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ULTRA CLEAN COAL PRODUCTION USING DENSE MEDIUM SEPARATION FOR THE SILICON MARKET

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ULTRA CLEAN COAL PRODUCTION
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THESIS

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

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ABSTRACT OF THESIS

ULTRA CLEAN COAL PRODUCTION USING DENSE MEDIUM SEPARATION FOR THE SILICON MARKET

The production of high quality silicon requires the use of ultraclean coal containing less than 1.5% ash. The magnetite used to clean the coal in a dense medium process is a contaminant that seriously impacts the quality of the final silicon product. As such, research has been conducted to evaluate the potential to substitute the magnetite with fine silica–based alternative material generated during the silicon production process. Dense medium cyclone tests were performed based on a statistically designed program to determine the optimum conditions that maximize organic efficiency and minimize probable error and low–density bypass. The results revealed that a clean coal product with less than 1.5% ash can be produced using a medium formed from the silicon production waste with an organic efficiency value of around 99% and a probable error value below 0.02. There was no measurable bypass of high density particles into the product stream or low–density particles into the reject stream.

Keywords: Dense medium cyclone, Medium stability, Medium rheology, Magnetite, Silicon

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DEDICATION

This work is dedicated to my mother Nahid Mousavi, and my father Ebrahim Amini, for their everlasting support, trust and love. Also, I dedicate this work to my sister Zahra, my brothers Hamed and Morteza as well, who I care about so much. Without my family this work would not have seen the light of day.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

1 INTRODUCTION

1.1 Preface

Silicon is the second most abundant element by weight in Earth's lithosphere. It occurs naturally, not in the free form, but in the form of oxides and silicates to create over 25% of Earth's crust (Dosaj, Kroupa, & Bittar, 2005; Yaroshevsky, 2006). Metallurgical grade silicon is obtained by the carbothermic reduction of silica–rich materials (i.e., quartz and quartzite) with carbonaceous reduction materials (i.e., coal, woodchips, charcoal and etc.) in a submerged arc furnace (O'Lenick Jr & Siltech, 2009; Anders Schei, Tuset, & Tveit, 1998).

The choice of carbon raw materials depends on the availability of carbon material, process parameters, product quality requirements, and environmental considerations. However, the quality of reduction material is perhaps the main concern to maximize silicon yield through an efficient smelting process and to satisfy the final silicon product specifications. Therefore, the amount and type of impurities, such as Fe, B, and P, contained in the carbon material is one of the main issues (Aasly, 2008; Gasik, 2013; E. Myrhaug, Tuset, & Tveit, 2004; Myrvågnes, 2008).

Coal is a fossil carbon material which is used fairly commonly as a reducing agent in silicon production. The main three properties for the characterization of coal are: coal grade or purity, coal type or maceral composition, and coal rank or maturity. The qualification of coal based on these factors is important since the type and amount of organic and inorganic materials in coals as well as rank of coal dramatically affect the silicon smelting process and final product specifications (Myrvågnes, 2008; Raaness & Gray, 1995; Speight, 2012).

Coal preparation technology plays a significant role in the silicon market by upgrading run–of–mine (ROM) coal to satisfy the particle size and quality specifications. Modern coal preparation plants consist of a complicated arrangement of solid–solid and solid–liquid separation processes with the objective of improving the purity of coal by reducing detrimental impurities; and the control of final product lump size to meet the designated product requirement which is dictated by the silicon production industry (Gluskoter, 2009; Meyers, 1981).

The dense medium cyclone (DMC) is one of the most widely applied gravity concentration units for cleaning ROM coal particles greater than 1 mm. The majority of the DMCs utilize a dense medium consisting of a suspension of finely ground magnetite to produce a low ash clean coal which is applied commercially in silicon production industry. The separation in a dense medium strongly depends on the weight distribution in the coal as a function of particle density, particle size and the stability and rheology properties of the medium (Gluskoter, 2009; YB He & Laskowski, 1994).

1.2 Problem Statement

Since the purity of the final silicon product is strongly determined by the type and level of contamination in the raw materials, the reduction in the amount of impurities is of the utmost importance in the silicon production. There are three main sources of contamination in silicon smelting process, i.e.:

1. Basic level of impurities present in the raw material composition;
2. External impurities which dilute the raw materials during preparation, handling and transportation;
3. Contamination by the material which is used for the construction of arc furnace.

The presence of magnetite in the final coal product, which has a significant effect on the purity of final silicon product, is the most common example of external impurities in the silicon smelting process. Small quantities of magnetite are present due to inefficiencies in rinsing the coal after cleaning in a dense medium separator. Due to electrical activity of iron, it is considered as the main impurity in silicon used in semiconductor industries. The critical level of iron impurity concentration in semiconductor industries is decreasing annually as sensitivity of devices yield to iron impurity is increasing due to the device dimension shrinkage.

1.3 Objectives

The goal of this research is to evaluate alternative materials that can be used to replace magnetite in dense medium cyclone circuit. Alternative materials include fine silica sand, fine silicon, and ultra–fine silica fume (micro–silica), of which the latter two materials are byproducts of the silicon production process. Silica contamination is not an issue since silica–rich materials are used as the raw materials in the silicon production. The ability of silica to be used to form the media is possible due to the low gravity cut points needed to achieve the required coal purity.

In summary, the itemized objectives of this study are to:

- Conduct the initial static stability tests to evaluate the medium stability that the alternative silica–based materials provided over a range of medium density values;
- Indirectly quantify the dynamic medium stability by measuring the difference in the pulp density in the underflow and overflow streams of the dense medium cyclone under various inlet pressures ranging from 3 to 8 psi (these pressures provide the recommended cyclone head for industrial units);
- Perform a detailed experimental program to determine the separation performance and efficiency achievable when treating 9×1 mm Blue Gem coal using a dense medium cyclone;

- Investigate the individual and interactive effects of various operating conditions on the DMC separation performance when alternative silica–based mediums are utilized;
- Identify the optimum conditions in which not only the production of clean coal containing below 1.5% ash is achievable but also the maximum separation efficiency can be obtained.

1.4 Thesis Organization

The body of this thesis is organized into four informative chapters, while an introductory and a concluding chapter complete the thesis.

Chapter 2 comprises a brief introduction to silicon production. An overview of raw materials and their specifications is given and the basic principle of silicon smelting process is described. This chapter describes the special emphasis on the source of contaminations in the silicon production and also presents the detrimental effects of iron, which is the main impurity in the silicon smelting process.

Chapter 3 provides a short review of the modern coal preparation plants in general and dense medium cyclone process in particular. The stability and rheological properties of dense mediums and their interactive effects with the operating conditions in determining DMC performance is described.

Chapter 4 introduces the experimental methods and conditions under which the various tests are conducted to determine the stability characteristics of the alternative mediums. The pilot–scale dense medium cyclone circuit is described and the basic definition of separation efficiency parameters is reviewed.

Chapter 5 provides the detailed discussions of the static and dynamic stability of silica–based alternative materials and the results of coal cleaning tests which are conducted based on a parametric design. The chapter presents the optimum conditions under which the maximum separation efficiency is achievable to produce clean coal containing below 1.5% ash is presented.

2 SILICON PRODUCTION

2.1 Introduction

In the liquid state, silicon characteristics are relatively similar to metals (high thermal and electrical conductivity); but in the solid state, silicon is a low–density element with nonmetallic properties. Silicon is a semiconductor and is called metalloid. Because of its optical similarities (especially in color) to metals and application for the production of several metals products, such as manganese alloys and chromium alloys, it is deceptively called silicon metal (Dosaj et al., 2005). Another common technical error is confusing silicon, which is used to refer to the elemental material (Si), with silicone, to refer to the material in which silicon is bonded to oxygen (O'Lenick Jr & Siltech, 2009). Before the 19th century, all elements were produced using reductants materials, such as hydrogen and carbon. In 1824, Swedish chemist Jons Jacob Berzelius discovered silicon and named it "silicium". Later on, the Scottish chemist Thomas Thomson renamed it to "silicon" since the new element has relatively similar characteristics to boron and carbon rather than magnesium and calcium (Gasik, 2013).

The development of the first industrial process of silicon production occurred nearly a century ago. The commercial production of silicon in the various forms of alloys began with the development of the electric arc and blast furnaces by Paul Héroult. In 1907, silicon could be produced in the first commercial plant in the United States by Frank Tone of Carborundum Corporation. In the 1920s and 1930s, the application of silicon in aluminum alloying and chemicals and silicones increased dramatically. After the 1950s, highly pure silicon was applied in the semiconductor industry. Currently, the most critical application of pure silicon is in the electronic industry. To use silicon in electronics, the metallurgical grade Si (MG–Si) must be further refined and purified to fulfill specifications. Among all metallic impurities, iron has always been considered the main impurity in silicon used in semiconductor industries due to its high electrical activity (Dosaj et al., 2005; Gasik, 2013;

Istratov, Hieslmair, & Weber, 1999, 2000; Moissan & Lenher, 1920; Anders Schei et al., 1998).

Silicon reaction with more than half of elements in the Periodic Table forms different types of silicides, which are unique materials both in terms of their chemistry and in their various applications. The refractory metal and noble metal silicides are used in the electronics industry. In the steel and iron industry, silicon is used to enhance mechanical characteristics of final product (Bimbo, 2012; Dosaj et al., 2005).

2.2 Properties of Silicon

Silicon is the second most abundant element by weight in Earth's lithosphere. It occurs naturally not in the free form but in the form of oxides and silicates to create over 25% of Earth's crust (Dosaj et al., 2005; Yaroshevsky, 2006). Abundances of some chemical elements in the upper continental crust are shown in Table 2.1.

Table 2.1: Abundances (10^{-4}wt %) of chemical elements in the upper continental crust.

Element	Vinogradov, 1962	Ronov et al., 1990
O	470000	479000
Si	295000	296300
Al	80500	79000
Fe	46500	40600
Ca	29600	29200
K	25000	23600
Na	25000	22400
Mg	18700	17100
Ti	4500	3220
Ca	230	2960
Si	470	1080
Mn	1000	670
P	930	620
Fe	660	530
Cl	170	220

Silicon is the 14th element in the periodic table and belongs to group IV with an atomic weight of 28.08. As an analog of carbon, silicon has oxidation states between 1 and 4, the latter being the most stable. Silicon lattice has a cubic, face–centered (FCC), diamond–type structure with lattice period 0.54307 nm at ambient conditions, giving silicon a density of 2.33 g/cm^3. The melting point of silicon is 1414°C, and the boiling point is 3250°C (Dosaj et al., 2005; O'Lenick Jr & Siltech, 2009). The density of liquid silicon is temperature dependent, as described as follows:

$$\rho_{liq} = 2552 - 0.45(T - T_m) \quad (1)$$

where T_m is the melting temperature.

Liquid silicon solubilizes most elements to a low degree, and hence the density will not greatly depend on dissolved elements (Gasik, 2013; Anders Schei et al., 1998). However, if iron is present, it will dramatically affect the density.

Table 2.2: Typical properties of Silicon (Dosaj et al., 2005).

Property	Value
Atomic weight	28.09
Boiling point, °C	2787
Density, g/cm^3	2.33
Hardness, Moh	7
Heat capacity, cal/g.mol °C	4.78
Liquid density at mp, g/cm^3	2.533
Liquid heat capacity at mp, cal/g.mol °C	6.755
Melting point, °C	1412±2
Poisson's ratio	0.27
Refractive index	3.4
Shear modulus, dynes/cm^2	7.55×10^{11}
Surface tension at mp, dynes/cm	736
Thermal conductivity cal/sec.cm. °C	0.353

2.3 Raw Materials in Silicon Production

Metallurgical grade silicon is obtained by the carbothermic reduction of silica–rich materials with carbonaceous reduction materials in a submerged arc furnace. The thermal reaction between these materials occurs at very high temperature and therefore is commonly carried out where low–cost power is available. The overall reaction for silicon production is expressed by reaction 2; however, the industrial production of silicon is more complicated than this reaction (O'Lenick Jr & Siltech, 2009; Anders Schei et al., 1998).

$$SiO_2(quartz) + 2C(coal, woodchips, coke) \rightarrow Si + 2CO \qquad (2)$$

Quartz and quartzite are the main sources of silica–rich materials in silicon production. Carbon reductant materials may be categorized in two main groups: fossil carbon materials, such as coal and petroleum coke, and biological carbon materials like woodchips and charcoal (Gasik, 2013; Myrvågnes, 2008).

Typical blend of raw material in the silicon production consists of 52 wt% quartz, 26 wt% coal, 10 wt% charcoal and 12 wt% wood chips. To produce one metric ton of MG–Si the electrical power consumption will be between 11 to 14 MWh (Dosaj et al., 2005; Myrvågnes, 2008).

2.3.1 Sources of Silica Raw Material

The fairly uncontaminated source of silica, which is referred to as mineral quartz, formed in pegmatite and hydrothermal bodies. Quartzite is a hard metamorphic rock which was originally quartz–rich sandstone. Pure quartzite is white to gray although type and level of mineral impurities would change its color to yellow, red or blue. From metallurgical point of view, quartz is used for low contamination silica source whilst quartzite is utilized for more impure raw material (Dosaj et al., 2005).

Considering availability, characteristics, and purity, quartz is the most common source of silica for the production of MG–Si. In order to fulfill the silicon product specifications and also optimize the process, the industry has considered the following properties for the quartz (Gasik, 2013):

- Chemistry (e.g., Fe, Al, Ti, B, P, and Ca content)
- Lump size (typically 10 to 150 mm)
- Mechanical and thermal strength (sometimes considered in combination)
- Softening behavior

Type and amount of impurities in the mineral quartz are possibly the most critical concern in the silicon smelting process. Some elements, such as iron, aluminum, titanium, boron, phosphor, and calcium, will finish up in the silicon final product (see section 2.5). Some of these elements may be removed from the silicon by oxidation; however, the most economical method to control the level of other impurities (e.g. Fe) is limiting the amount of detrimental elements in the raw materials. The presence of alkalis elements (e.g. Na and K) influences the melting point of Si, thereby affecting the silicon production process (Gasik, 2013). Typically, the requirements for the amount of contaminants in the quartz are: SiO_2 >99%, Al <0.5%, Fe <0.3%, alkali <0.25% and Ti <0.02% (Aasly, 2008).

The lump size requirement is the most significant variable affecting the operation in the arc furnace. Generally, quartz raw material, with particles between 10 to 150 mm, provides the most optimum conditions in the furnace even though each plant may require narrower or finer size particle distribution based on its product specifications and technological limitations (Aasly, 2008; Gasik, 2013).

Different types of mechanical loads are applied to the mineral quartz during drilling, blasting, transportation and handling of the raw materials. However the most detrimental mechanical load comes from the dropping stage, when a raw material is dropped from the vessel to the arc furnace (height is nearly 70 meter). The mechanical strength of the mineral quartz depends upon the type of quartz, mining and transportation methods. The low mechanical strength causes significant size reduction, in turn, creating difficulties in the silicon production process within the arc furnace. Mineral quartz with particle sizes below 2 mm not only is problematic in carbothermic operation but also is usefulness for other markets. Moreover, increasing the amount of fine particles decreases the permeability of charge in the furnace, thereby decreasing Si yield. Another problem is related to a popping effect where in some cases fragments of quartz may be thrown up into the air (Aasly, 2008; Gasik, 2013).

Thermo–mechanical strength of mineral quartz is one of the most important characteristics of raw material which has considerable influence on the operation. Generation of micro cracks in the quartz particles during mining and handling operations results in the reduction of the thermo–mechanical strength of the particles. Quartz with low thermal stability that disintegrates within the charge may also contribute to slag formation in the furnace. Ideally, the lumpy quartz should keep its original size as it moves down through the charge, until the quartz starts to soften and melt in the lower parts of the furnace (Aasly, 2008; Senapati, Maheswar, & Ray, 2007).

Although the chemical composition, particle size distribution, and the mechanical strength are the most applied characterization methods for the feasibility of quartz, other parameters, such as the softening and melting temperature of quartz must be evaluated. When the melting temperature of the quartz is low, it leads to the formation of a sticky mass with the rest of the charge materials. This will prevent gas flow through the charge causing gas channels to form, which results in a high SiO percentage loss and low Si yield (Aasly, 2008).

2.3.2 Carbon Reduction Material

The carbonaceous materials are used as a reducing agent in the silicon production. The choice of carbon raw materials depends on the availability of carbon material, process parameters, product quality requirements, and environmental considerations. The most prominent role of carbon materials is to react with SiO gas to produce SiC in the silicon furnace. SiO reactivity, which is defined as the ability of carbon reductants to react with SiO, is one the most important factors in the silicon production that dramatically affect Si recovery, energy consumption and profitability of silicon smelting process. Carbon reduction materials can be classified based on their reactivity towards SiO gas by conducting a SINTEF test (Gasik, 2013; E. Myrhaug et al., 2004; Myrvågnes, 2008).

The quality of reduction material is perhaps the main concern to maximize silicon yield through an efficient smelting process, and to satisfy the final silicon product specifications. Therefore, the amount and type of impurities, such as Fe, B, and P, contained with the carbon material is one of the main issues (Aasly, 2008; Gasik, 2013; Myrvågnes, 2008).

The particle size distribution of the carbon materials is one of the most important properties that have effect on the silicon smelting process. It affects the permeability of charge burden as well as SiO reactivity in the furnace. The typical sizes of the carbon reductants are between 1 to 30 mm. A high amount of fine particles reduces the permeability of gas and also increases the SiO reactivity. In addition, the gas flow may disperse the fine carbon particles into the air, thereby reducing the amount of carbon reduction materials available for Si reduction. Hence, carbon materials must have high amount of mechanical strength to avoid producing high amount of fine particles during preparation, handling and transportation (Myrvågnes, 2008; Anders Schei et al., 1998).

Some characteristics of carbon materials, such as high reactivity and thermal stability, result in reliable and complete conversion of oxide (SiO_2) to pure silicon (Si). Thus, carbon materials must be stable and highly reactive at the temperature of 2500°C and above (Strakhov, Surovtseva, Elkin, Elkin, & Cherevko, 2012).

Considering the availability, cost and quality of raw materials as well as different experiences, various blends of carbon materials are used in silicon smelting process worldwide. Among all carbon reduction materials, woodchips is the only common material that is used worldwide by all silicon producers. Comparison of the various compositions of the carbon materials for silicon production with basis on fossil materials and charcoal

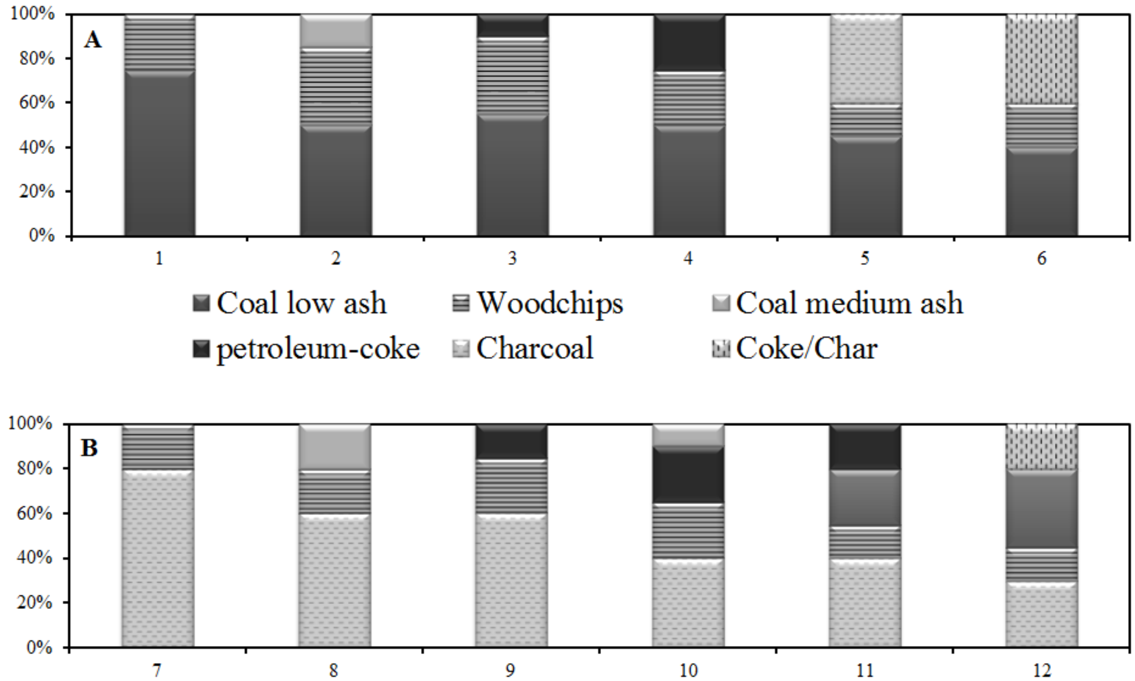

Figure 2.1: Different blends of carbon reductants with basis on (A) fossil materials and (B) charcoal (Myrvågnes, 2008).

are presented in Figure 2.1. The most common carbon reductants used in silicon processing are coal, charcoal, petroleum–coke, coke and char for reduction and woodchips for permeability purposes (Gasik, 2013; Myrvågnes, 2008).

Coal is a fossil carbon material which is used fairly commonly as a reducing agent is the silicon production. The main three properties for the characterization of coal are: coal grade or purity, coal type or maceral composition, and coal rank or maturity. The qualification of coal based on these three factors is important since the type and amount of organic and inorganic materials in coals as well as rank of coal dramatically affect the silicon smelting process and final product specifications (Myrvågnes, 2008; Raaness & Gray, 1995; Speight, 2012).

Metallurgical coke is a solid residue from the controlled carbonization of high rank coal. Majority of produced metallurgical cokes, which have high mechanical strength and low reactivity with CO_2, are applied in iron pig production. Due to the low correlation of metallurgical coke characteristics with the requirements of reduction material in silicon smelting process, char and reactive coke are utilized more frequently than metallurgical coke in silicon production (Myrvågnes, 2008).

Char is a production of controlled carbonization process of wood or special kinds of coal. Due to high amount of metal impurities in the char, majority of silicon producers prefer to not use char as a reductants (Myrvågnes, 2008). Typical properties of char are shown in Table 2.3.

Charcoal is a biological carbon reduction material in silicon production. Carbonization of wood at a moderate rate of temperature results in the production of charcoal. Special properties of charcoal, such as high reactivity and low amount of impurities, make this carbon material the primary source of raw materials comprising most silicon production circuits. Table 2.3 revealed the main characteristics of charcoal (Byrne, Marsh, & Patrick, 1995; Myrvågnes, 2008).

Table 2.3: Comparison of properties of various carbon reductants.

Parameter	Coke	Nut–Coke	Half–Coke	Petro–coke	Charcoal
Ash (% wt)	8–11	8–11	22–27	<1	<2
Volatiles (% wt)	<2	<1.5	4–6	7–9	10–15
Moisture (% wt)	<0.5	<1.5	<2	<1	1.5–2.5
Sulfur (% wt)	<1	<1.5	<1	1–5	<0.5
Solid carbon	>85	>85	>70	>85	>80
Reactivity at 1323 K, ml/(g.s)	>0.5	>0.9	>8	>0.3	>10
Resistivity, Ohm.m, for 3–6 mm	1–2	1–2	7500	3.10^6	2.10^6
Structural strength, %	>80	>85	>62	>62	>35
Density (g/cm3)	1.82	1.95	1.58	1.41	1.40
Ash composition (% wt):					
SiO2	33–37	35–37	75–78	45–48	<2
Al2O3	20–25	21–23	10–12	23–26	<4
CaO+MgO	3 to 5	3–5	3–5	9–11	40–45
Fe2O3	30–35	30–35	7–9	13–15	<1
P2O5	0.1–0.3	0.1–0.3	<0.05	0.5–1.0	5–6
K2O+Na2O	2–2.5	2–2.5	1–2	0.1–0.2	<0.5

Petroleum coke, which is also called petro–coke in some references, is a by–product of oil refining process. Petro–coke with very low ash content may be used in silicon production (Myrvågnes, 2008).

Woodchips is used most commonly as a reductant in silicon smelting process. Applying woodchips in the arc furnace results in high improvement in permeability, thereby providing excellent gas dispersion and minimum blowing phenomena (Myrvågnes, 2008).

2.4 Silicon Production Technology

2.4.1 Basic Principle of Operation

The most economically viable route to produce metallurgical grade silicon and possibly very high purity silicon is by carbothermic reduction of silica rich materials with carbon reduction agents in a three–electrode, a–c submerged electric arc furnaces. During the last century numerous developments have been occurred regarding safety and environmental aspects and computerization of the control system. However, fundamental of silicon smelting process is still the same. The overall ideal reaction of the silicon production was previously presented in Eq. 2. Although this process is basically simple, the industrial production of silicon is more complicated. In addition to the metallurgical part of the process, safety and environmentally aspects of the silicon production make it more complex (Gasik, 2013; Gribov & Zinov'ev, 2003; E. Myrhaug et al., 2004; Myrvågnes, 2008). The principle parts of a modern silicon production plant are illustrated in Figure 2.2.

As Shown in Figure 2.2, most silicon production plants can be taken apart into the raw material handling unit, furnace electrical system, energy recovery and gas purification unit, and the final product processing circuit(Myrvågnes, 2008; Valderhaug, 1992)**.**

According to the daily silicon production and specifications of the raw materials, silica and carbon materials are transported for weighting and mixing processes. Consequently, mixed

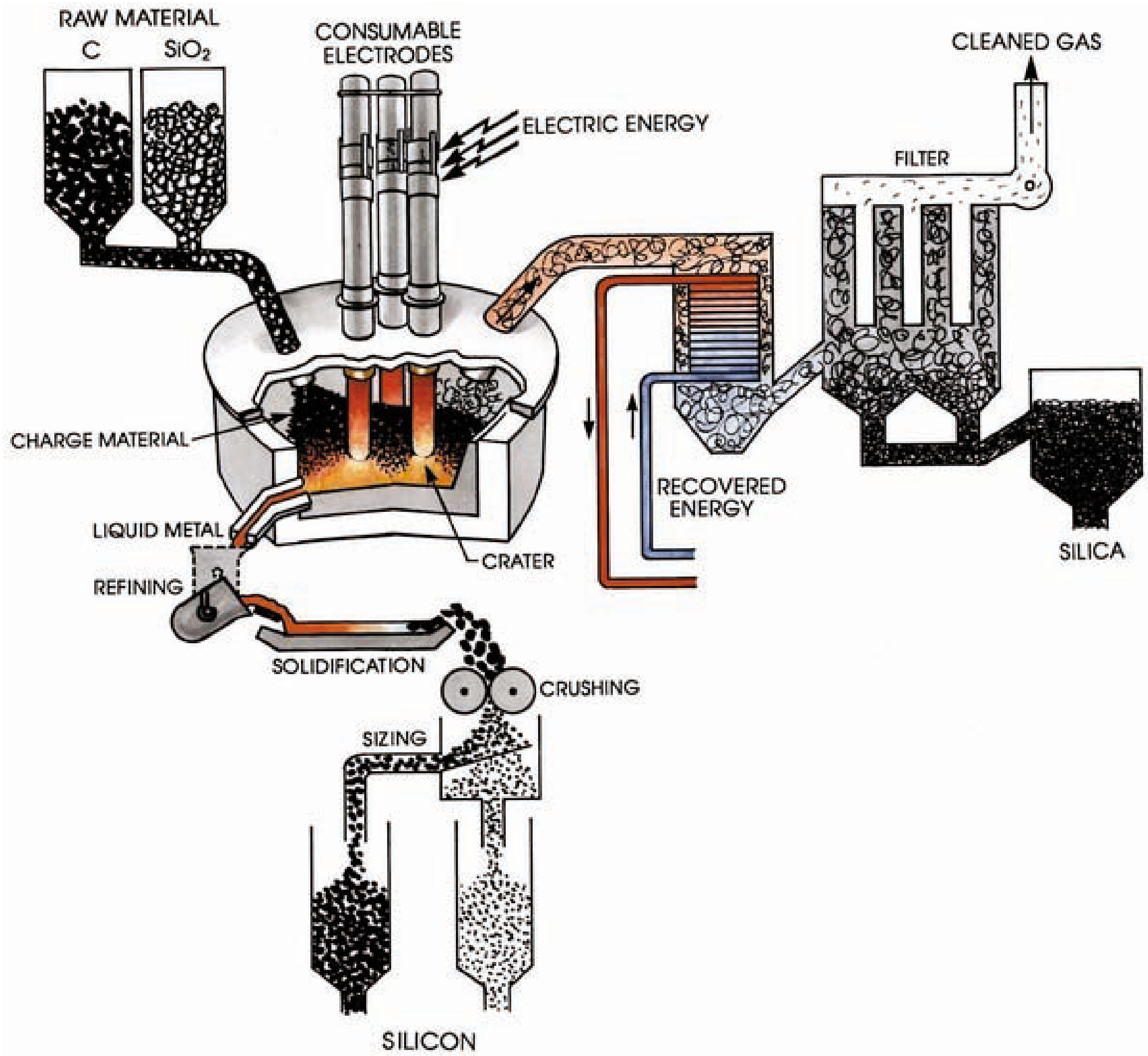

Figure 2.2: Principle parts of a modern silicon production plant (Schei, Tuset et al. 1998).

raw materials are moved into the submerged arc furnaces (Myrvågnes, 2008; Valderhaug, 1992).

