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Continuous Segregation and Removal of Biochar from Bubbling Fluidized Bed

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A thesis submitted in partial fulfillment of the requirements for the degree in Master of Engineering Science

CONTINUOUS SEPARATION AND REMOVAL OF BIOCHAR IN A LAB SCALE BUBBLING FLUIDIZED BED PYROLYZER.

(Thesis format: Monograph)

By

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Abstract

Biochar is a valuable co-product with increasing agronomic and environmental values produced during pyrolysis, which is a thermochemical conversion process of biomass in the absence of oxygen. Accumulation of biochar in the bed may result in poor fluidization performance and product quality. The efficient removal of the separated biochar from the fluidized bed is therefore crucial for economical reasons.

This thesis is structured in three sections. Section 1, provides an introduction and literature review on biomass pyrolysis covering the main types of processes and reactors focusing on fluidized bed technologies. It section addresses the mechanisms associated with mixing and separation processes in fluidized beds are briefly discussed. The significance of biochar recovery is also emphasized and the various current recovery methods are reviewed and discussed. Section 2 describes the methodology, the operating conditions and the variables investigated and measured in this work to generate the experimental data and ensure process reproducibility and accuracy experimental findings. Following this section, the section 3, addresses the experimental results and analysis leading to the conclusions and recommendations for future research directions.

The primary objective of this research is to describe, through experimental investigations, the effects of geometrical and operating parameters on the separation efficiency and the yield of the recovered biochar in a cold simulator laboratory scale bubbling fluidized bed. A section of the bed simulates the pyrolysis reactor bed, is fluidized at high gas velocity to provide intense mixing, and a second section operates at a lower gas velocity to promote particle separation. Biochar is continuously fed to the well mixed zone to simulate the production in the reactor bed. An automated pulsating feeding inlet regulated by sleeve valves allowed continuous feeding of biochar while overflow ports in the separation section allowed for the continuous discharge of the biochar.

The lab scale bubbling fluidized bed unit, made it feasible for continuous segregation and removal of biochar at an optimized separation efficiency range of 96.40 % to 98 % while operating at vigorous bubbling conditions, optimizing the separation efficiency of the biochar solids was necessary at a desirable superficial gas velocity along with the best fit submergence of the vertical baffle plate height (P_h) above the bed in preventing back mixing and bubbles from the segregation zone preventing biochar accumulation into the well mixed zone which might if not addressed result into defluidization quality in pyrolysis units.

These research experimental analyses produced better results with improved separation efficiency of 92 % unlike 80 % separation efficiency achieved in previous conducted experiment. In addition, the elutriation encountered in earlier experiments accounted for > 20 % fraction of the biochar fed, while the presently conducted and investigated findings successfully documented elutriation of < 3 % fraction of the biochar fed into the lab scale bubbling fluidized bed unit.

Following modifications of the bubbling fluidized bed pyrolyzer, the unit proved efficient in handling range of feed rates and also capable of processing different particle sizes attaining very high separation efficiency and stability (yield) states. Further optimization and adaptation for large industrial applications can be achieved through further research investigations and recommendations as suggested below.

Keywords: Fluidization, Biochar, Pyrolysis, Bubbling Fluidized Bed, Biomass, Segregation, Yield, Layer Inversion, Binary mixture, Elutriation, Gas velocity, Separation Efficiency

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Above all, I give glory to God, the beginning and the end of all things, the source of all wisdom and strength for seeing me through the journey of starting and completing my study at Western University.

Dedication

In loving and evergreen memory of my dearly beloved brother **Emmanuel Olusegun Awosolu**, and my Sweet Dad, **Barrister Christopher Olutosin Awosolu (S.A.N.)**
May their Gentle Souls Rest in Perfect Peace.

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ρ_f - fluidization gas density (kg/m^3)

ρ_p - particle density (kg/m^3)

$F_{biochar}$ - feedrate of biochar (kg/min)

U_{fs}- superficial gas velocity (m/s)

μ- viscosity of fluidization gas (Pa.s)

H_p - height of plate above bottom of bed (cm)

d_p - particle diameter (μm)

A_{bed} - area of fluidized bed (m^2)

U_{wm} - superficial gas velocity in well mixed zone (m/s)

U_{seg} - superficial gas velocity (m/s)

U_{mf} - minimum fluidization gas velocity of pure sand (m/s)

$A_{bed,pyrolysis}$ - bed area of pyrolysis unit (m^2)

A_{wm} - area of well mixed zone (m^2)

A_{sep} - area of separation zone (m^2)

VM - Volatile Matter

HHV - High Heating Values

FC - Fixed Carbon

M - Kramer Mixing Index

c – Average mass concentration

σ – Variance of the mixture

n - Total sample numbe

CHAPTER 1.

1. INTRODUCTION AND LITERATURE REVIEW

The dwindling supply of petrochemical resources and the rising environmental problems associated with emissions of greenhouse gases and other environmentally unfriendly gases like sulphur dioxide and nitrous oxide are perpetuating the quest for alternative, sustainable and clean alternative resources for the production of chemicals, fuels and energy. In recent years, as this quest for substituting the current chemical energy extraction platform is gaining momentum, production of energy and chemicals from biomass is gradually being seen as having the potential for shifting the paradigm from the traditional fossil route to a renewable, second generation production opportunity.

Biomass is an organic matter with trapped energy predominantly. This trapped energy can be released, exploited or converted into clean fuel, and high valued products. Accounting for about one third of the world-wide primary clean fuel production is biomass stream and for effective and maximum utilization of these energy streams, modern technological breakthroughs and approaches are essential in the conversion of biomass to energy. Biomass can be obtained from different sources and the inherent attributes of each biomass source influences the type of conversion process and processing challenges that may result.

Biomass can be classified as energy crops, non-food lignocelluloses based, woody, forestry and agricultural residues as well as organic animal matter and manures. The utilization of biomass derived from agricultural and forestry residues or waste stream through thermal cracking processes has the potential to mitigate waste management problem and also to produce clean energy, fuels, and chemical products. There are many potential drivers that make the applications of biomass immense summed up as:

- Climate mitigation: The high levels of greenhouse gases emission constitute potential threat influencing climate change, and a s a result biomass has turned out to be a major potential for renewable energy production.

- Economical high conversion technologies create a stimulus in the application of biomass for environmentally friendly fuel-energy generation and value chemicals.
- Agricultural policy changes, subsidies and bills in favor of Agricultural Land Use Acts offers a means of reducing the emission of green house gases by sources generating them.

1. 1. Biomass Conversion Technologies

Several technological pathways are now under development in which high value products and energy as well as fuels can be extracted from biomass. Increasingly accepted biomass conversion technologies include biochemical, physical and thermochemical conversions.

Biochemical conversion involves the application of enzymes and microorganisms mainly bacteria, and fungi in the treatment and conversion of biomass and organic wastes. Microbial principle of fermentation (anaerobic or aerobic) is used in the production of high value products, clean energy and fuels such as biogas and biodiesel.

Physical process involves mechanical extraction, and drying processes for high moisture containing biomasses so that they can be compressed into uniform sizes for the yield of high value products.

Thermochemical biomass conversion technology utilizes heat in the rapid degradation of biomasses to produce condensable gases, liquid and solids. Thermochemical processes further involve three technologies: biomass combustion, pyrolysis and gasification, all technologies requiring the use of fluidized beds to enhance efficient mixing between particles and gases[2]. The thermochemical processes: pyrolysis, gasification and combustion have been deemed as the most promising biomass conversion technology. Pyrolytic conversion technology utilizes heat in the presence of trace, or in the total absence, of oxygen for the degradation of biomass in producing condensable gases, liquid and solids.

Several researchers determined that biomass conversion technologies are greatly influenced by the high variability in the feedstock's composition, particle size, density, moisture contents to mention a few. Solid biomass combustion has limited applications for energy production owing to the low energy density, high bulk volume and handling cost and significant production of pollutants. Therefore, pyrolysis and gasification are promoted as the most efficient and promising conversion processes of solid biomass into value added products[3]. Pyrolysis however, has been of increased interest with several advancements in the process and operating conditions to maximize the yields of gases, chars that can be utilized in form of briquettes or applied in purification processes in form of activated carbon.

1.2. Biomass Pyrolysis: Products Applications and Reactor Types

The most promising technology applied in biomass utilization and conversion into derivatives of high value is thermal processing known as pyrolysis[4]. Pyrolysis is a thermochemical conversion technology, which in the absence or in trace amount of oxygen generates three major products: bio-oil (liquid), biochar (solid) and gases [5]. The proportion of these products depends on several factors including the process parameters and composition of the feedstock. Biomass pyrolysis is typically carried out at temperatures ranging from 450 °C to 600 °C with condensable vapors (i.e. liquid bio-oil), solid residues (biochar) and non-condensable gases products. When the pyrolysis temperature is at a very high temperature, the bio-oil yield is optimized constituting about 60-70 wt% yield from a typical biomass feedstock, with 15-25 wt% yields of bio-char while 10-15 wt% is syngas under fast pyrolysis conditions.

Bio oil has numerous significances including the following:

1). Bio oil is a renewable fuel.
2). Bio-oil is the dark brown free flowing liquid composed of complex mixture of oxygenated compounds can be stored and utilized as chemical for industrial purposes.
3). Bio oil generates reduced NOx emissions in gas turbines than light fuels and diesel fuel.

In spite of the significance of bio oil, there are also limitations to the wide use of this biomass pyrolytic product. In comparison to petroleum-based fuels, bio oil contains oxygen and water which reduces its ability to mix with other hydrocarbons. Furthermore, storage of bio oil on a long term basis is a challenge due to corrosion. Bio oil has low pH coming from organic acids within the degradation of biomass. With low pH, bio oil corrodes steels and can only be stored in containers that are resistant to corrosion like stainless steel.

Biochar is an organic carbon-rich by-product derived from the thermal cracking of biomass (woody crops, agricultural wastes, wood wastes, industrial residues, municipal solid wastes, food and organic wastes), has the following fundamental attributes of

- Enhancement of soil water and increased agricultural productivity
- Carbon sequestration and simultaneous production of Bioenergy, all which forms the platform for Biochar production through Pyrolytic process.

Several biomass can be used for pyrolytic processes, which can be of industrial residues, municipal wastes forestry or woody origin, agricultural energy crops as shown in Figure1.2.

Figure 1.1: Typical biomass of agricultural residues, forestry or woody origin used for pyrolysis

Pyrolysis may be a self sustaining thermochemical process such that the combustion of one or combination of the products provides adequate heat for the reaction. Figure 1.3 is a schematic representation of the pyrolysis process with the three major products being produced. The biochar produced from pyrolytic process offers positive effects in increasing agricultural productivity, carbon sequestration soil water enhancement and simultaneous production of energy. The pyrolysis of biomass is rapidly developing and could play a significant role in the future development of renewable energy production. Generally, a pyrolysis unit also known as the pyrolysis reactor specifically consists of a designed biomass pre-processing unit and processing downstream equipment.

1.3. Biomass Pyrolysis: Reactor Types

Pyrolysis reactors can be classified as either units targeted to produce biochar and bio-oils under fast pyrolysis operations or slow pyrolysis unit that produce biochar and heat as illustrated in Figure 1.3. Biomass pyrolysis consists of combination of a reactor, operating conditions and one or several analytical tools. Influencing the kinetics and mechanism of pyrolytic reactions, are several variables which include the bed temperature, ambient atmosphere present, substrate composition, heating rate and use of catalysts all which are relevant to understand profound effects on product yields formed.

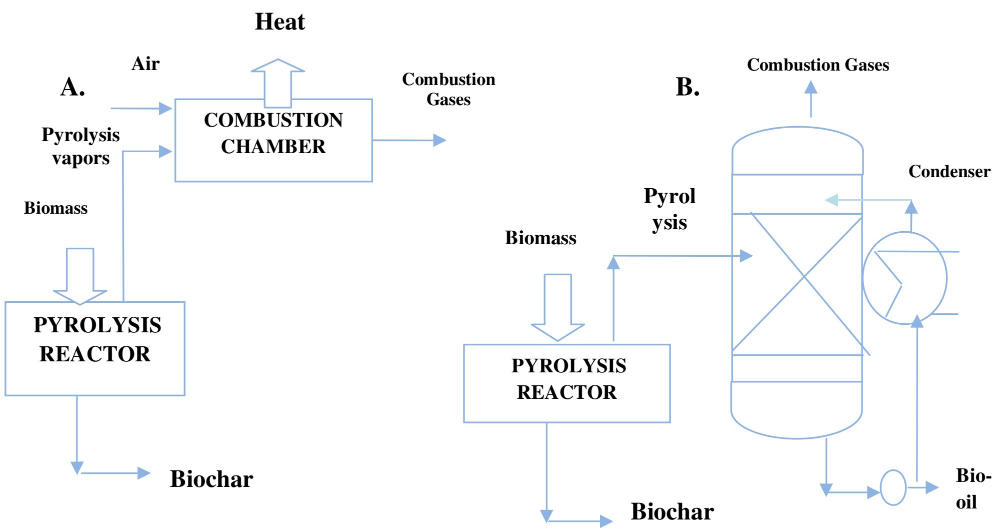

Figure 1.2. Pyrolysis Reactor for A) Biochar and Heat. B). Biochar, Bio-oil and Gases[3]

Relative to the operating conditions such as temperature, volatile residence times and heating rates, pyrolysis process can be classified into three subdivisions depending on operating conditions: flash pyrolysis, fast pyrolysis and flash pyrolysis. Based on the surrounding medium, pyrolysis can also be differentiated as catalytic pyrolysis, steam pyrolysis, vacuum pyrolysis, hydro pyrolysis or oxidative pyrolysis. Illustrated in Table 1.2., are the arrays of essential operating variables for pyrolysis processes.

In slow pyrolysis, there is higher char yields compared to liquid and gaseous products as a result of slow heating rate with varying vapor residence time between 5 minutes to 30 minutes. Slow Pyrolysis has been primarily used for charcoal production for several years. The long residence reactor time result in possibility of the gas phase yields to form char as a result of reaction with other products[6]. Fast pyrolysis thermochemical conversion technology adaptable for producing gases or liquid pyrolytic products and much improved than slow pyrolysis; however the liquid product converts to unwanted coke as a result of the rapid heating rates involving biomass compounds that are thermally unstable.

The heating rates for fast pyrolysis are approximately about 10 ˚C/s – 200 ˚C/s. Figure 1.4 is a schematic illustration of a fast pyrolysis process. Generally depending on the feedstock utilized, a fast pyrolysis processes yield 15 – 25 wt% of solid char, 10 – 20 wt% of noncondensable gases, and 60 – 75 wt% of liquid bio-oil[7, 8, 9].

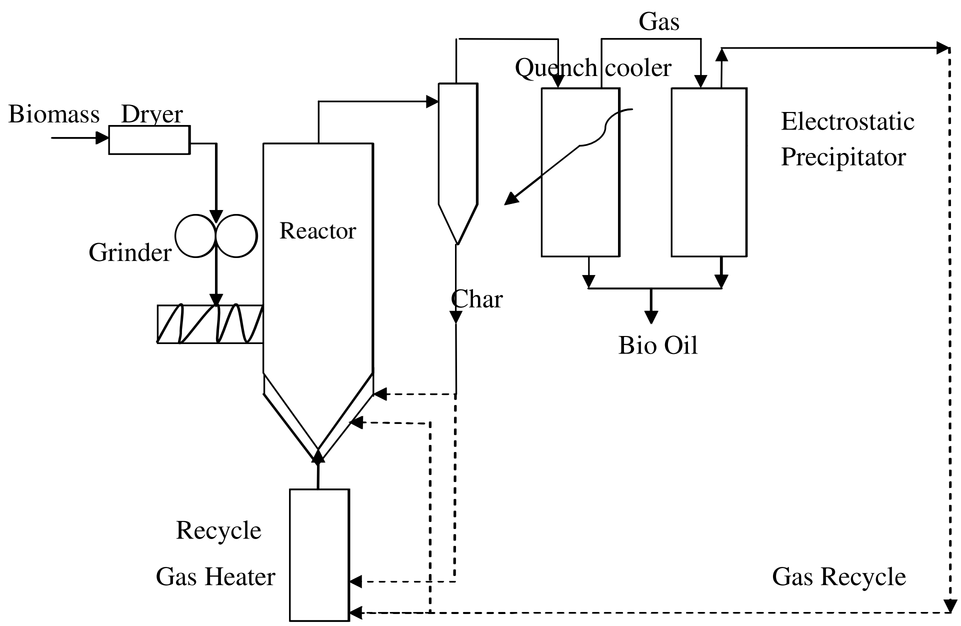

Figure 1.3. Conceptual Representation of a Fast Pyrolysis Process

Flash pyrolysis is an advanced method of fast pyrolysis involving very high heating rates. In other to achieve improved yields with the abrupt reaction times and spontaneous heating rates mode of operation, smaller particle size is required unlike other processes. Typically examples of flash pyrolysis reactors include vacuum pyrolysis reactor, ablative pyrolysis reactor, entrained flow reactor, blade or vortex reactor, twin screw reactors and fluidized bed pyrolytic reactors, with reaction times of few to several seconds. Among these numerous flash pyrolysis reactors, fluidized beds and entrained flow and fluidized-bed reactors are best preferred and most widely used in flash pyrolysis. Current reactors for flash pyrolysis include fluidized bed reactors, vacuum pyrolysis reactor, rotating cone reactor, entrained flow reactor, ablative, vortex or blade, twin screw reactors[10, 11]. Consequent on several advancements, the most feasible and practical pyrolysis technology preference is fast or flash pyrolysis at very short residence times and high temperatures. Recent developments on pyrolysis reactors have focused on designing essential features for successful reactor performances.

