the impact of nozzle size on the spray characteristics. The open-air experiments presented in Chapter 4 allowed us to adjust the spray systems so that each nozzle size, for the same fluxes of sprayed liquid and atomization gas, operated with the same pre-mixer pressure and provided a stable spray. In this chapter the sprays from different nozzle sizes were injected into a fluidized bed and their performance with agglomeration formation were evaluated and compared.

### 5.1 Pre-Mixer Coefficient of Variation

While performing the in-bed experiments no video analysis can be utilized so the relationship between pre-mixer pressure fluctuations and the spray stability was used to make sure all the sprays still matched with stability criteria.

The pre-mixer pressure was recorded during each in-bed injection and the pre-mixer pressure instability index was calculated. The spray duration was kept constant requiring each size greater in nozzle size to double the mass injected. 200 g was injected for the 1 mm diameter nozzle, 400 g for the 1.41 mm diameter nozzle, and 800 g for the 2 mm diameter nozzle. The conditions for each run are shown in Table 5.1, along with the instability index.
Table 5.1 In-Bed Run Conditions

<table>
<thead>
<tr>
<th>$P_{pre}$ (psig)</th>
<th>$F_L$ (g/(mm$^2\cdot$s))</th>
<th>GLR (wt%)</th>
<th>$D_N$ (mm)</th>
<th>Mass Injected (g)</th>
<th>$P_{pre}$ Instability Index</th>
</tr>
</thead>
<tbody>
<tr>
<td>319</td>
<td>29.3</td>
<td>1.51</td>
<td>1</td>
<td>200</td>
<td>0.5015</td>
</tr>
<tr>
<td>333</td>
<td>28.2</td>
<td>1.71</td>
<td>1.41</td>
<td>400</td>
<td>1.6090</td>
</tr>
<tr>
<td>285</td>
<td>24</td>
<td>1.90</td>
<td>1</td>
<td>200</td>
<td>0.5963</td>
</tr>
<tr>
<td>332</td>
<td>27</td>
<td>1.75</td>
<td>1.41</td>
<td>400</td>
<td>0.4377</td>
</tr>
<tr>
<td>308</td>
<td>21.9</td>
<td>2.15</td>
<td>1.41</td>
<td>400</td>
<td>0.4726</td>
</tr>
<tr>
<td>310</td>
<td>21.8</td>
<td>2.04</td>
<td>2</td>
<td>800</td>
<td>0.5026</td>
</tr>
<tr>
<td>287</td>
<td>20.1</td>
<td>2.20</td>
<td>1</td>
<td>200</td>
<td>0.5710</td>
</tr>
<tr>
<td>312</td>
<td>22.5</td>
<td>2.10</td>
<td>1.41</td>
<td>400</td>
<td>0.5510</td>
</tr>
<tr>
<td>310</td>
<td>22.2</td>
<td>2.00</td>
<td>2</td>
<td>800</td>
<td>0.4324</td>
</tr>
<tr>
<td>325</td>
<td>22.4</td>
<td>1.98</td>
<td>1.41 x 2</td>
<td>800</td>
<td>0.6917</td>
</tr>
<tr>
<td>290</td>
<td>24.2</td>
<td>1.83</td>
<td>1 x 2</td>
<td>400</td>
<td>0.6576</td>
</tr>
<tr>
<td>289</td>
<td>23.0</td>
<td>1.92</td>
<td>1</td>
<td>200</td>
<td>0.7268</td>
</tr>
<tr>
<td>289</td>
<td>22.5</td>
<td>1.96</td>
<td>1 x 2</td>
<td>400</td>
<td>0.4068</td>
</tr>
<tr>
<td>289</td>
<td>22.5</td>
<td>1.97</td>
<td>1 x 2</td>
<td>400</td>
<td>0.4807</td>
</tr>
</tbody>
</table>
These results were graphed against the stability criteria. Figure 5.1 shows that the sprays remained stable inside the fluidized bed.

![Figure 5.1: Pre-mixer pressure Stability Index for In Bed Experiments](image)

During the experiments one of the 1.41 (mm) nozzle runs was found to fall within the unstable range. This run had a higher liquid flux and, with closer analysis, was determined to be unstable since the pre-mixer pressure was not able to reach its maximum pressure until half the duration of the run. This run was rejected based on the fact that its liquid flux was far from the typical value for commercial Fluid Coker™ nozzles.