Based on the production capacity, silicon plant contains one or more arc furnaces. The raw materials are heated in the furnace supplying high amount of electrical energy through three electrodes which are placed at the corners of a regular triangle and submerged into the charged materials. Finally, molten pure silicon tapped out from the lower part of the furnace. Size of the furnace and the level of technology used for the silicon production may be different in silicon plants; however, equipment for transportation of raw materials, furnace body and equipment for tapping the molten silicon out of the furnace have the relatively same principle (Kadkhodabeigi, Tveit, & Johansen, 2011; Myrvågnes, 2008; Valderhaug, 1992).

During the silicon smelting process, at very high temperatures, large amount of waste gas containing high amount of silica fume (0.2 – 0.4 tons per produced ton of molten silicon) leaves the furnace. Loosing this amount of off–gas without purification, not only is problematic for the environment but also large amount of energy and silica fume (micro–silica) is wasted. Utilizing the heat in the off–gas, nearly a third of energy using in the plant can be regenerated. As energy is one of the main sources affecting profitability and sustainability of the process, energy recovery unit may be one of the main necessities in silicon production plants. The condensed silica fume, as the main by–product in the process, can be filtered in dust filter units to be used as a filter to concrete, ceramics, refractory, rubber and etc. (Gasik, 2013; Myrvågnes, 2008; Valderhaug, 1992).

The level and type of impurities in the molten free silicon is proportional to the type and amount of contamination in raw materials. While the silicon product is still liquid, the impurities less noble than silicon, such as aluminum and calcium, can be removed through the oxygen refining circuit. After the oxygen refining step, the molten silicon is cooled and solidified during the solidification process. In order to avoid adding contamination to the final product the casting process occurs in big bed lined with fine silicon particles. Consequently, the final solid silicon product is crushed and screened to produce the desired product (Gasik, 2013; Myrvågnes, 2008).

2.4.2 Chemical Reactions in the Furnace

The furnace is the heart of the silicon production plant. Typically, arc furnace consists of the furnace body, which constructed from the steel shells, and the furnace lining. The materials which are used for lining of the furnace body must not only withstand high mechanical stress and temperatures but also chemically resist when molten silicon and oxide flow through it. Most modern furnaces are semi–closed, which has a partly open above the furnace chamber. The most prominent advantage of these types of open–top furnaces is that materials inside the chamber are accessible during the production of silicon

(Gasik, 2013; Myrvågnes, 2008). The structure of typical submerged arc furnace showed in Figure 2.3.

After weighting and mixing raw materials, silica and carbon raw materials are charged into the submerged arc furnace. Immediately, inside temperature is increased to 700–1300°C by high temperature gases rising in the arc furnace. In this environment, different chemical reactions can occur, based upon the carbon coverage. Carbon coverage may be defined by parameter x in the reaction between silica–rich materials and carbon reduction agents as following: $SiO_2 + xC$ (Dosaj et al., 2005; Anders Schei et al., 1998). Different values of x results in producing desirable or undesirable products according to the reactions:

$$SiO_2(s) + 2C(s) = Si(l) + 2CO(g) \tag{3}$$

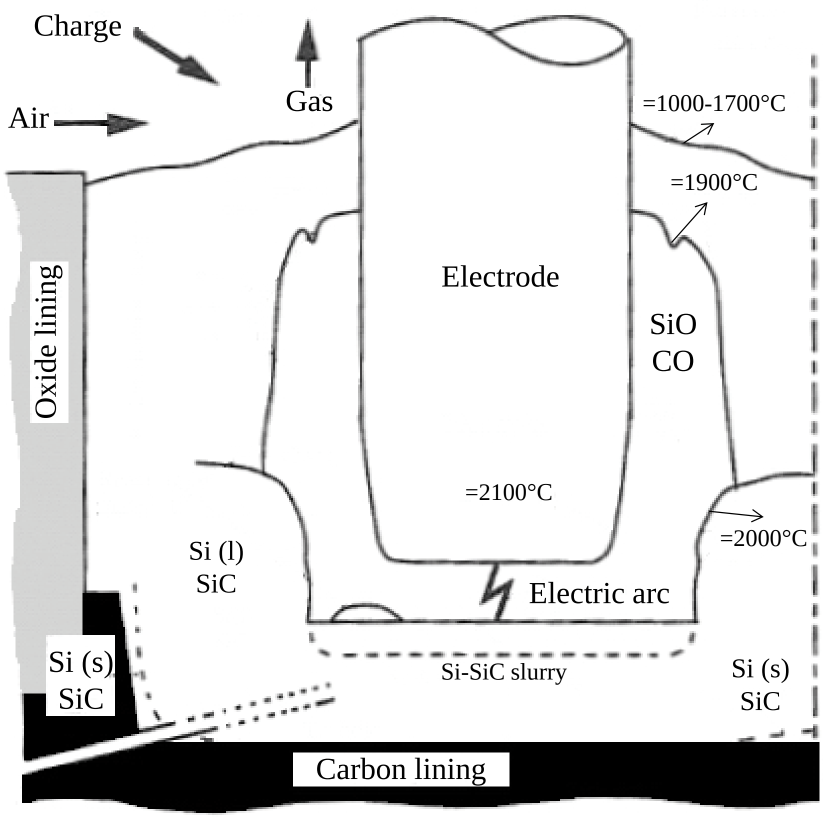

Figure 2.3: The inner structure of a silicon arc furnace (Schei, Tuset et al. 1998).

$$SiO_2(s) + C(s) = SiO(g) + CO(g) \quad (4)$$

$$SiO_2(s) + 3C(s) = SiC(s) + 2CO(g) \quad (5)$$

The ideal reduction process of the silica–rich material is given by reaction 3 with optimal carbon coverage of 2, which results in the production of free molten silicon. Silicon recovery is decreasing dramatically when carbon coverage is lower or higher than optimal carbon content as illustrated in Figure 2.4. According to reaction 4, when carbon content in the charge is lower than optimal (equal to 1) high amount of silicon in the form of SiO gas leaves the furnaces, thereby vividly reducing silicon recovery. An over–coked charge is characterized by carbon coverage higher than optimal value that results in accumulation of SiC at the bottom of the arc furnace as well as low Si yield. This is expressed by setting x equal to 3 in the reaction 5.

The liquid pure silicon is produced in the high temperature area of the arc furnace denoted the inner reaction zone. As oxygen is available at the outer reaction zone, which is the cooler top area of the furnace, SiO gas will oxidize producing silicon in the form of micro–silica (SiO_2) (Myrvågnes, 2008). In Figure 2.5, a stoichiometric model, in which the main chemical reactions in the inner and outer reaction zones as well as material flowing in the silicon smelting process occur, is demonstrated.

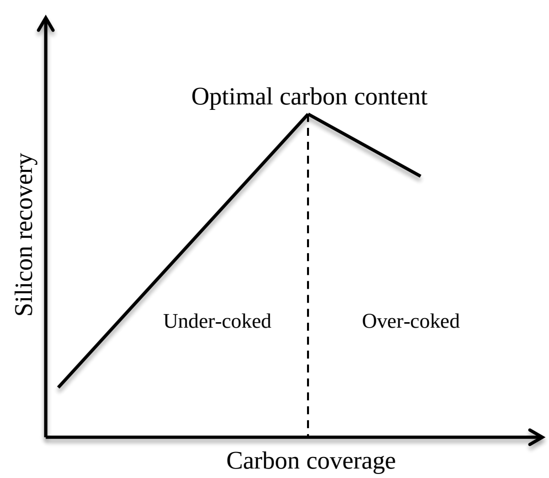

Figure 2.4: Silicon recovery as a function of the carbon content in the charge (Myrvågnes, 2008; Valderhaug, 1992).

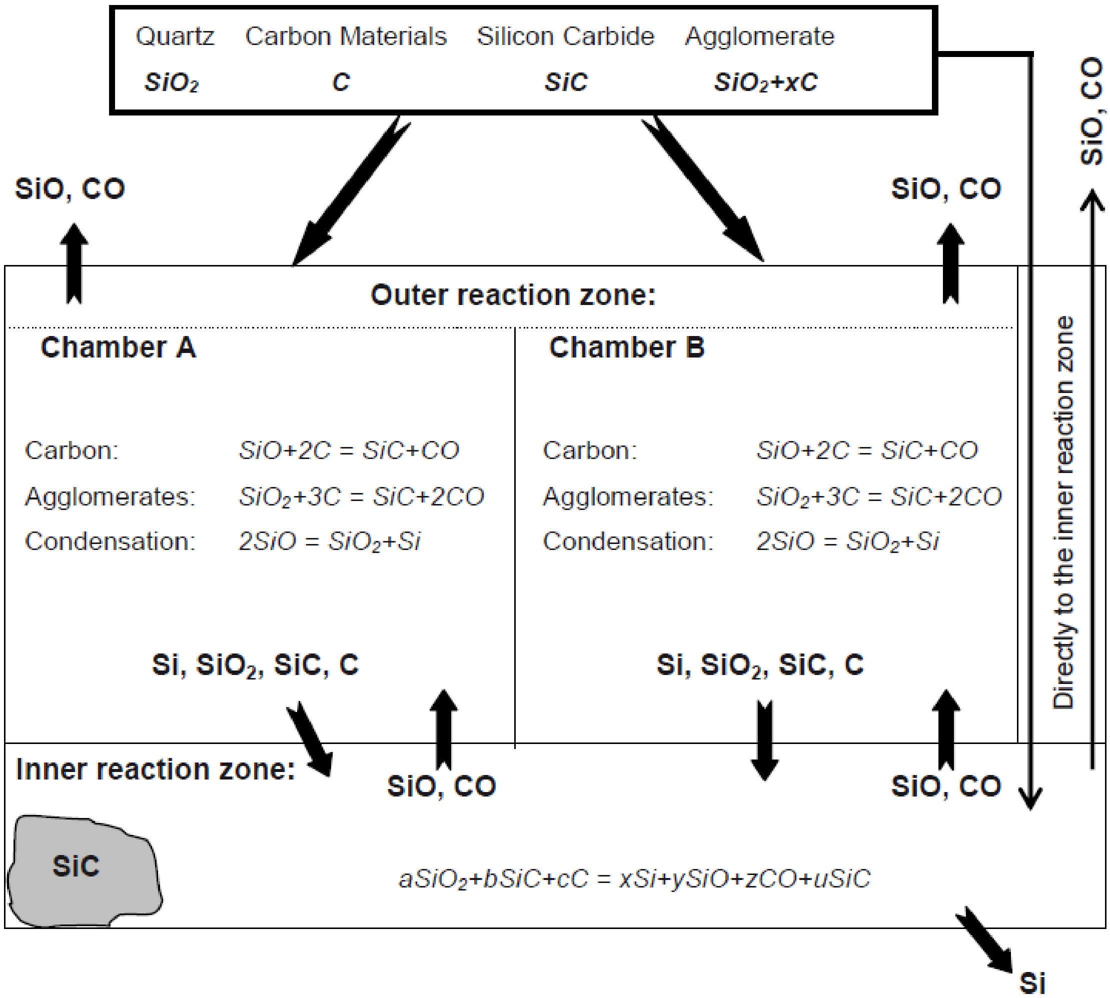

Figure 2.5: The stoichiometric model illustrating chemical reactions in the inner and outer reaction zones as well as material flowing (A Schei & Larsen, 1982).

According to reaction 4, SiO gas is produced and then rises into the furnace from the hottest parts (inner reaction zone) to the cooler top area (outer reaction zone). High level of energy is required for the formation of SiO; therefore, if SiO gas leaves the arc furnace, power consumption will increase dramatically. Also, recovery of silicon decreases considerably as elemental silicon go away from the smelting process. In the presence of high quality carbon materials (with high SiO reactivity and degree of conversion), the reaction between SiO gas and C results in the production of SiC that sinks down to the bottom of the furnace:

$$SiO + 2C = SiC + CO \qquad (6)$$

According to reaction 7, Even if the high quality carbon material is not available, there is a possibility of disintegration of SiO to the silica and pure silicon when resistance time is long enough.

$$2SiO = SiO_2 + Si \quad (7)$$

Finally, waste gas containing SiO and CO leaves the furnace and goes to the purification and energy recovery unit. Meanwhile, silica–rich and carbon reduction materials as well as silicon carbide and low amount of pure molten silicon sink down into the hottest part of the furnace. The overall unbalanced reaction in the inner reaction zone may be expressed as follows:

$$SiO_2 + SiC + C \rightarrow Si + SiO(g) + CO(g) + SiC(s) \quad (8)$$

At the very high temperature (~2000°C), the final silicon product is tapped out from the bottom of the furnace and then send to the casting and refining units (Gasik, 2013; Myrvågnes, 2008).

2.5 Contaminations in Silicon Production

The feed raw materials, such as quartz, coal and woodchips, contain different percentages of trace elements, which are introduced into the molten silicon product, silica slag, or/and gases. In other words, distribution of trace elements in the raw materials strongly determines the purity of final silicon product. An example of a trace element partition between the three flows passing out of the arc furnace is illustrated in Figure 2.6. Depending upon the type of raw materials and operation properties, the trace element distribution may be different from a furnace to another furnace (E. Myrhaug et al., 2004).

Most trace elements follow the boiling–point model, which states that the elements with the lowest boiling temperature will go to the gas phase, the elements with a slightly higher boiling temperature will condense on the micro–silica or stay in the slag, and the ones with the highest boiling points will mostly go into the final silicon product.

Production of highly pure silicon in silicon production circuits is strongly related to the type and level of contamination in the raw materials as well as in the submerged arc furnace. The average distribution of trace elements in quartz, carbon and electrode is illustrated in Table 2.4 and Figure 2.7. Obviously, the reduction in the amount of impurities results in improvement of the final silicon product quality. There are three main sources of contamination in silicon smelting process as following (Myrvågnes, 2008):

I. Basic level of impurities present in the raw materials composition (e.g. Al, Fe, B and P).
II. External impurities which diluted the raw materials during preparation, handling and transportation of silica and reductants.
III. Contamination in the material which is used for the construction of arc furnace.

Based on the location and geological properties of silica sand mine, the industrial quartzite or quartz frequently used in silicon production includes the considerable concentration of impurities (e.g. phosphorus and boron); For instance, highly pure silica deposits with 99.99% SiO_2 concentration may be found in some parts of Russia (Amick et al., 1985; Gribov & Zinov'ev, 2003). Reduction in the concentration of impurities through physical or chemical purification may be beneficial for the silicon production. Although chemical

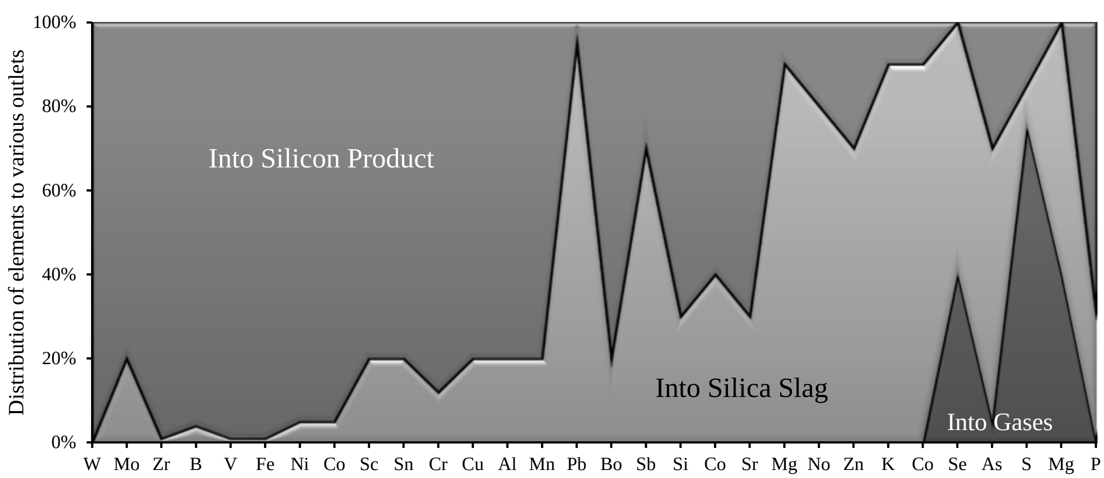

Figure 2.6: Example of a trace element partition between different products of silicon smelting process (E. H. Myrhaug, 2003).

refining of silica sand is highly efficient method, it is more expensive than conventional purification. Therefore, physical beneficiation, such as crushing, sieving and washing, is widely used to improve the purity of quartz and quartzite for the silicon smelting processes (Dal Martello, 2012).

Type and level of detrimental elements in the carbon reduction materials significantly depend upon the source of reductant materials, as discussed in section 2.3.2, as well as the effectiveness of coal cleaning process. The minimization of ash content in coal can be occurred through physical and chemical separation processes. In the coal preparation plants, gravity–based separations are the most effective methods to clean coal particles coarser than 1 mm, which is used for the silicon production.

External contamination during preparation, handling and transportation is one of the most critical issues in silicon smelting process. In coal preparation plant, magnetite is used to elevate the density of medium in dense medium separation circuits. Although magnetite is one of the most effective mediums to be used in dense medium concentrators, the presence of magnetite in final coal product considerably decreases the purity of final silicon product.

Table 2.4 Average impurity content (ppmw) in quartz, carbon and electrode in silicon production (Myrhaug and Tveit 2000, Aasly 2008)

Element	Quartz	Carbon	Electrode
P	<5–50	<5–300	<5–170
B	<10–45	<10–100	<10
Fe	1000–1500	<100–9000	100–3500
Al	300–3200	<50–10000	<50–4000
Ca	<75–160	<75–4000	200–1500
Ti	20–200	<0.5–4000	100
Mn	3–600	1–270	50–3500
K	<75–1700	<75–1800	<75–250
Mg	20–140	70–1400	<15–500
Na	50–170	<50–2100	<50–300

An ideal silicon production circuits, there is no addition of any type of contaminations into the final silicon product. Some necessary modifications in arc furnace design, to minimize contact between molten silicon and the parts of furnace, are required to reduce the impurities concentration in the final silicon product. These modifications can be divided into three main categories, as following: (i) lining up the bottom of the arc furnace, where the molten silicon accumulate, with ultra–pure graphite; (ii) using a core graphite electrode; and (iii) molten silicon constantly tapped off into the highly pure silica crucibles (Gribov & Zinov'ev, 2003).

In order to produce metallurgical grade silicon that meets the requirements of the chemical, primary aluminum markets, and electronics industry the silicon produced in the arc furnace requires further purification. The quality of silicon for the chemical silicones industry is critical with respect to the levels of aluminum and calcium present, and the primary aluminum grade of silicon requires low levels of calcium, iron, and phosphorus.

Typical trace elements that must be controlled in silicon are Fe, Ca, and Al. Both Ca, and Al elements are less noble than Si (have higher oxygen affinity), thus they can be removed

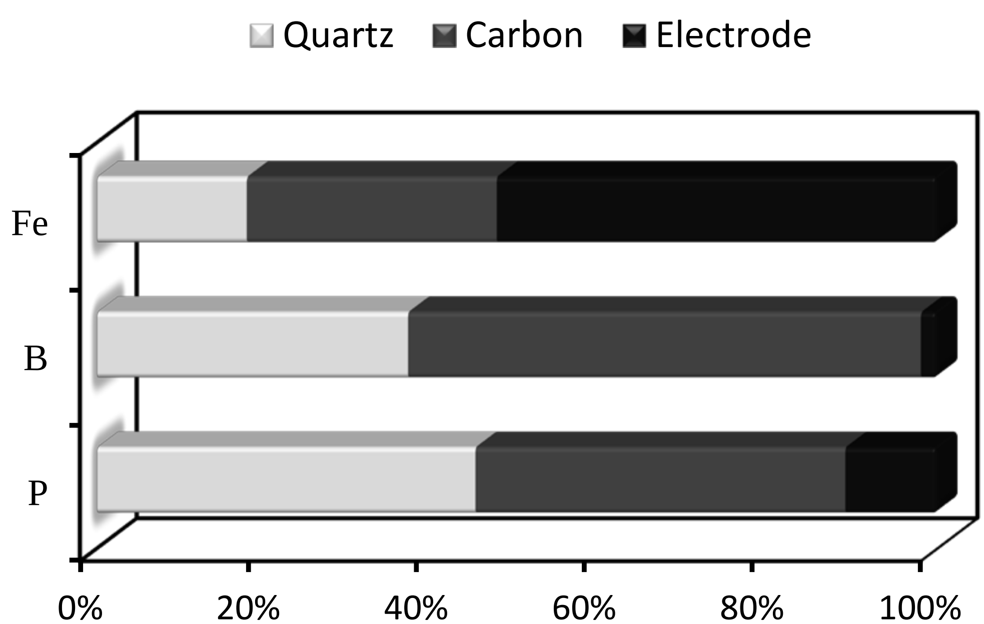

Figure 2.7: The distribution of Fe, B and P in the raw materials and electrode in silicon production (Myrhaug and Tveit 2000).

by oxidation. On the other hand, the most economical method to control the level of iron content in the final silicon product is the minimization of Fe content in the quartz and carbon reduction material.

2.5.1 Iron Impurity in Silicon Production

One of the most challenging issues in the silicon production regards to the type and level of metallic impurities in the final silicon product. The presence of the metal contaminations in silicon results in poor performances and efficiency degradation in devices and materials with basis of silicon. According to diffusion velocity of the metallic impurities in silicon, they may be categorized in very fast, fast and slow diffusers. Iron, which belongs to the fast diffusers group, is one of the most troubling impurities in the silicon structure (Dubois & Martinuzzi, 2008; Istratov et al., 2000; Peng, Lu, Zhang, & Lee, 2008).

Iron is one of the most abundant elements in nature and is difficult to entirely remove on a silicon production line. Consequently, the amount of accidental iron contamination in the final silicon product is higher than of other metallic impurities. Due to its electrical activity, iron has always been considered the main impurity in silicon used in semiconductor industries. The main detrimental effects of transition metals in general, and iron in particular, for silicon devices are as following:

I. In crystalline and polycrystalline photovoltaic devices, iron impurity causes the creation of recombination centers, reducing the minority carrier lifetime, thereby decreasing solar cell efficiency.
II. In reverse–biased junction, the generation–recombination centers associated with dissolved iron and its complexes increase leakage currents, consequently increasing power consumption and heat production.
III. Fast iron contamination of large wafer areas occurs, even from point sources and from the wafer backside, due to high diffusion coefficients at high processing temperatures.

The critical level of iron impurity concentration in semiconductor industries, that is 5×10^9 cm^{-2} of surface iron contamination, is decreasing annually as sensitivity of devices yield to iron impurity is increasing due to the device dimension shrinkage (Istratov et al., 1999, 2000).

2.6 Application of Silicon

Metallurgical grade silicon, defined as at least 96% silicon content, is an important raw material for various markets and applications, such as chemical industries, steel, copper and aluminum markets, and electronic industries. Some typical compositions of metallurgical grade silicon are shown in Table 2.5. As discussed in section 2.5, depending on the type and amount of contaminations in the silica and carbon raw materials, compositions of metallurgical grade silicon, especially in the case of minor impurities, may be different (Dosaj et al., 2005; Myrvågnes, 2008).

The utilization of silicon in chemical industry for the production of silicone and its by–product components is the most fast–growing and the largest application of silicon among all dependent markets. Typically, highly pure silicon (>99% Si) containing low amount of trace elements is used for the production of silicone compounds. Generally, silicone materials may be produced in the various chemical forms, which are chemically inert and stable at high temperatures (~400°C). It can be used for the materials range from fluid thinner than water to rigid plastics which may be clear or pigmented. One of the most important applications of silicones is in the medical and pharmaceuticals industries. Also, it can be utilize to produce electric insulators, protective paints and coatings, hydraulic fluids, and lubricants. In order to use silicon for chemical applications, type and amount of impurities as well as lump size of silicon are the most critical issues (Gasik, 2013; O'Lenick Jr & Siltech, 2009).

Table 2.5 Composition of metallurgical grade silicon

	Maximum wt. % (except for silicon)				
Grade	Si	Fe	Al	Ca	Fe+Al+Ca
Si99	99	0.4	0.4	0.4	1
Si98	98	0.7	0.7	0.6	2
Si97	97	1.0	1.2	0.8	3
Si96	96	1.5	1.5	1.5	4

Silicon in the form of alloying elements, such as ferrosilicon, is widely used in steel–making process. Ferrosilicon for steel–making and foundry uses have a silicon content of 14–95 wt%. Same as the silicon smelting process, ferrosilicon is produced in a submerged arc furnace by the reduction of silica–rich materials in the presence of high quality iron ore. In the steel industry, ferrosilicon is used to enhance elasticity, high–temperature oxidation resistance as well as mechanical properties in steel. In addition to the ferrosilicon, some other silicon ferroalloys (e.g. ferrosilicon manganese) are typically utilized in steel markets (Dosaj et al., 2005).

In addition to the chemical and steel–making industries, silicon as an alloying element is applied in aluminum–casting process to improve the mechanical characteristics of the aluminum. Level of the iron content in raw materials, which are used in aluminum–casting, must be controlled since the presence of iron can make problems in the process, structure of aluminum and its alloys (Gasik, 2013).

The relatively small portion (<10%) of very highly purified silicon is utilized in the electronic industries as a semiconductor. Due to very low amount of contamination requirements, use of silicon in electronic industries may be the most challenging application. After production of metallurgical grade silicon through carbothermic reduction process the impurity contents of the final silicon product is in the ppm level. Hence, further refining circuits is needed to reduce the amount of impurities to ppb level for the production of the solar grade silicon. One of the most efficient methods for the purification of metallurgical grade silicon is Siemens process in which the solid free silicon transformed to silicon–chlorine gases and then distilled and reduced back to very highly purified silicon to be used widely in the integrated circuits as the basis of most computers (Dosaj et al., 2005; Gasik, 2013). In addition to the Siemens process, other purification methods such as acid leaching, directional solidification and refining of silicon with reactive plasma are readily available as technologies for inexpensive and energy–efficient purification of silicon (Istratov, Buonassisi, Pickett, Heuer, & Weber, 2006).

In the copper industry silicon is applied for the production of silicon bronzes, which include 96% copper and the reminders are silicon and other alloys such as manganese, tin and iron. Presence of silicon in silicon bronze structure not only enhances the corrosion resistance and strength properties but also minimizes dross formation. Silicon bronze can be used in the variety of application, such as bearing cages, raceways and spacers especially for the aerospace industries (Dosaj et al., 2005; Gasik, 2013).

3 COAL PREPARATION

3.1 Introduction

Coal preparation technology plays a significant role in the silicon market by upgrading run–of–mine (ROM) coal to satisfy the size and quality specifications. Coal cleaning process is essential since run–of–mine coal contains organic (carbonaceous) and inorganic (mineral) matter such as shale, slate, and clay. These contaminations in unprocessed coal not only are undesirable for silicon smelting process but also dramatically increase the cost of transportation coal to the market (Gluskoter, 2009; Meyers, 1981).

Modern coal preparation plants consist of a complicated arrangement of solid–solid and solid–liquid separation processes with objective of improvement in the purity of coal by reducing detrimental impurities; and the control of final product lump size to meet the designated product requirement. These processes identify and employ the differences in physical properties (e.g. density), and chemical properties (e.g. wettability) to separate valuable carbonaceous material from waste rock. The main examples of separation technologies used by the coal preparation plants are screening, classification, gravity separation, froth flotation, filtration, and thickening (Gluskoter, 2009; Meyers, 1981).

Although design of coal preparation plants may vary among coal cleaning industries, the plant flowsheet can be generally represented by a sequence of operation units for particle sizing, cleaning, and dewatering, as shown in Figure 3.1. The efficiency of various types of separation processes is limited to a range of applicability regarding ROM particle size. Figure 3.2 summarizes some of the main available technologies in the coal preparation plants and the range of particle size in which they achieve maximum efficiency. Typically, Modern plants may consist of four separate processing circuits for treating the coarse (greater than 10 mm), intermediate (between one and 10 mm), fine (between one and 0.15 mm), and ultrafine (less than 0.15 mm) material. Depending on the properties of the coal and required specifications of the final product, different scenarios on the type and number of unit operation may be considered (Gluskoter, 2009; D. Osborne, 2013).

Typically, one of the most efficient concentrators for cleaning the coarse (greater than 10 mm) and intermediate (between 1 to 10 mm) ROM coal are the dense medium vessel (DMV) and the dense medium cyclone (DMC), respectively (Figure 3.3). However, the considerable increase in the dimension of DMCs allows coal preparation plants to eliminate the coarse cleaning stream, crush down ROM coal to a specific top size, and substitute DMVs with large diameter cyclones. There are many examples of large diameter DMCs (1000 to 1450 mm) widely operating in the modern coal preparation plants in Australia even though some plants are still using DMVs (Gluskoter, 2009; Mackinnon & Swanson, 2010; D. Osborne, 2013).