Fixed bed reactors have been widely used as for relatively uniform sized biomass particles, simply reliable and conventionally used for charcoal production. The fixed bed pyrolyzers conduct slow and poor heat transfer resulting in very low liquid fractions. Fixed bed reactors can be wither down draft fixed bed reactors or updraft fixed bed reactors, each of these classes of fixed bed reactors have peculiar mode of operations. In updraft fixed bed pyrolyzer, there is upward counter current flowing product gas stream that comes in contact with fast downward solid moving component in a vertical shaft [12, 13].There is a high level tar formation and a very dirty product gas stream; however tar crackers have been designed over the years for mitigation. In contrast, the downdraft fixed bed pyrolyzers, there is concurrent downward movement of both product gas and solid producing low tar with accompanying high carbon conversions and the gas is relatively clean. The solid and product gas move downward in co-current mode. A relatively clean gas is produced with low tar and usually with high carbon conversion.

In circulating fluidized bed reactors, the residence time for the vapors and gas is relatively the same for char. It is an established technology in which a hot circulating fluidized bed of hot sand, the biomass particles are introduced in circulating bed reactor units in which the hot sand, biomass particles and recirculated gas product all move together. As a result of the high transfer heat rates from the sand, there is rapid heating of biomass particles. Erosion or ablation inside the unit vessel is more typically persistent than with general fluidized beds [14, 15, 16, 17, 18]. Other limitations include the difficulty for catalytically operating process to be attained. In circulating fluidized bed reactors, increased gas velocities cause more spontaneous attrition of the char creating relatively high char proportions; the use of a cyclone generally separates the char contents generated. The Circulating Fluidized Bed operation array is fixed over that entrainment velocity. Above this velocity the bed material becomes entrained and with distribution of solids throughout the furnace, gradually decreasing density from the bottom to the top of the furnace. Unique attribute of circulating fluidized bed reactors is the good temperature control [19, 20, 21, 22, 23, 24, 25, 26, 27].

Schematic set up of circulating fluidized bed is represented in Figure 1.5 below.

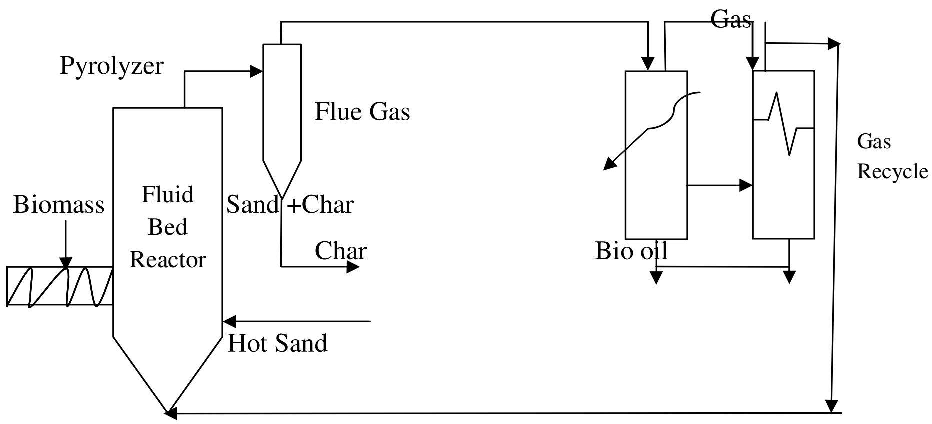

Figure 1.4. Schematic Circulating Fluidized Bed.

1.4. Bubbling Bed Fluidized Pyrolyzer and Applications

Bubbling fluidized beds have numerous advantages that justify their use as an acceptable technology within a pyrolysis process. Figure 1.5 is a general illustration of typical bubbling fluidized bed. Bubbling fluidized beds (BFB) is often preferred in small-scale applications, with biomasses having low heat value and high moisture content. For a reliable operation, the bubbling fluidized bed provides uniform and good mixing and heat distribution in the bed enhancing good fluidization performance[28, 29, 30, 31].

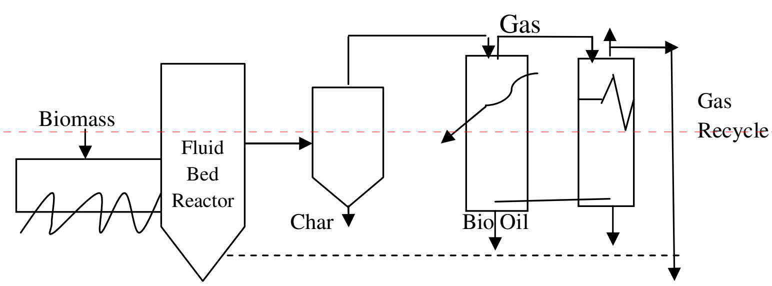

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Figure 1.5. Schematic Bubbling Fluid Bed

1.5. Biochar Recovery: Challenges and Opportunities

A crucial need during pyrolysis, especially fast pyrolysis, is separating the solid char immediately and totally from the vapors due to the fact that the solid by-product char acts as vapor cracking catalyst with possibility of significantly reducing the yields of liquid bio-oil. In addition, biochar is a valuable co-product that needs to be separated from the sand, which is usually utilized as inert bed material in pyrolysis reactors, and, consequently commercialized. Entrainment and elutriation followed by one or more cyclonic separation devices is basically the general char separation process when very fine char particles are produced. As a result, it is important to understand the hydrodynamics of the mixtures of sand-char and of sand-biomass.

Excessive biochar concentration in the bubbling fluidized bed leads to unpredictable and unstable fluidization resulting into poor mass and heat transfer. Therefore, designing a reliable, cost effective, and pilot scale bubbling fluidized bed reliable reactor on a pilot scale that operates in continuous mode is highly desirable. In addition to these features mentioned, such reactor system should have the potential capacity for processing several biomasses when fed into the bubbling zone and practical feasibility of segregating, removing and recovering biochar and other pyrolytic products independently from the bubbling fluidized reactor[32, 33]

Biochar acts as a vapor catalyst cracker, therefore the effective and rapid biochar removal from fluidized bed reactor is essential and highly desirable. Excessive biochar accumulation cracks vapors and diminishes yield to approximately 20%. Accumulation of biochar impacts the fluidization efficiency as well as the pyrolysis reactions and the quality and yield of the products. Consequent on these practical limitations, in order to attain optimal fluidization steady state and process performance and to recover the valuable biochar co-product, it is advisable that the concentration of biochar in the bed be maintained at low levels. Cyclone integration is very important in the designing of fluidized beds to achieve biochar removal from the product gases.

However, it should be noted that except filters are immediately in operation after using the cyclones, the bio-oil will erode and plug the channels. Cyclonic char removal processes are generally employed in char removal. The removal of fine particles from fluidized bed pyrolysis units is mainly achieved through elutriation of the fine particles.[34]

Elutriation is a process which involves fine particles that are carried out of a fluidized bed due to the inflowing gas stream through the bed. In bubbling fluidized bed pyrolytic units, elutriation is a major contributing factor of inefficiency encountered in applications of fluidizing bed technique when the fluidizing gas velocity exceeds the terminal velocity of the finer flotsam bed particles, although at lower gas velocities, elutriation can also occur. Elutriation of fines from binary particle fluidized bed is feasible through the efficient design of the fluidized bed with knowledge of the hydrodynamic behavior of the particles in the free board and subsequent elutriation variables from the fluidized bed [35]. In fluidizing systems designed mainly to purify granulated materials from the fines, maximum elutriation is essential and highly desirable.

However, when expensive solid particles are involved such as powdered particles, a high degree of elutriation is practically undesirable. The entrained fine flotsam particles present in the mechanical attrition of large particles or initial size particle of the feedstock as fed into the bed are separated and collected from the high velocity fluidizing gas stream using separating devices such as cyclones, hydrocyclones, filter bags or scrubbers.

In relation to fluidized bed application, elutriation operates for the removal of fine biochar particles at terminal velocity much lower than fluidization velocity, which is the superficial minimum gas velocity for removing a known particle size, density, sphericity and shape. According to experimental findings by some researchers, reported the removal of biochar with range of particle size distribution as shown in the Table 1.4 below, no data on the particle density is however reported. In addition, hot filter traps particles > 1µm and cyclones can only remove biochar particles >10µm.

Table 1.1. Elutriable Biochar Particle size distribution Mullen et al. (2010)

Particle size distribution of bio-chars (µm).							
	Mean	Std	<10%	<25%	<50%	<75%	<90%
Corn cob bio-char	708.8	513.3	71.58	265.5	651.3	1043	1479
Corn stover bio-char	373.6	420.9	30.35	93.69	212.3	477.8	1008

The hydrodynamics parameters of the fluidized bed, such as excess gas velocity above the minimum fluidization velocity, bubble diameter and bed heights can influence the rate of elutriation of ejected particles at the fluidized bed surface. Experimental studies conducted by some researchers, validated that fine particles cluster with other particles in the bed for example silica sand (when present in the binary mixture) and into large coarse particles, thereby rendering elutriation impracticable. As particle size becomes increasing smaller, elutriation rates progressively increases and as a result, smaller particles are favourably elutriated compared with larger particles of the same densities. Although elutriation can be applicable to selective removal of fine particles from fluidized beds in general cases, it does not offer a practical solution for the complete removal of larger biochar particles during pyrolysis reactions.

In fact, it is desirable to pyrolyze biomass feedstock particles that are not excessively small, to minimize the grinding costs as well as to produce biochar particles that are easier handling in order to utilize them in most of the practical applications mentioned earlier. In the case of relatively coarse biochar particles, the most practical biochar recovery process could be the segregation coupled with an efficient removal system. This removal system won't be practicable if cyclones can be used to capture and efficiently collect the particles.

Process intensification involving efficient high selectivity of operating parameters and cost effective procedures are continuously mandatory for efficient pilot scale process conversions. A steady continuous system has numerous advantages compared to batch systems associated with energy losses, periodically shutdowns, unstable inconsistent operation conditions and reduced yields. Non-uniform and varying concentration of biochar in the bed can result in excessive accumulation of biochar in the bed which invariably influences the unsteady fluidization operations resulting in decreased heat and mass transfer. It is therefore highly essential that designing a cost effective fluidized bed pyrolysis unit with potential feasibility of continuous industrial operation with capacity of handling wide range of feedstocks and effective simultaneous removal of biochar from other pyrolytic by-products is inevitable.

1.6. Fluidized Bed Technologies (Mixing and Segregation Phenomena)

This review deals with the state-of-the-art fluidized beds that have been extensively researched in attempts to improve various industrial and chemical processes that depend on their applications[36]. Fluidized beds have a key advantage of promoting unique, uniform mixing of particles, which allows for continuous processing at very low temperature gradients for large and small scale operations. Fluidized beds also provide a method for particle separation based on density difference and constitute an established technology for particulate processing. The most important phenomenon that makes fluidization unit operation practicable is that the fluid-like behaviour of solids facilitates easy and rapid transportation ability with direct gas contact. However, there are several stages that involve the bed undergoing fluidization process[37, 38].

Fluidization is a well-known industrial mechanism for mixing and segregation of binary mixture of particles. It is a process in which solid particles achieve a fluid-like behaviour while suspending them in a gas or a liquid upwards through a solid-filled reactor vessel. It involves fluidized beds, which provide uniform mixing of feedstock with heat carrier solids, and good transfer of heat and mass. Fluidization mechanism operates on contact between a mixture of solid materials and a fluid stream with process variations. In depth understanding of the mixing and segregation operations in fluidized beds are of particular relevance to related industrial applications that utilizes fluidization. In fluidized bed, the segregation pattern of a binary mixture is a very critical factor which influences mass and heat transfer properties, bed expansion, and process parameters in the fluidized bed.

Fluidized beds have their applications in metallurgical, pharmaceutical, food and chemical related industries [39]. Gas velocity strongly affects segregation phenomenon as the bubbles create an intricate effect strongly influencing the varying mixing and segregation phenomena in fluidized beds[40]. While quantifying degree of mixing using the mixing index, final degree of mixing is quantified by the segregation index. Mixing and segregation sequentially take place together often overlapping each other. In attempts to quantify the various mixing indexes, there are documented literatures developed to quantify the mixing degree. A reference example is Kramer's mixing index calculated as

$$M = \frac{\sigma_0^2 - \sigma^2}{\sigma_0^2 - \sigma_r^2} \qquad \mathbf{1}$$

in which, σ^2_r measures the variance of the mixture close to perfectly mixed state, σ^2 expresses the variance at actual mixed phase and $\boldsymbol{\sigma_0^2}$ indicates at totally segregated state the variance of the biomass particle concentration.

However, σ can be deduced by the mathematic representation

$$\sigma = \sqrt{\sum_{i=1}^{n} \frac{cb_i - \overline{c}}{n-1}} \qquad \mathbf{2}$$

where, c measures the average mass concentration, and n is the total sample number. At totally segregated state, the mixing index is zero when $\sigma^2 = \sigma_0^2$, and at perfectly mixed state, M = 1 when $\sigma^2 = \sigma^2_r$. It important to know that decreasing segregation, gives increasing potential to mixing index with increasing homogenity. According to Gyenis and Zhang et.al., at a mixing index of zero, a complete segregation state is achieved[41].

Although, Marzocchella et al. did not apply statistical correlation analyzing mixing index of their experimental findings, instead adopted a correlation in determining the absolute bed. The distinctive attribute of a binary fluidization is particle suspension characterized as either size segregating or density segregating. The overall efficiency of fluidization of binary mixtures is been influenced by segregation tendency of the component of the binary mixtures. Binary mixtures of solid particles of varying sizes and or densities tend to segregate in vertical condition under fluidized conditions. Uneven distribution of dissimilar solid particles in fluidized beds results into segregation phenomenon as shown in Figure 1.6.

Comprehensive understanding of segregation dynamics is a prerequisite for proper optimisation of fluidized beds of binary mixtures. General phenomenon associated with the mixture of dissimilar solids is the ability of such solids to segregate which is primarily due to particles differing in densities or sizes. Practical investigation reported that density ratio has been shown to have a greater influence than the size ratio. [42, 43, 44, 45, 46]. In a freely bubbling fluidized bed, the superficial gas velocity sensitively influences the segregation rate of binary mixture of particles, and is at its peak when gas fluidization velocity is between the minimum fluidization velocities of the flotsam and jetsam.

Separation efficiency (%) can be influenced by altering the fluidization conditions, installing bed internals or changing the particles characteristics. A practical example is the use of sieve-like baffles in optimizing segregation potential in fluidized bed. Larger bubbles break into smaller bubbles up by the sieve like baffles and these smaller bubbles create a mixing phenomenon with the feasibility of conveying to the bed surface the flotsam light particles. The segregation phenomenon that typifies the fluidization pattern of binary mixtures is based on the effect and action of gas bubbles that are generated immediately as the bed leaves the packed state.

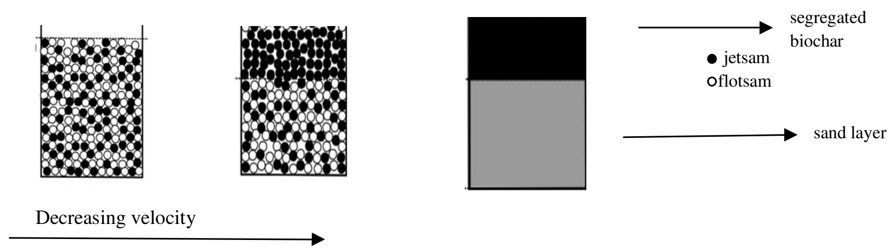


Figure 1.6. Representation of mixing and segregation processes of dissimilar solids

Regardless of the extensive literature findings on the mixing and segregation phenomena of dissimilar binary mixture of solids in fluidized beds, still lacking is the comprehensive understanding of segregation phenomena dynamics. Several experimental data already exists in literature concerning fluidized bed and segregation of simple binary mixtures. Rowe et al. were the first to describe the terms flotsam and jetsam. Rowe et al., findings showed that one of the components of the binary mixture becomes selectively dragged upward in the bubble wake to form the upper layer of the bed and this component is referred to as flotsam.