The rest of the experiments inside the bed all showed good stabilities. There were no clear trends between the different nozzle sizes or with dual injections experiments in which 2 nozzles were operated simultaneously at different locations in the bed. The stability index fluctuated slightly even between runs with similar \( F_L \).
5.2 LIQUID DISTRIBUTION

The performance of each nozzle inside the fluidized bed was analyzed using its liquid distribution. To determine the liquid distribution of the spray inside the bed two main methods were employed, the Gum Arabic Method and the Thermal Balance Method. The Gum Arabic Method was developed in a previous study by Reyes & Andrea (2015) as described in Section 2.4 and the Thermal Balance Method, which was developed for this study, utilized the monitoring of the bed temperature to predict the amount of liquid evaporated during and immediately after an injection.

5.2.1 Methods

The Gum Arabic Method was used in this study to determine liquid distribution. Results obtained with this method correlate well with results obtained when injecting bitumen into a fluidized bed of hot coke particles (Reyes & Andrea, 2015).

The fluidized bed was heated up to 130 (°C). At time zero the fluidization gas velocity was set to $V_g = 0.55$ (m/s). At time = 3 (s), the Gum Arabic solution was injected into the bed. The injection lasted about 11.4 (s) depending on set $P_{PRE}$. At time = 60 (s) the fluidization velocity was set to minimum fluidization velocity. The bed was then dried until time = 660 (s) or for 10 (min). The bed setup and experimental procedure was explained more thoroughly in Chapter 3.3.

The Gum Arabic solution, as explained in Chapter 3.4.2, was injected in place of water and left to dry after the injection finished. The agglomerates formed from the Gum Arabic solution that bonded with the sand were sieved, sized, and analyzed to determine their dry Gum Arabic content.

The Thermal Balance Method was developed to allow for a quicker solution to determine liquid trapped inside the agglomerates. This alternative method involved a Thermal Balance to determine how quickly water evaporated during the injections. The Thermal method can be performed independently or in conjunction with other methods like the Gum Arabic method. Equation 5.1 was developed to calculate how much energy was absorbed by the fluidized bed from the heated fluidization gas, before the liquid injection. Although the Thermal Balance
Method could be used with any liquid, all the thermal balance results reported in this thesis were obtained with the Gum Arabic solution, for easy comparison with the results obtained from the Gum Arabic Method.

Equation 5.1 Energy Balance: Energy going into the Bed

\[
C_p \cdot \dot{m} \cdot (T_G - T_B) = U A (T_B - T_S) + m_s C_{ps} \frac{dT_B}{dt}
\]

Heat capacity and mass flow of fluidization gas is constant. \((T_B - T_S)\) assumed constant.

\[
\alpha = C_p \cdot \dot{m} \\
\beta = U A (T_B + T_S) \\
\gamma = m_s C_{ps}
\]

\[
\frac{\alpha \cdot (T_G - T_B)}{\gamma} = \frac{\beta}{\gamma} + \frac{dT_B}{dt}
\]

\[
\frac{\alpha}{\gamma} \cdot (T_G - T_B) = \frac{\beta}{\gamma} + \frac{dT_B}{dt}
\]

\[
\alpha_2 \cdot (T_G - T_B) = \beta_2 + \frac{dT_B}{dt}
\]

\[
\alpha_2 \cdot (T_G - T_B) - \beta_2 = \frac{dT_B}{dt}
\]

\[
\frac{dT_B}{dt} = \alpha_2 \cdot (T_G - T_B) + \beta_3
\]
In Equation 5.1 the heat capacity and mass flow of gas into the system were assumed constant since the gas temperature does not change significantly to affect its heat capacity and the flow of gas was kept the same for all experiments. The temperature difference between the bed and the surroundings was considered constant since the bed temperature changed only slightly during injection and the temperature of the surroundings did not vary. The heat transfer coefficient between the column wall and its surroundings was assumed constant, as was the bed mass. The final equation consisted of the temperature gradient of the fluidization gas, the bed temperature, and the change of bed temperature with time.