Generally, dense medium separators utilize a suspension of magnetite to elevate the medium density to a value that is between coal and the ash material. Due to the high density of medium, lighter particles (coal) float and the particles with higher density (waste rock) sink in the suspension. Finally, the clean coal and reject materials pass over the drain–and–

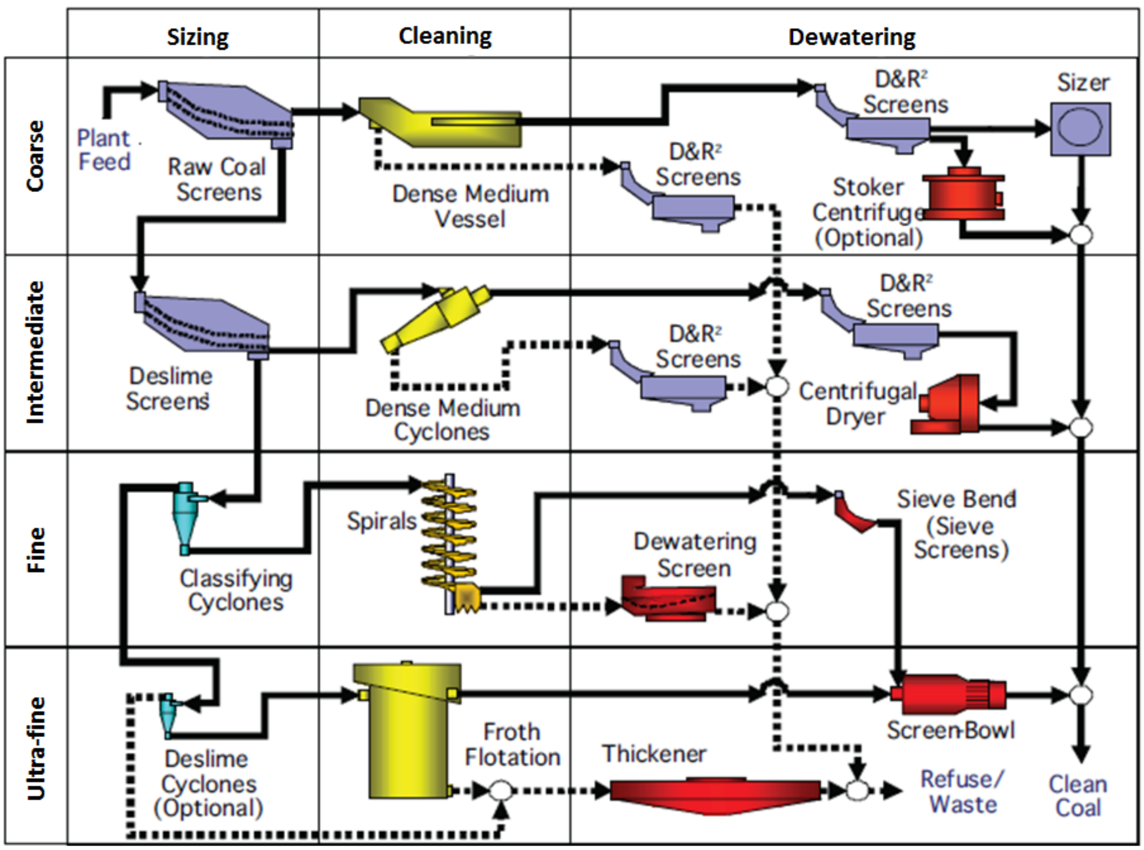

Figure 3.1: Simplified flowsheet matrix for a modern coal preparation plant incorporating four parallel streams for different ROM size fractions (Gluskoter 2009).

rinse screens to wash and recover magnetite uses shower boxes and water sprays. The separation in a dense medium strongly depends on density differences between light and heavy particles, their size and the stability and rheology properties of the medium (Gluskoter, 2009; YB He & Laskowski, 1994).

As section 2.5 speculated on the source of contaminations in the silicon product, external contaminations during preparation of carbon raw material are of serious concern to the silicon market. Although magnetite is one of the most effective suspensions used in the dense medium processes, it dilutes final coal product since it is not possible to wash all the ultra–fine magnetite particles, say between 10–60 µm, from the surface of the final coal product.

As the ROM particle size decreases, cleanability enhances because of liberation issues; however, the separation efficiency provided by the fine and ultra–fine cleaning separators are inferior to those achieved by the dense medium processes used on coarse and intermediate coal. Moreover, operating and capital cost of fine and ultra–fine units are considerably higher than coarser units due to lower capacity, chemical reagent

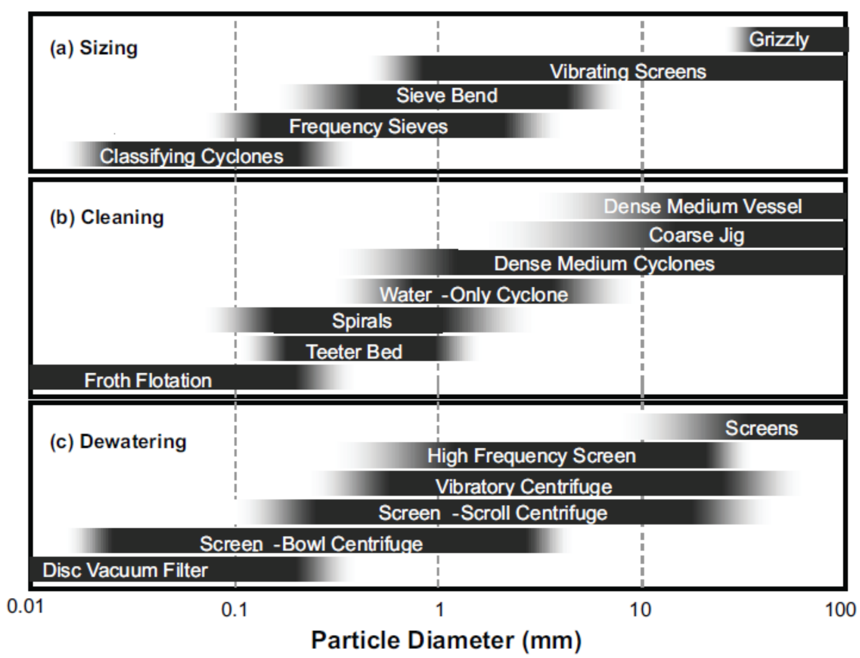

Figure 3.2: Application of various coal preparation processes as a function of particle size (Gluskoter, 2009).

requirements, complicated and expensive dewatering technologies; Therefore, one of the major purposes of the coal mining and processing is to minimize the production of below 1 mm coal particles (D. Osborne, 2013).

Variety of density–based separators are available for treating fine size coal, which range between 0.2 and 1 mm. Considering a number of developments over the past decades, spiral concentrator, reflux classifier, and water–only cyclone are the most widely used technologies. Conventional and column flotation units, which exploit the inherent differences in the surface wettability of coal and rock, are the only feasible methods for commercially cleaning ultra–fine coal (Gluskoter, 2009; D. Osborne, 2013).

As section 2.3.2 focused on the carbon raw materials and their particle size specification for the silicon market, the typical sizes of the carbon reductants are between 1 to 30 mm.

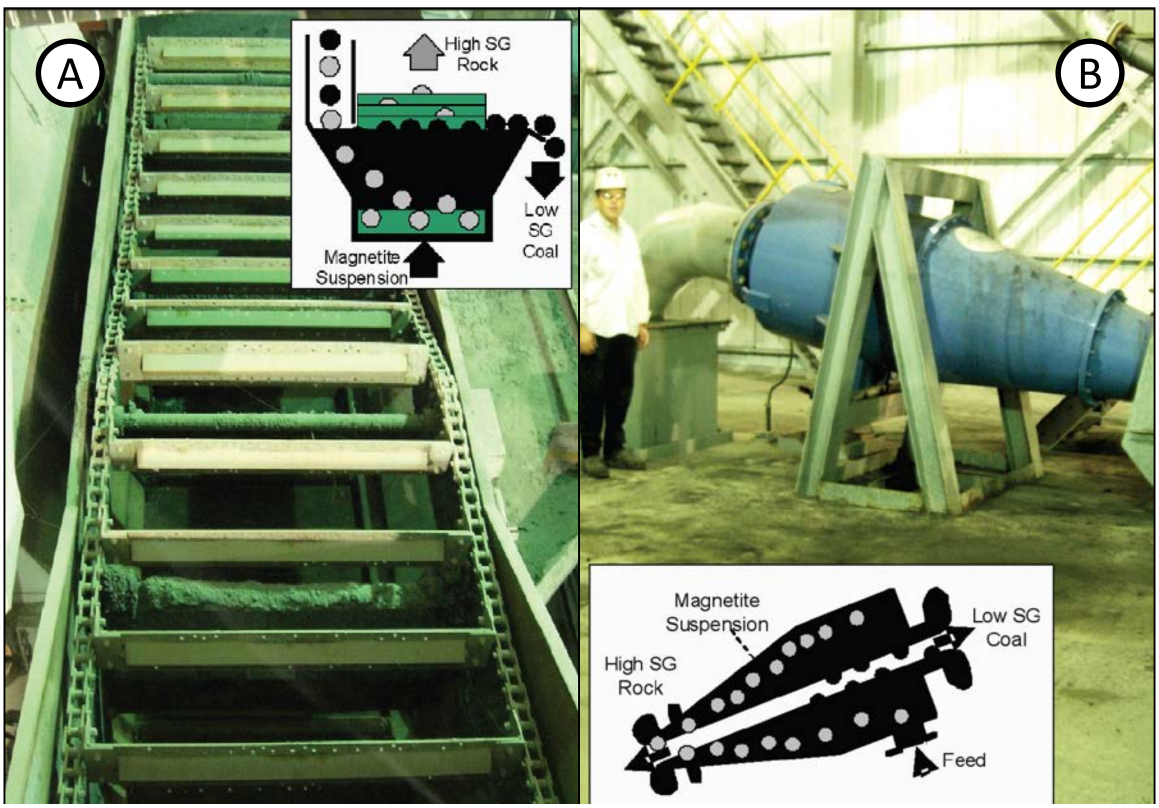

Figure 3.3: Examples of dense medium separators for treating coarse and intermediate size fractions of coal; (A) dense medium vessel, and (B) dense medium cyclone (Gluskoter, 2009).

Consequently, although the new technologies in fine and ultra–fine coal cleaning may be capable of producing a high quality coal, the final coal product below 1 mm is not saleable to the silicon market (Myrvågnes, 2008).

3.2 Dense Medium Separation

Dense medium separation is basically a density–based process which cleans ROM coal by applying a fluid having a density intermediate between the lighter constituent (coal) and the heavy constituent (ash–forming impurities). As there is a clear correlation between ash content and specific gravity, the high ash reduction is achievable where an adequate media is used. Generally, dense medium separation has significant superiority over other coal cleaning processes as following:

1. Ability to have a sharp separation at wide range of specific gravities even in the presence of high amount of near gravity materials in the ROM coal.
2. Ability to clean coarse to ultra–fine coal feed using gravitational or centrifugal forces in the dense medium separators.
3. Comparatively low capital and operating costs when considered in terms of high capacity and small space requirements with no grinding involved.
4. Ability to produce high quality final products to fulfill different market specifications even when considerable fluctuations occur in the ROM coal, both in terms of quality and quantity.
5. Environmentally more friendly; disposal of coarse rejects on dumps, as backfill (Backus, 2007; Leonard & Humphreys, 1979).

In the early 1800s, the first idea of applying dense media to separate the low–density materials from the high density particles began as a result of observation of float–sink phenomenon in early jig washers, although the precise date of development and source of dense medium separation concept is not completely obvious. From then on, numerous remarkable advancements in fundamental of the process as well as the types of dense media and dense medium separators have been taking place for upgrading coal (D. G. Osborne, 1988).

3.3 Types of Media Used in Dense Medium Processes

Application of the dense medium process in the coal preparation plant is principally a commercial scale of the laboratory float–sink (washability) test. However, a practical extension of the float–sink separation is not viable because: (i) fine solid suspension, rather than true high density liquid, is utilized as dense media for coal cleaning; (ii) introduction of disturbances in the dense media is not avoidable due to immersion of raw feed and collection of clean coal and refuse; (iii) the need for high throughput capacity of the dense medium separator results in imperfect separation of near gravity materials because of insufficient retention time (Leonard & Humphreys, 1979).

An efficient dense medium operation, to economically produce a saleable product at high recovery value requires the dense media with desirable properties. An ideal dense media would be a true liquid having the following properties: inexpensive, miscible with water, capable of adjustment over a wide range of density fractions, non–toxic and environmentally friendly, non–corrosive, chemically inert, easily separated from both products after processing, readily recoverable to reuse, stable over the required range of densities, and low in apparent viscosity. Although no ideal medium exists, different types of dense medium have been developed commercially for coal cleaning processes. Generally, there can be three types of media to be used industrially as a separating liquid (Leonard & Humphreys, 1979; Sanders, 2007):

- Organic liquids, same as utilized in the laboratory float–sink test
- Aqueous solution, dissolved solid in water
- Dense solid suspended in water

3.3.1 Organic Liquids

High density organic liquids (0.86–2.96 RD) used for coal cleaning processes are the most attractive media from the point of view that they are easy to be regulated at the exact specific gravity required, by mixing high and low specific gravity liquids, and have high stability and low viscosity which allows the separation of fine particles. Previous attempts at industrial applications failed due to high cost and consumption of separating media; however, pretreating of the raw coal in a solution of active agents may decrease the high consumption of organic liquids through surface adsorption of the coal particles (Leonard & Humphreys, 1979; Sanders, 2007; Yan & Gupta, 2006).

There are small number of industrial applications of organic liquid in the coal preparation plant such as a pilot plant built by the DuPont Co with 50 tph capacity for cleaning anthracite coal, some continuous float–sink separators used for quality control, and the Otisca process (20 tph) which applied nonpolar organic liquids. With the exception of these, organic liquids have not been utilized commercially for dense medium processes (Keller Jr, Smith, & Burch, 1977; Leonard & Humphreys, 1979).

Considering the increased awareness of the importance of environmental, safety and health issues, it would be expected that no further research about the commercial applications of organic liquids will be carried out. Nevertheless, Various Organic liquids, such as gasoline, benzene, perchlorethylene, carbon tetrachloride, bromoform, acetylene tetrabromide, and lithium metatungstate are used for laboratory float–sink test (Leonard & Humphreys, 1979; Sanders, 2007; Yan & Gupta, 2006).

3.3.2 Aqueous Solutions

Several dense medium processes applying aqueous solutions of inorganic salts, such as such as calcium chloride ($CaCl_2$) and zinc chloride ($ZnCl_2$), were industrially used in Europe in the 1930s. Due to the relatively low specific gravity of inorganic liquids (calcium chloride and zinc chloride solutions have the density of 1.35 RD and 1.8 RD, respectively), a mechanically induced upward currents are needed to perfectly separate coal from rock at higher selected density cut–points (Leonard & Humphreys, 1979; Sanders, 2007).

The WEMCO–Belknap calcium chloride separator, based on the Lessing process, is the only process that commercially applied a high density organic liquid as a separating medium. The Belknap coal washer employed calcium chloride medium, with actual density varied from 1.14 to 1.25, to achieve an affective cut–point density ranging from 1.4 to 1.6 with the assistance of upward currents. The Belknap concentrator was able to achieve an effective separation of coarse coal particle ranging from 6.3 mm to 200 mm. (Leonard & Humphreys, 1979).

The corrosive nature of aqueous solutions to metals and their low specific gravity are the major disadvantages of the inorganic solutions. In addition, the medium recovery procedure is complicated and expensive; and at densities greater than 1.4 solutions become unstable without applying of upward currents (Sanders, 2007).

3.3.3 Dense Solid Suspensions

The problem associated with the utilization of organic liquids and aqueous solutions encourage the development of dense media using a suspension of finely divided high density solid particles in the water. To achieve a selected separating density, which ranges from 1.30 to 1.90 for coal cleaning processes, it is essential either to select high density solids or to apply upward currents to elevate the separation density where low–density material is used. As the usually accepted volumetric concentration of high density solid is between 25% and 45%, their specific gravity and particle size distribution must be selected to obtain the dense medium with high stability properties. The coarser the solids, the higher the settling rate, the lower the viscosity, and the more difficult it is to recover the medium. Clearly, the utilization of higher density solid results in the low volumetric concentration for a given specific gravity; therefore, it is more viable to generate a dense medium that obtain optimum performance by selecting the effective specific gravity, size distribution, and volumetric concentration of the suspended solid (Leonard & Humphreys, 1979; Sanders, 2007; Yan & Gupta, 2006).

Proper control of density, stability and rheological properties of the suspension is necessary for an efficient dense medium separation. Eq. 9 determines the specific gravity of suspension:

$$\rho_m = \frac{100}{(100 - c) + \frac{c}{\rho_s}} \tag{9}$$

where ρ_m is the density of medium (g/cm^3), ρ_s is the density of solids (g/cm^3), and c is the percentage of solid concentration by weight. As Eq. 9 revealed, the density of suspension can be varied by adjusting the proportion of medium solids present in the liquid. However, there is a natural limitation of solid content due to viscosity issues. The limiting volumetric concentration is similar for all suspension with particles of similar size range, but can be modified to a limited extend by change in the size distribution of solid. Eq. 9 also indicates that the medium density is a function of solid particles specific gravity; and at the same solid concentration, the media composed of higher specific gravity solids have higher density than that of lower gravity solids (Leonard & Humphreys, 1979).

A variety of substances, such as silica sand (2.65 RD), magnetite (5.0 RD), ferrosilicon (2.33–7.87 RD), barytes (4.5 RD), pyrites (5.0 RD), shale (2.67), have been used as the solid suspend in water to be utilized in the dense medium process. Among all of these, only sand and magnetite are applied extensively in the United States for dense medium separation of coal (Leonard & Humphreys, 1979; Sanders, 2007).

3.3.3.1 Magnetite and Ferrosilicon

The most popular manufactured media used in coal preparation is a fine magnetite suspension in water (1.25 to 2.2 RD), ferrosilicon suspension (2.9 to 3.4 RD), or a mixture of the two (2.2 to 2.9 RD), depending on the selected separating density. The effective density of such slurry can be controlled by adjusting the solid concentration of suspension. The size of the particles that generate the suspension must be much smaller than the particles that are to be separated easily. As Table 3.1 demonstrated, the limiting specific gravity values of each dense media is a function of solid particle size distributions, amount of magnetite content in suspensions, and also the percentage of silicon in the ferrosilicon alloys (King, 2001; D. Osborne, 2013; Yan & Gupta, 2006).

The first reported use of suspension containing finely divided magnetite was by Conklin in 1922. Commercial development was unsuccessful due to the lack of an efficient method of medium recovery. Later in 1937, Butler Brothers ore concentrator pilot plant was successful in incorporating magnetite medium recovery. Finely divided steel and ferrosilicon was used as a dense media, which were recovered using cross–belt magnetic separators. American Cyanamid for the first time applied the magnetic separator to recover and clean magnetite medium in a coal preparation circuit (Leonard & Humphreys, 1979).

After the development of wet drum magnetite separators, magnetite became the first choice of solid material for dense medium separations. Various grades of magnetite, which is determined by its fineness, are produced for coal cleaning processes. The grade of magnetite is typically marketed based on the percentage of ultra–fine particles smaller than 325 mesh. The most common grade of magnetite used in coal preparation technology is grade B type which is normally over 95% below 325 mesh. Compared to magnetite, ferrosilicon is rarely utilized as a dense media in coal preparation plants due to high solid density and cost (D. Osborne, 2013; D. G. Osborne, 1988; Yan & Gupta, 2006).

The physical and rheological properties are of the considerable importance in determining the effectiveness of magnetite as a medium. Since the relative density of magnetite is relatively high (about 5.0), the generation of low viscosity dense medium over a wide range of density, even in the presence of slimes contamination, is possible due to low degree of solid concentration. Furthermore, high effective medium recovery and regeneration is achievable due to the magnetic characteristics of magnetite (D. G. Osborne, 1988; Sanders, 2007).

Table 3.1 Limitation of media density values using various solids (Yan & Gupta, 2006).

Solid	Limiting Media S.G.
Magnetite	2.5
Ferrosilicon (15% Si)	2.5–3.5
Ferrosilicon (<150 micron)+10–20% Magnetite	2.65–2.9
Ferrosilicon (<150 micron)	2.8–3.0
Ferrosilicon (<212 micron)+Magnetite	2.8–3.0
Ferrosilicon (<212 micron)	>3.0

The physical and rheological properties are of the considerable importance in determining the effectiveness of magnetite as a medium. Since the relative density of magnetite is relatively high (about 5.0), the generation of low viscosity dense medium over a wide range of density, even in the presence of slimes contamination, is possible due to low degree of solid concentration. Furthermore, high effective medium recovery and regeneration is achievable due to the magnetic characteristics of magnetite (D. G. Osborne, 1988; Sanders, 2007).

Contrary to the common use of magnetite in typical coal cleaning industries, the coal preparation plants, which upgrade saleable clean coal to the silicon market, prefer to not apply magnetite as a dense media due to following reasons: (i) the relatively low separating density is needed since the silicon production plants utilizes very low ash coal as a raw material; and generation of low–density magnetite medium with high stability properties is hardly possible. (ii) Magnetite contamination has been identified as a serious problem in the silicon market since it strongly affects the quality of final silicon product (as discussed in section 2.5.1).

3.3.3.2 Silica Sand

In the early development of dense medium processes, silica sand was one of the most popular suspensions applied for coal cleaning. The silica sand, primarily mineral quartz, has a relative density of around 2.6. Alluvial or beach sands, which are comprised of rounded grains, provide an effective medium to separate coal and mineral matters at the separation densities which range from 1.25 to 1.80 (Leonard & Humphreys, 1979; Sanders, 2007).

In the Chance Cone process (Figure 3.4), which was the first patent in 1917, the ROM coal is fed into a conical separating chamber, which contains a mixture of silica sand (between 0.5mm and 0.125mm) and water, to separate coal from rock based on the differential density. Since these sizes of sand are unstable in water, the silica sand was kept in suspension in the water by upward water current and by a revolving agitator. The clean coal floats near the surface of the fluid mixture and moves around the cone until it reaches the discharge point, while the reject material sink through the separating medium to report into the refuse gates. Finally, the cleaned coal and refuse products, mixed with sand and water passes over the designed screens for the removal and recovery of silica sand. Under ideal conditions, Chance cone is capable of achieving a highly efficient separation, even in the presence of high amount of near gravity materials, with Ep values of 0.02–0.03 (Leonard & Humphreys, 1979; Sanders, 2007).

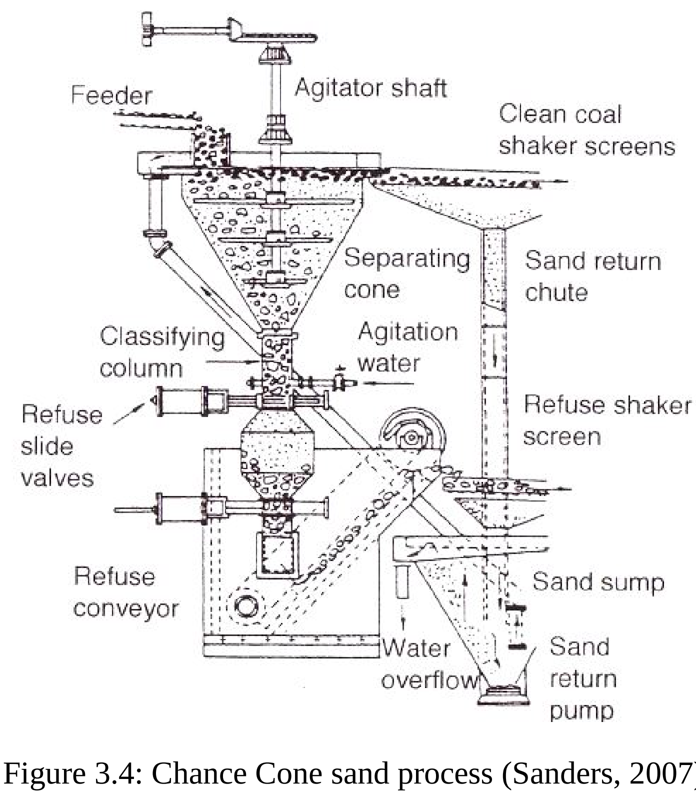

Figure 3.4: Chance Cone sand process (Sanders, 2007).

Although the main cause for the demise of the Chance Cone equipment is not completely clear, it seems that the reason was its conical geometry, which was the serious disadvantage of the system. This geometry results in the velocity variations within the separating vessel, thereby declining the sharpness of separation of ROM coal with a wide size distribution. Another drawback with the Chance Cone separation was the availability of sufficiently closely graded silica sand materials (Sanders, 2007).

In 2013, Honaker and Bimpong performed a study to evaluate the potential of fine sand to be used as a dense medium for coal–cleaning applications. Results of the conducted pilot–scale dense medium cyclone tests revealed that the high separation efficiency (Ep value around 0.04) can be achieved by applying the fine sand dense media with relatively coarse particle size distribution. The coarseness of the sand minimizes the detrimental effects of medium rheology, while the relative high solid concentration at the selected medium densities maximizes the dense medium stability (Honaker & Bimpong, 2009).

3.4 Particle Settling Phenomena

The fundamental basis of dense medium separators is governed by differential settling velocities of particles within a given medium. A solid particle settling rate in a liquid may be determined based on acceleration force due to gravity and a drag force that opposes motion (D. Osborne, 2013; Yan & Gupta, 2006). Considering these two forces, the terminal velocity of the particle in a liquid can be expressed as following:

$$v_t = \sqrt{\frac{\left(4d\left(\rho_s - \rho_p\right)\right)}{3C_D\rho_p}g} \qquad (10)$$

Where v_t is the terminal velocity of the particle, d is the spherical particle diameter, ρ_s is the particle density, ρ_p is the density of pulp or medium, C_D is the dimensionless drag coefficient, and g is the gravitational force. The drag coefficient is a function of the Reynolds number of particle ($C_D = f(\mathrm{Re}_p)$). The particle Reynolds number is expressed as:

$$Re_p = \frac{\rho v d}{\eta} \quad (11)$$

where η is the viscosity of the liquid.

For spherical particles finer than 100 micron the drag coefficient is defined as:

$$C_D = \frac{24}{Re_p} \quad (12)$$

By substituting equation 12 into equation 10, Stokes' law is obtained:

$$v_t = \frac{d^2(\rho_s - \rho_m)}{18\eta} g \quad (13)$$

For spherical particles coarser than 1 mm the drag coefficient is considered about 0.44; thus, substitution of C_D value is into the equation 10 results in Newton's equation as following:

$$v_t = \sqrt{\frac{10d(\rho_s - \rho_p)}{3\rho_p} g} \quad (14)$$

A comparison between Stokes' and Newton's equations indicates that the settling rate of fine solid particles ($d < 100\mu m$) largely depends on the viscosity of the medium, while the settling velocity for the spherical particles coarser than 1 mm is not affected by medium viscosity. The differential density between solid particles and medium ($\rho_s - \rho_p$) is another affective parameter in the particle settling rate determination. Considering the Stokes' and Newton's expressions, the movement of near gravity particles, which are within ±0.1 RD of the medium density, is slow for both fine and coarse particles (D. Osborne, 2013; Yan & Gupta, 2006).

Dense medium bath (static dense medium separation) utilizes gravitational driving force for sedimentation of solids in fluid; while in a dense medium cyclone, the acceleration of gravity is substituted by a centrifugal acceleration:

$$F_c = \frac{\pi d^3}{6}(\rho_s - \rho_m)\frac{v_t^2}{r} \qquad (15)$$

Where F_c is the centrifugal force, d is the particle size, V is the tangential velocity, and r is the radius of cyclone.

In a typical cyclone, the centrifugal force in the inlet region is considerably greater than the gravitational force acting on a particle in a static dense medium (about 20 times). Since $\frac{v_t^2}{r}$ is always constant in a cyclone, the centrifugal acceleration increases dramatically in the conical section of a cyclone and may be elevated to over 200 times greater than gravitational driving force at the apex of the cyclone. These large forces in the dense medium cyclone not only increase the capacity of the cyclone remarkably but also explain its ability to process fine coal particles (D. Osborne, 2013).

3.5 Dense Medium Cyclone

Development of the dense medium cyclone occurs during the World War II at the laboratories of the Dutch States Mines (DSM), now known as Stamicarbon. In 1940s, M G Driessen constructed the first dense medium cyclone pilot plant with capacity of 13.6 tph. McNally Pittsburgh in the USA commercially implemented the concept of dense medium cyclone processing by development of the cycloid which was considerably different from the DSM. In Australia, both the DSM and McNally designs were introduced in 1960s for the preparation of Australian coal. By development of large diameter dense medium cyclones, the process has become the most common and efficient technique for coal cleaning around the world (D. Osborne, 2013; Sanders, 2007).

Details of the standard dense medium cyclone of the DSM design are shown in Figure 3.5. The most significant parameter that defines the cyclone capacity and upper size limit is the

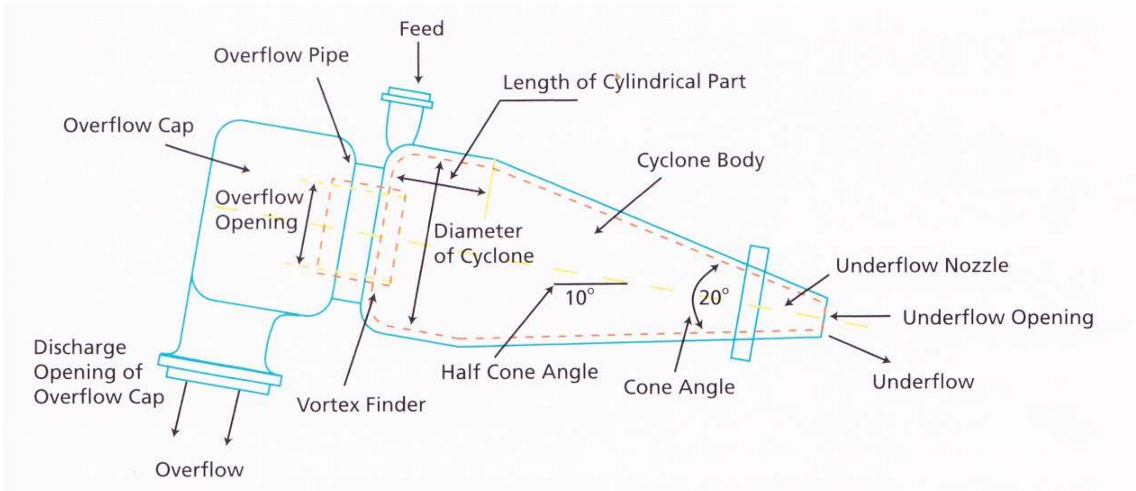

Figure 3.5: Typical dense medium cyclone that originally developed by DSM (Sanders, 2007).

cyclone diameter (D_c). The feed inlet diameter (D_i), the vortex finder diameter (D_o), and the apex diameter (D_u) are typically from 0.15 to 0.25, 0.43 to 0.50, and 0.3 to 0.4 times the cyclone diameter, respectively. An optimum performance is achieved with D_o/D_u ratio of around 1.8. The cyclone manufacturer specifies these components to provide the desired capacity and separation characteristics (Mular, Halbe, & Barratt, 2002; D. Osborne, 2013; Sanders, 2007).