Further, the second component, which is heavier, sinks to the bottom of the column, is called the jetsam. Rowe et al. conclusion, buttressed the suggestion by that the segregated components were consequence of the bubbling motion effect of the gas responsible for the segregation pattern [46]. Hoffmann et al. analyzed that particles conveyed by the bubble wakes to the top of the fluidized bed percolate back down under the action of gravity through the emulsion phase, then segregating out into flotsam and jetsam.[47].

Investigation on the effect of increasing velocity on segregating particles of differing densities, that is, particles of heterogeneous densities, there is a decreasing extent of segregation of the denser particles behaving as the jetsam. The criterion of separation of various particles can be best described on Reynolds number, which is a dimensionless number that gives a measure of the ratio of inertial forces to viscous forces and consequently quantifies the relative importance of these two types of forces for given flow conditions provided the fluidization occurs in a solid-fluid medium. Another criterion that can validate the separation of various particles as jetsam or flotsam is Archimedes principle, which is the upward buoyant force that is exerted on a body immersed in a fluid, whether fully or partially submerged, is equal to the weight of the fluid that the body displaces. On the other hand, particles with homogenous densities, there is tendency for the smaller particles segregating as flotsam [48, 49, 50.] The behaviour of segregation of particles consisting of different sizes, equal density was investigated by Wu et al.

Binary mixture with dissimilar size an equal density distribution in bubbling fluidized bed were studied by Marzocchella et al. tested the particle size distribution in the equal density and dissimilar size of binary mixture in a bubbling fluidized bed [51, 52]. Mixing and segregation behaviour of spherical solids in silica sand bubbling fluidized bed were studied by Manfred et al. [53]. In a sand fluidized bed that consists of biochar this constituting a binary mixture, the biochar pose few limitations in negatively influencing the heat transfer rates and invariably accumulation of biochar in the bed might eventually influence the fluidization overall efficiency owing to the excessive biochar build in the reactor and can contribute to increased sand losses with adverse effect of energy losses resulting from reheating the sand and sand recirculation the sand.

The excessive build of biochar resulting in its accumulation disrupting the system stability needs to be addressed. At first fixed bed state, the fluid operates at very low velocity, and void spaces are filled between the stationary particles and proceeds through the bed slowly. Increasing the low velocity rate, the particles tend to vibrate and move apart resulting in bed expansion. Generally, there are two types of fluidization systems which are both considered as dense phase fluidizing systems as the bed upper surface can be clearly defined: the solid-gas fluidizing systems and solid-liquid fluidizing systems.

These fluidizing systems have peculiar practical applications, advantages and disadvantages. Heat and mass transfer rates are relatively higher in gas-solid fluidizing systems. In addition, the fluid like particles transport makes it easy to handle and suitable for large scale operations[54]. In contrast, the gas-solid fluidizing systems, non-uniform residence times of the solids in the bed arise from the rapid mixing especially in bubbling fluidized beds and as a result solids particles become friable and easily entrained. The recirculation of the entrained solid particles is necessary to ensure the solid particles back into the system with resultant attrition effect. It is imperative to implement cost effective, operationally simple, set up that promotes and allow continuous operation by selectively removing from the bed the accumulated char during the pyrolysis process and avoiding or drastically minimizing sand removal from the Pyrolytic reactor. In addition, extended attributes of this set up should encompass its simplicity in optimization and versatility in handling different biomass.

Previous experimental findings performed by Mara et.al 2010 focused on effects of physical properties, geometry and bed configuration in analyzing the separation efficiency and yield of the continuous segregation of biochar particles from cold scale bubbling fluidized bed. However the findings by Mara et.al experimental results attained close to steady state and the good purity levels that need to be enhanced and improved. Based on these limitations, this experimental study was designed to improve both separation efficiency and biochar purity by analyzing the geometrical effects, physical properties and operating conditions and identifying their optimal values.

In order to achieve enhanced separation efficiency and shorter duration to attain steady state (yield), some modifications put in place in to the geometry, physical properties and bed configurations in the same bubbling fluidized bed used by ensuring the segmented sections totally prevents sand splashing from the well mixed zone from entering into the segregated regime affecting the purity levels, installing better automated feeding unit for controlled paced continuous feeding thereby minimizing error in feeding intervals and ensuring well fitted filter bag is mounted to trap elutriated biochar particles in form of dust emitting out thereby avoiding dust particles in the air.

This report is structured into three main sections. Section 1, provides an introduction and literature review on biomass pyrolysis covering the main types of process and reactors with some in-depth to fluidized bed technologies. In this section also, the mechanisms associated with mixing and segregation processes are briefly discussed. Review on the significance for biochar recovery is also emphasized and the various recovery methods briefly discussed. Section 2 describes the methodology and experimental procedures involved in the generating of data for the process along with reference examples. Following this section, section 3 is the final section that addresses the conclusions of the experimental investigations and suggested some recommendations for future research directions that can be explored. Continuous biochar removal is crucial in preventing poor fluidization quality that would result from high concentration of biochar in the sand bed. Where biochar is not continuously removed or segregated in a reactor, the fluidization quality of the biochar and the overall performance of the reactor is low affecting process cost and profitability.

1.7. Research Objectives.

There are numerous research findings on separation phenomenon, however there are lack of experimental findings on separation and removal of biochar from a binary mixture of biochar and sand in a continuously bubbling lab-scale fluidized bed. As a result of this insufficient data, this makes this research investigation of practical relevance and significance. The main objective of this study was the development of a practical removal system based on separation to recover larger, mm or cm sized particles of biochar from a bubbling fluidized bed pyrolyzer. Experimental investigations on the effects of geometrical and operating parameters (effects of separation zone area, rate of feed rates, effects of particle size, effects of plate height, and effect of the removal ports) on the separation efficiency of the removed biochar in a lab-scale bubbling fluidized bed were conducted.

A key feature in determining biochar separation efficiency stemmed from the quantitative feasibility in analyzing the separation efficiency attained under the operating conditions and parameters that ensured process reproducibility and accuracy. The modified lab-scale bubbling fluidized bed used in previous finding by Mara 2010, was remodified to meet better potentialities of simple and easy of operation with adjustment feasibility, minimal biochar accumulation biochar and reduced sand removal, and continuous mode operation in attaining steady state such that influx of biochar should be the same as the biochar removed from the system to prevent biochar accumulation.

The output that is the removed biochar recovered from the separation zone will be analyzed during the ongoing fluidization process. The mass fraction of biochar in these solids, which represents the purity of the removed solids, will be determined using sieving. Any removed sand will be recirculated back into the well mixed zone operating at a vigorously high gas velocity (that is, above minimum fluidization velocity) bubbling conditions during the continuous fluidization process. In achieving optimized biochar separation, it is very important to maintain gently bubbling conditions similar to laminar flow at much lower superficial gas velocities in the bubbling bed reactor.

This research presents the description of continuous biochar segregation and recovery in bubbling bed fluidized bed. This set up was developed for the continuous removal of biochar while investigating the effects of geometry, physical characteristics and operating conditions on the separation technology in determining the purity of the segregated biochar. The experimental investigations further aim to determine feasibility of achieving steady state with potential reproducibility of the process in attempt of attaining good accuracy with negligible deviations. In achieving optimized biochar separation, it is very important to maintain gently bubbling conditions occurrence at much lower superficial gas velocities in the bubbling bed reactor.

CHAPTER 2.

EXPERIMENTAL APPARATUS AND METHODOLOGY

2.1. Introduction.

In this section, the methodology and experimental set up are described. The main controllable process parameters and their effects on yields and separation efficiency are discussed. The main aim of these experimental findings is to explore the effects of geometrical variables, physical properties and operating parameters on the purity of segregated and biochar in a modified segmented cold simulator laboratory scale bubbling fluidized bed having a well mixed zone at high gas velocity with evidence of gas bubbles and vigorous mixing and segregation zone operating at a lower superficial gas velocity.

Extensive experimental runs were conducted to determine the practical feasibility and probable reproducibility conditions in ensuring consistent purity levels under all operating parameters and variables with negligible deviations. The characteristics, behaviour and dynamic parameters of the apparatus (the cold lab-scale bubbling fluidized bed reactor) will influence the geometrical, physical and operating parameters effects on the purity of the segregated biochar. The essential characteristics required for the apparatus must ensure that simultaneous mixing and segregation phenomena are feasible in continuous operation at ambient temperature and atmospheric pressure.

Desirable gas velocities are crucial parameters required to facilitate well mixed state and segregation conditions that is, achieving good mixing between the solids and the suspending gas in order to measure the variable effects mentioned above. Interesting characteristic feature mandatory for the apparatus is the potentials for allowing extremely high surface area contact between the sand and biochar particles in the well mixed zone within very short interval following the onset fluidizing gas flow. Another remarkable characteristic for the apparatus centers handling capacities and adaptabilities for wide range and large quantities of feedstocks and sand to maximize profit within very short period. On the scale of profitability, the most important characteristic of the bubbling fluidized bed reactor builds on simplicity in construction with maintenance and potentials for upgrading. Installation of features to prevent loss of inert sand and trap the release of excessive dust load must aligned with the attributes of the cold simulator lab-scale bubbling fluidized bed.

2.1.1. Plan of Experiments.

The experimental runs were all conducted in a segmented bubbling fluidized bed unit to facilitate enhanced biochar recovery method. The fluidization column measures approximately 0.14 m high, and with a square cross section of 0.04 m^2 (0.20 m x 0.20 m). The cold simulator lab-scale fluidized reactor operates simultaneously and synergistically serial combination of two processes of mixing followed by segregation. Preliminary Experiments: In this experimental study, two different gas distributors were employed and preliminary experiments were conducted for the selection of superficial gas velocities in each section of the segmented fluidizing reactor.

It was ensured that in all the experimental runs, steady states conditions were attained and purity levels determined. This work was done under a lab-scale designed fluidized bed unit composed of two sections fluidized at different velocities which are separated from each other by a vertical baffle**.** One section simulated the pyrolysis reactor bed, and it was fluidized at high gas velocity to provide intense solid mixing, whereas, the second section operates at a lower gas velocity to promote particle segregation. A vertical baffle divided the two zones from each other so that two different operating conditions could be set into action and contents prevented from mixing with each other.

Trial and error attempts were made to determine the numerical factor of the well mixed gas velocity relative to the segregation velocity, that is the ratio of the numerical exponent of the well mixed velocity to the segregation velocity which is slightly above minimum fluidization velocity deduced as (U_{wm}= 2.5U_{seg}), as well as the numerical factor to the minimum fluidization velocity and it was deduced that the U_{wm}= 3.2$U_{mf.}$ The well mixed velocity is a combination of the fluidizing gas flow through the spargers in addition with the gas flow through the porous distributor plates. Findings and analyses of tested variables are presented and discussed later in this chapter to support the criterion for continuous separation of biochar in binary mixture with sand with references made to purity.

2.2. Materials.

The feedstock used in this research consists of milled wood charcoal in form of briquettes initially purchased in large bags from Lowes stores. The wood charcoal was milled within the size range of 850 µm, 1400 µm, 2000 µm, and 2300 µm respectively. The sand constitutes the inert component of the binary mixture used and has a density of 2650 kg/m^3. Table 2.1 summarizes the physical properties of each material. The physical appearances of the individual solid particles used in this experimental study are shown in Figure 2.1.

Samples of the solids used in this study (a) Sand particles A (white) (b) Biochar particles B (black)

Figure 2.1. Solid components in the Binary Mixture used in this study.

Parameters	Component A: Coarse Silica Sand	Component B: Black Biochar
Mean particle diameter d_p (µm)	200	850, 1400, 2000, 2300
Particle density ρ_p (kg/m^3)	2650	250
Geldart's size classification	B	B

Table 2.1. Physical Parameters of the Binary Mixture Components used in this study.

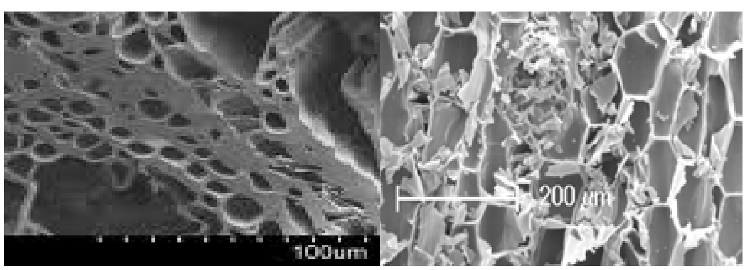


Figure 2.3. Micrograph of biochar used at 100x magnification measuring 100 µm and 200 µm.

2. 3. Equipment set-up

In an attempt to achieve low vapour residence times, high heat transfer rates and good mixing of hot sand particles and biomass particles, a bubbling fluidized bed pyrolyzer unit has to operate under vigorous mixing conditions. Unlike fluidization to obtain a good mixing achieved at high gas velocity, biochar - sand binary mixture segregation demands smooth gentle bubbling operating at reduced gas velocities slightly just above the binary mixture minimum fluidization velocity. The bubbling fluidized bed used in this experiment was initially designed by Mara 2010; with a square area of 0.04 m^2 having 3 drilled overports 0.025 m width on a side of the fluidized bed positioned at 0.2 m distance above the bed. Regulated by a voltmeter and a calibrated 3 mm diameter calibrated sonic nozzle by Omega PX181 pressure transducer, is the fluidizing gas streaming through a porous polypropylene gas distributor.

Elutriated biochar fine particles are captured from the existing air from the fluidizing column when passed through a filter fabric bag. Figure 2.3 and Figure 2.4 below shows the schematic illustration of the previously designed fluidized bed, comprising of two zones: a well mixed zone operating at highly bubbling vigorous velocity and a segregation zone operating with a gas velocity slightly above the minimum fluidization velocity. In order to achieve a good and improved segregation of the biochar particles from the bubbling fluidized lab-scale reactor, the previously described two zoned fluidized bed is modified to improve and enhance better overall purity and efficiency. The elutriated fines from the bed exiting the fluidization columns are captured in a fabric filter bag.

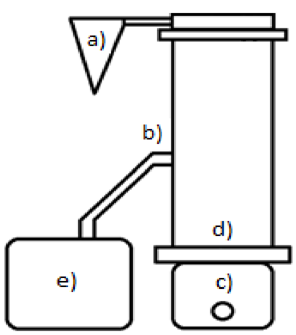


Figure 2. Fluidized bed used for segregation and removal experiments: a) filter bag b) overflow ports c) windbox d) porous plate distributor e) solids collection container (Adapted from Mara et al., 2010)

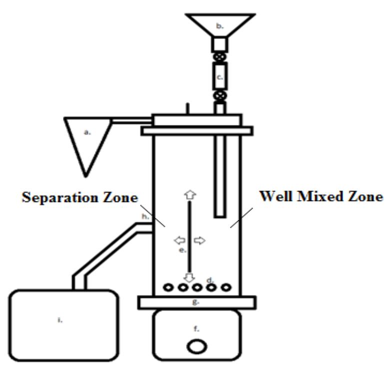


Figure 1. Novel fluidized bed set up used for continuous biochar separation. A) filter bag, b) solids hopper, c) biochar feed system, d) sparger distributor e) movable vertical plate f) wind box g) porous plate distributor h) removal ports, i) solids

A schematic diagram and set up of the lab-scale cold fluidized unit designed for this experimental investigation is shown in captured image in Figure 2.5. The lab scale bubbling fluidized bed reactor system used in all the experimental investigations pictured on this page includes the following key components. This reactor provides

- Automated pulsating feeding system or inlet for easy loading and removing of biomass (feedstocks or solids) of interests. This is advantageous when the solids bed must be removed and replaced frequently. This controlled automated feeding enhances accurate timely continuous feeding of the biochar reducing time lapse errors.

- Segmented sections that promotes continuous vigorously bubbling well mixing zone at high gas velocity and fluid like segregation phase at slightly above minimum fluidization velocity.

- Filter bag install to capture biochar fines resulting from particle attrition due to high gas velocity impacts. The exiting air from the fluidization column passes through the fabric filter bag which traps all elutriated particles from the bed.

- Gas regulator inlets (porous plates and spargers) regulating the flow of fluidizing gas through the bottom of the reactor into the segmented zones. An Omega rotameter regulates and controls the fluidizing gas flow rates for low superficial gas velocities measuring up to 2.7 cm/s and three sonic nozzles of 4 mm, 6 mm and 7 mm throat diameter to achieve high fluidization velocities ranging up to 100 cm/s.

- Vertical baffle plate preventing back mixing in the segmented zones caused by gas bubbles. The desirable and proper positioning of the vertical baffle relative to the axial discharge port is of paramount importance in determining the degree of purity of the segregated biochar and the time taken to reach steady state.