To obtain the constants for this equation, the bed was heated up to 130 (°C) and the fluidization gas was increased to $F_i$ (fluidization for injection). The temperature gradient between the fluidization gas and bed were recorded for multiple runs and plotted against changes of bed temperature.

$$\frac{dT_B}{dt} = \alpha_2 \cdot (T_G - T_B) + \beta_3$$

$$y = 0.000244x - 0.006939$$

Figure 5.2 Bed Temperature Change vs. Temperature Delta of Heating Gas and Bed

Figure 5.2 shows the correlation between the rate of change in bed temperature and the temperature gradient between the inlet gas and the bed. This equation was used to predict the temperature change in the absence of an injection.
If the fluidized bed was a closed system, with perfect insulation, the intercept (B) in Figure 5.2 would be expected to be zero and no heat would be lost to the surroundings. The runs with no injection started with a bed temperature of 130 °C and surroundings at ambient temperature (~21 °C). This difference of temperature of the bed and surroundings was responsible for the constant heat loss of the system (T_B – T_S). During injection the maximum temperature drop of the bed was 12 °C. This resulted in an average change of 6 °C in the bed temperature making (T_B – T_S) range from 109 to 103 °C. This was less than 6 % change in the driving force of heat loss to the surroundings through an insulated fluidized bed concluding that ΔB can be assumed to be zero.

From previous calculations the amount of heat entering the system based on the temperature difference between the fluidization gas and the bed was obtained. This was used to create the predicted temperature evolution without injection. This is shown in Figure 5.3 with an increasing line that was gradually flattening due to a shrinking difference between inlet gas temperature and the bed temperature as the bed temperature increased.

![Graph showing calculated temperature without liquid injection](image)

Figure 5.3 Calculated Temperature Without Liquid Injection, F_l: 20.1 (g/(mm²·s)), GLR 2.2 (wt%), D_N: 1 (mm), D_C: 1.55 (mm)
A second energy balance was needed to calculate the amount of water that was evaporated. Equation 5.2 shows the calculations used to find the mass of water evaporated per change in bed temperature during an injection and Table 5.2 shows all the values used and obtained in the calculation.

Equation 5.2 Mass of Water Evaporated per change of Bed Temperature

\[
\Delta H_{Total, \text{ Water}} = \Delta H_{\text{Evap, Water}} + \Delta H_{\text{G, Water}}(130 - 100 \degree C) + \Delta H_{\text{L, Water}}(100 - 20 \degree C)
\]

\[
C_{Bed} = c_{Sand} \cdot M_{Bed}
\]

\[
\frac{M_{\text{Evap, Water}}}{\Delta T_{Bed}} = \frac{C_{Bed}}{\Delta H_{Bed, \text{ Water}}}
\]
Table 5.2 Energy Balance 2: Energy needed to Vaporize Water in the Bed

<table>
<thead>
<tr>
<th>Substance</th>
<th>Specific Heat Capacity, J/(kg· °C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand</td>
<td>830</td>
</tr>
<tr>
<td>Air</td>
<td>1005</td>
</tr>
<tr>
<td>Water</td>
<td>4182</td>
</tr>
<tr>
<td>Steam</td>
<td>1864</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Bed Mass, kg</th>
<th>Heat Capacity, J/°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>150</td>
<td>124 500</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Substance</th>
<th>Heat of Vaporization J/kg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>2 257 000</td>
</tr>
</tbody>
</table>

Mass of water vaporized, per degree change of sand temperature. Assume steam is at 130 °C.

<table>
<thead>
<tr>
<th>Gram Water</th>
<th>20 to 100</th>
<th>335</th>
<th>J/g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steam</td>
<td>100 to 130</td>
<td>56</td>
<td>J/g</td>
</tr>
<tr>
<td>Gram Water Vaporize</td>
<td>2 257</td>
<td>J/g</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>2 647</td>
<td>J/g</td>
<td></td>
</tr>
</tbody>
</table>

Bed Heat Capacity 124 500 J/°C

Grams Water/°C Sand 47.03 g/°C

The water during injection was assumed to start at ambient temperature 20 (°C), evaporate at 100 (°C), and leave the bed as steam at 130 (°C). Figure 5.4 shows the calculated liquid evaporated with time. Liquid evaporation started with the injection, at 30 (s), and was calculated until the end of the high fluidization velocity period at 60 (s). The calculation was stopped here since the calculated heat into the system was based on a high fluidization velocity (0.55 m/s) and would not be accurate at minimum fluidization. The rate at which liquid evaporated reached a maximum value near the end of the injection and tapered off later. This was due to the highest amount of unevaporated liquid being present in the bed at the end of the injection.
Figure 5.4 Calculated Liquid Vaporization during Injection \( F_L: 20.1 \text{ (g/(mm}^2\text{·s))}, \text{ GLR 2.2 (wt%), D}_{N}: 1 \text{ (mm), D}_{C}: 1.55 \text{ (mm)} \)

5.2.2 Results for Injection Nozzles

The outcome from the Gum Arabic Method provided the proportion of the injected liquid that was trapped inside agglomerates of injected liquid and bed particles (Total Liquid Trapped % inside the agglomerates). These values were graphed and compared for different nozzle sizes and liquid fluxes through the nozzle tip (\( F_L \)).