The optimal operating pressure of a DMC is around 9 D_c, and it is important to control the pressure value since any changes in the inlet pressure may result in the low efficiency performance. Table 3.2 summarizes the required inlet pressure values to maintain D_c at various media density values when 15–cm cyclone is used. Dense medium cyclones can operate at any angle since the effect of the gravitational force, compare to centrifugal force in DMC, is negligible. However, to ease the drain on shutdown and also to make the medium split less susceptible to operating variables including inlet pressure fluctuations, DMCs are mounted with the longitudinal at an angle to horizontal (e.g. 10° to 15°) (D. Osborne, 2013; Sanders, 2007).

Table 3.2: Dense medium cyclone pressure required to maintain 9 D_c at various media density values for 15–cm cyclone.

Medium S.G.		1.00	1.10	1.20	1.30	1.40	1.50
Required Inlet Pressure	Psi	1.95	2.15	2.34	2.54	2.73	2.93
	kPa	13.45	14.79	16.14	17.48	18.83	20.17

A mixture of medium and raw coal enters tangentially near the top of the cylindrical section of the dense medium cyclone, therefore forming a vortex down the center of the vessel. As the flow pattern of the materials demonstrated in Figure 3.6, the high density reject material move along the wall of the cyclone, and finally reports to the underflow stream. On the other hand, the air core is formed in the center of the vessel of which there is an inward flow of medium towards the vortex, thus, the lower density coal particles migrates towards the longitudinal axis of the cyclone and finally report into the overflow stream (Sanders, 2007).

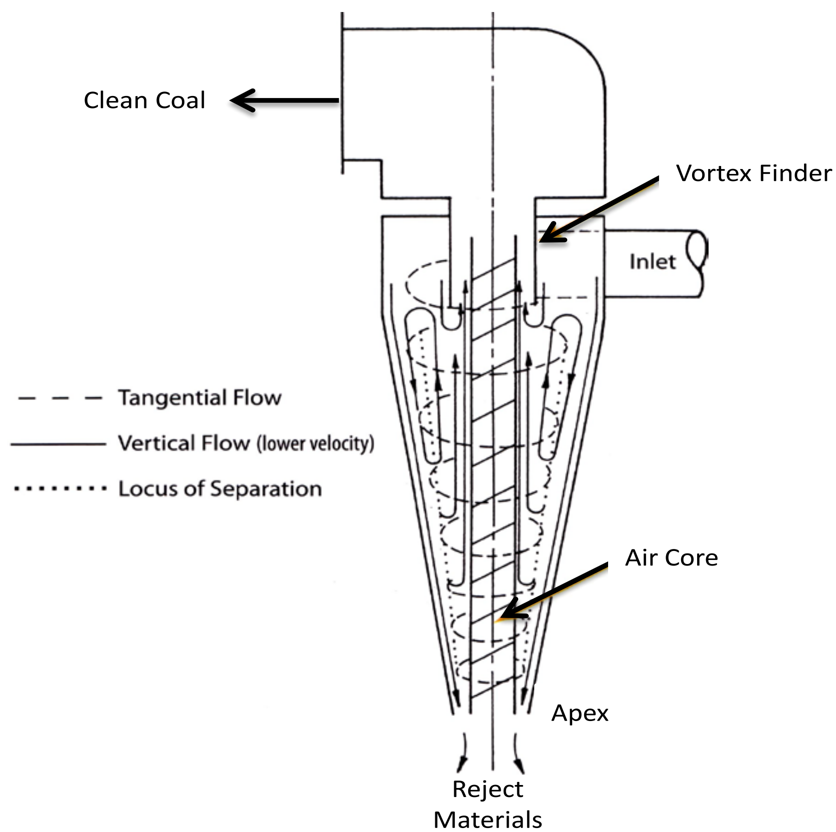

Figure 3.6: Flow pattern in dense medium cyclone (Sanders, 2007).

As a common dense medium circuit illustrated in Figure 3.7, raw coal feed is passed over a screen to remove the fine and ultra–fine particles. The oversize materials are mixed with the dense media in the cyclone feed sump and then the mixture is pumped directly to the cyclone. After the separation of high and low–density particles in the cyclone, the clean coal is passed over a drain and rinse screen, and then the final product is sent for dewatering stage. The same procedure is established for the reject materials before disposal.

Undulated (corrected) dense medium, which is drained from the initial part of the product and reject screens, is directly sent to the correct medium sump, whilst the reminder of the medium is washed out from the product and reject materials and send to the magnetic separator to recover magnetite and reuse it as the dense media.

All ultra–fine particles of magnetite cannot be recovered from the fine coal particles. It is speculated that the medium is more easily recovered from coarse coal, and also the medium

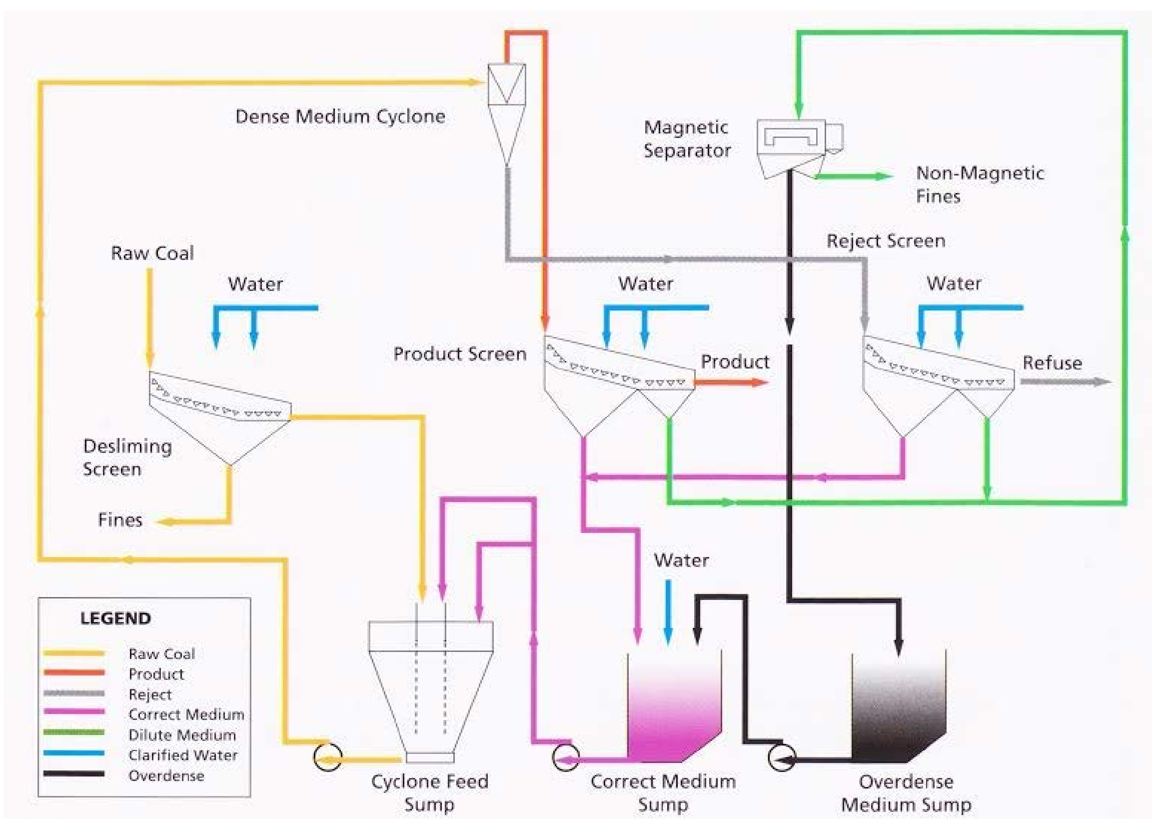

Figure 3.7: A typical dense medium cyclone circuit in the coal preparation plant (Sanders, 2007).

including coarse solid particles is much easier to recover. The reminder of the ultra–fine magnetite on the surface of the clean coal particles causes not only medium losses, which represent 10–20% of operating costs of a dense medium circuit, but also the main problem in the silicon smelting process. The present iron content in the charge materials of the silicon production is one of the major detrimental impurities in the silicon production (D. Osborne, 2013; Sanders, 2007; Sripriya et al., 2006).

3.6 Factors Affecting Dense Medium Cyclone Performance

Efficiency of the dense medium cyclone performance is affected and controlled by dense media properties in combination with raw coal feed characteristics as well as DMC operating conditions. Medium rheology and stability, which are affected by medium composition, play an integral role in DMC separation. In addition to the DMC feed properties, operating conditions of a DMC are of considerable importance in separation performance. Figure 3.8 illustrates the performance indicators and the variables that influence dense medium separation performance.

In dense medium cyclone, the medium composition affects separation performance by varying the stability and rheology characteristics of the dense media. Medium stability is a measure of tendency of the medium, while rheology is a measure of medium resistance to fluid flow. An ideal medium needs to have a low apparent viscosity and high stability to maximize cyclone separation and pump efficiency. By reducing the terminal velocity of fine particles in the suspension, medium rheology affects separation efficiency in the dense medium cyclone process due to the high probability of particle misplacement.

Improving medium stability by changing medium composition often adversely affects medium rheological properties, and vice versa. These medium properties are strongly influenced by parameters such as medium density, particle shape, particle size distribution, solid concentration, and level of slimes contamination. The factors controlling the rheological and stability properties of dense medium may be summarized as following:

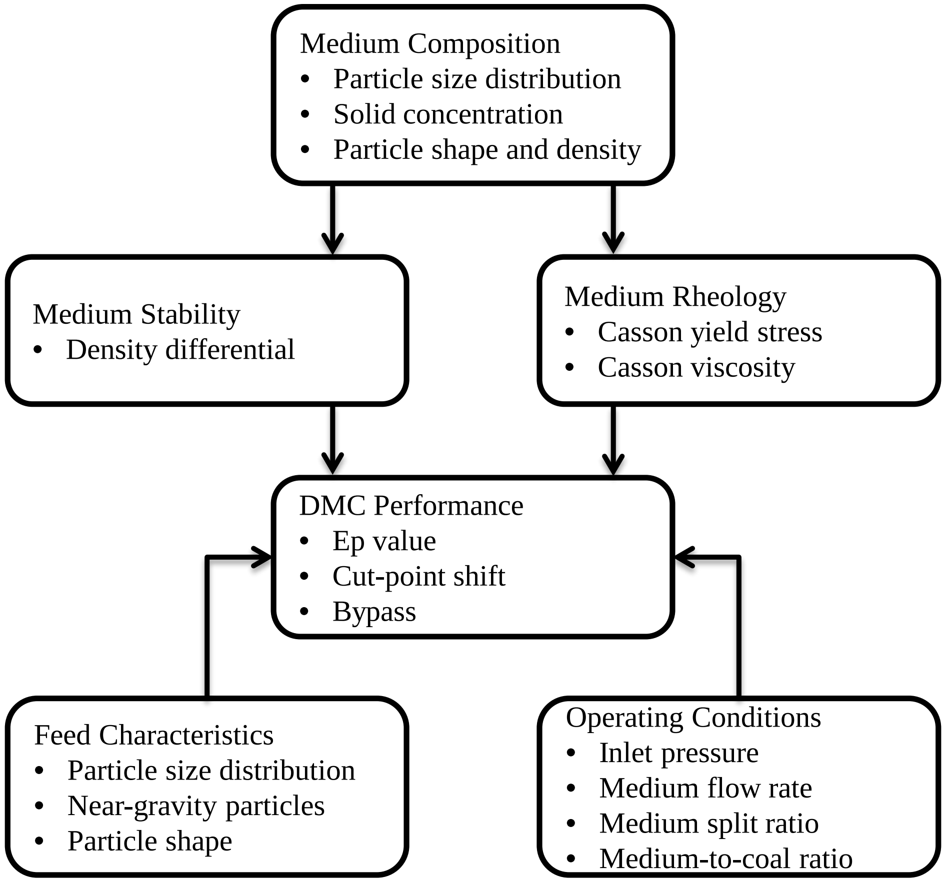

Figure 3.8: A schematic diagram of the performance indicators and the factors that affect dense medium separation performance.

- Medium specific gravity: both the medium apparent viscosity and stability increase with solid concentration and thus with medium density.
- Solid density: based on the specific gravity of solid particles, different degrees of solid concentrations is required to obtain a given medium density. Obviously, high density solids require a lower solid content (and thus a lower apparent viscosity and stability) to achieve a given medium density.
- Particle size distribution: finer medium particles produce higher stability and apparent viscosity values.
- Particle shape: spherical or smooth particles produce lower viscosity and stability values compared to angular or rough shape particles.
- Slime contamination: contaminants, such as slimes from the ore or coal, usually increase viscosity and stability due to both their lower solids density and fine

particle size (Chedgy, Watters, & Higgins, 1986; B. Collins, Napier–Munn, & Sciarone, 1974; Fourie, Van Der Walt, & Falcon, 1980; YB He & Laskowski, 1994; Y. He & J. Laskowski, 1995)

The investigation of the effect of medium properties on the DMC performance is complicated due to the contradicting influences of medium rheology and medium stability which, in the DMC separation process, are also related to the operating conditions. In addition, the individual and interactive effects of feed particle size with medium properties play an integral role in determining the cyclone performance efficiency. With fine medium particles, the effect of medium rheology dominates the separation efficiency. Under this condition, a decrease in medium density and an increase in centrifugal acceleration result in achieving a more efficient separation performance. On the other hand, with coarse medium particles, the effect of medium stability plays a main role. Consequently, the separation efficiency is improved by higher medium density or lower centrifugal acceleration (YB He & Laskowski, 1994; Y. He & J. Laskowski, 1995).

In addition, the DMC separation performance strongly depends on feed particles size. The separation of fine feed particles is considerably affected by the rheological properties of dense medium. Therefore, the separation efficiency and cut point shift is optimized when the medium apparent viscosity has an acceptable level. On the other hand, the separation of coarse feed particles is significantly affected by the stability properties of dense medium; thereby, the separation efficiency is improved and cut point shift is minimized when highly stable medium is applied (Y. He & J. Laskowski, 1995).

3.6.1 Medium Stability Effects

The ultrafine solid particles tend to settle out when the medium is left to stand without agitation due to their high density. The speed at which the particles settle is an indication of the medium stability. Medium stability directly affects separation performance by determination of the density gradient of the high–density suspension in the separation zone due to the effect of centrifugal acceleration. The degree of classification depends on the solid particle shapes and size distribution as well as medium specific gravity. The formation of density zone in the dense medium cyclone results in a poor separation efficiency and high cut point shift. At a constant medium density, the cut point may be decreased when the medium is stabilized by the addition of low–density slimes contamination or by the use of fine and irregularly shaped particles. In spite of static stability in dense medium bath, the direct measurement of the medium stability under dynamic conditions in a dense medium cyclone is difficult. The dynamic stability can be quantified indirectly as the density differential between overflow and underflow streams which is an indicator of the density gradient inside the separator. The medium stability parameter is industrially acceptable if the differential density is smaller than 0.40 (Bozzato, Bevilacqua, & Ferrara, 2000; D. Collins, Wright, Turnbull, & Ngan, 1983; Davis & Napier–Munn, 1987; YB He & Laskowski, 1994).

The factors affecting the density differential in the order of their significance include: the shape and size of solid particles, relative medium density, solid population, the apex diameter, and the cyclone inlet pressure. The separation efficiency could be increased by decreasing the density differential (that is by increasing suspension stability) (D. Collins et al., 1983).

He and Laskowski (Yingbin He, 1994) conducted a detailed rheological and stabilization study of the dense medium using magnetite with six different size distribution. The particle size distributions of the tested samples, as demonstrated in Table 3.3, are well described by the Rosin–Rammler–Bennett (RRB) particle size distribution: $d_{63.2}$ and m represent the size modulus and the distribution modulus, respectively.

Table 3.3: Particle size distributions of the tested magnetite (Sanders, 2007).

Sample	$d_{63.2}$ (micron)	M
Mag #1	30.5	3.2
Mag #2	18.0	1.6
Mag #3	33.0	4.1
Mag #4	4.3	1.9
Mag #5	2.7	2.5
Mag #6	35.0	3.9

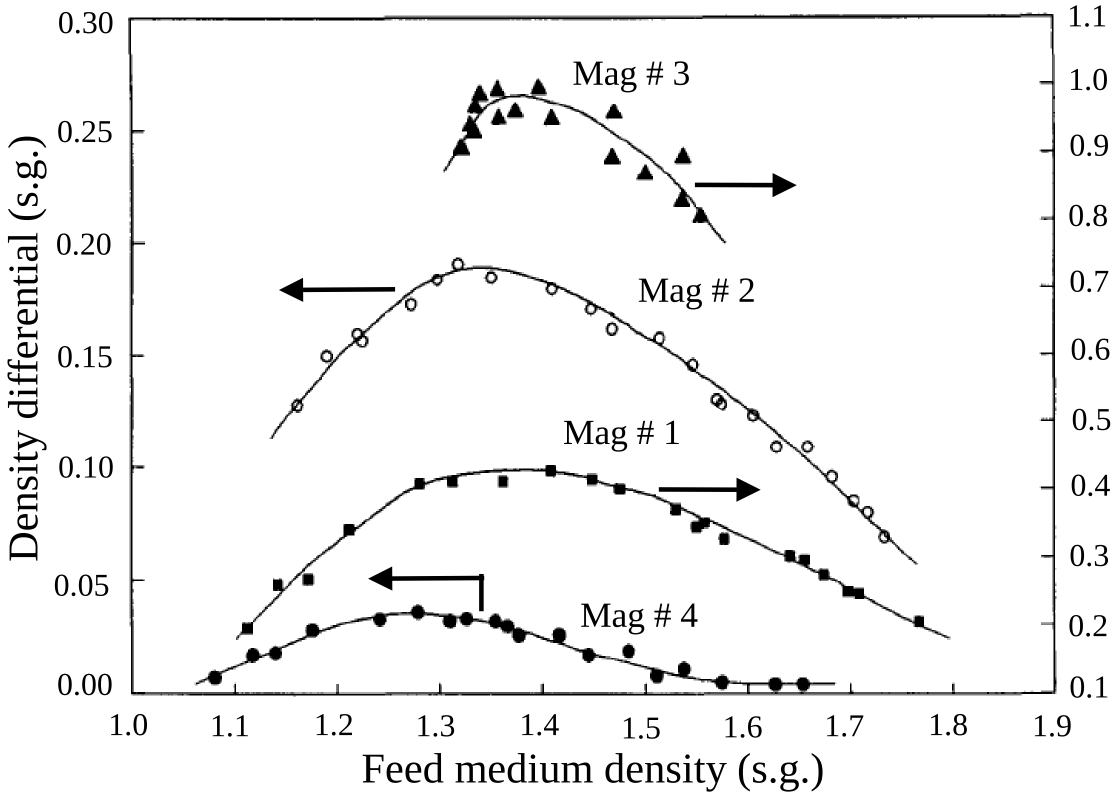

Figure 3.9: Effect of medium composition on the medium stability parameter (YB He & Laskowski, 1994).

The results of the medium stabilization tests, as shown in Figure 3.9, revealed that the medium stability, which is characterized by the density differential between underflow and overflow streams, was initially declined with a raise in medium relative density but then improved at higher medium density values. This observation can be explained by the greater impact of medium rheology at higher relative density values with elevation of the volumetric solid concentration in the medium. Moreover, at the same medium relative density, the stability of the medium with finer particle size distribution, such as Mag #2 and Mag #4, are higher than the medium with coarser particle size distribution (YB He & Laskowski, 1994; Sanders, 2007).

3.6.2 Medium Rheology Effects

The medium rheology can be determined by Casson viscosity (η_c) and Casson yield stress (τ_c) parameters. Although these two parameters are independent, they jointly define the rheology behavior of the dense medium. The apparent viscosity of solid suspensions can be expressed as following (YB He, Laskowski, & Klein, 2001; Y. B. He & J. S. Laskowski, 1995):

$$\eta_a = \left(\sqrt{\frac{\tau_c}{D}} + \sqrt{\eta_c}\right)^2 \tag{16}$$

where η_a is the apparent viscosity, τ_c is the Casson yield stress, η_c is the Casson viscosity, and D is the shear rate.

As Figure 3.10 demonstrates, the Casson yield stress is very susceptible to the changes in the size of solid particles, solid concentration, and medium relative density, while the Casson viscosity is only a subordinate parameter and turned out not to be very sensitive to these changes. It is believed that the studies, which correlate separation efficiency with apparent viscosity without considering apparent yield stress, may provide incomplete data about the relationship (YB He & Laskowski, 1994; YB He et al., 2001; Napier–Munn &

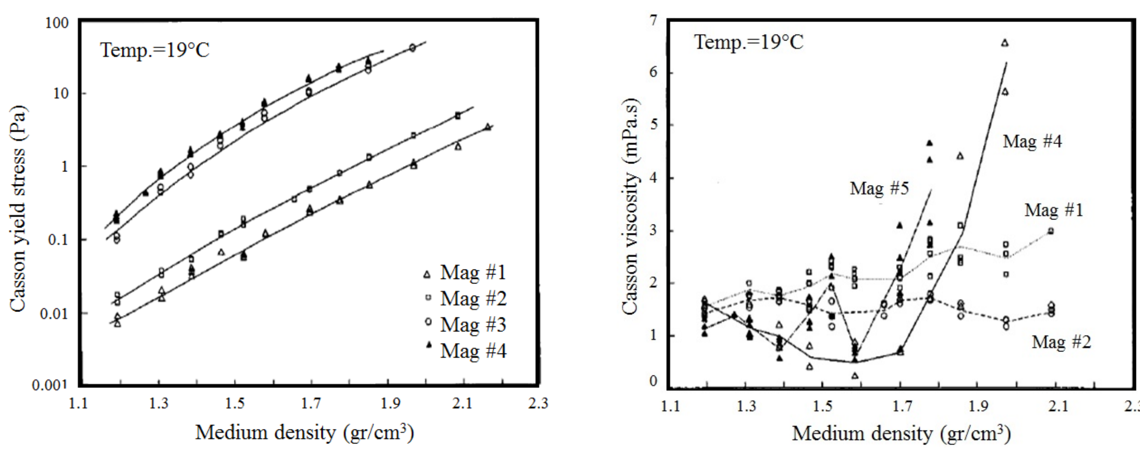

Figure 3.10: Effect of magnetite particle size distribution and medium density on the (A) Casson yield stress and (B) Casson viscosity (YB He & Laskowski, 1994; YB He et al., 2001).

Scott, 1990). The Casson viscosity is relatively constant over the medium relative density from 1.2 to 1.7. The Casson yield stress and viscosity values for the magnetite samples at the medium relative density of 1.45 are given in Table 3.3.At the same medium density the Casson yield stress values are considerably greater than the viscosity. In addition, the Casson yield stress is strongly dependent on the fineness of the magnetite samples and significantly increases as medium specific gravity elevates; but The Casson viscosity is not very sensitive to the changes in magnetite particle size or solid content over broad medium density ranges (YB He & Laskowski, 1994; YB He et al., 2001).

Napier–Munn proposed an empirical relationship between the separation efficiency (E_p) and the medium apparent viscosity to describe the effect of medium rheology on dense medium cyclone performance (Napier–Munn, 1990), as follows:

$$E_p = \Phi d^n \qquad (17)$$

where E_p is the probable error, Φ is a function of apparent viscosity and cyclone geometry, d is the feed size, and n is constant value.

Table 3.4: Measured Casson yield stress and viscosity values for the magnetite samples at a medium density of 1.45 (YB He et al., 2001).

Sample	Casson Yield Stress (mPa)	Casson Viscosity (mPa.s)
Mag #1	62	1.85
Mag #2	118	1.50
Mag #3	2110	0.70
Mag #4	2660	21.50

3.6.3 Bimodal Dense Media

The belief is that, at any given solid content, suspensions with bimodal particle size distributions of 25% to 40% fines of the total solid content have significantly much lower apparent viscosities than the monomodal suspensions. The unique rheological characteristics of bimodal suspensions may have a great potential for application in dense medium separation. The bimodal dense medium composed of a coarse and a fine narrow size fractions may decrease the detrimental effect of medium rheology parameter and, at the same time, enhance the medium stability (Barnes & Hutton, 1989; Farris, 1968; Yingbin He, 1994; Y. He & J. Laskowski, 1995).

He and Loskowski (Y. He & J. Laskowski, 1995) conducted a detailed experiment to study the effect of the solid content and magnetite particle size distribution on rheology of magnetite suspension. The magnetite dense medium, at the wide range of density values from 1.2RD to 1.8RD, composed of the mixture of Mag #4 and Mag #3 as the fine and coarse size fractions, respectively. The determined apparent viscosity for such bimodal magnetite samples (combination of Mag #4 and Magnetite #3 at various ratios) are shown in Figure 3.11. As results of the rheological tests confirm, at lower specific gravities of the bimodal medium the apparent viscosity stays relatively constant. At higher medium densities, up to about 35% of fine magnetite fraction, the apparent viscosity value stays low irrespective of the medium density (magnetite content) and increases only when the fine particle concentration exceeds this value.

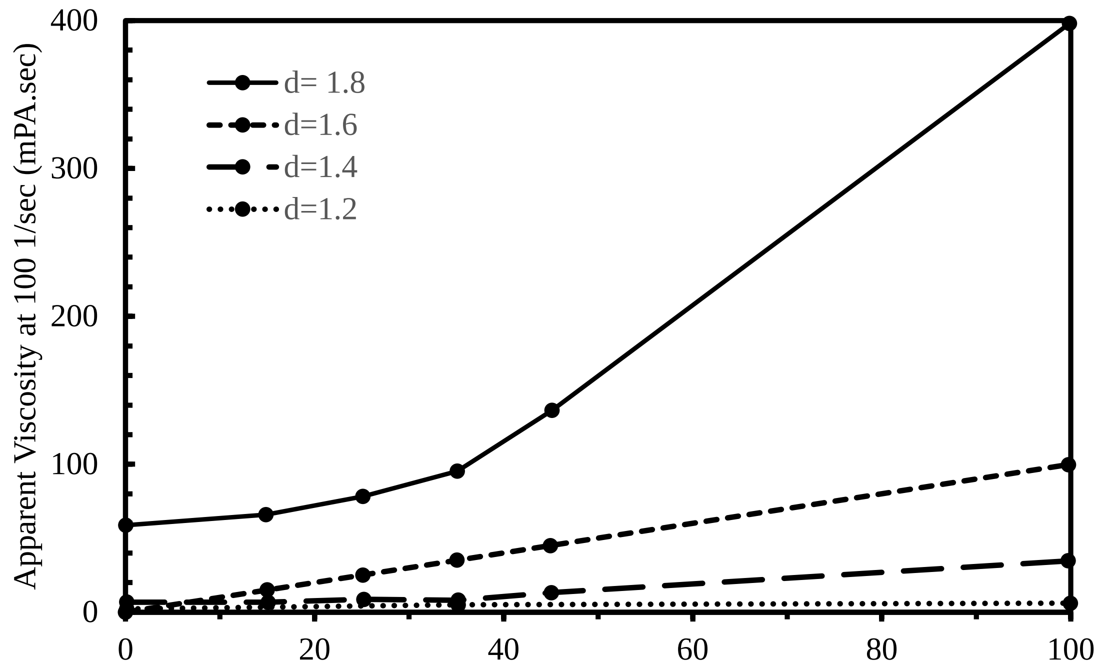

Figure 3.11: The apparent viscosity as a function of the proportion of fine magnetite (#4) in bimodal medium with Mag #3 at the range of medium densities (Y. He & J. Laskowski, 1995).

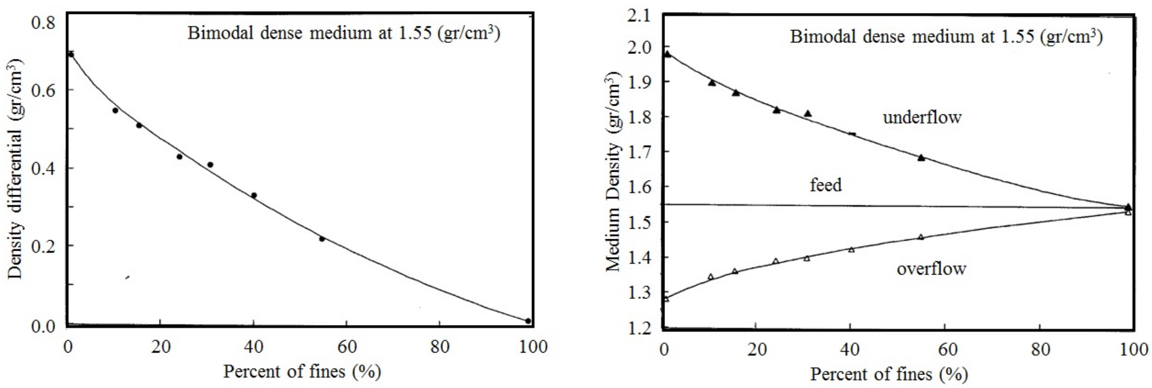

Figure 3.12: Effect of the bimodal dense medium composition on the medium stability (Y. He & J. Laskowski, 1995).