- Transparent fluidized bed that can easily be seen through and the walls regularly emptied and cleaned of sand and biochar particles after completion of the process so as to proceed unto another experimental runs without impurities.

- The novel modified cold simulator lab-scale bubbling fluidized bed reactor is compact and can be readily mounted on a wheel platform or other mobile surfaces promoting easy movement in various locations of the laboratory and operates at atmospheric pressure room temperature.

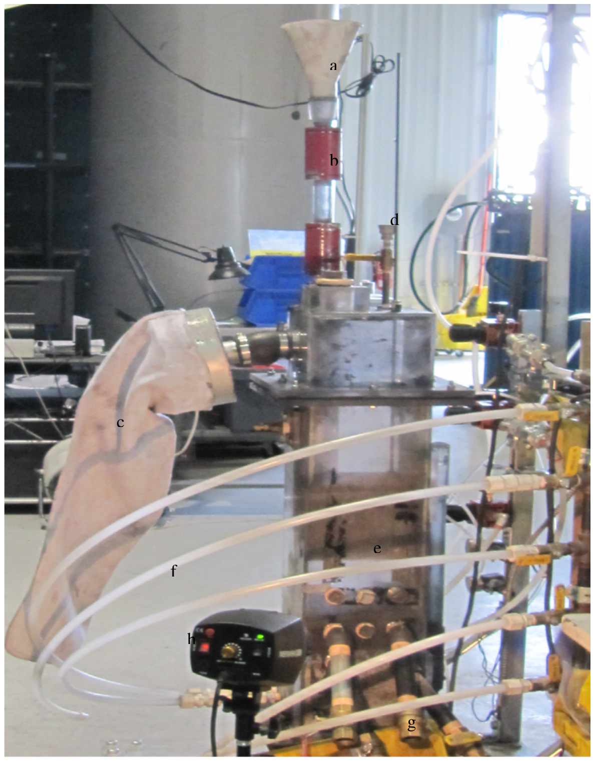


Figure 3 Set up view of the Modified Lab scale BFB (a-Biochar feeding inlet b-Pulsating pinch valve c-Filter bag d-Sand Inlet e-Vertical plate f-Gas sparger g-Axial Discharge Port h-light camera).

The unit was equipped with set of two gas distributors comprising of sparger pipes and porous plate to supply different gas velocities to the well mixed zone and the segregation zone respectively. The superficial gas velocity at which minimum fluidization was achieved was supplied through the porous plate. The vigorous bubbling condition to promote desired quality good mixing in the well mixed zone primarily required for good mixing in fluidized bed pyrolysis unit was supplemented with additional air supply by the set of two sparger tubes.

Five installed sparger pipes with approximate diameter of 0.127m, each sparger has 12 holes per sparger pipes measuring relatively 0.002m and positioned at the base of the bed. The spargers supply extra fluidizing gas to the porous plate gas stream to facilitate and achieve vigorously bubbling regime in the well mixed zone in the bubbling fluidized bed. Installed mounted ball valves regulate the on and off gas flow through each sparger, while sonic nozzles calibrated with Omega PX181 pressure transducers and regulated pressure controls fitted to a voltmeter supplies gas flow through each distributor.

Gas velocity slightly above the minimum fluidization velocity brings biochar that freely distributes under the submerged internal baffle into the segregation zone from the well mixed zone. The pressure drop across the bed and the distributor were measured by the U-tube manometers. The binary mixture materials (sand and biochar), to be fluidized are fed from the top of the fluidised bed reactor by set of regulated feeding unit. In addition, internally and partially submerged into the bed was a moveable simple baffle with vertical plate located at a certain axial distance from the distributor plate in a way to allow movement of solids between the two zones but, at the same time, segregation of particles to allow their removal and partitions the well mixed zone from the segregation zone.

The internal baffle plate functionally prevented the migration of large bubbles from the well mixed zones into the segregation zone and also restricted back mixing flow into the well mixed zone from the segregation zone. The designed biochar feeding system by Mara et.al, 2010, was initially manually controlled by two valves that opens and closes and connected in series beneath the hopper, however the modified upgraded biochar feeding system is automated pulse feeding system with pinch valves that opens and closes at desired duration.

An automated biochar feeding system is fitted with a transparent plastic tube through which the biochar is fed into the vigorously bubbling fluidized zone of the bed. A transparent plastic tube is located at about 0.1m above bed height on top of the well mixed section, discharging the biochar fed directly into the bubbling well mixed zone of the bubbling bed unit. In order to remove the segregated biochar, the discharge or axial removal ports have to be positioned at height within the proximity of the segregated biochar layer to ensure and obtain high purity that is to attain maximum separation efficiency. Ball valves are positioned 0.30 m, 0.23 m, and 0.16 m above the bed base, and primarily function to either allow or discontinue the recovery of the segregated biochar in the segregated zone.

Crucial to note, in this project, the ball valves 0.25 m and 0.16 m are maintained in operational mode while the 0.30 m ports were never used. These series of ports were never discontinued, but left opened for continuous recovery of the biochar unlike the previous research conducted findings[33]in which the ball valves are discontinued and opened periodically. The segregated biochar discharging through the ports were then received in a container and analyzed for separation efficiency. Therefore in designing a bubbling fluidized bed, it is crucial to carefully determine and select the parameters such as geometrical variables, the operating parameters and the physical properties in order to achieve well mixing and segregation mechanisms

2.4. Experimental Procedures and Operating Parameters.

Experimental investigations on the continuous segregation and removal of biochar in bubbling fluidized bed were conducted with air as the fluidizing gas and inert silica sand as the sand forms the jetsam-rich component of the binary mixture of particles. The feed was the flotsam biochar particles of different sizes, but same density. The fluidized bed was loaded with sand mass (8 kg) barco silica 71 sand produced by Optima minerals (ρ_s 2650 kg/m^3, d_{psm} 0.0002 m, ρ_b 650 kg/m^3) and initial 0.500 kg of biochar to accelerate the time to attain steady state. Validating the use of 8kg mass of sand throughout the experiment is based on the fact that the 8kg sand mass is a critical quantity because the amount of sand relates to the location of the withdrawal ports. Too much sand would overfill the bed in relation to the exit ports and prevent the formation of a sufficiently thick layer of segregated biochar that can be collected with a high degree of separation efficiency.

The amount of sand and the biochar holdup and the thickness of the layer at steady state in relation to the location of the exit ports have direct implications on biochar purity. Increasing quantity of the sand mass which results in increased sand level around the axial port and as a result contaminating the biochar layer formed above such sand which will ultimately result in detrimental influence on the segregated biochar purity. Biochar particle sizes 850 µm, 1400 µm, 2000 µm and 2300 µm were fluidized with air in the presence of inert silica sand. Findings and analyses of tested variables is presented and discussed later in this chapter to support the criterion for continuous separation of biochar in binary mixture with sand emphasis on achieved purity, time taken to reach steady state during continuous separation and removal of biochar from the bed. The fluidized bed is segmented into two zones operating at different gas velocities under different conditions. In all the experimental runs, a mixing velocity of $V_{well\ mixed}$*=0.20 m/s* and segregation velocity of V_{seg}*=0.0963 m/s, each of these velocities were* established after series of trial and error of several ranges and these velocities resulted in good performance of the process. A mass of 8 kg of barco silica sand 710 microns was loaded into the well mixed zone and the biochar continuously fed at the desired feed rate, both of the binary mixture fluidized with air through a set of five sparger pipes with dimensions of 5/8" in diameter, 2 mm holes, 12 holes per tube were positioned at the bottom of the bed.

Table 2.1. Experimental Operating Parameters.

Experimental Operating Parameters							
Bed Width (m)	Bed Height (m)	Bed Area $(m)^2$	Minimum fluidization velocity U*mf* (m/s)	Segregation velocity U*seg* (m/s)	Vigorous mixing velocity U*wm* (m/s)	Gas Viscosity μ_g (kg/ms)	Gas Density ρ_g (kg/m^3)
0.2	0.74	0.04	0.0683	0.0963	0.2	0.0000178	1.225

Table 2.2. Experimental Parameters.

<table>
<tr><td rowspan="2">Physical Properties Parameters</td><td colspan="4">Particle Size
(d_p, μm)</td><td colspan="4">Feed rate
($F_{biochar}$, kg/min)</td><td colspan="2" rowspan="2"></td></tr>
<tr><td>850</td><td>1400</td><td>2000</td><td>2300</td><td>0.34</td><td>0.36</td><td>0.38</td><td>0.40</td></tr>
<tr><td rowspan="2">Geometrical Parameters</td><td colspan="4">Separation Zone Area
(A_{sep}/A_{bed}, %)</td><td colspan="4">Plate Height
(P_h , m)</td><td colspan="2">Axial Port height (z, m)</td></tr>
<tr><td>19.2</td><td>29.2</td><td colspan="2">39.2</td><td>0.14</td><td>0.18</td><td>0.22</td><td>0.26</td><td>Base (0.16)</td><td>Top (0.23)</td></tr>
</table>

For each experimental run, the desired segregation gas velocity (U_{seg}) is achieved through gas velocity channeled through the porous plate. The gas velocity in the well mixing zone is augmented with additional air at a fixed velocity (U_{wm} = 0.20 m/s) which simulates conventional value in bubbling bed pyrolyzer. During all experimental runs, the bed was fluidized at set desired vigorous mixing velocity and segregation velocity as illustrated in Table 2.2. Biochar is fed at intervals of 60 s into the well mixed bubbling regime through transparent tube connected to a plastic hopper positioned 0.15 m above the bed height of the well mixing zone. Trial attempts of feeding durations of 15s, 30s, and 45s were tested but all yielded no sufficient time for proper mixing and segregation.

However, the duration of 60 s was allowed enough time to ensure good mixing with the sand and duration biochar particles to be drifted by gas bubbles and into the segregation zone. Similarly the feed rate of 0.30 kg/min which amounted to 2 kg/hr was also established to be the desirable feeding rate to avoid excessive accumulation of biochar in the bed. The biochar feeding system is connected to and controlled by an automated pulsating feeding system beneath a transparent plastic hopper. Caution must be taken however to ensure that the biochar is completely discharged into the well mixing bubbling zone of the fluidized bed pyrolyzer.

The biochar fed into the well mixing zone, circulates through the gas drifting of bubbles, thereby accumulating in almost pure layer of biochar in the segregation zone. Upon uniform mixing in the bubbling zone of the bed, successful segregation of biochar is achieved with the segregation velocity greater than the minimum fluidization velocity (U_{mf}). Continuous biochar segregation is ensured to achieve accumulation of well-defined biochar thickened layer in the bed on top of the sand in the segregation zone and at every 15 minutes intervals the accumulated segregated biochar layer was measured.

The bed height of sand-biochar binary mixture of both well mixing and segregation zone at the beginning of each experimental run before fluidization was recorded and noted to be 0.20 m with biochar height of 0.8 m and sand height 0.12 m in the bed. In each of the experimental runs before injecting fluidizing gas, the separation zone area A_{sep} to the wall of the fluidizing pyrolyzer is set to the appropriate lateral position and the plate height (p_h) is also adjusted above the base of the bed to preferred height that will prevent back mixing of biochar from the segregation zone into the well mixed zone as a result of the effect of the gas bubbles in the vigorously bubbling well mixed zone. The preferred height location was achieved through series of attempts positioning the plate at various heights and then purity of the segregated biochar following the mixing and segregation mechanism. The thickened biochar layer segregated layer results from continuous biochar feeding into well mixing zone which is drifted through gas bubbles into the segregation zone. The axial discharge ports remained continuously opened for the removal of total solids consisting of biochar and traces of sand. The sand mass fraction removed through sieving is recirculated back into the well mixing zone.

The separation efficiency (%) of the biochar fraction is a measure of the recovered biochar mass fraction from the total solids mass fraction analyzed through sieving.

$$\textbf{\% Separation Efficiency} = \frac{\text{Flow rate of Biochar Recovered}}{\text{Flow rate Fed}} \qquad \textbf{(3)}$$

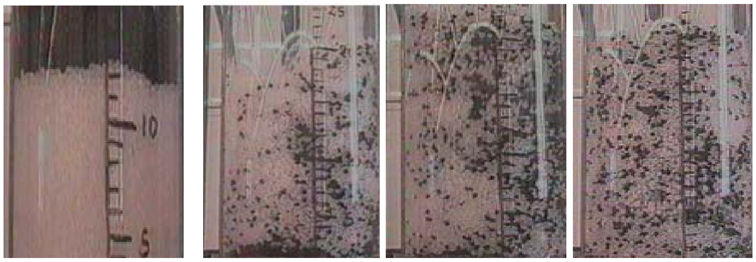
Yield is best defined as the ratio of the biochar recovered to the biochar loaded and when the biochar recovered equals to the biochar loaded, a steady state is reached in the system.

$$\textbf{Yield (ŋ)} = \frac{\text{Biochar recovered}}{\text{Biochar loaded}} \qquad \textbf{(4)}$$

The steady state was achieved just before the entire the entire bed was completely loaded and the duration was between 50 minutes and 75 minutes. In each experimental run, 2.0 kg/hour of biochar was continuously fed into the bubbling fluidized bed. It should be noted that in repeating the study made by Mara et.al 2010, the same biochar feed rate was used so that there will be no deviation in the biochar fed into the bed in order to achieve improved purity state and steady state. This feed rate will allow small mass of biochar to be continuously fed over long duration reducing the chances of overcrowding the bed. About 0.05 kg biochar elutriated was collected in the filter bag elutriated which amounts to about 2.5 % of the amount of biochar fed.

Although small fraction of biochar was elutriated into the filter bag, this was considered negligible. The continuous biochar separation and removal process of the bed purity and time to reach steady state (that is when there is an equal flow rate in the biochar fed rate and the removed biochar flow rate) were determined for each experimental runs in the bubbling fluidized bed. All the experimental runs achieved complete good fluidization performance. Replicates were performed for reproducibility and near to accuracy purposes of steady state removal and high purity.

2.5. Mixing Profiles of Sand-Biochar at Different Time Intervals.



t =0 s t=15 s t=30 s t= 60 s

Figure 4. Mixing profiles of biochar particles in the cold simulator Bubbling Fluidized Bed

The Figure 2.7 shows captured digital images of the mixing process profile of the biochar particles and sand particles with superficial gas velocity (U_{fs}) of 0.0683 m/s of the fluidizing gas into the well mixed regime (U_{wm} 0.20 m/s), that is, Uwm = 2.93* U_{fs}. The bubbling fluidized bed was initially loaded with 500 g of biochar to accelerate the segregation process and allow shorter duration for the biochar to form distinct thick layer in the segregated zone.

2.6. Separation Profile of Sand-Biochar in Bubbling Fluidized Bed

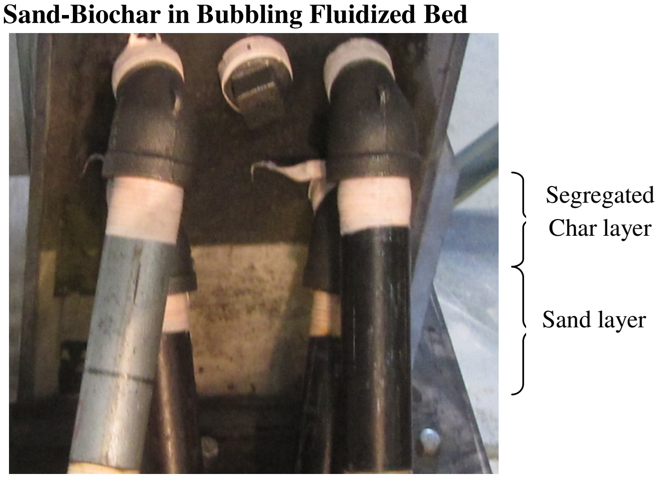


Figure 5. Visual observation of the segregated biochar layer at steady state (P_h = 18 cm, t = 75 min)

Figure 2.8 illustrates an image of the transition towards the steady state of the segregation process for biochar particles. These biochar particles are drifted at high gas velocity of (U_{wm}

0.20 m/s) from the well mixed zone by the fluidizing gas streaming through the porous plate and extra gas supplied by the spargers into the segregation zone. Calibration trial attempts were performed to determine the desirable gas velocities to achieve steady state, and it happened that that the minimum fluidization velocity of 0.0683 m/s, well mixed velocity of 0.2 m/s and segregation velocity 0.0963 m/s used in the experimental runs in resulted in steady states in all the conducted experiments.

Biochar feed rate constitutes a crucial variable essential to be taken into consideration in designing a pyrolyzer unit based on the fact that varying biochar feed rates into the bed influence and affects the overall efficiency and fluidization quality as well as the associated kinetics and heat transfer dynamics involved in the fluidization process. Through the fluidization and segregation operations, noticeable differences in bed heights of the two zones were taken into account.