Figure 5.5 indicates that the proportion of liquid trapped for the 1 (mm) and 1.41 (mm) nozzles increased as the liquid flux increased. As the gas flux through the nozzle tip (\( F_G \)) was kept constant the GLR decreased as the liquid flux increased. It is known that decreasing the GLR will increase the proportion of liquid trapped (Portoghese, 2007). A linear regression was found for the 1 (mm) and 1.41 (mm) nozzles with r-squared values of 0.862 and 0.958 respectively. The
proportion of liquid that is trapped in agglomerates can be accurately estimated based on the linear relationship of liquid flux and liquid trapped for each nozzle size.

Figure 5.5 shows that as the nozzle size increased the proportion of liquid trapped decreased when scaling up from the 1 (mm) to the 1.41 (mm) and then the 2 (mm) nozzle for the same GLR.

The first explanation is that larger nozzles result in jet cavities that penetrate further into the fluidized bed reaching bed regions with more intense fluidization. The fluidization regimes for this bed were previously measured by Bhatti (2017). Using Bhatti’s (2017) measurements for superficial gas velocities of 0.50 and 0.60 (m/s) an interpolated graph for 0.55 (m/s) was formed and is shown below in Figure 5.6. The gas bubbles flux peaks near the middle of the bed and drops near the walls. The larger nozzles can be seen to hit the higher fluidization velocity regions causing more energy from the impact of the gas helping agglomerates break up. The larger
nozzles that have deeper jet penetrations and hit higher fluidization zones also have lower liquid trapped percentages as seen in Figure 5.6.

An additional explanation is that the vaporization of the injected liquid significantly increases the average superficial gas velocity. Table 5.3 shows how much the fluidization velocity would change for each nozzle if all the liquid was instantaneously evaporated during injection for a liquid flux of 22.3 (g/(s·mm²)). Although, as shown in Figure 5.4, not all the injected liquid is vaporized during
the injection. Most of the liquid is vaporized in the regions of the bed where agglomerates are
formed, increasing the fluidization in these regions. Since changing the fluidization in the region
of the bed where agglomerates are formed has the most significant impact on preventing
agglomeration it can be concluded that the evaporation can have a significant impact on reducing
agglomerates. A graph with the added impact of fluidization velocity from liquid evaporation is
shown in Figure 5.7.

<table>
<thead>
<tr>
<th>( D_N (\text{mm}) )</th>
<th>Number of Nozzles</th>
<th>Mass Injected (g)</th>
<th>Fluidization Velocity Increase (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>200</td>
<td>0.065</td>
</tr>
<tr>
<td>1.41</td>
<td>1</td>
<td>400</td>
<td>0.129</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>800</td>
<td>0.259</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>400</td>
<td>0.129</td>
</tr>
<tr>
<td>1.41</td>
<td>2</td>
<td>800</td>
<td>0.259</td>
</tr>
</tbody>
</table>

Table 5.3 Fluidization Velocity Increase due to Liquid Vaporization at \( F_L : 22.3 \, (g/(s \cdot \text{mm}^2)) \)
The larger nozzles also had a higher liquid flowrate increasing the fluidization due to evaporation. They had the combination of penetration deeper into the fluidized bed to zones of higher fluidization and more fluidization due evaporation. To test if the fluidization due to evaporation had a significant impact on agglomeration the tests needed to be done at the same mass injected. To achieve this a second nozzle was inserted into the fluidized bed opposite the first nozzle. This nozzle had its own identical injection system and matched the first nozzles flow conditions including $F_L$, GLR, mass injected, and injection time. They identical nozzles were also sprayed simultaneously so to represent two half sizes nozzles vs. one double sized nozzle. Results from the dual injections are shown in Figure 5.8.