Another study was performed to investigate the stability behavior of the bimodal magnetite dense medium, at 1.55 RD, composed of the mixture of Mag #4 and Mag #6 as the fine and coarse size fractions, respectively. As demonstrated in Figure 3.12, the stability of the bimodal dense medium was not correlated well to the rheology parameter. Clearly, the classification of coarse magnetite particles determines the density differential, while the fine magnetite suspension serves as the medium for the coarse magnetite fraction; therefore, increasing in the percentage of the fine particles prevent the classification of the presented coarser magnetite fraction (Y. He & J. Laskowski, 1995).

3.6.4 Effect of Operating Conditions

One of the most important operating variables affecting dense medium cyclone performance is the cyclone overflow to underflow flow rate ratio (O/U ratio). The fundamental property of the O/U ratio is reflected by its close correlation with medium stability, separation efficiency, and cut–point shift. At constant medium specific gravity value and composition, as illustrated in Figure 3.13, the differential density values decreases remarkably when O/U ratio increases (YB He & Laskowski, 1994).

Under ideal conditions, when medium–to–coal ratio is very high, a higher O/U ratio leads to obtain a lower Ep value. An optimum O/U ratio for the dense medium cyclone processes in coal preparation, in which lower Ep value can be achieved, is recommended to be around 2.0. With fixed circuit and cyclone configurations and, more importantly, with a constant

O/U ratio of around 1.8 and a constant medium flow rate (inlet pressure), any change in the dense medium cyclone performance can be related to the change in the stability and rheological properties of dense media.

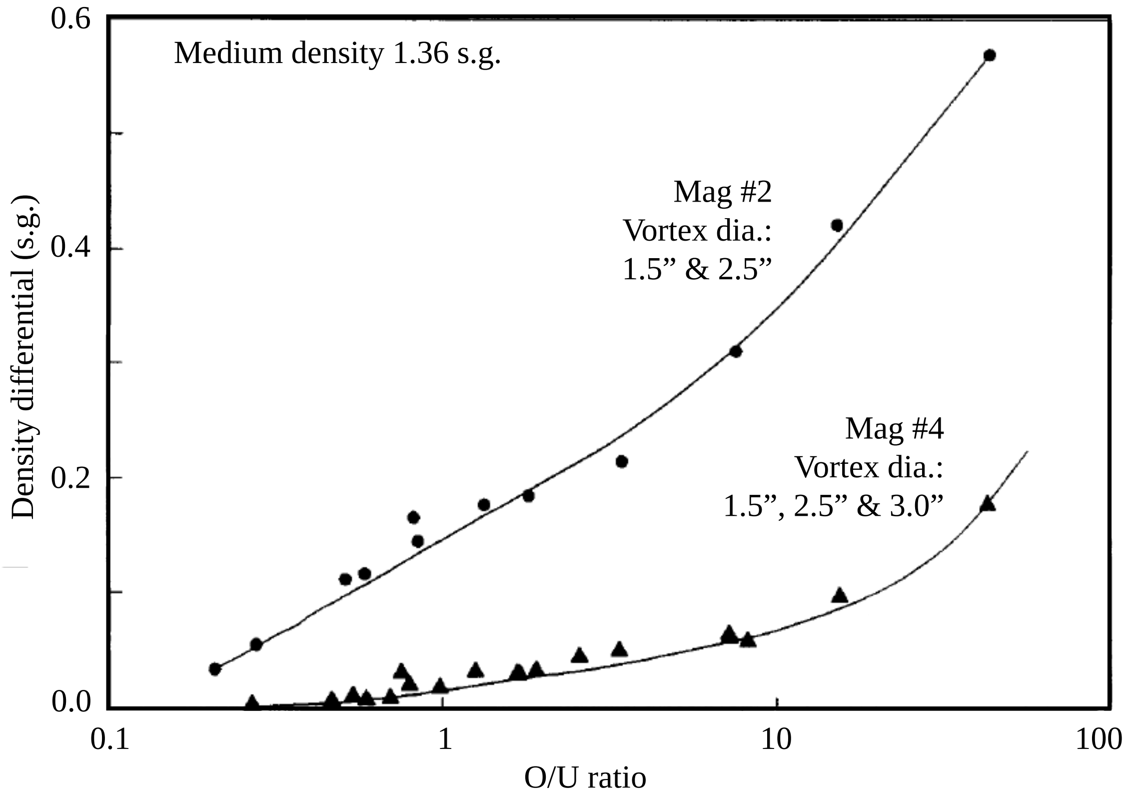

Figure 3.13: The effect of the overflow to underflow ration on the density differential (YB He & Laskowski, 1994).

4 EXPERIMENTAL

The aim of this investigation was to evaluate the potential of substituting magnetite in dense medium processes with silica–based materials which would represent an acceptable contaminant in the feed coal. Potential substitutes include fine silica sand, fine silicon, and ultra–fine silica fume (micro–silica), of which the latter two are by–product of silicon manufacturing. The experimental program was conducted in two phase; initially, medium stability evaluations were performed on all types of medium over a range of medium density including blends of the three silica sources. The stabilization study involved initial static and dense medium cyclone tests with no coal added in the suspension. Afterward, coal cleaning experiments were performed based on a statistically designed program utilizing the silica–based solids to achieve the following objectives:

1. Since the silicon production plants utilizes very low ash coal as a raw material in the silicon smelting process, the production of clean coal containing less than 1.5% ash was the main requirement.
2. As the value of organic efficiency is strongly affected by the stability and rheological properties of the dense medium, achieving the high organic efficiency (greater than 95%) was one of the most significant purposes.
3. Similar to organic efficiency, probable error value is a reliable indicative of dense medium properties; therefore, the minimization of probable error was one of the main purposes of this research work.
4. Under desirable conditions, the alternative dense mediums must yield no low–density bypass to maximize profit.

4.1 Coal Characteristics

4.1.1 Particle Size–by–Size Analysis

The coal sample was obtained from the Alden Resources coal preparation plant treating the Blue Gem coal. The Blue Gem coal bed located within the central Appalachian coal region and the south eastern Kentucky. It is a high quality (low ash and low sulfur content) coal resource that is categorized as high volatile bituminous B according to the American coal classification. Due to the high quality of the coal, Blue Gem coal is heavily recognized as a suitable source of carbon raw materials in the silicon smelting process (Myrvågnes, 2008).

The dense medium cyclone feed sample was screened using 6.35 mm and 1 mm screens. The materials coarser than 6.35 mm were crushed using a laboratory jaw crusher and then screened to obtain the 6.35×1 mm particle size feed material. The coal sample was mixed and split into 10 kg lots and then used as the dense medium cyclone feed material. A representative sample was collected for particle size–by–size and float–sink analyses.

The particle size–by–size analysis of the dense medium cyclone feed (Table 4.1) revealed that the majority of particles (37.40%) were in the 6.35×4.00 mm size fraction. The sample had a low inherent moisture content of 1.66 with a dry ash content of 13.22%, which was uniformly distributed in each size fraction.

Table 4.1: Size–by–size analysis of the Blue Gem dense medium cyclone feed sample.

	Incremental				Cumulative	
Particle Size (mm)	Actual Weight	Weight (%)	Moisture (%)	Ash (%)	Weight (%)	Ash (%)
6.35 × 4.00	98.55	37.40	1.50	14.36	37.40	14.36
4.00 × 2.83	72.49	27.51	1.78	12.23	64.91	13.46
2.83 × 2.00	43.79	16.62	1.84	13.78	81.53	13.52
2.00 × 1.00	48.67	18.47	1.62	11.87	100.00	13.22
Total	263.50	100.00	1.66	13.22		

4.1.2 Washability Analysis

The dense medium separation exploits the differential densities between coal and mineral matter particles to achieve the desirable specific gravity cut–point which provides the acceptable clean coal quality. Therefore, the feed float–sink analysis data was essential to evaluate the potential cleanability of the coal and access the theoretical yield and recovery achievable at a given product quality.

The float–sink test was conducted by submerging the coal sample into a medium of lithium metatungstate (LMT) and distilled water. By controlling the blend of LMT and distilled water, liquids having density values of 1.25, 1.35, 1.45, 1.55, and 1.65 relative densities were generated. The test was initiated by floating the coal sample in a liquid having a relative density value of 1.25. The float material was collected and sink fraction was transferred to the 1.35 density medium bath. This procedure, as shown in Figure 4.1, was carried out until the float–and–sink materials from the highest density medium (1.65 RD) were collected. The materials from six separate fractions were collected, as following:

- Fraction 1: material with relative density less than 1.25;
- Fraction 2: material with relative density between 1.25 and 1.35;
- Fraction 3: material with relative density between 1.35 and 1.45;
- Fraction 4: material with relative density between 1.45 and 1.55;
- Fraction 5: material with relative density between 1.55 and 1.65;
- Fraction 6: material with relative density more than 1.65.

Next, the collected materials were thoroughly washed to remove all of the salt–based liquid, dried, and then analyzed for weight, moisture and ash content. The float–sink data for the feed of dense medium cyclone are shown in Table 4.2.

Based on the results of the float–sink analysis in Table 4.2, the coal can be classified as easy–to–clean as indicated by a cleaning index (C.I = 1.3 cumulative weight % float / 1.6 cumulative weight % float) of 0.98 for the dense medium cyclone feed. High quantities of the coal mass were floated in the fractions 1 and 2 (85.48%) in which 52.00% existed in the 1.25×1.35 density fraction.

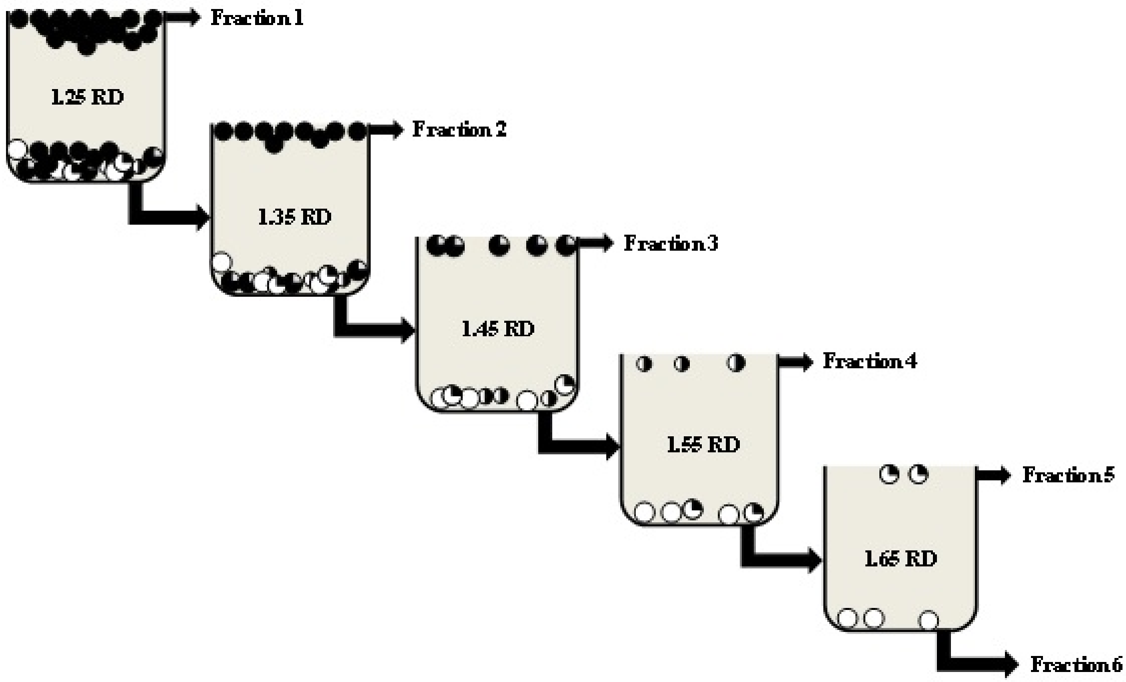

Figure 4.1: Schematic diagram of the float–sink analysis.

The washabiliy curves (Figure 4.2) revealed that the amount of near gravity particles decreases as the specific gravity increases, and the lowest amount of near gravity particles are in the 1.45×1.65 density fractions. It is also concluded that production of the clean coal with less than 1.50% ash for silicon production is theoretically achievable if separation occurs at the cut point below 1.50 RD.

Table 4.2: The washability data of the dense medium cyclone feed.

Specific Gravity		Individual Fractions		Cumulative Float		Cumulative Sink	
Float	Sink	Wt. (%)	Ash (%)	Wt. (%)	Ash (%)	Wt. (%)	Ash (%)
——	1.25	33.48	0.81	33.48	0.81	100.00	13.28
1.25	1.35	52.00	1.61	85.48	1.30	66.52	19.55
1.35	1.45	0.75	12.38	86.23	1.39	14.52	83.79
1.45	1.55	0.47	18.53	86.70	1.49	13.77	87.68
1.55	1.65	0.08	30.20	86.78	1.51	13.30	90.13
1.65	——	13.22	90.49	100.00	13.28	13.22	90.49
Total		100.00	13.28				

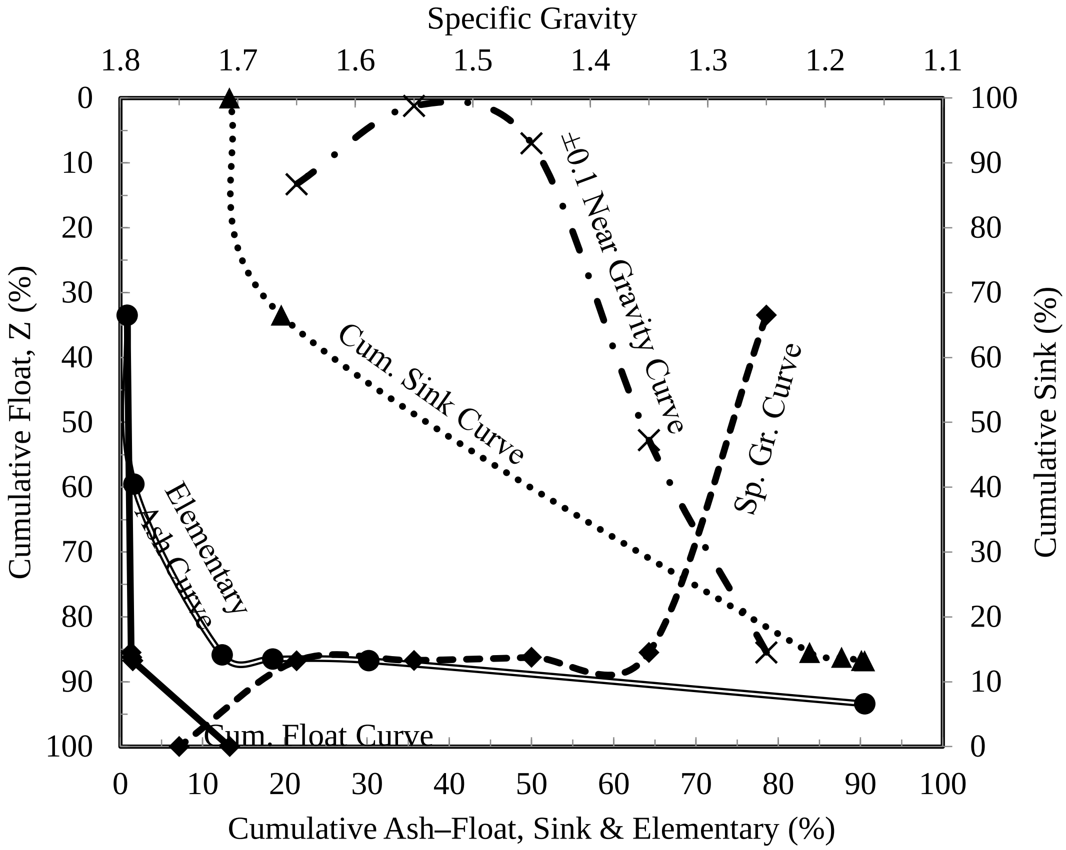

Figure 4.2: Washability curves for 6.35×1 mm fraction size of the Blue Gem coal sample.

4.2 Alternative Materials Characteristics

In addition to the requirement for a low ash content clean coal product, magnetite contamination has been identified as a problem in silicon production. Thus, this research was conducted to evaluate the potential of alternative materials that can be used to replace magnetite in dense medium cyclone circuit. Since quartz is the main raw material in the silicon smelting process, alternative silica–based materials, which included fine silica sand, silica fume, and fine silicon, were used to generate a dense medium for upgrading coal in the dense medium cyclone process.

Fine silica sand was purchased from an outside source as a potential alternative. The belief was that viscous issues that are typically associated with a silica sand based medium would not be significant due to the low medium specific gravity values used to produce the low ash content coal. Based on the particle size distribution data in Table 4.3, silica sand is significantly coarse as 83.28% of the particles are greater than 200 mesh. Moreover, there is a high population of relatively spherical particles in the sand sample, as demonstrated in Figure 4.3. Based upon these observations and also the relative density of sand particles (2.40), silica sand suspension at lower density values would likely be unstable.

Ultra–fine silica fume was obtained to be used as a medium stabilizer when silica sand was utilized to generate a low–density medium. Silica fume, which is also called micro–silica, is an ultra–fine powder generated during the production of silicon. It consists of spherical particles with 96.38% of the particles finer than 325 mesh. Obviously, silica fume, by itself, may not be applied as a dense medium within a wide range of density values due to high surface area and the high amount of ultra–fine particles, which causes viscosity problems (Figure 4.4 and Table 4.3). However, it might be used as a stabilizer to elevate the stability of medium formed using the coarser fine silica sand.

Table 4.3: Particle size distribution and specific gravity of the alternative silica–based materials.

Particle Size (mesh)	Weight Distribution (%)		
	Sand	Silica Fume	Silicon
Plus 60	0.77	0.23	0.65
60×100	13.75	0.55	1.48
100×200	68.76	1.60	3.03
200×325	11.47	1.24	48.21
Minus 325	5.25	96.38	46.63
Relative particle density	2.40	2.00	2.28

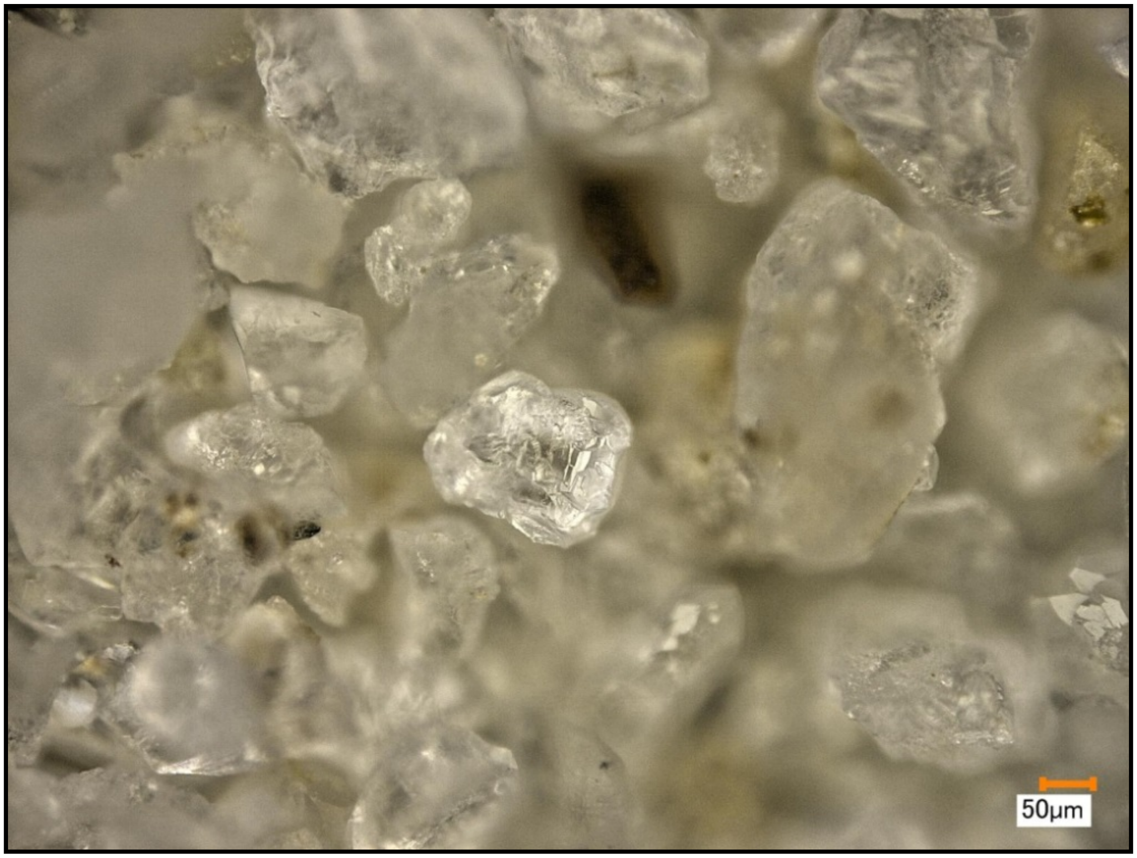

Figure 4.3: Scanning electron microscope images showing spherically of the sand particles.

Fine silicon material, which was purchased from the silicon production facility, was another silica–based material evaluated for the generation of dense medium. Obviously, silicon contamination is not an issue since the clean coal product is used to produce silicon. The fact that majority of the particles were irregular in shape (Figure 4.5) and have a size that is greater than 45 microns (325 mesh) as shown in Table 4.3 indicate that the silicon material could be used to generate an effective medium for low–density separations.

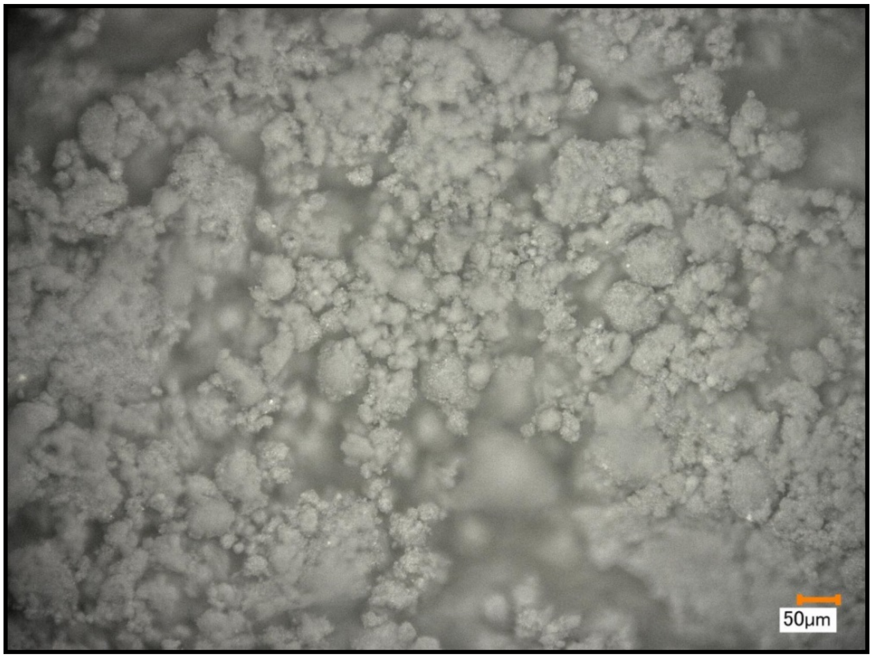

Figure 4.4: Scanning electron microscope images showing irregularity of the silicon particles.

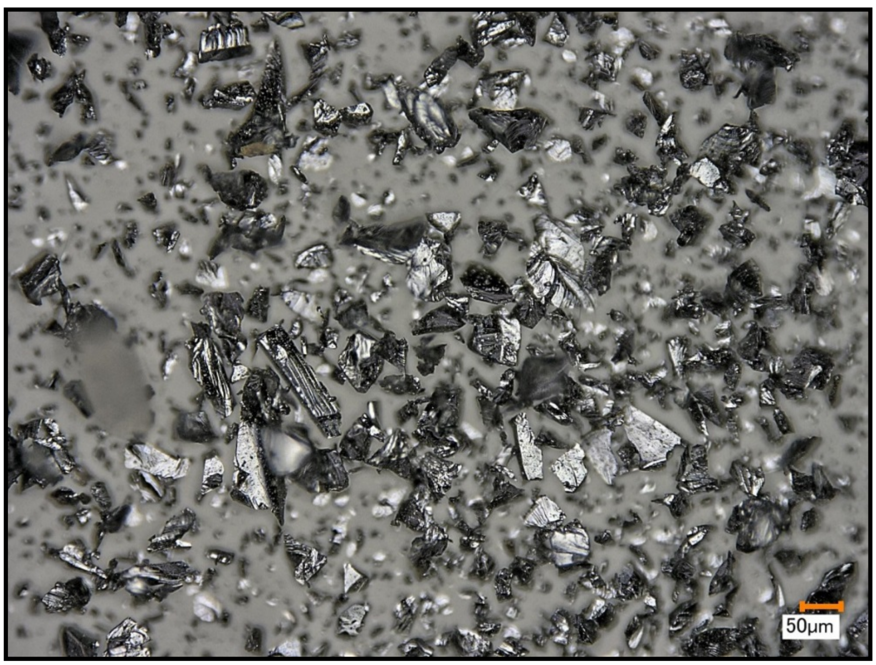

Figure 4.5: Scanning electron microscope images showing irregularity of the silicon particles.

4.3 Pilot–Scale Dense Medium Cyclone Circuit

Tests utilizing the silica–based suspensions as dense medium for cleaning coal were conducted using a 15–cm (6–in) diameter dense medium cyclone with 6.3–cm diameter vortex. The cone angle of cyclone and the angle of inclination of the set up were 20° and 10°, respectively. The inlet pressure operated between 3 (20 kPa) and 8 psi (50 kPa). Various apex sizes (40, 45, and 50 mm) were investigated. As shown in Figure 4.6, the dense medium cyclone was operated in a closed–loop circuit set with the overflow and underflow streams of the cyclone reporting back to the feed sump. A feed bypass stream also reported to the sump, which was used to collect feed samples. Figure 4.7 and Figure 4.8 demonstrate the actual pilot–scale laboratory set up of the dense medium cyclone test.

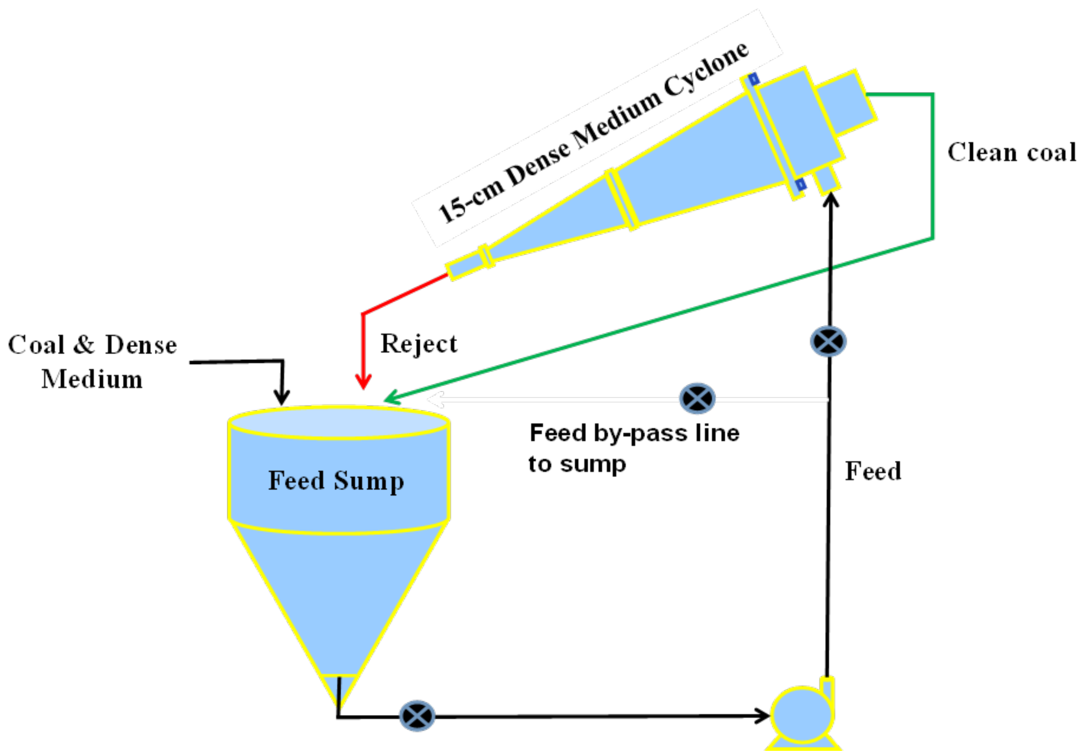

Figure 4.6: Schematic diagram of the dense medium cyclone circuit.

Figure 4.7: Dense medium cyclone with 15–cm diameter used in the experimental test program.