At steady state, there exists steady equilibrium between the evolved flotsam and the residual fed biochar when the both sand and biochar solids are subjected to fluid-like state as in the case of density segregation of solids. At steady state, the two zones attain different bed height influenced by variation in the positioning of the vertical plate and different gas velocities in the bed. The observable bed height difference was due to differing densities in the two partitioned zones. Below are illustrative depictions of the well mixed zone during and after steady state shown in Figure 2.9 also illustrates the segregation zone during and at steady state is reached.

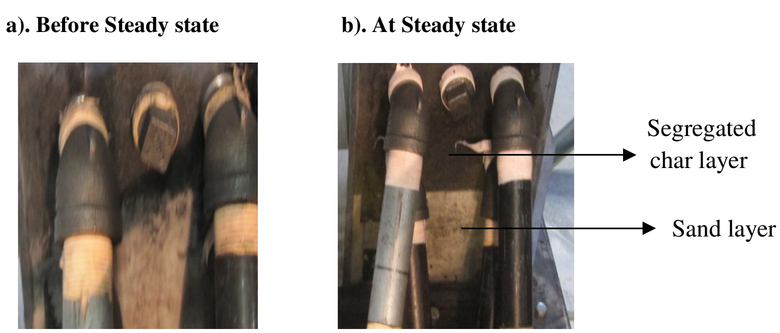


Figure 6: Biochar Segregation before, and at steady state in a Bubbling Fluidized Bed.

The initial bed height H_b of the sand biochar binary mixture loaded into the well mixed zone and segregation zone before fluidization was measured. Verifications of the reproducibility and accuracy of the experimental findings of the operating parameters to attain optimal yield and purity several replicates were conducted. Figure 2.9a shows schematic profile of segregation of biochar process with transition towards steady state in one of the experimental findings Initially at time (t_1), there was no fluidizing gas passed into the fluidizing column of the well mixed region and the height of the binary mixture of sand and biochar were the same in both well mixed and segregation zone of the fluidizing bed.

However, in order to accelerate the time to reach the steady state in all the experiments conducted, an initial mass of 0.5 kg biochar is added into the well mixed zone to 8 kg of sand. The bed is allowed to fluidize over desired time t_2 at high gas velocity as shown with evidence of gas bubbles formation creating a vigorous mix of the sand with the biochar and the bubble effect causing a fair increase in the height of the sand and biochar binary mixture in the well mixed zone, however few biochar particles are drifted by the bubble effects into the segregation zone and shortly following a duration time t_3, there is considerable increase in the height of the biochar layer formed on top of the sand in the segregation zone creating a concentration difference of the biochar in both well mixed and segregation zone.

This effect continued over time t_3 with concentration difference gradient created and the biochar layer thickened above the sand in the segregation zone with increased height. At this stage when time t_3 is reached, there is probability that there could be char hold if there is excessive continuous feeding or increased feeding rate into the well mixed zone of the bed as a result of back mixing caused by the gas bubbles generated at high fluidizing gas velocity in the well mixed zone. It's also crucial to stress the fact that sand removed along with the biochar at the segregation zone through the discharge port is kept continuously recirculated back into the bed maintaining the mass of the sand constant and this is very beneficial to prevent sand loss and enhance process profitability.

However, the height of the removal port will be based on the feed rate of biochar loaded into the well mixed zone. Increasing the flow rate, height of biochar layer formed in the segregation zone of the fluidizing column increases. There will be a situation where limitation may be reached if there is excessive feeding and back mixing created in the bed leading to concentration difference between the two zones. At steady state, this is the time the flow rate of biochar feed equals the same flow rate of biochar removed from the bed when $\eta = 1.00$. As the process progress towards the time t_4 at steady state condition, the biochar formed layer increased above the overflow height and is removed through the axial port. Biochar is continuously fed at a feed rate F_1 of 30 g/s which corresponds in each experimental run to approximately 2 kg biochar fed over an hour, thus ($F_{biochar}$ = F_1 = 2 kg/h). Figure 2.10 illustrates the biochar segregation mechanism with continuously mixed sand biochar layer formed in the well mixed zone and

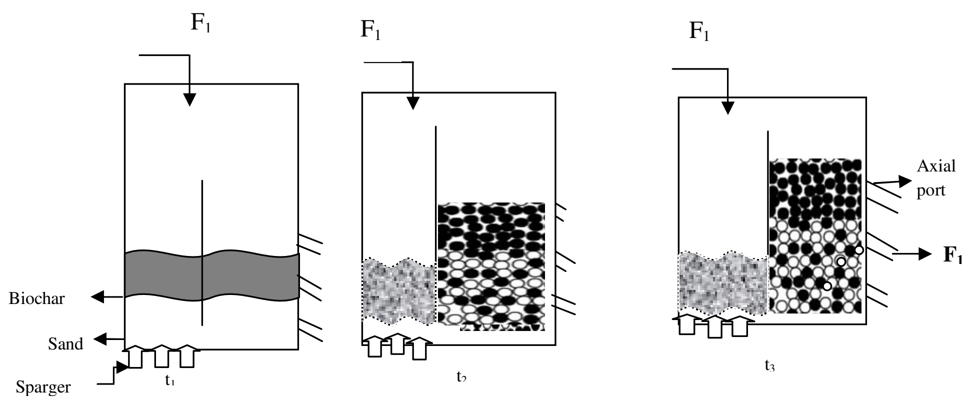


Figure 7. Visual observation of segregated biochar layer formed with decreasing biochar in the well mixed zone and increasing biochar in the separation zone U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, P_h=0.18 m

progressively increasing biochar height in the segregation zone. Steady state is reached when the flow rate of biochar recovered at the axial discharge port equals the flow rate of biochar fed. Figure 2.11a shows a steady state reached, with simultaneous effects observed in progressive increase in purity level over time on the effects of the separation zone area A_{sep}/A_{bed}) in the continuously bubbling fluidized bed reactor.

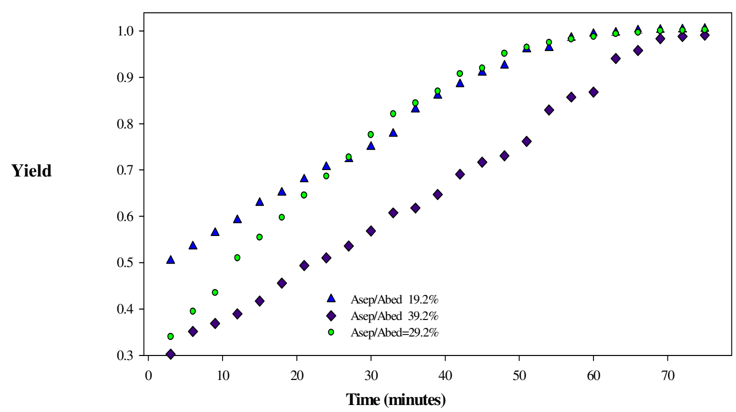


Figure 8a. Comparing separation zones effects on efficiency U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s

However, when the area of separation zone A_{sep}/A_{bed} was decreased, this prevented back mixing effect of biochar from the segregation zone into the well mixed zone thereby improving the purity levels and allowing steady state to be reached within short time.

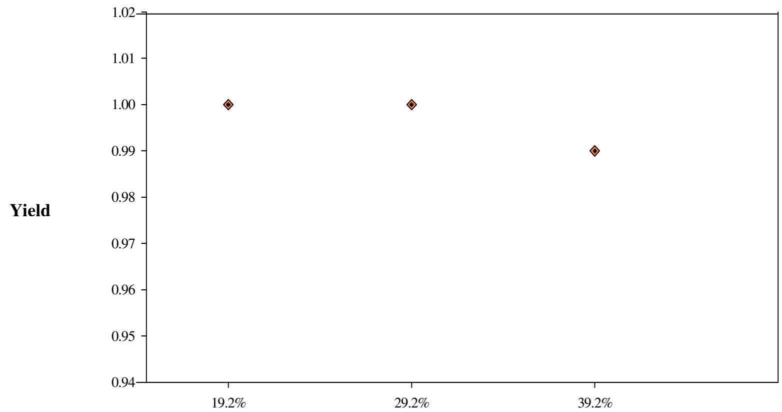


Figure 9b. Comparing separation zones effects on efficiency U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s

However the inventory of the sand in the fluidized bed is a determining factor in the location of the axial discharge port effects on the purity of the biochar layer formed in the segregation zone. When the biochar feed rate was increased, that is $F_{biochar}>$ 0.38 kg/h overloading state, a notably poor fluidization quality was established whereby the steady state process was not practically achieved or feasible. Trial experimental runs with biochar feed rate was less than 0.034 kg/h, that is, $F_{biochar} <$ 0.34 kg/h under loading state were conducted in which the bed was fed at a biochar feed rate of 0.028 kg/h, 0.030 kg/hr and 0.032 kg/h resulted in similar effects of undesirable steady state and very poor purity levels in the process thus creating a poor system performance.

CHAPTER 3.

3.1. EXPERIMENTAL RESULTS AND DISCUSSION.

In this chapter, the results of the experimental investigations conducted at ambient atmospheric pressure and temperature are reported and discussed. The table in chapter 2 shows the variables that were used in determining the effects of the geometrical parameters, and physical properties operating conditions on the efficiency of the segregation and the purity of segregated biochar in a bubbling fluidized bed.

A). Effects of Geometrical Parameters

3.1.1. Effect of Plate Height (P_h) on Purity and Steady State

Relative to the bottom of the bubbling fluidized bed, the vertical positioning of the plate height (h_p) had significant effect in the attaining steady state and high purity in the continuous separation and removal of biochar. It must be noted that formation of sufficient biochar layer must be allowed with the plate height positioning and has to be relative and dependent on the binary mixture solids (biochar and sand) loaded into the bed. Experimental runs investigating the effect of the pate height involves initial loading the bed with Barco silica sand 71 into the bed followed by the biochar. Different plate heights were tested to determine the position in which the purity level was optimal and steady state was achieved at a faster duration.

Therefore optimizing the system operation was paramount to avoiding poor fluidization performance of the various parameters tested. Poor fluidization performance usually brings about either the accumulation of excessive biochar in the well mixed zone (as a result of recirculation of biochar from the separation zone) or very close proximity of the plate to fluidized bed creating insufficient surface area for particle segregation eventually leading to overloading of the bed. The plate heights investigated ranged from 0.14 m, 0.18 m, 0.22 m and 0.26 m respectively. The experimental runs, results showed that following segregation, increased purity levels of the removed biochar was as a result of increasing the plate height. There was however a limitation reached as separation efficiency (%) decreased with submergence in the bed at 0.22 m and 0.26 m onwards initially before the continual removal of the segregated biochar from the segregation zone as shown in Figure 3.1. The separation efficiency was undesirable and this resulted in a process failure as the plate height exceeded beyond 0.18 m.

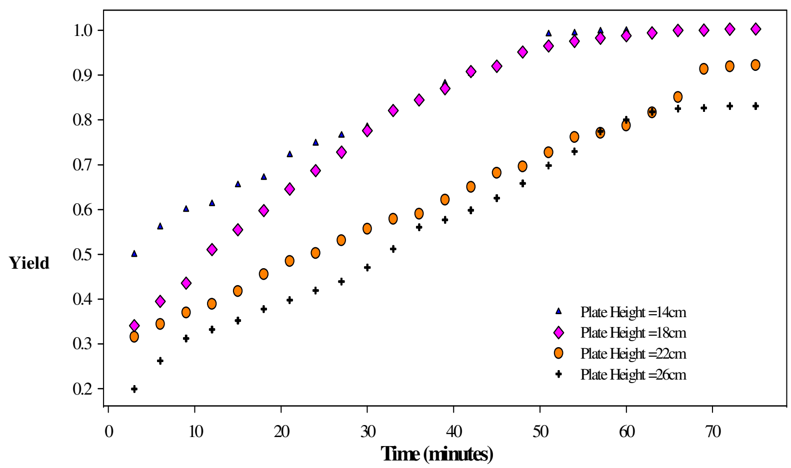


Figure 10. Biochar removal at varying plate heights (transient to steady state), U_{seg}=0.09 m/s, U_{wm}=0.16 m/s

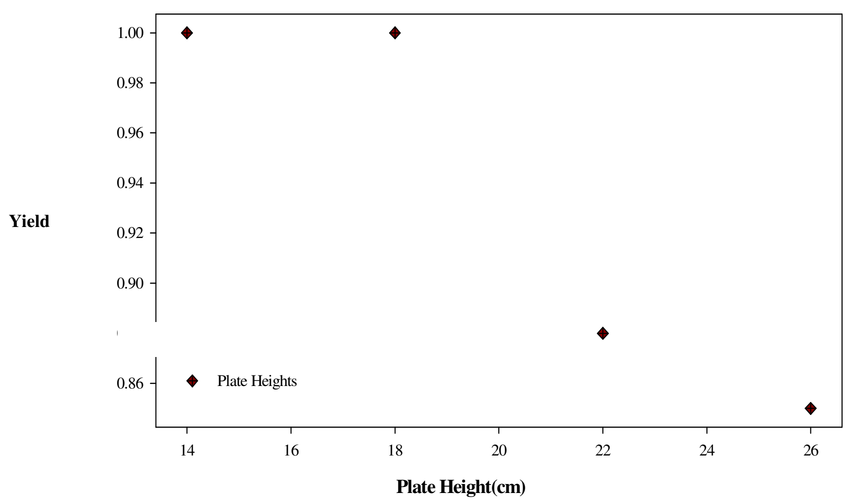


Figure 11. Biochar removal at varying plate heights at steady state, U_{seg}=0.09 m/s, U_{wm}=0.16 m/s

Increasingly adding biochar into the bed decreases the separation efficiency and the yield. Although the removed sand is recycled back into the system, there was no additional sand leading to biochar build up accounting for the difference in the binary mixture and creating accumulation and overloading of biochar in the well mixed zone.

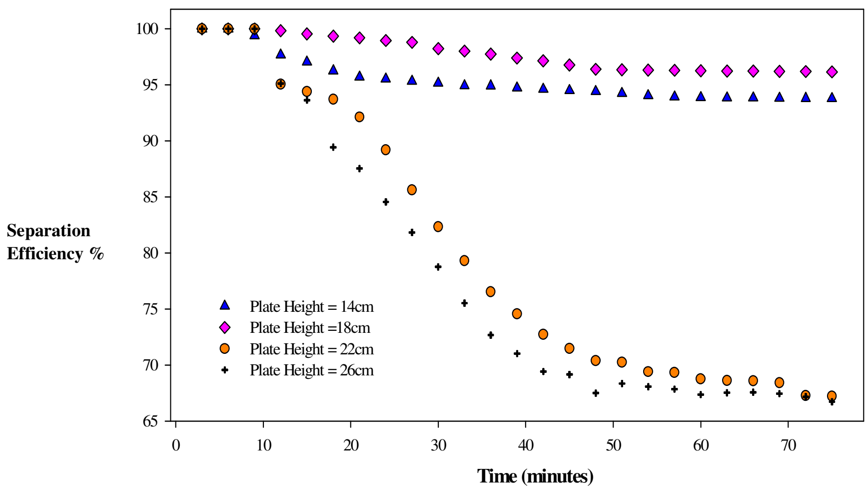


Figure 12. Plate heights effects on separation efficiency U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, Fbiochar=0.036 kg/s

Furthermore, the experimental runs demonstrated that the steady state could be attained more quickly and separation efficiency greatly enhanced when the plate height of the vertical baffle was submerged between 0.14 m to 0.18 m in the fluidized bed using the axial removal port positioned at 0.23 m on the segregation side of the fluidizing bed.

At plate height of 0.14 m with feedrate of 0.036 kg/s, the separation efficiency accounts for approximately 96.40 % at 66 minutes with steady state (ŋ = 1) and when the vertical baffle plate height separating the two zones (well mixed and separation zone) was submerged at plate height of 0.18 m, the steady state (ŋ=1) was attained within approximately 60minutes shorter period of with increased separation efficiency of 98.00 %. Experimental findings by Mara et.al, 2010 illustrated in Figure 3.4, proved progressive purity increase with increasing plate heights in the bed followed by system failure when the plate height is submerged at a further height increase beyond 0.20 m, approximately at 0.22 m onwards. The placement of the plate height is influential to prevent back mixing that might affect the purity thus resulting in system inefficiency.