Figure 5.7 Single Nozzle Jet Penetration and Liquid Trapped % with Adjusted Velocities for Evaporation
The experiments showed a decrease in liquid trapped when a second 1 mm nozzle was added. An experiment done with two 1.41 mm nozzles showed very little change in liquid trapped only a slight increase. The change in liquid trapped at commercial conditions and the relative fluidization impacts are shown in Figure 5.9.
The 1.41 (mm) dual injection had similar results for the proportion of liquid trapped as the single injection 1.41 (mm). This would show that both nozzles hit similar fluidization zones. There are two possible explanations of these results. Both explanations are related to previous studies by Li (2016) that have indicated that the local fluidized bed hydrodynamics have a strong impact on the distribution of a sprayed liquid on the fluidized particles.

The liquid trapped % of 2.5 % for adding a second 1.41 mm nozzle is insignificant. It can be accounted for in experimental error. The change in liquid trapped for the 1 mm was more significant and validated by multiple runs. The second 1 mm nozzle doubled the mass injected from 200 g to 400 g, while the 1.41 mm doubled from 400 g to 800 g. If this impact was due to
higher fluidization from more evaporation of liquid and more significant reduction in liquid trapped would have been seen for the 1.41 mm dual injection.

Each run performed inside the fluidized bed utilized both methods simultaneously. The temperature of the bed was recorded while using Gum Arabic solution for the injected liquid. The results obtained with the Thermal Model and the Gum Arabic Method were compared and found to agree with each other. Figure 5.10 shows the mass of evaporated liquid at 60 (s), as determined with the thermal model, agreed well with the mass of liquid trapped within agglomerates, as determined with the Gum Arabic Method, for all the conditions shown in Table 5.1.

---

**Figure 5.10 Gum Arabic vs. Thermal Method for Free Liquid**
As seen in Figure 5.10 there is not a 1 to 1 fit for fit between the Gum Arabic Method and the Thermal Method. This is due to them measuring liquid trapped different parts of the agglomeration process. The Gum Arabic method measures how much liquid was trapped inside the agglomerates from their initial formation only missing the amount of liquid instantaneously evaporated at injection, the Free Liquid. The Thermal method measures all the liquid that evaporates up until 60 s run time of high fluidization. This captures the initial free liquid evaporation and the liquid evaporating in the agglomerates leaving only the liquid still trapped in the agglomerates at 60s. An example of the breakdown of liquid measured for both models is shown in Figure 5.11

![Figure 5.11 For a 1.41 mm Nozzle run with 400g of liquid injected](image)

The correlation between the two methods means that, in future studies, the Thermal Method could be used to quickly identify the most promising conditions for further testing with the Gum Arabic Method.
5.3 CONCLUSION AND DISCUSSION

The $P_{PRE}$ Stability Method from Chapter 4 was utilized for the in-bed experiments to determine stability of the sprays. Sprays at the same and similar conditions as the open-air sprays both resulted in stable sprays. This shows a good translation between the open-air and in-bed experiments and that the sprays have the same stability when transferred to the bed. This is a good indication, that along with consistent Liquid Flux and GLR, the sprays will retain their stability characteristics.

The development of the Thermal Balance Method introduced an alternative to the Gum Arabic method for estimating the free liquid inside the fluidized bed after injection. This quick method performed well and could be used to complement the Gum Arabic method. For example, it could be used to identify the most promising conditions for further testing with the Gum Arabic method.

The results for the liquid trapped show that the larger nozzles perform better than the smaller nozzles. The key contributing factor may be the penetration of the jets of the nozzles. Deeper penetrations hit areas of the bed with higher local fluidization velocities, which is known to reduce agglomerates formation and promote their breakup (Bhatti, 2017). The large change from the 1 to 1.41 (mm) nozzle is likely due to the jet produced by the 1 (mm) nozzle being too small to be impacted fully by the rising bubbles of the bed. The improved performance of the larger nozzles was also found to be insignificantly impacted by the increased evaporation due to more liquid being evaporated in the bed for high liquid flowrates.
6. **CONCLUSIONS AND RECOMMENDATIONS**

In this research it was found that the injection system used for a Fluid Coker™ can be altered to maintain the Pre-Mixer pressure, liquid flux, gas-liquid ratio, and stability to achieve proper scaling of nozzle size. From properly scaled nozzles the effect of nozzle size on agglomeration was found.

**6.1 CONCLUSIONS**

1. The Pre-Mixer pressure of the injection system can be changed by changing the size of the conduit. It is possible to match the Pre-Mixer pressure of different sized nozzles to obtain similar conditions for nozzle scale comparison.