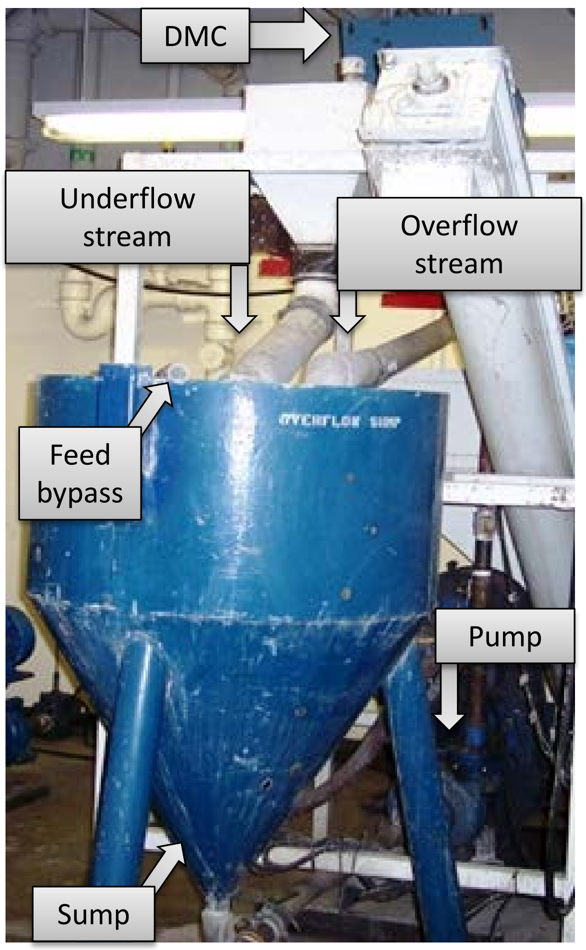

Figure 4.8: Feed sump and pump arrangement used in the closed–loop circuit.

4.4 Experimental Procedure

4.4.1 Medium Stabilization

To evaluate the potential of the silica–based materials for dense medium applications, an initial stability study was performed using different types of ultra–fine solids over a range of medium specific gravity values (Figure 4.9). Initially, alternative materials were mixed with water in a 4–liter beaker to generate a dense medium; and then the suspension was transferred to a 2–liter glass cylinder. Next, the alternative medium was pumped from the cylinder at the one liter height. Finally, the suspended material was filtered, dried, and weighted. Medium stability, S, was measured by the ration of the suspended solid particles (M_S) to the total solid mass (M_T):

$$S = 2 * \left(\frac{M_S}{M_T}\right) \tag{18}$$

Subsequently, the medium stability is assessed in a 6–in (15–cm) dense medium cyclone by measuring the pulp differential density values between the overflow and underflow

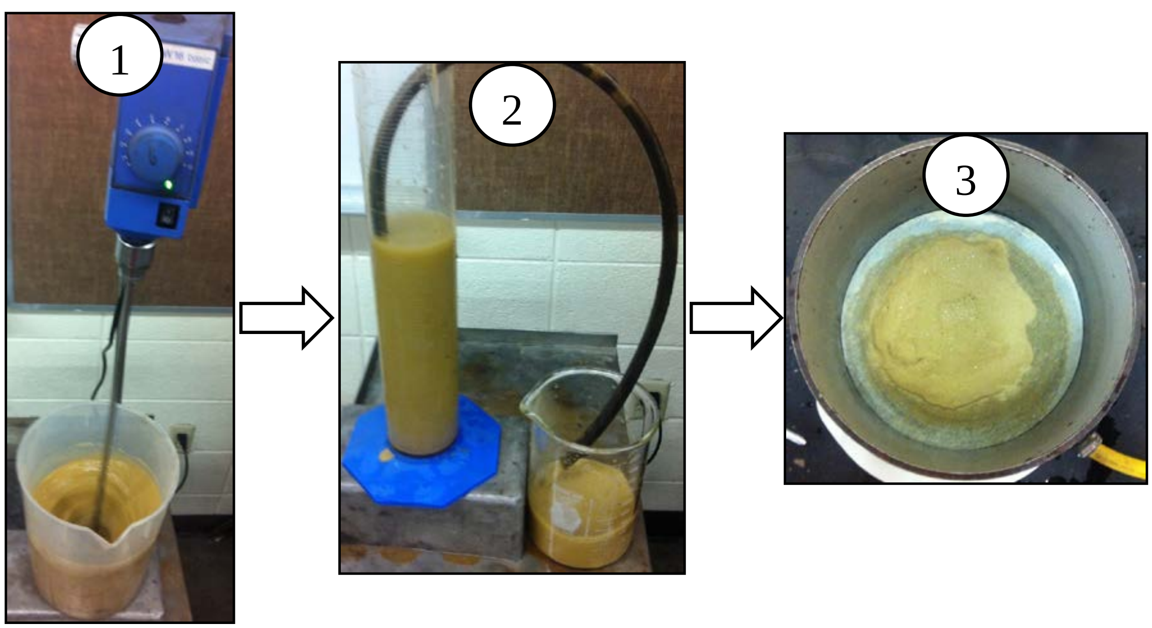

Figure 4.9: Medium stability assessment procedure on the silica–based alternative dense medium.

stream densities (i.e., ρu– ρo). The general standard for ensuring optimum performances is that the difference density should be less than 0.4 units. The final stability study was performed at various inlet pressures ranging between 3 and 8 psi over the selected medium specific gravity values.

4.4.2 Dense Medium Cyclone Test

Experiments were conducted based on the Box–Behnken and 2–factorial design to determine the optimum content of silica fume while also varying apex diameter and medium specific gravity, as shown in Table 4.4. The main purpose of the test program was to quantify the effect that the process factors exert on separation performance. As such, a total of five tests involving relative medium density and apex diameter parameters conducted while fine silicon alternative material was used as a dense medium. Similarly, test program based on a Box–Behnken statistical experiment three individual parameters (i.e., medium density, percentage of silica fume content, and apex diameter) was performed. A total of 13 tests were conducted. There were three additional trials, in which all factorial levels were set at the middle level (Table 4.5).

At the onset of each test, the relative density of the silica–based medium was adjusted to the selected value using a Marcy scale. Next, Blue Gem coal sample was added to the feed sump in an amount that provides a medium–to–coal ratio of 4:1. The coal and high density alternative medium were pumped to the cyclone at the rate that provides a static equivalent to 9–cyclone diameters, which is the standard value for dense medium cyclone operation. After a time interval of 15 minutes, which is necessary to ensure sufficient mixing and steady state conditions, samples from the overflow (clean coal and medium), underflow (reject and medium), and bypass feed (raw coal and medium) streams were collected simultaneously. Afterward, the samples were wet screened using a 16 mesh (1–mm) sieve to separate medium from the clean coal, reject, and feed materials. Finally, the detailed float–sink analysis on each sample was carried out to determine data analysis and performance assessment of each dense medium cyclone test.

Table 4.4 Dense medium cyclone test parameters and their value ranges used in the experimental test program based on a Box–Behnken design.

Parameter	Level					
	Low		Medium		High	
Alternative Medium	Sand/Fume	Silicon	Sand/Fume	Silicon	Sand/Fume	Silicon
Medium SG.	1.35	1.25	1.4	1.35	1.45	1.45
Silica Fume (%)	30	——	40	——	50	——
Apex diameter (mm)	40	40	45	45	50	50

Table 4.5: A total of 16 tests based on a Box–Behnken statistical experiment design involving three individual parameter using fine sand and silica fume dense medium.

	Parameters		
Trial Order	Medium (S.G.)	Silica Fume (%)	Apex diameter (mm)
1	1.40	50	50
2	1.40	50	40
3	1.35	40	50
4	1.35	50	45
5	1.45	50	45
6	1.40	40	45
7	1.45	30	45
8	1.40	40	45
9	1.45	40	50
10	1.40	30	40
11	1.45	40	40
12	1.40	40	45
13	1.40	40	45
14	1.35	40	40
15	1.35	30	45
16	1.40	30	50

4.5 Separation Efficiency Determination

4.5.1 Mass Yield and Combustible Recovery

Mass yield or mass recovery, is defined as the ratio of the amount of clean coal produced to the feed coal mass entering the process. The mass yield factor determines what portion of the raw coal feed can be recovered in the product stream to achieve a clean coal with certain specifications. It can be quantified by the "two product formula" expression, as follows:

$$Y = \frac{C}{F} = \frac{f - t}{c - t} \tag{19}$$

where F is the feed mass or rate (tph), C is the clean coal mass rate (tph), and f, c, and t are the ash content of feed, product and tailings, respectively.

In a dense medium cyclone process, the mass recovery is highly dependent on the feed characteristics, operating parameters of the cyclone, and the medium properties. A high efficiency process provides the required clean coal quality specifications while maximizing mass yield, which means no displacement of the valuable materials in the reject stream, is achieved.

In addition to the mass yield, combustible recovery is also one of the main performance factors which is directly associated with the amount of non–ash (combustible) material. The combustible recovery (R) can be determined by:

$$R = \left[\frac{(100 - c)}{(100 - f)}\right] * Y \tag{20}$$

4.5.2 Performance Efficiency

In a dense medium cyclone separation, raw feed particles are divided into two components (product and reject) based on their density differences. Under ideal conditions, all particles lighter than the selected separation density report into product A, as illustrated in Figure 4.10, while particles having specific gravity higher than the selected density report into product B. Based on the fact that practically no separator performs so accurately, some particles report into the wrong product stream, which is called misplacement of the particle. In other words, particle misplacement occurs when some particles having a density close to the point of separation report to the wrong product stream (Figure 4.10).

The partition curve is a tool for determination of coal cleaning performance efficiency which may be defined as: “a curve, which gives, as a function of physical property, the proportions in which the different elemental classes of raw feed having the same property are split into separate products” (Sanders, 2007). It provides several measures of efficiency, as provided in Figure 4.11, as follows:

- High density bypass (R_1): sometimes high density particles whose densities are far from the selected separation density fail to report into the reject stream due to the forces greater than the density–based forces. The causes of this performance error, which is called high density bypass, may include: vortex finder wear, low inlet pressure, high overflow–to–underflow ratio, and viscous medium.
- Low–density bypass (R_2): occasionally light particles, having the specific gravity

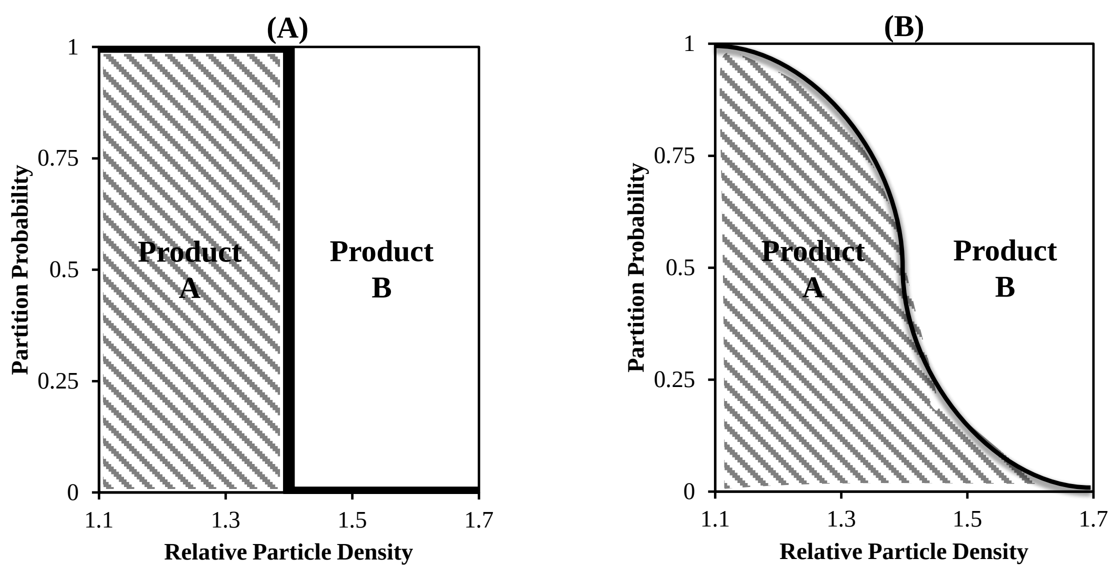

Figure 4.10: The partition curve of the (A) ideal dense medium separation and (B) imperfect dense medium separation when the particle misplacement occurs.

that is considerably far away from the separating density, report into reject stream.

This inefficiency performance (low–density bypass) may occur due to over loaded vortex finder, apex wear, unstable medium, or low feed pressure which provides low feed volume flow rate which results in low medium–to–coal ratio in overflow.

- Cut–point (ρ_{50}): it is the relative density at which the dense medium cyclone separates the coal and rock. The partition number at the cut point is 50%, as it is shown in Figure 4.11.

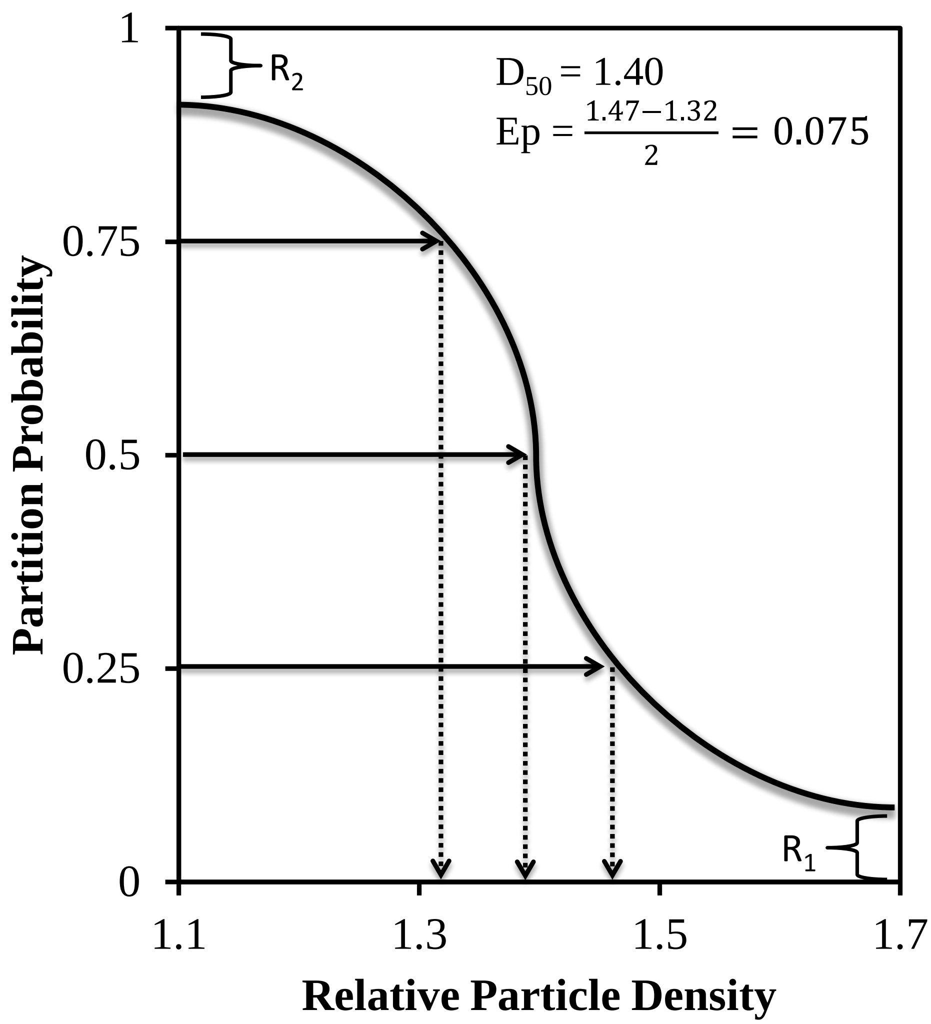

Figure 4.11: Calculation of the separation performance parameters using partition curve.

Probable error or Ecart probability (Ep): the parameter describes the slope of the partition curve centered around the cut–point and is determined by half the differential relative density of particles having a 75% and 25% probability of reporting to the product stream:

$$Ep = \frac{\rho_{75} - \rho_{25}}{2} \tag{21}$$

The more efficient dense medium separation is represented by the steeper partition curve (lower Ep value). Under ideal conditions, where the partition curve is vertical, the Ep value is zero. For dense medium cyclone processes, the separation efficiencies are generally at a high lead as indicated by Ep values in the range of 0.01 to 0.05.

In addition to the separation efficiency parameters that can be determined using partition curve, organic efficiency (OE) is widely used as an indicator of the separation efficiency in coal preparation processes. The OE value is calculated (typically as a percentage) as a ratio of the actual clean coal energy recovery to the theoretical maximum recovery achievable according to washablity data analysis (Eq. 22). It is also can be determined simply as the ratio between measured and theoretical clean coal yield at the same feed ash content. The fact that organic efficiency value reflects the impact of both probable error and high/low–density bypass makes the parameter a very effective measurement of overall process efficiency.

$$Organic\ Efficiency\ (\%) = \frac{Actual\ Energy\ Recovery}{Theoritical\ Energy\ Recovery} * 100 \tag{22}$$

5 RESULTS AND DISCUSSIONS

5.1 Introduction

The dense medium cyclone (DMC) is one of the most widely applied gravity concentration units for upgrading the coarse and intermediate run–of–mine coal in coal preparation plants. The process uses a dense medium consisting of a suspension of finely ground magnetite to produce a clean coal containing below 1.5% ash when applied commercially to produce pure silicon. Magnetite contamination, primarily from the dense medium process, has been identified as the most detrimental impurity in the silicon production process. In response to concerns regarding iron contamination, a study has been performed to evaluate the potential of alternative silica–based materials that can be used to generate an effective dense media for coal–cleaning applications. Alternative silica–based materials were used as a substitute for magnetite medium since silica–rich material (i.e., quartz and quartzite) is a feedstock for the production of metallurgical grade silicon and by–products of the production process are readily available.

Dense medium cyclone performance efficiency may be determined using various indicators such as probable error, organic efficiency and low–density bypass parameters which are strongly affected by medium stability and rheological properties. The individual and interactive effects of medium compositions (i.e., particle size distribution, particle shape, and solid density) and operating conditions (i.e., inlet pressure, overflow/underflow rate ratio) control the rheological and stability properties of dense medium.

Medium stabilization studies were performed on all dense media types over a range of medium density. The tests involved initial static stability experiments in a 2–liter glass cylinder and dynamic stability tests with no coal added in the suspension. In the first phase, medium stabilization was assessed qualitatively by determining the percentage of suspended solid mass, in a certain time, within the suspension. In the next phase, dynamic medium stability was indirectly determined by the difference in the medium density in the overflow and underflow streams.

Based on the medium stabilization tests results, coal cleaning tests were conducted based on a parametric design using the alternative silica–based mediums. Washability analyses were performed on the feed, product and tailings material from each test to provide the needed data to construct partition curves. Finally, the results of the dense medium cyclone performance were used to develop empirical models that characterize the overall process. The empirical models describe the response variables (i.e. organic efficiency, probable error, and high density bypass) as a function of operating parameter values (i.e., apex diameter and medium relative density).

5.2 Medium Stabilization

5.2.1 Static Medium Stability

The ultrafine magnetite particles tend to settle out when the medium is left to stand without agitation due to its high density. The speed at which the particles settle, or the relative movement of the solid particles in the liquid under gravitational force, determines the suspension's degree of homogeneity and is a reliable indication of the medium stability.

In the absence of coal, an initial stability test was conducted to evaluate the medium stability that the alternative silica–based materials provide over a range of medium density values. Medium stabilization was assessed qualitatively by determining the percentage of suspended solid mass within the suspension. Based on the qualitative information, valuable preliminary information was obtained about the suspension stability and its variation over a selected range of medium specific gravity values was generated (Table 5.1).

Table 5.1: Qualitative classification of medium stability characteristics based on the suspended mass (%).

Mass Suspended (%)	Level of Stability
75–100	Completely Acceptable
50–100	Relatively Acceptable
25–50	Relatively Unacceptable
0–25	Completely Unacceptable

As indicated in Figure 5.1, at the medium density fraction between 1.20 and 1.25, the stability of fine silicon suspension can be classified within the relatively acceptable range. As the medium relative density increased, the amount of suspended silicon particles raised due to a decrease in the settling rate of the solid particles. It is speculated that the silicon suspension was significantly stable over a wide range of medium specific gravity from 1.25 to 1.50. As Table 4.3 revealed, fine silicon suspension, by itself, can be considered as a bimodal suspension with compositions of 45% fines of the total solid content. The bimodal dense medium of coarse and fine narrow size fractions may provide high medium stability

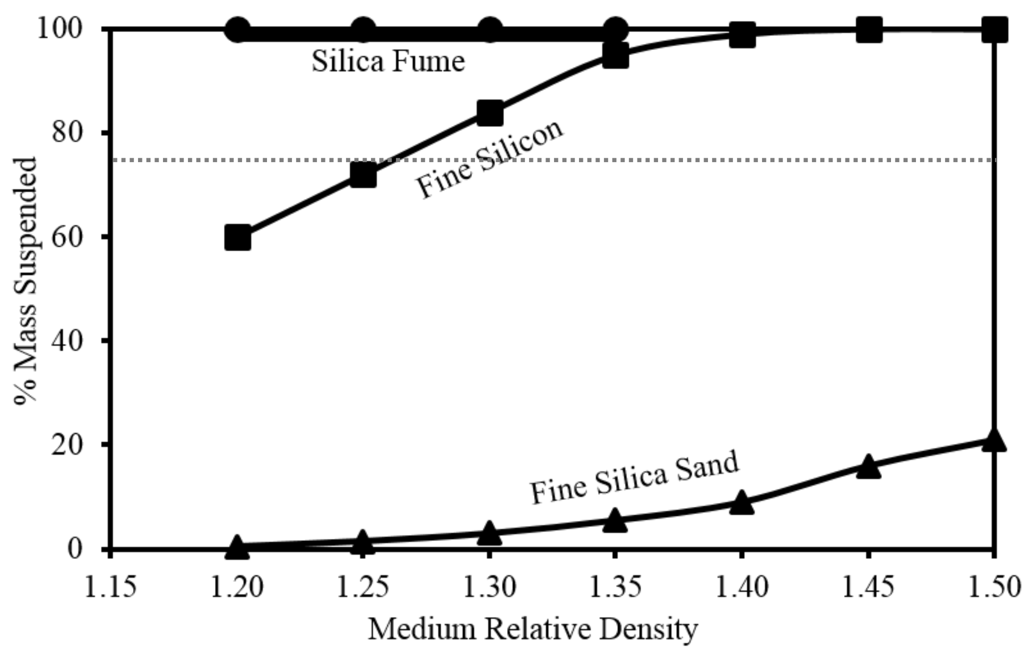

Figure 5.1: Static stability test results using various alternative silica–based materials.

properties while eliminating the adverse effect of medium rheology. Moreover, high volumetric concentration (due to low particle density) of irregularly shaped particles (Figure 4.5) cause the elevation of turbulent drag forces, thereby achieving highly stable medium even at relatively low specific gravities. Therefore, it was expected that the fine silicon medium could potentially be used as an effective high density suspension in dense medium cyclone separations over a wide range of medium specific gravity values.

On the other hand, the fine silica sand suspension was completely unstable at the medium density fraction between 1.20 and 1.50; while the ultra–fine silica fume medium was completely stable (Figure 5.1). The instability property of sand medium was a result of the high presence of nearly spherical coarse particles and relative coarse size distribution which provided fast–settling rates in the suspension. On the contrary, the high population of ultrafine low–density silica fume particles with its large surface area causes very high stability and viscous medium characteristics. It can be concluded that both fine silica sand and ultrafine silica fume suspensions, with monomodal compositions are not applicable by themselves in dense medium separations.

To overcome the instability of the fine silica sand suspension, a bimodal dense medium composed of a coarse (sand) and a fine (fume) size fractions was generated at the different proportions of silica fume to the total solid concentration. Results of the static stability analysis (Figure 5.2) demonstrated that the mixture of sand and fume suspension, as a bimodal medium, was considerably stable over the density range of 1.30 to 1.50 by adding different amount of silica fume. At 75% mass suspended, a sufficiently stable medium occurs at around 14% silica fume concentration at a medium density of 1.50. The silica fume concentration required to meet acceptable suspension stability increases to 24% for a 1.40 S.G. medium and 41% for a medium S.G. of 1.30. This trend reflects the ability of material with a significantly smaller particle size distribution to stable a material having slightly coarser distribution. It was also showed that, at the same ratio of silica fume, with an increase in medium specific gravity the percentage of the suspended mass remarkably raised due to the elevation of turbulent drag forces on the particles.

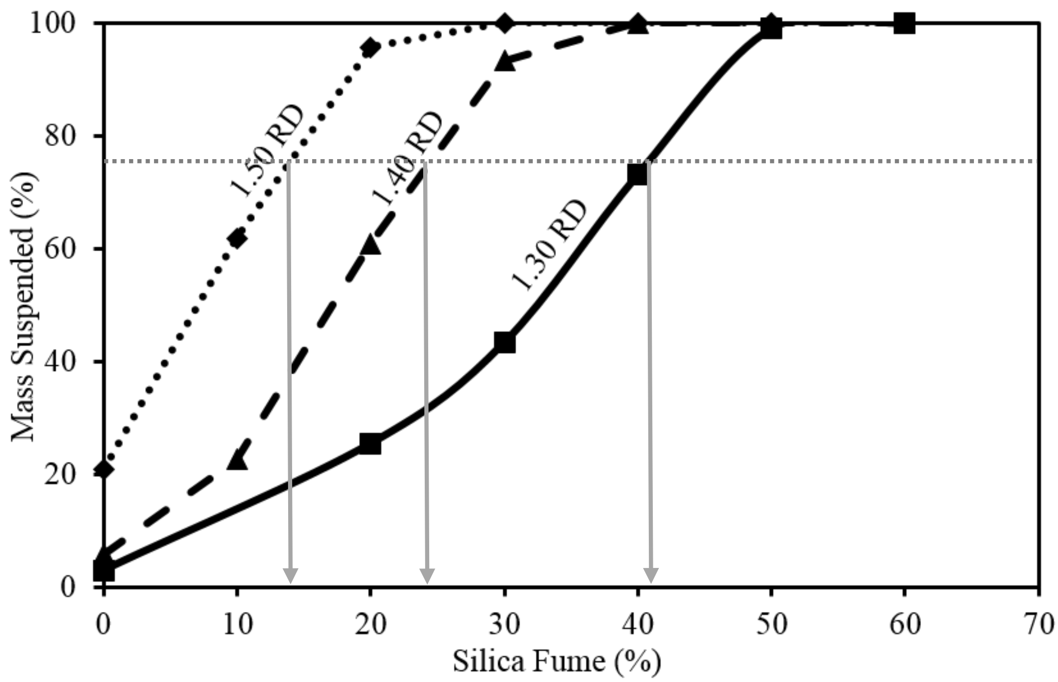

Figure 5.2: The preliminary static stability study on the bimodal suspension composed of fine silica sand and ultrafine silica fume.

Consequently, based on the results of the initial stability study, it was assumed that fine silicon suspension and the blend of fine silica sand and silica fume medium can be effectively applied in dense medium cyclone process. To verify this finding, dynamic stability tests were conducted in a dense medium cyclone using medium in the absence of coal.

5.2.2 Dynamic Medium Stability

Medium stability directly has influence on separation performance through the creation of density gradients within the cyclone due to the effect of centrifugal acceleration in the dense medium cyclone. The existence of density gradients make the separation performance sensitive to the apex diameter which is typically not variable in an on–line manner. In the absence of coal, dynamic medium stability was quantified indirectly by measuring the difference in the pulp density in the underflow and overflow streams of the dense medium cyclone under various inlet pressures ranging from 3 to 8 psi. The density differential is a reliable indicator of the amount density gradation inside the separator, and is industrially acceptable if the density differential is smaller than 0.40. However, the detrimental effects of medium rheology slightly appear when the differential density becomes smaller than 0.15. The most significant factors affecting the density differential include: the shape and size of solid particles, relative medium density, solid particle population, and the cyclone inlet pressure.

As shown in Figure 5.3, the fine silicon material generated a stable medium over a wide range of inlet pressures (3 to 7 psi) and the target medium relative density values (1.2 to 1.45 RD), which is in agreement of the findings at the static test results. The stability of the fine silicon medium improved with the increase in medium specific gravity, which was reflected by a decrease in density differential values. Obviously, this trend was due to a raise in solid concentration, which hindered the silicon particle settling rate thereby providing a stable medium over a wide range of medium density values. As shown in Figure 5.3, the density differential increased as inlet pressure was raised which was due to the elevated centrifugal forces in the cyclone. In other words, increasing feed pressure raised the classification degree of the fine silicon particles which caused particle concentration near the apex of the cyclone. Moreover, the density differential values

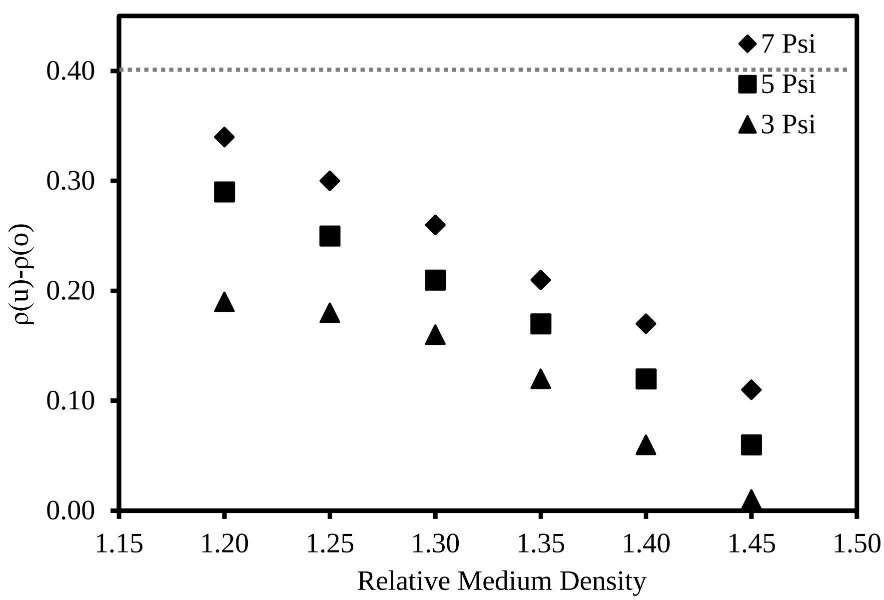

Figure 5.3: Medium stability achieved using the fine silicon material over a range of inlet pressures and medium density values.

smaller than 0.15 manifested the possibility of high apparent viscosity at medium specific gravity values greater than 1.40.