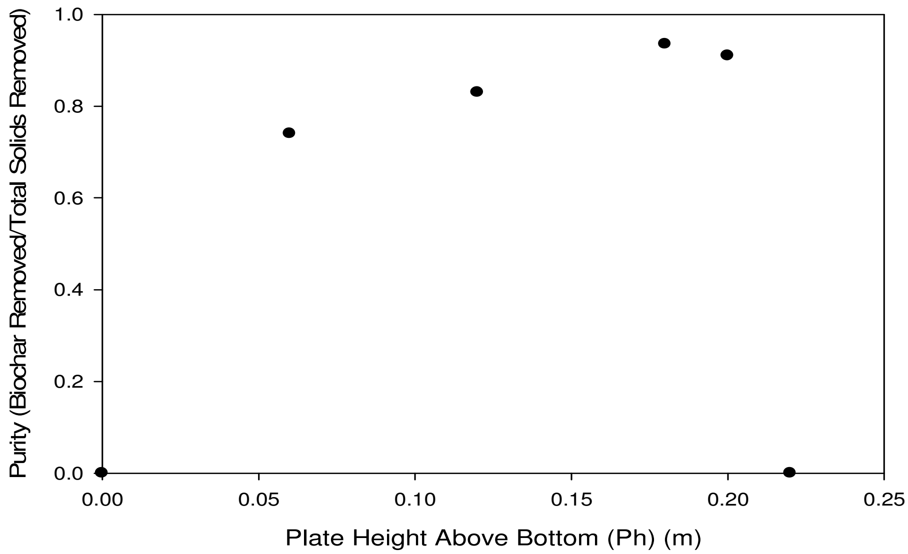


Figure 13. Plate height effects on separation efficiency of biochar removed, Mara et.al 2010

3.1.2. Effect of Axial Removal Port on Purity and Steady State

The novel cold simulator lab-scale bubbling fluidizing unit used in the experimental investigation in determining the effects of continuous segregation and removal of biochar from bubbling fluidized bed pyrolyzer unit consisted of three different axial positions of removal ports at heights of 0.16 m, 0.23 m and 0.30 m respectively. However only two ports were investigated z = 0.16 m, which is the base port and z = 0.23 m which is the top port. The uppermost port z = 0.30 m was not investigated in the experimental runs as it would result in huge sand losses.

The performance in terms of separation efficiency and yield was investigated as shown below. There was system failure with zero purity and instability when tested with particle sizes 850 μm and 2300 μm. Comparison between the separation efficiency and yield levels for particle sizes 1400 μm and 2000 μm were explored and analyzed as shown below in Figures 3.5 and Figure 3.6 respectively.

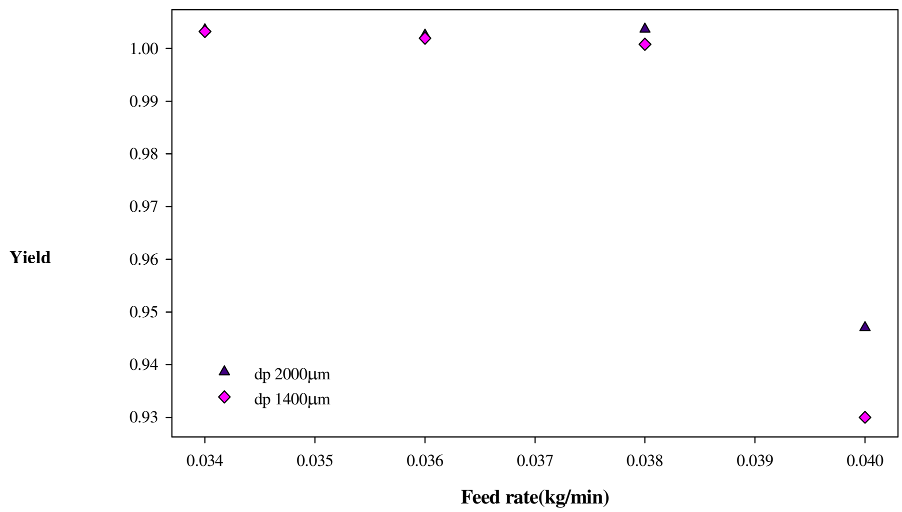


Figure 14. Effect of axial removal ports on yield U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, z=0.16 m

The lower axial port presented a reduced or decreased biochar purity performance of binary mixture vigorously fluidized in the well mixed zone proceeded by the removal of the biochar from segregation zone.

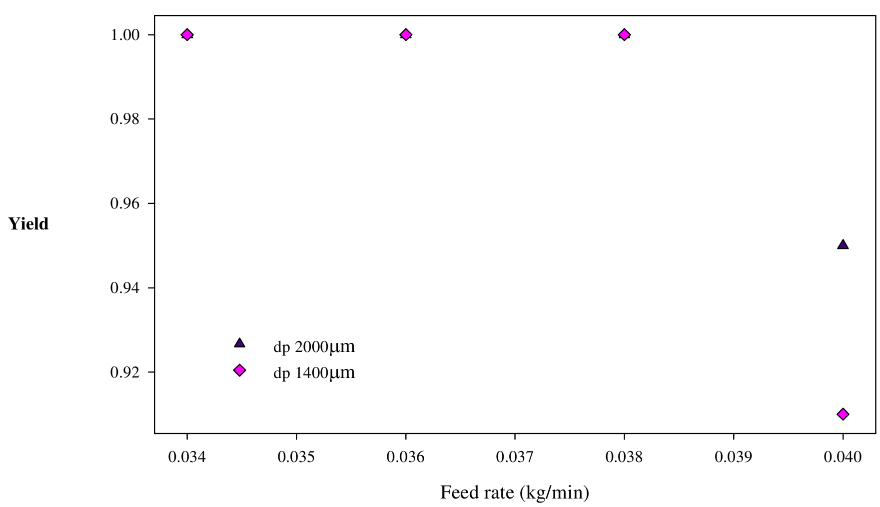


Figure 15. Effect of axial removal ports on yield U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, z=0.23 m

Comparatively, the top layer axial port offers improved removal purity efficiency in the continuously bubbling fluidized bed unit with an increased overall removal performance for the federates of 0.034 kg/s to 0.038 kg/s. Above these federates, there was a system failure of defluidization as shown in Figure 3.6 above. Same profile in Figure 3.7 was noticed with the purity results.

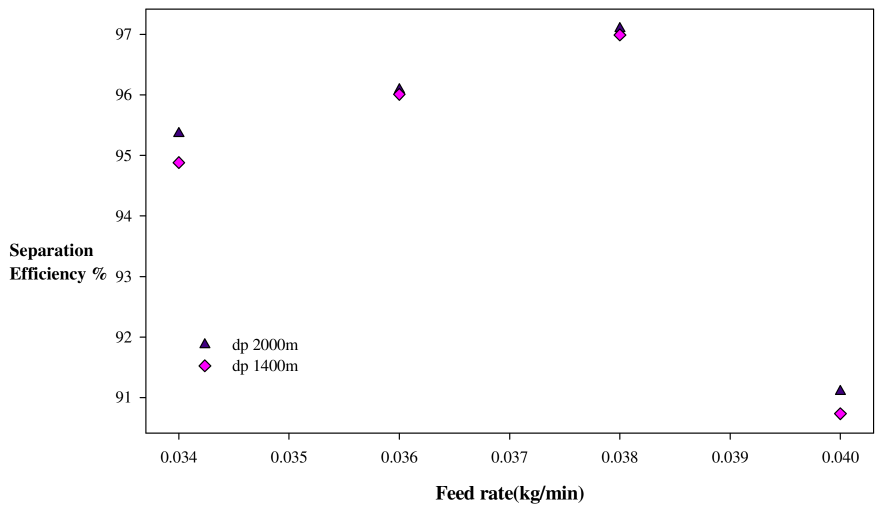


Figure 16. Effect of axial removal ports on yield U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, z=0.23m, P_h =0.18m

The upper port z = 0.23 m yielded higher purity and attained steady state faster than the base port z = 0.16 m when tested with particle sizes 1400 µm and 2000 µm as seen in figure 3.7, while figure 3.8 explained the comparison with the axial ports relative to corresponding feed rates.

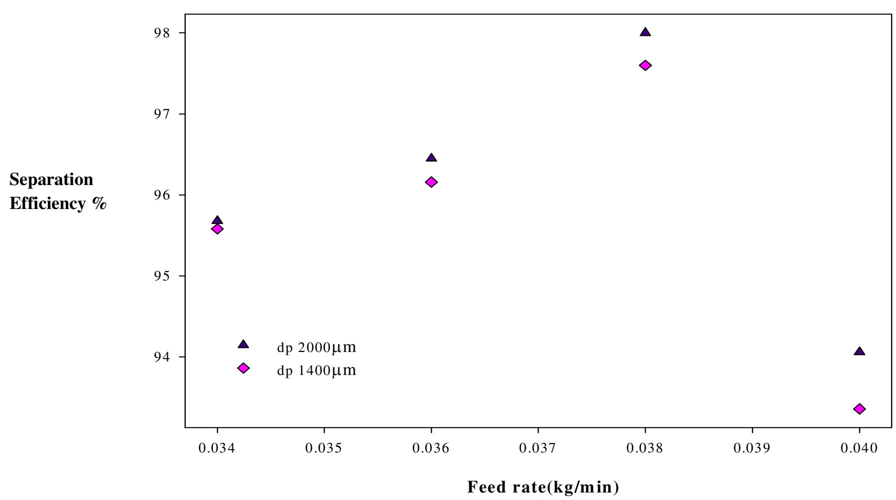


Figure 17. Comparing the effects of axial ports yield: Useg=0.0963 m/s, Uwm=0.20 m/s, P_h=0.18m, z=0.16 m

Similar trend was observed with particle size of 2000 μm and the result was that increasing the plate height led to higher separation efficiency in the removal performances shown in Figure 3.9 and Figure 3.10 respectively. The experimental findings proved that the relative position of the port with respect to the thickness of the segregated biochar layer is a determining factor that enhanced more selectivity with less sand removal.

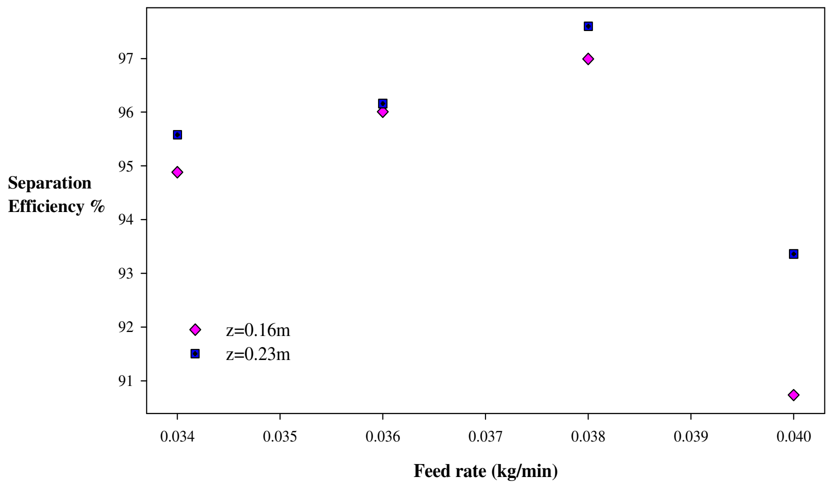


Figure 18 Comparing effects of axial lower port (z = 0.16 m) and axial upper axial (z = 0.23 m) port on separation efficiency U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s , P_h=0.18m, d_p 1400 μm

The report findings of previous experiment by Mara et.al 2010 shown below indicated that yield was less than ŋ = 1.00 was not reached in the conducted experimental runs while comparing the effects of the separation zone on the separation efficiency of the separated biochar

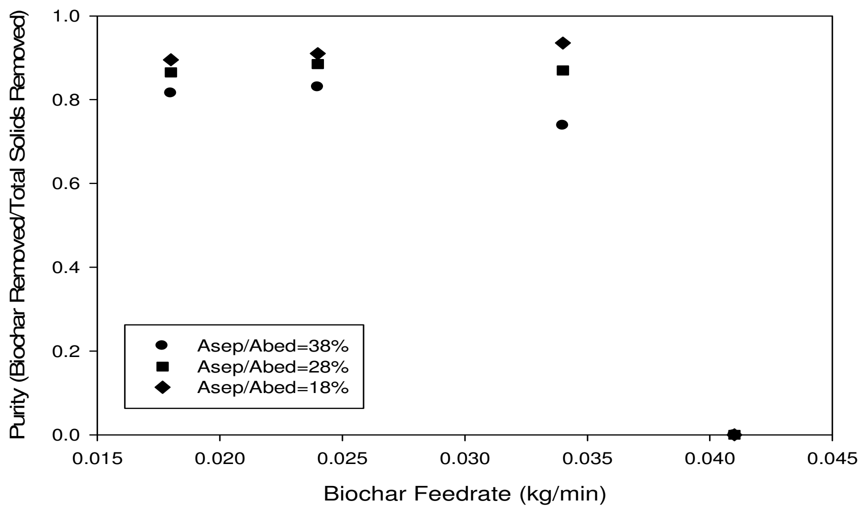


Figure 19 Effect of separation zone on biochar separation efficiency (%), Mara 2010

Moreover, the new findings shown in figure 3.11 proved that steady states were achieved in all the experimental runs with the axial ports; the only exception is unsteady state encountered with both ports when feed rate of 0.040 kg/s due to excessive biochar bed accumulation.

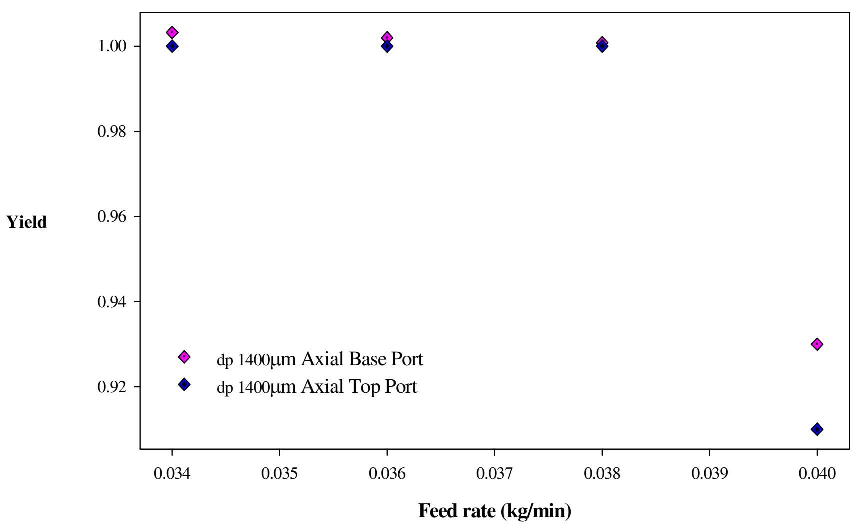


Figure 20. Comparing effects of axial ports) U_{seg} = 0.0963 m/s, U_{wm} = 0.20 m/s, P_h = 0.18 m, d_p 1400 μm

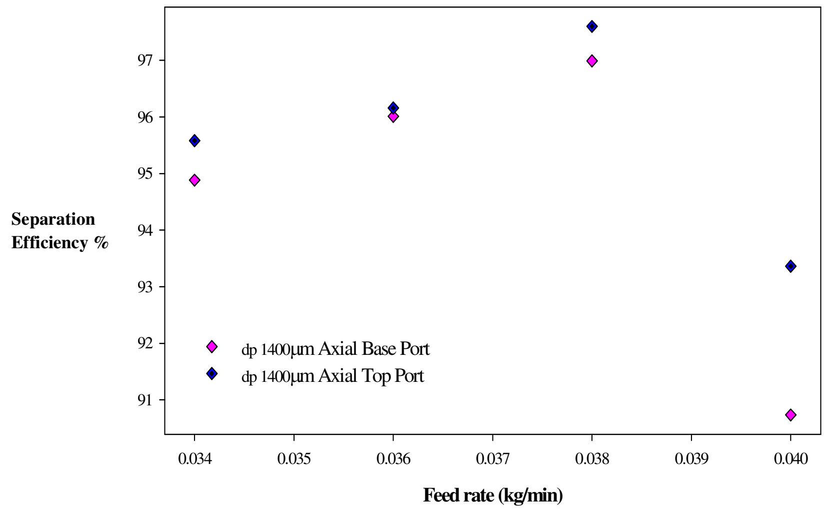


Figure 21. Axial ports effects on separation efficiency of biochar U_{seg}=0.0963 m/s, U_{wm}=0.20m/s, P_h=0.18m

All the experimental findings indicated a steady state in efficiency and purity attained with a trend of increased separation efficiency of the axial ports and a noticeable sharp decline of separation efficiency and yield at feedrate of 0.040 kg/minute, effect of which might be validated by the fact that the critical point is reached in the bed that cannot further attain process stability and performance optimization.