2. The length of the conduits at the scale of testing show little effect on Pre-Mixer pressure.

3. Using a high-speed camera and MATLAB a stability method was developed to analyze stability and set a stable criterion for spray in open air.

4. The conditions of the 3 small scaled nozzles were all verified as stable sprays. Along with this, the commercial scaled nozzle was also found to be stable at similar conditions. This confirmed the use of scaling for the open-air stability method.

5. A stability method using Pre-Mixer pressure was also developed to confirm stability inside the fluidized bed. Using this method, the chosen conditions were verified to remain stable after being placed inside the fluidized bed.

6. A thermal model was developed and was determined to be a reliable method for estimating the amount of free liquid inside the fluidized bed. This method is viable for temperatures above the injected liquids boiling point and is a quicker alternative to the Gum Arabic Method.
7. Increasing the nozzle size was shown to decrease the amount of liquid trapped inside agglomerates. The prominent reason being that the tip of the jet reached a zone of higher fluidization inside the bed.

6.2 RECOMMENDATIONS

To achieve similar performance for smaller nozzles as the larger nozzles they should be inserted farther into the fluidized bed to reach the same zones of fluidization.

Since the conclusion that the improved performance of the larger nozzles is mainly due to the jet spraying into a region of increased fluidization velocity, the tests should be repeated using a bed with an even fluidization throughout the bed. This would show if the nozzle has any remaining impact beside reaching farther inside the bed to the higher fluidization zones.

It may be more viable to determine how to choose a nozzle size that reaches the desired high fluidization zones of the reactor than seeing the impact of nozzle size.
7. REFERENCES


Bhatti, Muhammad. (2017). Preliminary Study on Behavior of Agglomerates formed by Liquid Injection. *University of Western Ontario*


Sanchez, F. (2013). Hydrodynamics in Recirculating Fluidized Bed Mimicking the Stripper Section of the Fluid Coke. The University of Western Ontario


8. **Appendix**

**Stability Method Scaling Factors**

To scale the stability methods to an instability cutoff of 1 stability scaling factors were used. By taking the original values and dividing them by these factors we can obtain the comparable values used in the thesis.

<table>
<thead>
<tr>
<th>$P_{\text{pre}}$</th>
<th>Gray Sum</th>
<th>Gray Bin</th>
<th>Gray Diff</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0055</td>
<td>0.03</td>
<td>0.048</td>
<td>0.11</td>
</tr>
</tbody>
</table>

**Sonic Nozzle Calibration for Fluidized Bed**

The slope and the Y-int for the calibration of the sonic nozzles of the fluidized bed fluidization system is shown below. The table shows each individual sonic nozzle’s calibration and every pair of nozzles.

<table>
<thead>
<tr>
<th>Side</th>
<th>Valves</th>
<th>Slope</th>
<th>Y-int</th>
</tr>
</thead>
<tbody>
<tr>
<td>Left</td>
<td>1</td>
<td>0.12</td>
<td>-0.09</td>
</tr>
<tr>
<td>Left</td>
<td>2</td>
<td>0.054</td>
<td>-0.04</td>
</tr>
<tr>
<td>Left</td>
<td>3</td>
<td>0.033</td>
<td>-0.03</td>
</tr>
<tr>
<td>Left</td>
<td>1,2</td>
<td>0.179</td>
<td>-0.13</td>
</tr>
<tr>
<td>Left</td>
<td>1,3</td>
<td>0.158</td>
<td>-0.12</td>
</tr>
<tr>
<td>Left</td>
<td>2,3</td>
<td>0.088</td>
<td>-0.06</td>
</tr>
<tr>
<td>Left</td>
<td>1,2,3</td>
<td>0.223</td>
<td>-0.17</td>
</tr>
<tr>
<td>Right</td>
<td>1</td>
<td>0.032</td>
<td>-0.03</td>
</tr>
<tr>
<td>Right</td>
<td>2</td>
<td>0.053</td>
<td>-0.04</td>
</tr>
<tr>
<td>Right</td>
<td>3</td>
<td>0.120</td>
<td>-0.09</td>
</tr>
<tr>
<td>Right</td>
<td>1,2</td>
<td>0.087</td>
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</tr>
<tr>
<td>Right</td>
<td>1,3</td>
<td>0.158</td>
<td>-0.13</td>
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<tr>
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<td>Right</td>
<td>1,2,3</td>
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<td>-0.18</td>
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