As expected, the medium formed by the silica sand material was completely unstable over a wide range of medium density and feed inlet pressure (Figure 5.4). At the lower medium density fraction between 1.25 and 1.40, nearly all of the silica sand particles reported to the underflow stream due to low solid concentration and, thus, minimum hindered particle settling in the suspension. With an elevation in medium density above 1.40, particle population increased, which improved the stability of the medium due to the development of hindered settling conditions. However, the silica sand medium remained unacceptably stable even at medium density values as high as of 1.55 RD, when the solid concentration was around 60%. Consequently, the dynamic stability study verified the results of the preliminary static test that the spherical shape of the silica sand particles and their relatively coarse size creates an unacceptable material for using by itself to form a dense medium.

To improve the stability of silica sand suspension, varying amounts of silica fume was blended with the fine silica sand to generate dense mediums stabilized due to the effect of

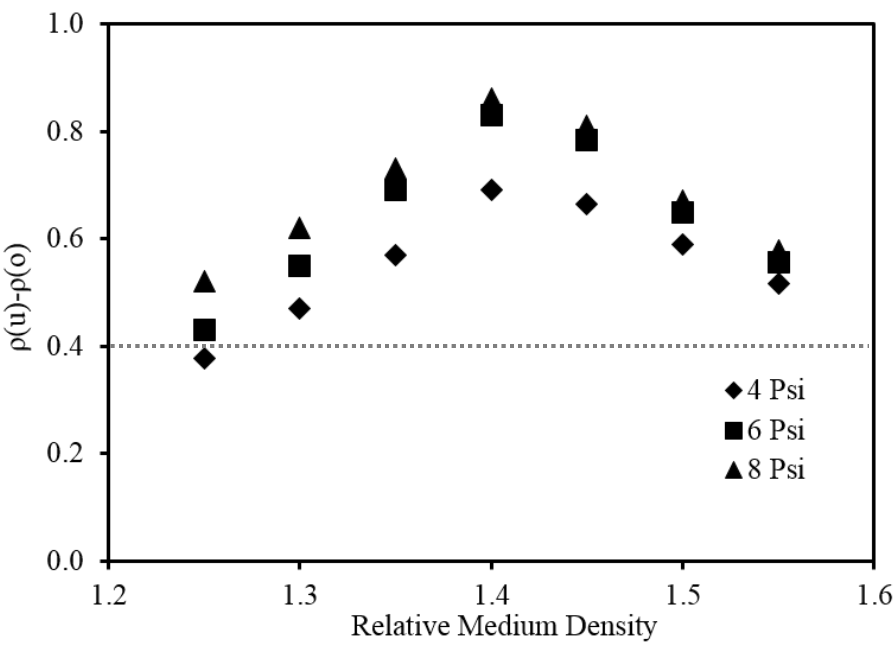

Figure 5.4: Medium stability achieved using the fine silica sand material over a range of inlet pressures and medium density values.

a bimodal particle size composition. Dynamic stability was evaluated over a range of blend proportions and medium density values. As shown in Figure 5.5, blends that range from 30% to 50% silica fume, as suggested by the results of the initial stability study, were found to provide relatively stable medium within the medium density target range. The density differential decreased and the medium became more stabilized with an elevation in the ultrafine silica fume particle concentration in the suspension. It is speculated that the classification of silica sand particles determined the density differential, while the ultrafine silica fume suspension served as the medium for the coarse sand fraction; therefore, increasing in the percentage of the ultrafine particles effectively prevented the presented coarser silica sand fraction from classification. The bimodal suspension composed of 50% silica fume and 50% fine silica sand provides acceptable medium stability over the entire range of medium density values tested. However, viscosity is likely an issue for medium density values above 1.35. A concentration of 40% silica fume provides an ideal medium for medium density values between 1.35 and 1.45. The use of 30% silica fume is not sufficient to stabilize the fine silica sand at medium density values below 1.40. However, the medium is sufficiently stable at a 1.40 medium density and higher values.

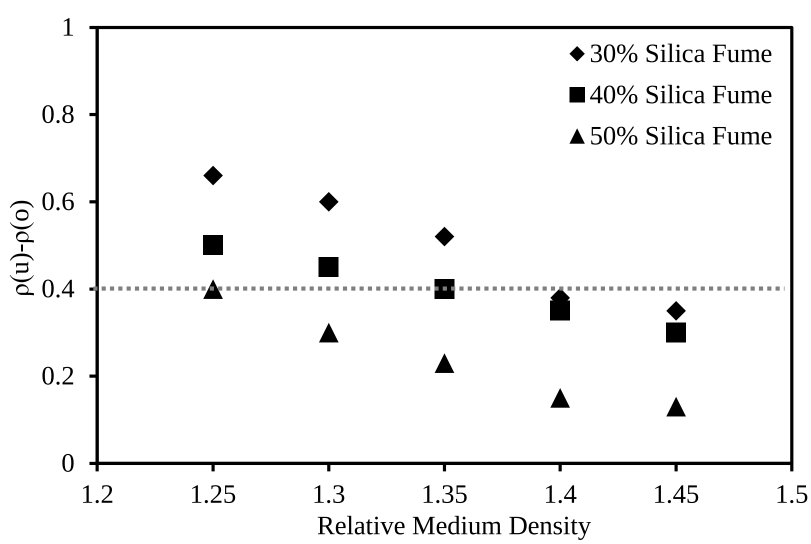

Figure 5.5: Medium stability achieved using the blend of fine sand and silica fume silicon material over a range of inlet pressures and medium density values.

5.3 Dense medium Cyclone Separation

5.3.1 Parametric Design Using Silicon Medium

The objective of the parametric test program was to evaluate the potential use of fine silicon medium as a replacement for magnetite in the establishment of a dense medium. A detailed experimental program was conducted to determine the separation performance and efficiency achievable when treating 9×1 mm Blue Gem seam coal using a dense medium cyclone. The test program involved a range of medium density values from 1.25RD to 1.45RD and different sizes of apex diameter (40, 45, and 50 mm).

Float–sink study was performed on the feed, product, and tailing material from each test to construct partition curves shown in Figure 5.6 over a range in medium density values from 1.25RD to 1.45RD. Afterward, the partition curves were used to determine the coal cleaning efficiency parameters in each dense medium cyclone test. The parametric test results, as shown in Table 5.2, revealed that the production of clean coal containing around 1.00% was possible over a range of selected medium density values using the fine silicon as a medium in a dense medium cyclone process. Moreover, the maximum organic efficiency of 97.19% and the minimum probable error value of 0.005 were achievable. There was no measurable bypass of high density particles to the product stream or low–density particles in the reject stream.

Density offset, which is the difference between the actual cut–point density and the medium density, reflects the medium stability and viscosity properties. At medium density values between 1.25RD and 1.45RD, the density offset values were in a range between –0.4 and +0.2 density units indicating that the dense medium composed of the fine silicon material was significantly stable over a wide range of medium specific gravity values from 1.25RD to 1.45RD. At medium density values of 1.35RD and 1.45RD, the density offset

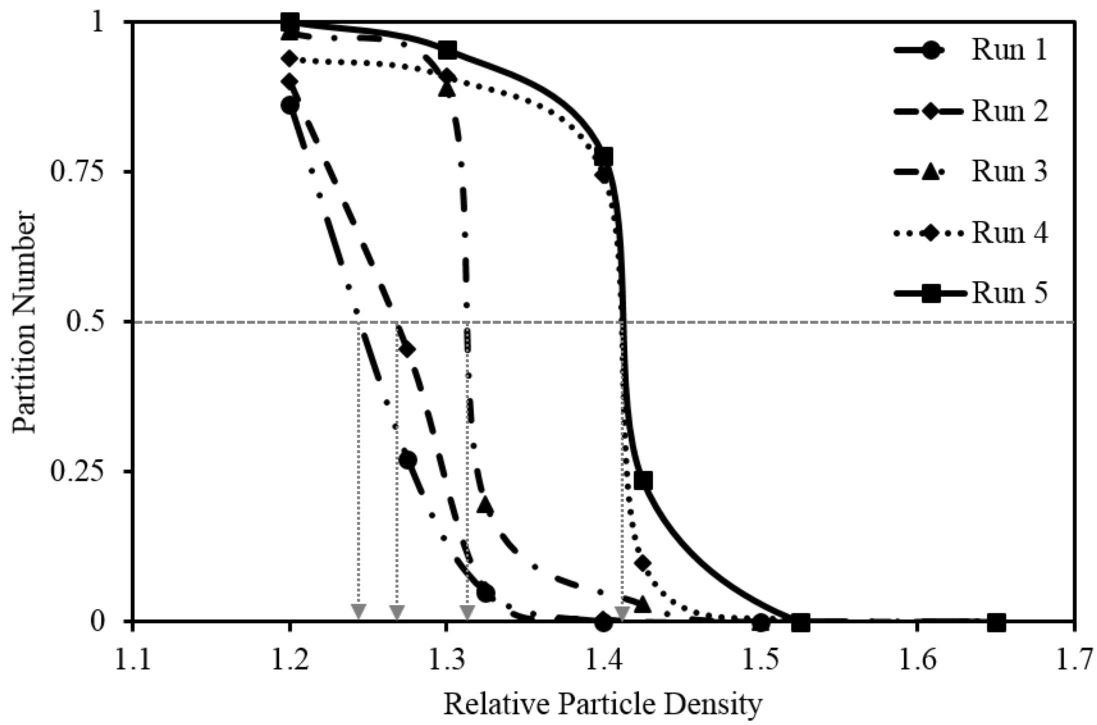

Figure 5.6: Partition curves generated from the separation achieved by fine silicon medium at 9–cyclone diameters inlet pressure over a range of medium density.

dropped around –0.4 density units indicating the possibility of detrimental effects of medium viscosity. However, these effects are negligible due to the relatively small amount of difference between medium density and actual cut–point.

As discussed in section 5.2.1 and 5.2.2, at the medium density of 1.25 RD, the stability of fine silicon medium can be classified within the relatively acceptable, not completely, range. At this medium density, the existence of density gradients make the separation performance susceptible to the apex diameter. As a result of apex enlargement, the actual cut–point decreases and results in the less efficient separation. As shown in Figure 5.6 and Table 5.2, shrinkage of the apex diameter (from 50–mm to 40–mm) in the test number 2 resulted in decreasing low–density bypass and probable error thereby achieving a higher organic efficiency.

Table 5.2: Summary of the DMC performance achieved using fine silicon medium.

Trial Order	Medium (S.G.)	Apex diameter (mm)	Ash Content (%)			Mass Yield (%)	Combustible Recovery (%)	Ep	OE (%)	R_2 (%)	Cutpiont (S.G.)	Density Offset
			Feed	Product	Tailing							
1	1.25	50	12.31	0.78	30.79	61.58	69.68	0.035	94.03	13.77	1.25	0.00
2	1.25	40	12.31	0.82	35.45	66.82	75.58	0.013	95.47	9.95	1.27	0.02
3	1.35	45	10.96	0.99	64.00	84.18	93.60	0.005	95.11	1.71	1.31	–0.04
4	1.45	50	9.26	1.02	45.89	81.64	89.05	0.008	89.69	10.18	1.41	–0.04
5	1.45	40	9.26	1.10	71.76	88.45	96.41	0.018	97.19	0.00	1.41	–0.04

At a relative medium density of 1.35, when medium is completely stable, more efficient separation was obtained due to the balanced stability and rheological properties of the dense medium. At the trial order 3, highly stable medium and a presence of lower amount of near gravity particles in the feed resulted in achieving lower probable error value. However, high amount of particles reporting to the overflow stream (87.90%) resulted in the considerable decline in medium–to–coal ratio in the overflow stream, thereby misplacement of coarse low–density particles took place (Table 5.2).

The comparison between trial order 4 and 5 revealed that highest values of Ep were achievable in the presence of low amount of near gravity particles (close to zero). At a medium density of 1.45RD, since high amount of particles (>90%) reported to the overflow stream, using smaller apex increased the population of dense medium in the overflow stream; therefore, a considerable elevation of medium–to–coal ratio in the overflow stream resulted in no displacement of the low–density materials. The separation efficiency measured for the medium density of 1.45RD when using apex diameter of 40–mm are excellent with organic efficiency value around 97%, probable error value of 0.018 and no measurable low–density bypass.

The results from a statistically designed test program based on the 2–Factorial model were used to identify the most significant process parameters that characterize the overall process. The empirical model describes the response variables (i.e. organic efficiency, probable error, and high density bypass) as a function of operating parameter values (i.e., apex diameter and medium relative density). The relationship between operating parameters could be represented as a 3–dimentional surface, which allowed the one to study the individual and interactive effects of factors on the response variables.

The coefficient of determination (R^2) was used to evaluate the accuracy of each empirical model. R^2 is introduced by following expression:

$$R^2 = 1 - \frac{\sum (y_i - \hat{y}_i)^2}{\sum (y_i - \bar{y}_i)^2}$$

where $\bar{y}$ the mean value obtained from available experimental results and $\hat{y}$ is the predicted responsible variable. An empirical model with a R^2 value greater than 0.90 is considered an adequate empirical model.

Also, adjusted coefficient of determination (R^2_{adj}) which is utilized to evaluate experimental models with large number of parameters, expressed as follows:

$$R^2_{adj} = 1 - \frac{n-1}{n-(k+1)}\left(1 - R^2\right)$$

where n is the number of data points and k the number of parameters.

The significance of the individual parameter and associated interactions effects can be measured by testing the hypothesis that the corresponding coefficients in the empirical expressions are zero. Table 5.3 summarizes the analysis of variance and p–value (Prob > F), which represents the probability of falsely rejecting the null hypothesis and is generally used as an indicator for the level of significance. Lower Prob > F values indicate a stronger effect on the response variable with values around 0.1 or less being associated with terms considered to have a significant effect on the response variable.

The linear expressions (Eq. 22, Eq. 23, and Eq. 24) were found to be adequate to describe the relationship between the response variables and the operating variables, as follows:

$$OE = -60.91 + 129.80 * D + 3.64 * A - 3.03 * AD \quad (23)$$

$$Ep = +0.017 - .0055 * D + 0.003 * A - 0.008 * AD \quad (24)$$

$$R_1 = 2.15.11 - 176.95 * \mathrm{D} - 3.59 * \mathrm{A} + 3.18 * \mathrm{AD} \quad (25)$$

where OE is the organic efficiency (%), Ep the probable error, R_1 the low–density bypass (%), A the apex diameter (mm), D the medium relative density.

Table 5.3: Analysis of variance and p–value (Prob > F) for 2–factorial models.

Organic Efficiency Model						
	Sum of		Mean	F	p–value	
Source	Squares	df	Square	Value	Prob > F	
Model	61.76	3	20.59	112.39	< 0.0001	significant
A–S.G.	3.43	1	3.43	18.74	0.0075	
B–Apex	39.96	1	39.96	218.19	< 0.0001	
AB	18.36	1	18.36	100.25	0.0002	
Residual	0.92	5	0.18			
Lack of Fit	0.92	1	0.92			
R–Squared	0.9854		Adj. R–Squared	0.9766		
Probable Error Model						
	Sum of		Mean	F	p–value	
Source	Squares	df	Square	Value	Prob > F	
Model	8.26E–04	3	2.75E–04	8.5	0.0208	significant
A–S.G.	2.42E–04	1	2.42E–04	7.47	0.0411	
B–Apex	7.20E–05	1	7.20E–05	2.22	0.1962	
AB	5.12E–04	1	5.12E–04	15.8	0.0106	
Residual	1.62E–04	5	3.24E–05			
Lack of Fit	1.62E–04	1	1.62E–04			
R–Squared	0.8360		Adj. R–Squared	0.7377		
Low–Density Bypass Model						
	Sum of		Mean	F	p–value	
Source	Squares	df	Square	Value	Prob > F	
Model	209.89	3	69.96	8.6	0.0203	significant
A–S.G.	91.67	1	91.67	11.27	0.0202	
B–Apex	98	1	98	12.05	0.0178	
AB	20.22	1	20.22	2.49	0.1757	
Residual	40.68	5	8.14			
Lack of Fit	40.68	1	40.68			
R–Squared	0.8376		Adj. R–Squared	0.7402		

As shown in Table 5.3, the organic efficiency and low–density bypass models suggested that the effects of both operating parameters and their interaction were highly significant as revealed by the associated p–value smaller than 0.05. On the other hand, the individual apex diameter variable was not significant factor in determining of the response variable of Ep.

Figure 5.7 shows the interactive effect of medium density and apex diameter on organic efficiency as predicted from the empirical models. The contour lines represent the tendency of the organic efficiency with respect to the two operating parameters. The optimum medium density and apex diameter appeared to exist at 1.25RD and 40–mm, for which the maximum organic efficiency value was achieved.

According to Table 5.3, the medium density and apex diameter were significant factors in estimating the organic efficiency of the dense medium cyclone performance using fine silicon suspension as the dense media. As shown in Figure 5.7 at lower medium density values when the medium is not completely stable, the effect of apex diameter played an important role in obtaining an effective separation performance. As a result of apex shrinkage, the actual cut–point increases, thereby more near gravity and low–density particles reported to the overflow stream. It can be concluded that, at lower medium density values, higher organic efficiency achieved when smaller apex (40–mm) was utilized.

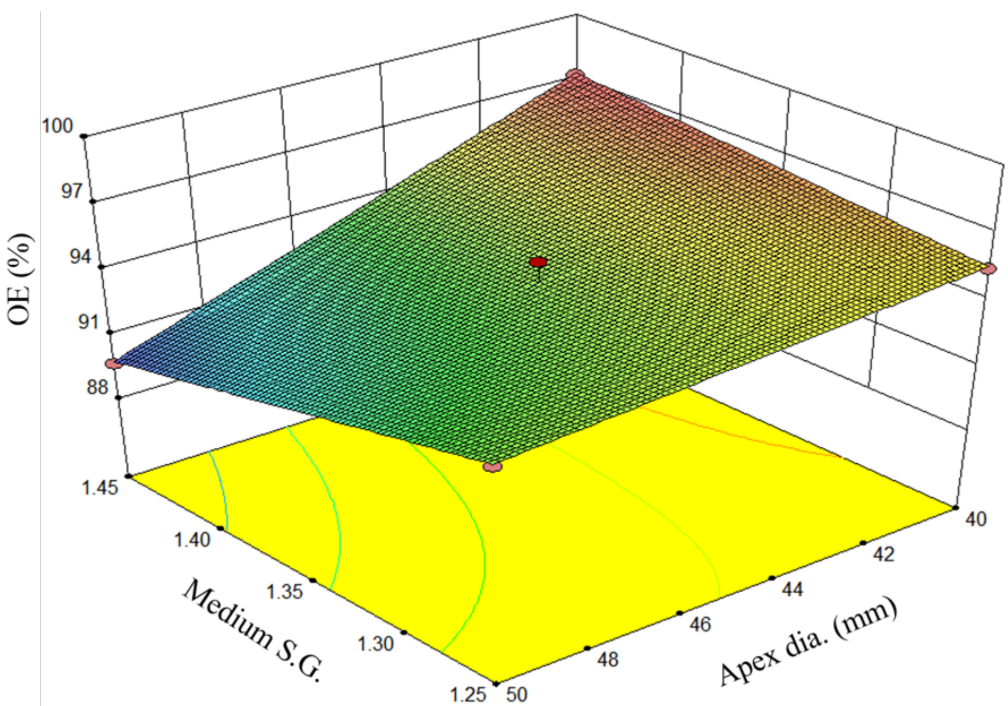

Figure 5.7: Interactive effects of medium density and apex diameter on organic efficiency.

With a raise in medium density, due to an increase in the concentration of irregularly shaped fine particles, as reflected by density differential values in Figure 5.3, medium stability improved. At this point, the apex diameter factor strongly affected the achieved organic efficiency values, by varying the overflow/underflow (O/U) ratio. As apex diameter increased, which led to decrease of the O/U ratio, the population of medium in the overflow stream diminished. As the medium density increased, amount of particles presented in the overflow stream elevated. Therefore, apex enlargement resulted in an inferior separation efficiency, which was reflected with the lower values of organic efficiency (Figure 5.7).

The dense medium separation using the fine silicon suspension as the medium over the targeted specific gravity values (1.25–1.45) provided high separation efficiency, with probable error of below 0.035, when treating 9×1 mm Blue Gem coal. Based on the statistical analysis of Ep empirical model, the apex diameter is an insignificant factor in the estimation of probable error, while the changes in medium density lead the probable error to vary in the range of 0.008 to 0.035. Also, the interaction (AD) effect of apex diameter and the medium density appears to be important in determining probable error. Figure 5.8 shows the interactive effect of medium density and apex diameter on organic efficiency as predicted from the empirical models.

This observation can be explained by the fact that, based on the washability data of the dense medium cyclone feed, very low amount of coal particles have density within 1.45 to 1.65 fraction. The low amount of particles in the intermediate density fraction raises the probability of error in the analysis which is used to generate the partition curves. It can be concluded that organic efficiency model can describe the effects of operating factors on the separation efficiency more significantly.

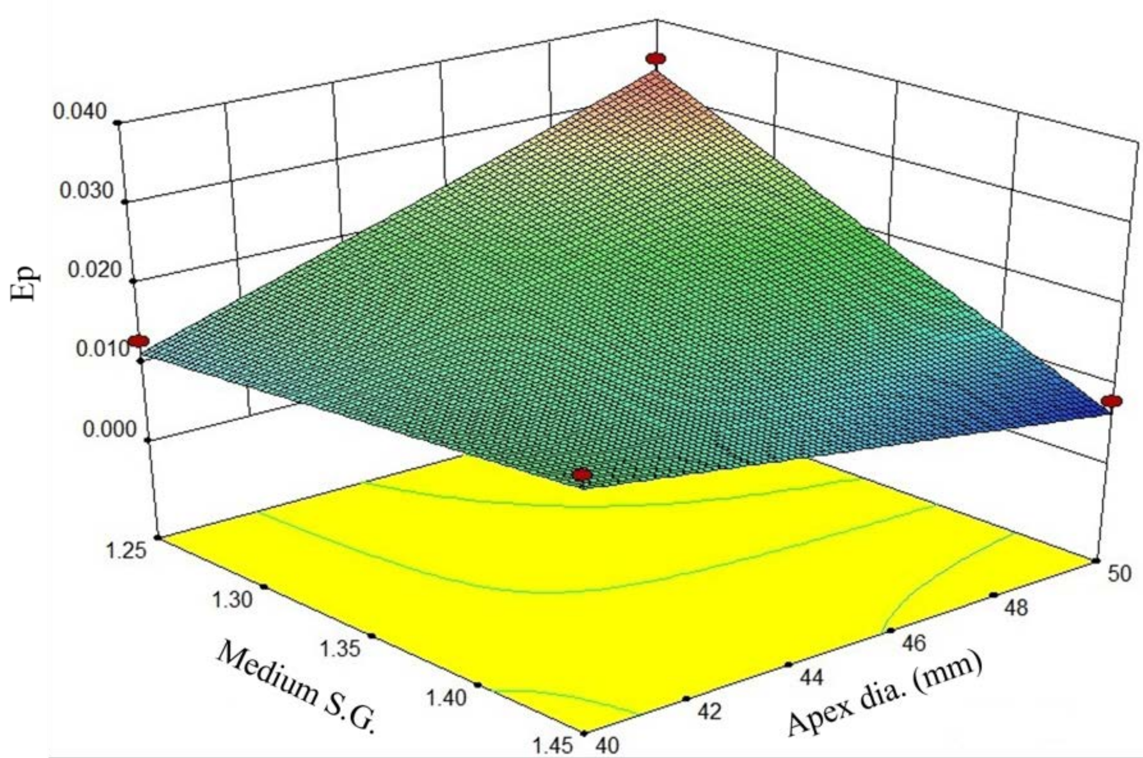

Figure 5.8: Effects of medium density and apex diameter on the probable error value.

Low–density bypass, which indirectly affects the organic efficiency, is one of the main parameters to describe the dense medium cyclone separation efficiency. As illustrated in Table 5.3, the estimation of low–density bypass is considerably affected by both operating parameters (medium density and apex diameter) as well as their interaction variable. With an increase in medium density, the medium stability was improved; thereby lower amount of low–density coal particles reported into the underflow stream. In other words, the elevation of medium density positively had influence on the bypass of low–density particles.

The major causes of low–density bypass include: over loaded vortex finder, apex wear, unstable medium, low–density split to overflow, and/or low medium–to–coal ratio in the overflow stream. As it was previously explained, a raise in the apex diameter significantly affects the density gradients in a cyclone, thereby increased the low–density bypass value. Vortex finder overloading exist when the vortex finder is too small to accept all of the floatable material reporting in the feed stream. Since the vortex finder diameter remained constant during parametric experimental tests, its overloading may not be applied to explain bypass phenomenon.

On the other hand, when a low volumetric amount of medium reports to the overflow stream, as a result of apex enlargement, coarse low–density particles are unable to exit; thus low–density bypass takes place. In addition to medium split factor, medium–to–coal ratio in the overflow stream also considerably controls the value of low–density bypass variable. To minimize the low–density bypass in the dense medium cyclone separation, medium–to–coal ratio in the overflow stream should be maintained above 3:1. With an increase in the apex diameter, medium–to–coal ratio in the product stream decreases thereby significantly increases the amount of floatable materials that reports into the reject stream. As shown in Figure 5.9, when medium is not completely stable, the amount of low–density bypass was strongly affected by apex diameter factor. Also, at higher medium density values, apex diameter shrinkage increase the separation efficiency by balancing the medium–to–coal ratio in the overflow stream.

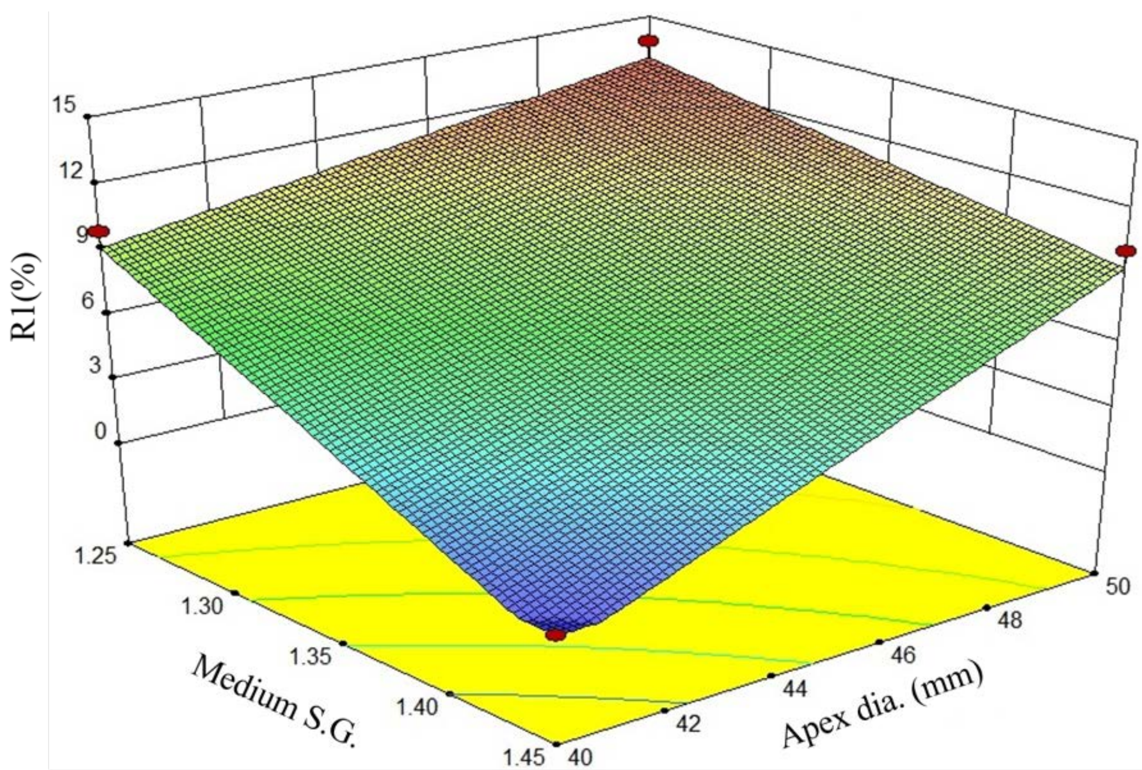

Figure 5.9: Effects of medium density and apex diameter on the low–density bypass value.

In summary, an optimum value of low–density bypass, which equals to zero value, was achievable when a 40–mm diameter apex was used to separate valuable materials from the rock at the medium density of 1.45. Under this conditions, the high separation efficiency, with organic efficiency of 97.19% and probable error of 0.018, was achieved to produce clean coal containing 1.10% ash.

5.3.2 Parametric Study Using Sand and Fume Blends

To assess the potential of the silica–based alternative material application in the dense medium cyclone circuit, a detailed parametric evaluation, using the silica sand and silica fume blend as a dense medium, was carried out. As such, the test program based on a Box–Behnken statistical experiment design involving three individual parameter (i.e., medium density, percentage of silica fume content, and apex diameter) was performed. The test program involved a range of relative medium density values from 1.35 to 1.45, 30% to 50% silica fume content, and three different sizes of apex diameter (including 40, 45, and 50–mm).