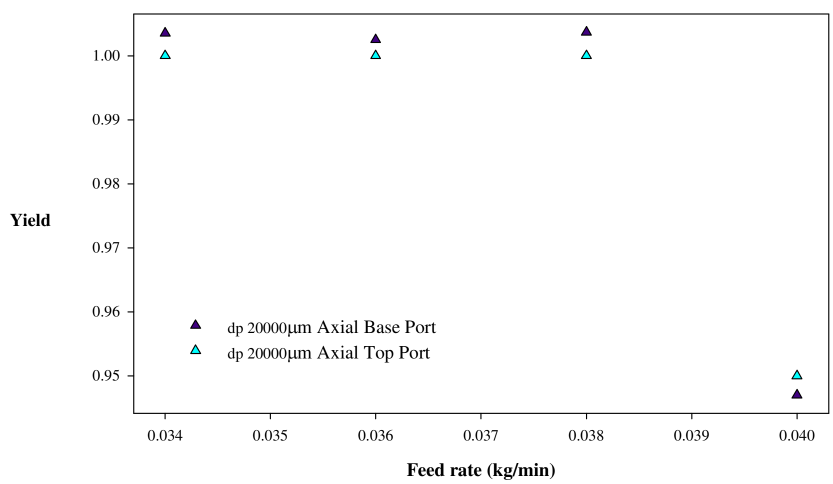


Figure 22. Axial port lower (0.16 m) and upper axial (0.23 m) effects on separated biochar U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s , P_h=0.18 m, d_p=2000 µm

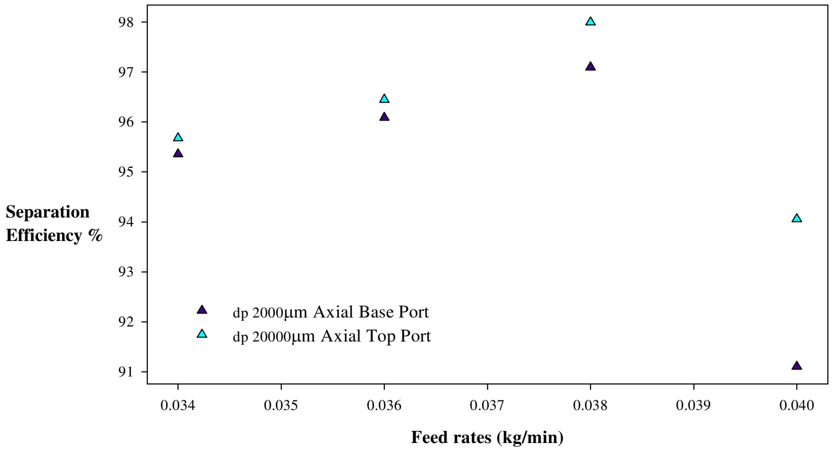


Figure 23. Effects of axial lower port (z = 0.16 m) and axial upper port (z = 0.23 m) on separation efficiency U_{seg}=0.0963 m/s, U_{wm}=0.20m/s, P_h=0.18m, d_p 2000 μm

3.1.3. Effect of Separation Areas on Separation Efficiency and Yield.

Effect of cross sectional separation area to the fluidized pyrolyzer wall was investigated. When the cross sectional separation area was decreased by half, sand loss was multiplied by a factor of about 2.5. This led to worse purities and accounted for poor fluidizing quality. Replicates to validate optimal performance and the high purity levels of continuously segregated and removed biochar as a result of the effects of cross sectional separation area were measured and documented over duration of 75minutes with negligible deviation attained in purity.

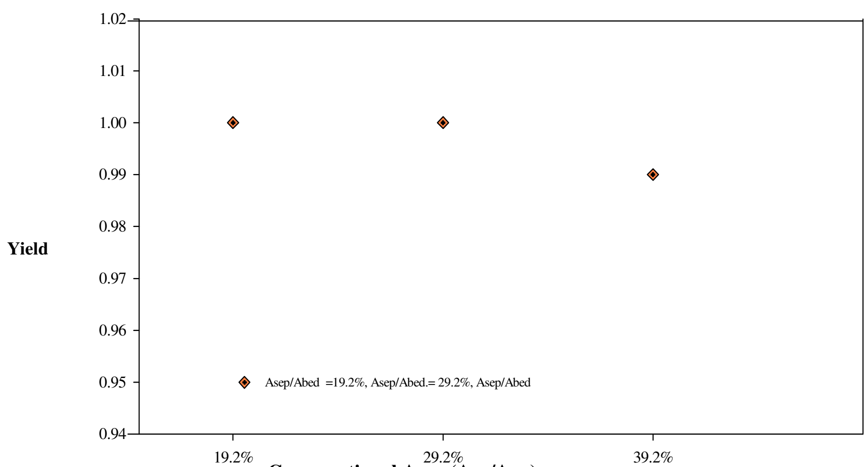


Figure 24 Comparing the effects separation zones of biochar removed U_{seg} = 0.0963 m/s, U_{wm} = 0.20 m/s

Using particle size 1400 µm with top axial discharge port (z = 0.23 m) and increasing the A_{sep}/A_{wm} led to a large area for increased layer of biochar accumulation in the segregated zone. With decreasing of A_{sep}/A_{wm} however, there was system failure with overloading of biochar accumulated in the well mixed zone as a result of back mixing effects from the segregation zone. This can be deduced from Figure 3.16 and Figure 3.17. Eventually, the steady state and purity levels in the continuous segregation and removal process of biochar were affected.

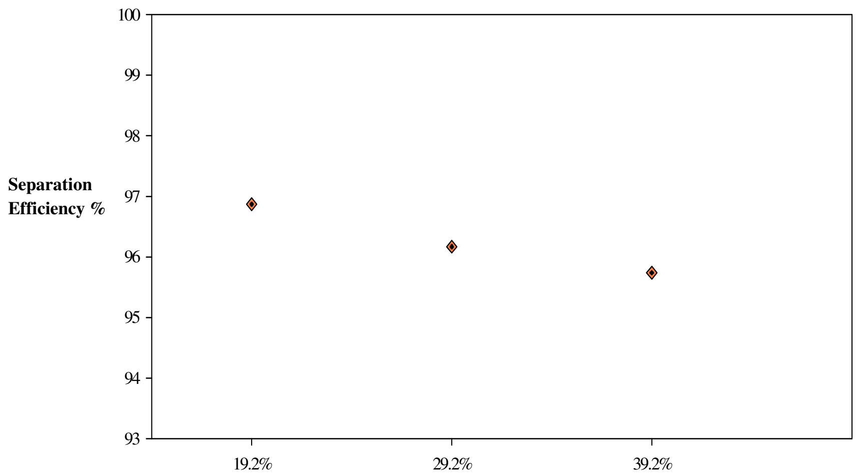


Figure 25. Effects separation zones on biochar removed U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s

Figure 3.16 indicates that when the cross sectional area (A_{sep}/A_{wm}) ratio was 19.2 %, a purity level of 96.99 % was achieved. However, when the cross sectional area (A_{sep}/A_{wm}) ratio was 29.2 %, a much reduced separation efficiency of 96.28 %was achieved. Furthermore, with cross sectional area (A_{sep}/A_{wm}) ratio of 39.2 %, the least unsatisfactory purity level of 95.4 %was obtained. However, figure 3.17 below shows the time involved in the transient to steady on the effect of separation zone on purity of the segregated biochar under in a bubbling fluidized bed pyrolyzer.

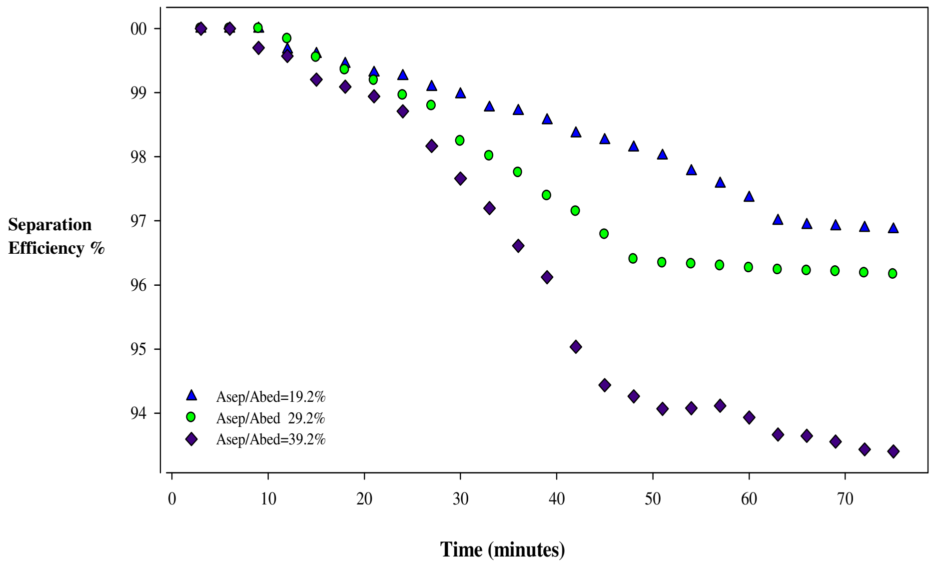


Figure 26. Comparing separation zones effects on efficiency U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s P_h 0.18m

B). Effect of Operating Conditions

3.1.4. Effects of Biochar Particle size.

Different pyrolysis processes utilize different properties of feedstocks such as size, density and shape. Biochar particle size creates significant effect on continuous segregation and removal of biochar in bubbling fluidized beds. In this experiment, the effects of biochar particle sizes of 850 µm, 1400 µm, 2000 µm, and 2300 µm were investigated in evaluating the performance of the system in terms of separation efficiency and yield in a continuous process of segregation and removal of biochar from the system.

The yield performance and separation efficiency obtained can best be explained from the result findings as seen in Figure 3.18. The biochar particles of 850 µm were very fine and almost entirely elutriated when the stream of fluidizing gas was passed into the well mixed zone. As a result there was no progress in the fluidization and the entire process was discontinued. This validated the fact that elutriation is best applicable in complete removal of very fine biochar particles and inefficient in removing larger particles of biochar from a fluidizing bed.

Thicker separated biochar layer were formed with biochar particle size of 2000 μm corresponding to an improved removal of biochar with increased separation efficiency during the segregation process. On the other hand, the biochar particle size of 1400 μm through visual observation created a thinner segregated layer.

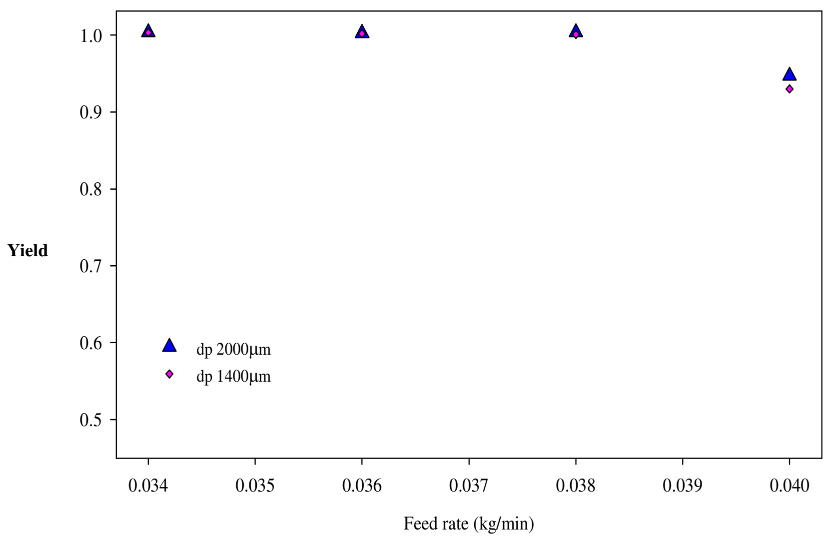


Figure 27. Effect of Particle Size on yield: U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, P_h=0.18 m

Besides, the smaller biochar particles created slightly lower purities compared to the larger biochar particle sizes, although the difference in the purity levels between these two different particle sizes was fairly significant. The decreasing efficiency resulting with increasing feed rate can be justified with the fact that there is back mixing from the segregation zone into the well mixed zone leading to excessive biochar overload in the well mixed zone thereby progressively causing poor fluidization quality and lessened separation efficiency.

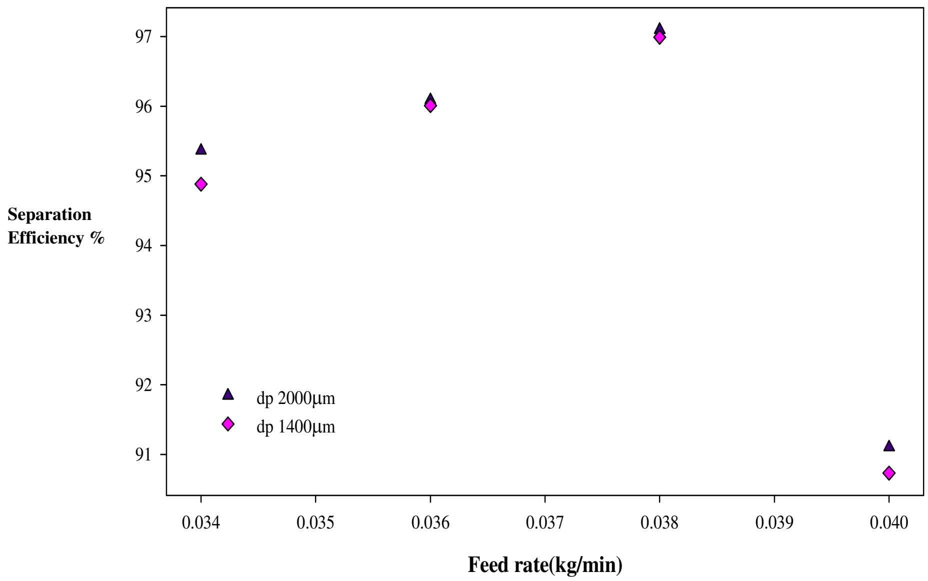


Figure 28. Effect of Particle size on removed biochar: U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, P_h = 0.18m

It was also observed that larger biochar particle sizes of 2300µm created much reduced purity level and unsteady state during the biochar removal as there were more sand impurities in the segregated fraction. Using biochar particle sizes of 850µm which were relatively fine, majority of these biochar particles were elutriated generating very poor fluidization quality and zero separation efficiency with unsteady state in the bench scale bubbling fluidized bed pyrolyzer. The 850µm biochar particles created a thinner segregated layer.

Steady state was achieved with corresponding increase in the feed rates but at much higher feed rates for both biochar particle sizes, removal efficiencies were less than 1.00, and the purity much more reduced as a result of excessive accumulation of the respective biochar particle sizes in the well mixing zone.

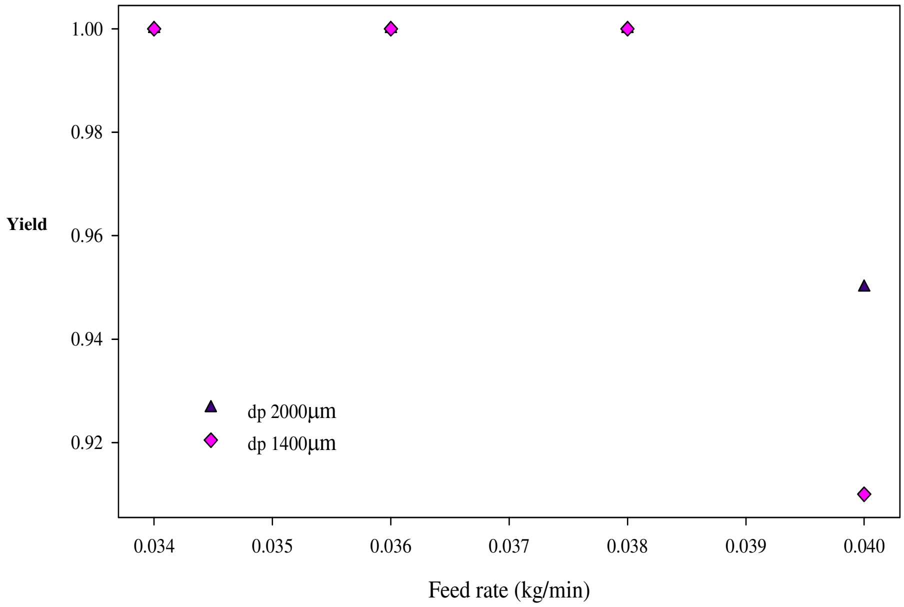


Figure 29. Effect of Particle Size 1400 µm, 2000 µm, U_{seg} = 0.0963 m/s, U_{wm} = 0.20 m/s, z = 0.23 m

Figure 3.20 above shows a steady state in the continuous segregation of biochar from the bubbling fluidized bed using the particle sizes. At a feederate of 0.040 kg/s, there was a noticeable disruption in the steady state of the system caused by unbalanced state in overloading of the biochar in the well mixed zone and removal process thereby causing the equal, thus indicating accumulation of more biochar in the well mixed zone which are unable to be drifted by the fluidizing gas into the segregation zone. However, the separation efficiency of the system not attaining steady state as the amount of biochar fed and biochar recovered are not larger 2000 µm biochar particle size fairly increased than the 1400 µm biochar particle size at steady states values

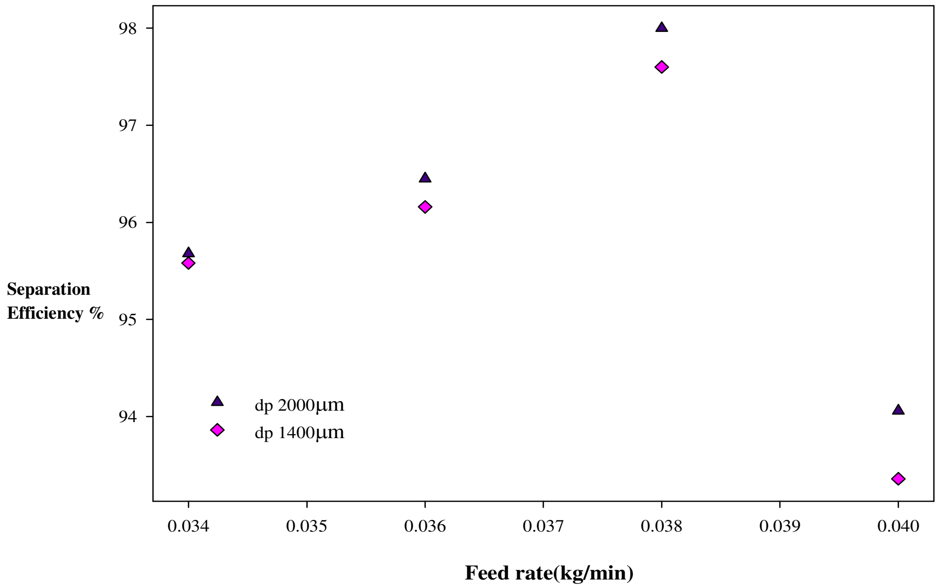


Figure 30. Effect of Particle Size on steady state U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, P_h=0.18m, z= 0.23 m

3.1.5. Effects of Biochar Feed rate.

A range of four different biochar feed rates were tested in determining the steady state yield and separation efficiency in a bubbling fluidized bed pyrolyzer in a continuous segregation mode. These feed rates 0.034 kg/min, 0.036 kg/min, 0.038 kg/min, and 0.040 kg/min were respectively used to investigate and analyze the peak operating process condition efficient for segregation of binary mixtures in bubbling fluidized bed pyrolyzer. Previously conducted experiment by Mara et.al 2010, it was also documented that increased biochar feed rates resulted in poor efficiency and decreased purity in figure 3.22

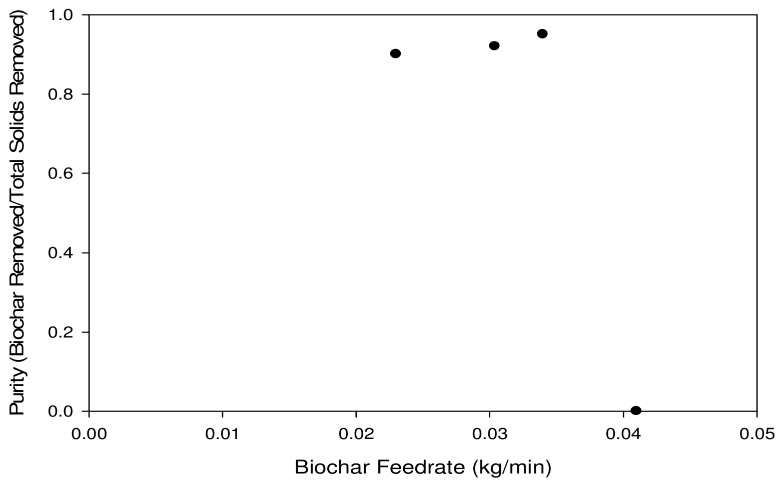


Figure 31. Effect of biochar feedrate on purity of removal (Mara et al, 2010)

Biochar feed rate constitutes a crucial variable essential to be taken into consideration in designing a pyrolyzer unit based on the fact that varying biochar feed rates into the bed influence and affects the overall efficiency and fluidization quality as well as the associated kinetics and heat transfer dynamics involved in the fluidization process. Undoubtedly proven from the experimental investigations conducted, the removal efficiency improved with biochar at feed rates between 0.034 kg/min to 0.038 kg/min resulted in increasing higher purity and with fairly noticeable decrease in the removal efficiency as a result of differences in bed compositions.

In the biochar sand binary mixture, biochar feed rate at 0.040 kg/min created general overall poor fluidization and inconsistent separation-removal quality with resulting decreased yield in biochar segregation at the segregation zone and removal through the discharge axial ports. Excessive loading of the bed with higher feedrate beyond the optimal limit of 0.038 kg/min, resulted into inconsistent yield (steady state) and poor separation efficiency outcome. Figure 3.23 below, shows transition towards achieving purity with ranges of biochar feed rates analyzed. It shows that increasing separation efficiency corresponding to increased feed rate up till 0.038kg/min beyond this point, at 0.040 kg/min, the was a noticeable poor fluidization quality in the bed.

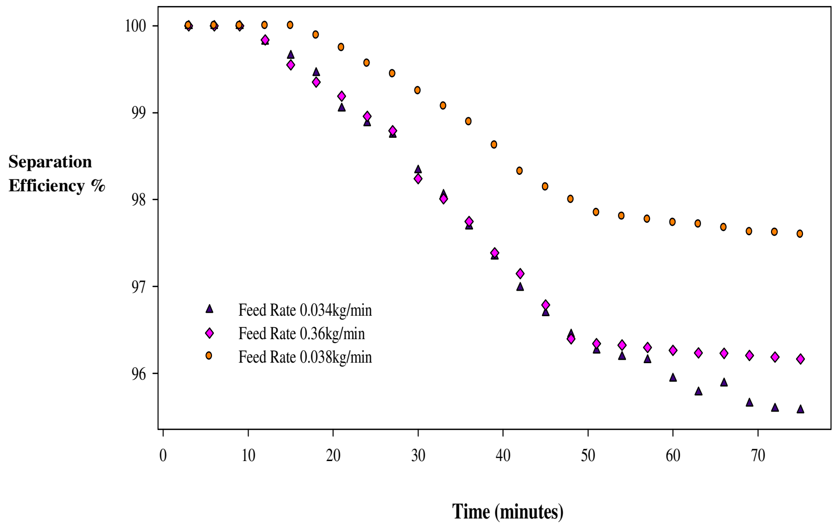


Figure 32. Effect of biochar feed rate on separation efficiency: U_{seg}=0.0963 m/s, U_{wm}=0.20 m/s, d_p 1400 μm

Although, at initial stage involving excessive biochar loaded into the system, normal performance of the fluidization process was achievable, eventually resulting to defluidization state generated as a result of increasing biochar composition before attaining the steady state. Fig 3.23 depicts a practical condition of negative and unsuccessful steady state in the biochar removal and segregation state with high biochar feed rate at 0.040 kg/min using particle size 2000 μm, similar result was achieved with biochar particle size d_p1400 μm.

The separation efficiency was decreased greatly with unreliable and unstable efficiency indicating overloading. The system reached optimum separation efficiency at a feed rate of 0038 kg/min with very high purity attaining a steady state. The system performed poorly at a feed rate of 0.040 kg/min for the two biochar particle sizes 1400 μm and 2000 μm tested. Feed rate of 0.040 kg/min biochar feed rate yielded an unsuccessful steady state with inconsistent and unsteady efficiency causing instability in the system with failure in accounting for the actual percentage of the biochar removed and remaining fraction built up in the bed. This unreliability associated with the biochar feed rate of 0.040 kg/min was attributed to negative impacts of overloading the fluidized bed pyrolyzer.

CHAPTER 4

CONCLUSIONS AND RECOMMENDATIONS.

4.1. Conclusions.

Biomass pyrolysis is fast becoming an attractive technology applied in the production of three valuable products; a rich flowing bio-oil, a solid carbon rich biochar and the pyrolytic gases. The Biochar have agronomic and environmental potentials in climate mitigation and a clean source of fuel. Fluidized beds have been widely utilized in several industrial, metallurgical, foods, pharmaceutical, and chemical applications. Fluidized beds have been extensively researched in attempts to improve various industrial and chemical processes that depend on their applications. Fluidized beds have a key advantage of promoting unique, well-mixed effects of particles which allows for continuous processing at very low temperature gradients for large and small scale operations.

Mixing and segregation are simultaneous processes that occur in fluidized beds of binary gas-solids mixtures. Uneven distribution of dissimilar solid particles in fluidized beds results into segregation phenomenon. The solids however can be of dissimilar sizes or have different densities. Non-uniform and varying concentration of Biochar in the bed can result in excessive accumulation of Biochar in the bed which invariably influences the unsteady fluidization operations resulting in decreased heat and mass transfer. In order to attain optimal fluidization steady state and process performance and to recover the valuable biochar co-product, it is advisable that the concentration of biochar in the bed be maintained at low levels.

Unlike batch systems associated with energy losses, periodically shutdowns, unstable inconsistent operation conditions and reduced yields, practical advantages of continuous solid separation removal system include Consistent Operation Conditions, Minimized Energy Losses, Cost effective Maintenance, Process Intensification, Improved Quality Yields, and High Profit Returns. There are documented researches on segregation of binary mixture of solids; however, lack of literature review on experimental data and findings on separation of biochar from binary mixture of sand-biochar mixture in a bubbling fluidized bed constitutes the practical significance in this experimental research.

Experimental investigations on the effects of geometrical and operating physical variables on separation efficiency and yield were conducted in this research and the analyses and results discussion emphasised in chapters 2 and 3. The lab scale bubbling fluidized bed unit, made it feasible for continuous segregation and removal of biochar at an optimized separation efficiency range of 96.40 % to 98 % while operating at vigorous bubbling conditions, optimizing the separation efficiency of the biochar solids was necessary at a desirable superficial gas velocity along with the best fit submergence of the vertical baffle plate height (P_h) above the bed in preventing back mixing and bubbles from the segregation zone preventing biochar accumulation into the well mixed zone which might if not addressed result into defluidization quality in pyrolysis units.

It also provided consistent and uniform bed composition of solids height unlike if the process was operated in batch mode that might result in decreasing bed solids with decreasing concentration of biochar, which is not practical for continuous segregation and removal process to be optimized efficiently resulting into poor fluidization quality. The experimental findings in previous study by (Mara et.al 2010), explained that the geometrical effects of plate height showed a qualitative depiction of both sides of the bed at near steady state with apparent density difference in both zones resulting into different bed heights. This result is comparative to the result findings in this experimental with enhanced purity levels attained and shorter duration to reach steady state. However, steady state of ŋ = 1.00 was not attained compared to a steady state reached in the recent finding.

In these experiments, the positioning of the plate height was relative to the location of the axial port so as to allow for the formation of thicker segregated biochar layer which will result in the removal of higher pure biochar with less sand. Biochar through visual observation closer to the upper layer were more pure than biochar at the interphase of the sand-biochar boundary. With reference to the effect of feedrate, (Mara et al 2010) deduced unsuccessful steady state removal with feed rate of 0.041 kg/min which is comparative to the findings conducted in the study on effects of Feedrate. There was stability in the efficiency to 80 % following the process as 80 % of biochar fed into the reactor is being removed leaving behind 20 % of the biochar accumulating in the bed by With reference to (Mara et al 2010). This however can be consider impractical as there must be significant portion of the biochar elutriated accounting for more loss of biochar in the process.

The new result findings demonstrated greater than 92 % of the fed biochar was recovered following the mixing and segregation process with steady state reached and about 3 % elutriated fraction of biochar lost and the remaining accumulated in the bed. This improved efficiency can be attributed to modifications put in place to ensure loss of biochar is avoided or practically reduced to negligible level. The modified lab-scale bubbling fluidized bed however validated the capacity of handling wide range of feedrates with increasing feedrates increasing purity achieved, exception of which is at feedrate 0.40 kg/min, there were fairly increased biochar particles in the bed and this might be probably overcome by allowing the process run for longer duration of more than 3hours or longer and not less.

The bed reached a critical limit at which the separation efficiency level dropped drastically indicating overcapacity, however the cold simulator lab-scale bubbling fluidized bed reactor offers continuous high purity removal of the segregated biochar layer formed. The effects of the cross sectional area or separation zone area can be best discussed as a result of loss of free space for the biochar to migrate to the segregation zone impairing a negative effect on the fluidizing reactor unit creating reduced biochar concentrations in the segregation zone.

In addition, the plate's prongs may also induce detrimental effects in fragmenting bigger bubbles flowing from the sparger tubes in the vigorously bubbling well mixed zone thereby causing decreased gas flow and preventing maximum purity level to be reached. The lab scale bubbling fluidized bed system was shown to work over a range of many feedrates (0.034 – 0.038 kg/s) which are in respect with to biochar generation rates present in pyrolysis units. The segmented fluidizing system was able to operate under two different gas velocities promoting intense particle mixing at high gas velocity and lower gas velocity to achieve biochar particle segregation and removal. Decreasing the $A_{sep}/A_{wm} < 38\%$ showed reduced system performance.

However, the lab-scale bubbling fluidized bed permitted higher biochar removal separation efficiency greater than 96% from the fluidized bed, compared to 93 % separation efficiency findings by Mara et.al, 2010. In determining the acceptable limits of biochar feed rates feasible for continuous segregation and removal of biochar in a bubbling fluidized bed pyrolyzer, feed rates of 0.034 kg/min of particle size 2000 μm resulted in 95.68 % separation efficiency, 0.036

kg/min gave a biochar separation efficiency of 96.45 %, a fairly higher purity of 98.00 % with 0.038 kg/min biochar feed rate. Low percentage separation efficiency of 94.06 % was obtained in higher feedrate of 0.040 kg/min as a result of excess biochar accumulation in the well mixed zone, leading to system failure and creating unsteady state.

This can also be compared with increasing separation efficiencies encountered with biochar particle size of 1400 μm yielding a separation efficiency of 93.63 % with 0.034 kg/min, 96.16 % purity with 0.036 kg/min, and 97.60 % separation efficiency with 0.038 kg/min and eventual process failure with higher feedrate of 0.040 kg/min at 93.36 % separation efficiency. Satisfactory system performance was validated effective with achievable steady state and purity levels in feed rates between 0.034 kg/min and 0.038 kg/min in both particle sizes of 1400 μm and 2000 μm. Through the fluidization and segregation operations, noticeable differences in bed heights of the two zones were taken into account.

At steady state, there exists steady equilibrium between the evolved flotsam and the residual fed biochar when the both sand and biochar solids are subjected to fluid-like state as in the case of density segregation of solids. At steady state, the two zones attain different bed height influenced by variation in the positioning of the vertical plate and different gas velocities in the bed. The observable bed height difference was due to differing densities in the two partitioned zones. The fluidized bed facilitated an acceptable continuous removal of biochar at a consistent gas velocity.

Furthermore, this fluidized bed unit demonstrated the capability of operating over wide range of federates of 0.034 kg/min-0.040 kg/min, segregating biochar at continuous and steady degree as encountered in pyrolysis operations, an indicator to desirable process stability, design reliability and dependability for increased quality and economic profitability. The built and optimized novel unit has capability of high purity levels in the removal of separated biochar at consistent rate in relation to the generation rates similar to pyrolysis unit. Following modifications of the bubbling fluidized bed pyrolyzer, the unit proved efficient in handling range of feed rates and also capable of processing different particle sizes attaining very high separation efficiency and stability states.

In summary, ccomparisons with (Mara et al 2010) previous findings, the present experimental analyses with Adegboye 2013, produced better results with improved separation efficiency of 92 % unlike 80 % separation efficiency achieved with (Mara et al 2010) result. The maximum flow char rate was much enhanced to 0.038 kg/min while that of (Mara et al 2010) was capable of a maximum char rate of 0.036kg/min. In addition, the elutriation encountered in (Mara et al 2010) accounted for > 20 % fraction of the biochar fed, while the presently conducted and investigated findings successfully documented elutriation of < 3 % fraction of the biochar fed into the lab scale bubbling fluidized bed unit.

4.2. Future Work Recommendations.

1. The use of Particle Tracking Technology (tagging labelled simulated char particles) to investigate the behaviour of individual particles in mixing and segregation zones.

2. Analyzing the biochar loading concentrations on both well mixed zone and segregation zone.

3. Extensive Process Optimization with varying wide range of Particle Sizes of binary mixture (Sand and Biochar) with individual mass balances following segregation.

4. Modelling the effects of various factors such as using different sand masses, different char from other biomass sources, might be an interesting area of focus on in determining the influence of heat and mass transfer characteristics in the pyrolysis unit.

5. Experimental investigations on procedures to scale up the unit and outcomes of scaling effects.

6. Further optimization and adaptation for large industrial applications can be embarked on with further research investigations.

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