A total of 13 tests were conducted and then the obtained results from the washability analysis were used to develop the partition curves (Figure 5.10) and then to determine the separation performance and efficiency achievable when treating 9×1 mm Blue Gem coal using a 15–cm dense medium cyclone. There were three additional trials, in which all factorial levels were set at the middle level. Table 5.4 summarizes the conditions associated with each of the 16 tests conducted, product ash content and the separation efficiency values were obtained for each test.

As illustrated in Table 5.4 , dense medium cyclone tests for the treatment of the Blue Gem coal resulted in production of clean coal containing less than 1.5% ash and organic efficiency values ranging from 1.03% to 99.67%. A wide range of low–density bypass and probable error values were realized, which are indicative of the evaluated parameter value ranges.

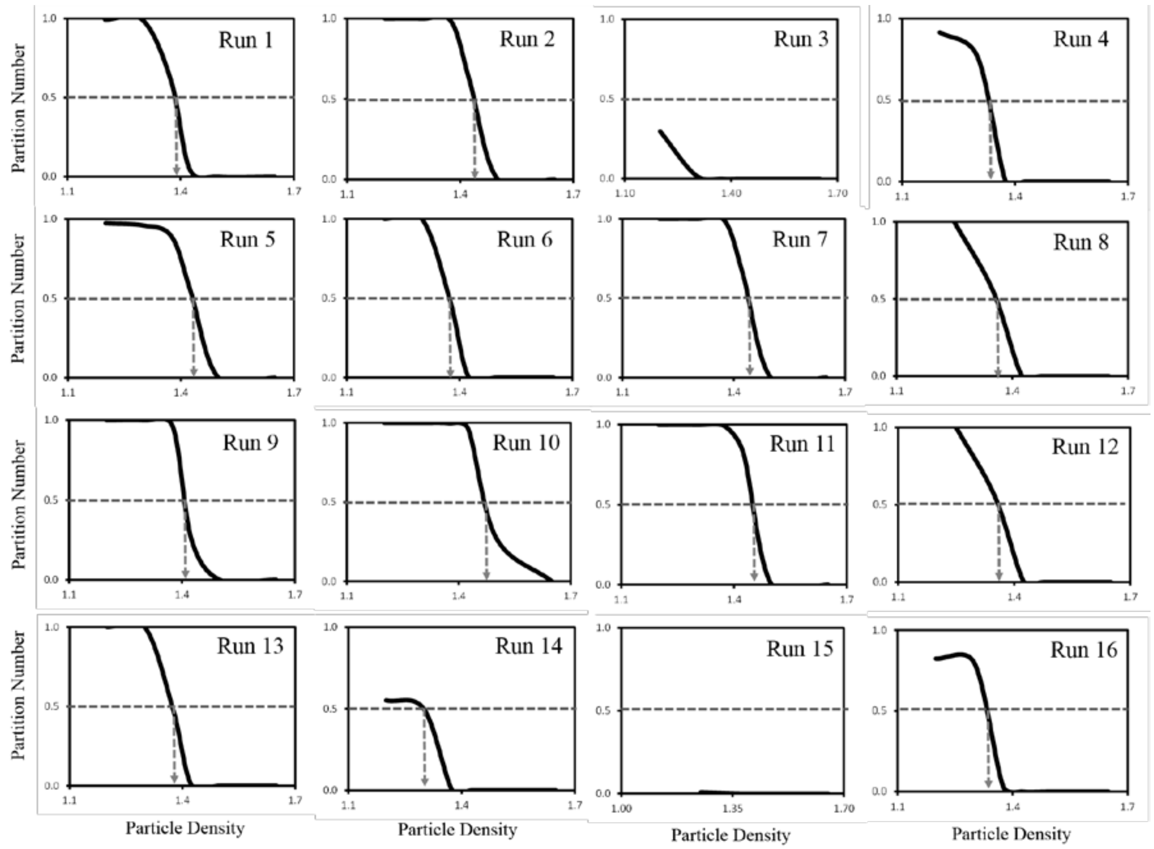

Figure 5.10: The partition curves generated from the separation achieved when using the sand–fume blend over a range of medium density at 9–cyclone diameter inlet pressure.

The performance data presented in Table 5.4 clearly shows the ability of the silica sand and fume blend to produce a high quality coal product containing less than 1.5% ash over a broad range of conditions. The tests involving a 1.35 S.G. medium resulted in a significant amount of 1.30 float material being bypassed to the reject stream. The bypass was especially large when the medium material was comprised of 30% and 40% silica fume. It should be noted that an increase in the silica fume concentration improved the separation efficiency. This outcome is indicative of the medium instability which causes significant gradation of the medium and performance sensitivity to the apex diameter. This is the reason why a smaller apex, at lower medium density values, led to more efficient separation; but as medium relative density and silica fume concentration increased the impact of apex diameter considerably decreased.

Table 5.4: Summary of performances achieved using silica sand and silica fume blend at 9–cyclone diameters inlet pressure.

Trial Order	Parameters			Product Ash (%)	Mass Yield (%)	Combustible Recovery (%)	Response Variable			Cutpoint (S.G.)	Density Offset
	Medium (S.G.)	Silica Fume (%)	Apex diameter (mm)				Ep	OE (%)	R2 (%)		
1	1.40	50	50	1.07	92.25	97.78	0.029	99.08	1.18	1.39	–0.01
2	1.40	50	40	1.18	93.57	99.06	0.025	99.20	0.04	1.44	0.04
3	1.35	40	50	0.86	23.74	25.95	0.090	35.84	70.44	——	——
4	1.35	50	45	0.87	79.56	86.34	0.065	89.55	20.20	1.33	–0.02
5	1.45	50	45	1.09	94.14	96.45	0.030	97.05	2.65	1.44	–0.01
6	1.40	40	45	1.00	90.31	98.19	0.028	99.59	0.10	1.38	–0.02
7	1.45	30	45	1.05	94.77	99.27	0.025	99.13	0.03	1.44	–0.01
8	1.40	40	45	0.97	88.34	97.50	0.040	98.16	0.22	1.36	–0.04
9	1.45	40	50	1.12	96.29	99.43	0.019	98.99	0.08	1.41	–0.04
10	1.40	30	40	1.19	91.53	99.10	0.018	99.67	0.00	1.47	0.07
11	1.45	40	40	1.17	96.30	99.37	0.027	99.36	0.05	1.45	0.00
12	1.40	40	45	0.97	88.12	96.85	0.031	98.02	0.33	1.37	–0.03
13	1.40	40	45	0.99	91.08	98.75	0.035	99.04	0.15	1.36	–0.04
14	1.35	40	40	0.85	47.57	52.59	0.029	80.61	38.58	1.30	–0.05
15	1.35	30	45	0.80	0.67	0.74	3.250	1.03	99.24	——	——
16	1.40	30	50	0.84	73.46	79.82	0.023	95.31	28.15	1.34	–0.06

To describe separation efficiency of dense medium cyclone performance as a function of operating parameter values (i.e., apex diameter, percentage of silica fume content, and medium relative density), empirical models were developed using the results obtained from the parametric evaluation tests programs. The relationship between operating parameters could be represented as a 3–dimentional surface, which allowed the one to study the individual and interactive effects of factors on the response variables.

The significance of the individual parameter and associated interactions effects can be measured by testing the hypothesis that the corresponding coefficients in the empirical expressions are zero. The analysis of variance and f–statistic values, which measured the coefficient of each parameter and interaction term. A prescreening of term was conducted and any parameter with the p–value (Prob > F) greater than 0.1 considered to have an insignificant effect on the response variable and rejected from the model. Consequently, the significant models were achieved by elimination of insignificant interactive parameters in each model.

According to Table 5.5, the individual effect of apex parameter was not significant in determining of the response variable of Ep. Furthermore, the probable error model may not be considered adequate empirical model since the coefficient of determination (R^2) value was 53.24%. The inability of probable empirical model to describe Ep as a function of medium relative density, silica fume concentration, and apex diameter can be explained by the fact that, based on the washability data of the dense medium cyclone feed, very low amount of coal particles have density within 1.45 to 1.65 values. The low amount of particles in the intermediate density fraction raises the probability of error in the analysis which is used to generate the partition curves.

Table 5.5: Analysis of variance and p–value (Prob > F) for Ep model.

Probable Error Model						
Source	Sum of Squares	df	Mean Square	F Value	p–value Prob > F	
Model	5.18	3	1.73	4.93	0.0167	significant
A–S.G.	1.39	1	1.39	3.97	0.0679	
B–Fume (%)	1.25	1	1.25	3.57	0.0812	
AB	2.54	1	2.54	7.26	0.0184	
Residual	4.55	13	0.35			
Lack of Fit	4.55	9	0.51	17625.51	< 0.0001	significant
R–Squared	0.5324		Adj R–Squared	0.4245		

Next, the dense medium cyclone tests results obtained from the Box–Behnken design were utilized to develop an empirical equation to describe organic efficiency parameter as a function of operating parameters. A cubic expression was found to be suitable for the prediction of the response variable. Table 5.6 summarizes the analysis of variance and f–statistics values, which determined the coefficient of each parameter and interaction terms. Any term with p–value smaller than 0.1 will be considered significant factor in determining organic efficiency value and not rejected from the empirical model.

Interestingly, the analysis indicates that relative medium density, percentage of silica fume, apex diameter and their interactive effects are significant parameters for the estimation of organic efficiency values. Considering the complexity of the cubic model, the effects of operating parameters were still difficult to judge directly from the empirical equation. Thus, the relationship between organic efficiency value and different variables were illustrated straightforwardly in a series of 3–D response surface plots.

Table 5.6: Analysis of variance and p–value (Prob > F) for OE model.

Organic Efficiency Model						
Source	Sum of Squares	df	Mean Square	F Value	p–value Prob > F	
Model	11775.78	12	981.32	2399.42	< 0.0001	significant
A–SG.	1676.9	1	1676.9	4100.21	< 0.0001	
B–Fume (%)	2.72	1	2.72	6.66	0.0613	
C–Apex (mm)	5.02	1	5.02	12.27	0.0248	
AB	2052.09	1	2052.09	5017.58	< 0.0001	
AC	492.84	1	492.84	1205.05	< 0.0001	
BC	4.49	1	4.49	10.99	0.0295	
A^2	2263.44	1	2263.44	5534.36	< 0.0001	
B^2	53.68	1	53.68	131.25	0.0003	
C^2	49.81	1	49.81	121.79	0.0004	
A^2B	864.03	1	864.03	2112.65	< 0.0001	
A^2C	206.65	1	206.65	505.29	< 0.0001	
AB^2	70.21	1	70.21	171.67	0.0002	
R–Squared	0.9999		Adj R–Squared	0.9994		

$$\begin{aligned} OE = &-14237.4 + 18740.86 * A + 1827.669 * B - 1669.49 * C \\ &- 2468.02 * AB + 2321.36 * AC + 0.0212 * BC - 5936.2 \\ &* A^2 - 1.695 * B^2 + 0.137C^2 + 831.4A^2B - 831.2 * A^2C \\ &+ 1.185 * A * B^2 \end{aligned} \tag{26}$$

where OE is the organic efficiency (%), A the medium density (S.G.), B the concentration of silica fume (%), C the apex diameter (mm).

According to Figure 5.11, at lower medium density values, in which the medium stability is a dominant factor, poor separation efficiency was achieved due to instability property of the silica sand–fume suspension. At the same ratio of silica fume, by the elevation of medium density, medium stability properties was enhanced which resulted in better separation efficiency. In addition to relative medium density, the percentage of silica fume in the mixture of silica sand and silica fume is a significant factor in determining efficiency of dense medium cyclone performance. As discussed in section 5.2.2, at the same medium specific gravity, increasing the proportion of the ultra–fine silica fume in the dense medium reduced the density differential resulting in a continuous improvement in the medium stability, thereby enhancing efficiency of the process. However, if the silica fume concentration exceeds the specific limitation, detrimental rheological effects may potentially reduce the separation efficiency.

In addition to the individual direct effects, the associated interactive effects of medium specific gravity and proportion of silica fume are important in determining the efficiency of separation performance (Figure 5.11). At lower medium density values, increasing the silica fume concentration in the bimodal suspension of fine sand and silica fume remarkably elevated the medium stability characteristics; thereby continuously improved the separation performance efficiency. This observation can be explained by the fact that, in a dense medium with low specific gravity, which is not very viscous, the effect of medium stability plays an integral role to obtain highly effective separation performance.

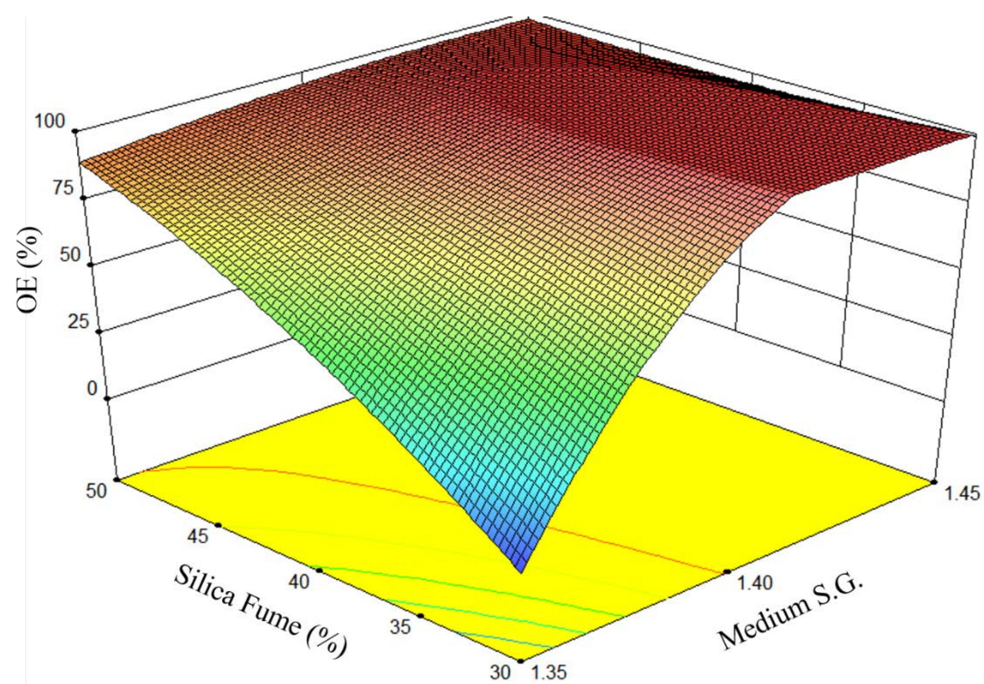

Figure 5.11: Predicted interactive effects of medium density and percentage of silica fume content on organic efficiency.

At intermediate medium density values, when the effects of medium rheological properties moderately were increased, an optimum concentration of micro–silica materials, which provided balanced stability and rheological properties of the dense medium, maximized the organic efficiency. It can be concluded that to obtain an optimal separation performance, the optimization of medium composition is essential in order to achieve a balanced stability and viscosity properties.

As previously explained (see section 5.2.2), the use of 30% silica fume is sufficient to acceptably stabilize the fine silica sand at medium density values above 1.40RD. As shown in Figure 5.11, at higher medium density values fine sand silica–fume medium was completely stable. Therefore, a raise in the silica fume concentration had nearly no influence on the separation performance as reflected by achieved organic efficiency values.

According to Table 5.6, the apex diameter was significant factor in estimating the organic efficiency of the dense medium cyclone performance using fine sand and silica fume blend as the dense media. As shown in Figure 5.12, when dense medium is not completely stable (lower medium density values and lower silica fume concentration), the effect of apex diameter played an important role in obtaining an effective separation performance. As a result of apex shrinkage, the actual cut–point increases, thereby more near gravity and low–density particles reported to the overflow stream; thereby higher organic efficiency achieved when smaller apex was utilized.

With a raise in medium density and silica fume proportion, as reflected by density differential values in Figure 5.5, medium stability considerably improved. At this point, the effect of apex diameter factor in estimating organic efficiency dramatically decreased. As shown in Figure 5.12, at the relative medium density of 1.45, the achieved organic efficiency values were completely independent of apex diameter factor. Similarly, the apex diameter effect was significantly lower when higher proportion of silica fume was used.

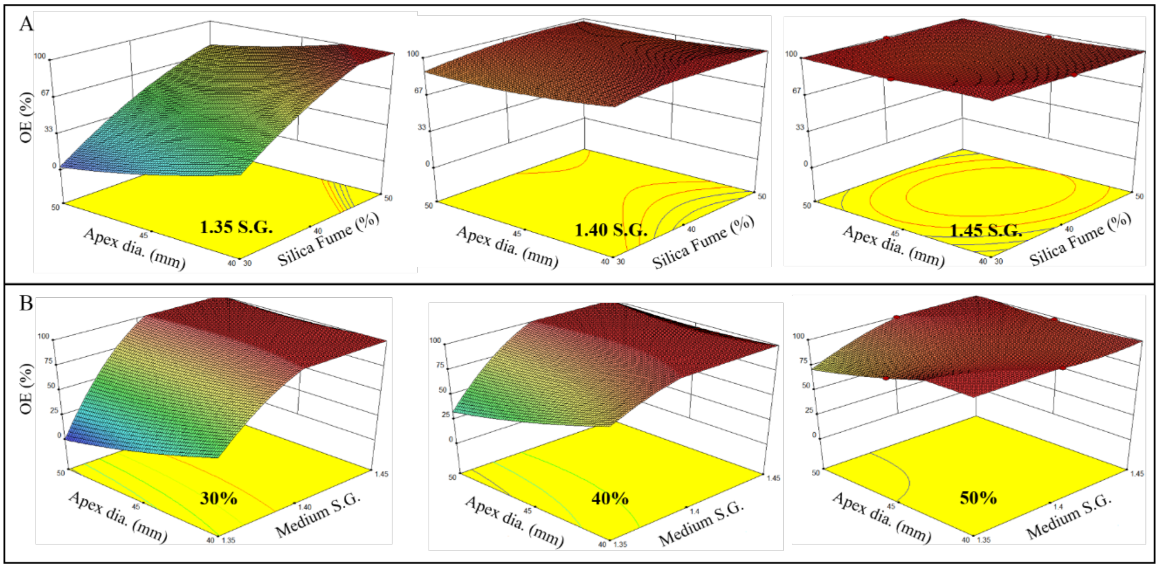

Figure 5.12: Predicted interactive effects of (A) apex diameter and silica fume concentration at different relative medium density values, and (B) apex diameter and relative medium density at different proportion of silica fume on achieved organic efficiency values.

Low–density bypass is one of the main parameters to describe the dense medium cyclone separation efficiency. Based on the p–values for both the individual and interactive terms, all of the operating parameters (medium relative density, silica fume concentration, apex diameter) are significant factors in determining the amount of low–density bypass (Table 5.7). This observation verified that the amount of high value coal particles that report to the reject stream can be minimized by utilizing stable medium and adequate sizes of apex. With an increase in medium relative density or silica fume concentration, the medium stability was improved; thereby lower amount of low–density coal particles reported into the underflow stream. In other words, the elevation of medium density or silica fume proportion positively had influence on the bypass of low–density particles. A quadratic expression was found to be suitable for the prediction of the low–density bypass value. Table 5.7 shows the analysis of variance and f–statistics values, which determined the coefficient of each parameter and interaction terms. Any term with p–value smaller than 0.1 will be considered significant factor in determining low–density bypass value and not rejected from the empirical model.

Table 5.7: Analysis of variance and p–value (Prob > F) bypass model.

Low–denisty Bypass Model						
Source	Sum of Squares	df	Mean Square	F Value	p–value Prob > F	
Model	13023.54	7	1860.51	37.45	< 0.0001	significant
A–SG.	6364.74	1	6364.74	128.13	< 0.0001	
B–Fume (%)	1335.15	1	1335.15	26.88	0.0006	
C–Apex (mm)	467.87	1	467.87	9.42	0.0134	
AB	1667.09	1	1667.09	33.56	0.0003	
AC	253.29	1	253.29	5.1	0.0503	
BC	182.39	1	182.39	3.67	0.0876	
A^2	2753.01	1	2753.01	55.42	< 0.0001	
Residual	447.07	9	49.67			
Lack of Fit	446.93	5	89.39	2575.96	< 0.0001	significant
R–Squared	0.9668		Adj R–Squared	0.9410		

$$R_1 = 20802.54 - 29319.8 * A - 52.377 * B + 51.493 * C + 40.83 * AB - 31.83AC - 0.135 * BC + 10198.17 * A^2 \quad (27)$$

where R_1 is the Low–density bypass (%), A the medium density (S.G.), B the concentration of silica fume (%), C the apex diameter (mm).

As it was previously explained, when dense medium is unstable, a raise in the apex diameter significantly affects the density gradients in a cyclone, thereby increased the low–density bypass value. On the other hand, when a low volumetric amount of medium reports to the overflow stream, coarse low–density particles are unable to exit; thus low–density bypass takes place. In addition, medium–to–coal ratio in the overflow stream also considerably controls the value of low–density bypass variable. To minimize the low–density bypass in the dense medium cyclone separation, medium–to–coal ratio in the overflow stream should be maintained above 3:1. With an increase in the apex diameter, medium–to–coal ratio in the product stream decreases thereby significantly increases the amount of floatable materials that reports into the reject stream.

As shown in Figure 5.13, at lower medium density of 1.35RD when 30% silica fume was used, dense medium was unstable, thereby high amount of low–density particles were misplaced. With an elevation of relative medium density and silica fume concentration, the stability properties of the medium improved which resulted in achieving high separation performance as reflected with lower value of low–density bypass. At higher medium density values (1.45RD), high amount of particles (>90%) reported to the overflow stream; therefore, a considerable decrease in medium–to–coal ratio in the overflow stream resulted in displacement of the low–density materials.

As the medium relative density and silica fume concentration decreased, the medium became unstable, thereby creating density gradients in the cyclone with significantly higher medium densities close to the cyclone walls. This action restricted the movement of the low–density particles to the overflow stream and resulted in higher misplacement of high value coal particles. The existence of density gradients, as a result of medium instability,

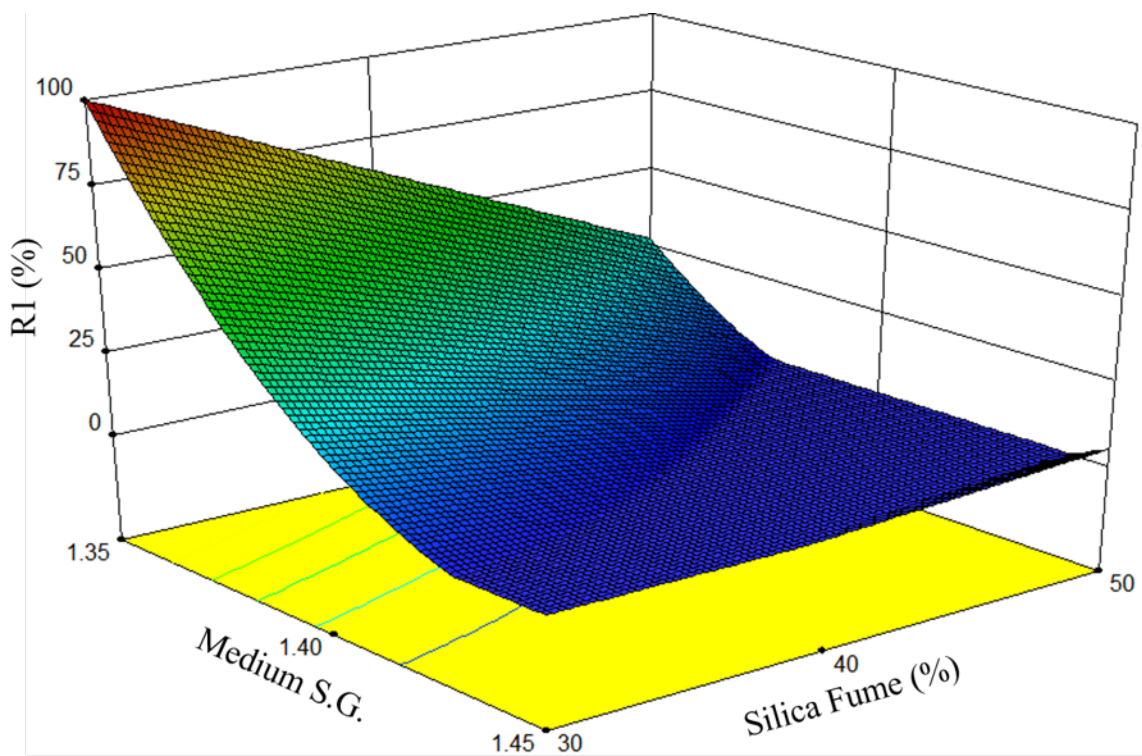

Figure 5.13: Predicted interactive effects of relative medium density and percentage of silica fume content on low density bypass.

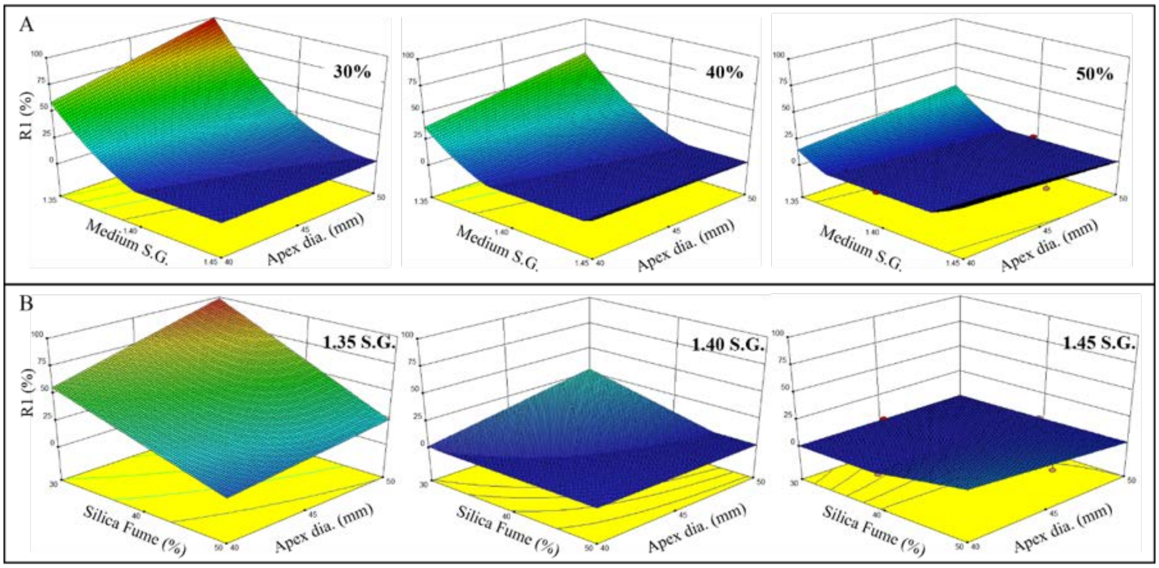

Figure 5.14: Predicted interactive effects of (A) apex diameter and relative medium density at different proportion of silica fume, and (B) apex diameter and silica fume concentration at different relative medium density values on low-density bypass variable.

makes the separation performance sensitive to the apex diameter which is typically not variable in an on–line manner. As revealed in Figure 5.14, at lower medium density values or silica fume concentration, shrinkage of the apex changed the position of vertical velocity border in the cyclone and increased the probability of low–density particles carried into the product stream. As medium density increased the gradation of dense medium in the cyclone was decreased and medium became more stabilized. Therefore, the effect of apex diameter in determining low–density bypass became negligible.

5.4 Dense Medium Cyclone Circuit

An efficient dense medium operation, to economically produce a saleable product at high recovery value requires the dense media with desirable properties as follows: inexpensive, capable of adjustment over a wide range of medium density values, easily separated from both products after processing, readily recoverable to reuse, stable over the required range of densities, and low in apparent viscosity.

The silica–based alternative materials, which are inexpensive and available sources, were stable over the wide range of relative medium density values. As discussed in the previous sections, a clean coal product with less than 1.5% ash can be produced using a medium formed from the silicon production waste with an organic efficiency value of above 95% and probable error value below 0.02. Therefore, these alternative materials can be utilized to replace magnetite in the dense medium cyclone separation. However, a typical dense medium cyclone circuit need to be modified to be able to recover and reuse dense medium in the coal preparation plant.

A modified dense medium cyclone circuit that incorporated the use of the silica–based materials illustrated in Figure 5.15. Raw coal feed is passed over a desliming screen with 1–mm aperture size to remove the fine and ultra–fine particles. The oversize materials are mixed with the dense media in the cyclone feed sump and then the mixture is pumped directly to the cyclone. After the separation of high and low–density particles in the cyclone, the clean coal and rock are passed over a drain and rinse screen, and then the final product is sent for dewatering stage. Undulated (corrected) dense medium, which is drained from the initial part of the product and reject screens, is directly sent to the correct medium sump and then is pumped to main cyclone feed sump. The diluted dense medium, which has a lower medium specific gravity flows to the dilute medium sump and is pumped to classifying cyclone. The underflow material from the classifying cyclone, which has a high solid concentration, gravitates to over–dense medium sump, while the overflow material, with low solid concentration, is sent to thickener. In the thickener, high density ultrafine particle are settled and reports to the over–dense medium sump.

A number of modifications were made to the new dense medium cyclone utilizing silica–based materials as the dense medium. The most significant of these was to eliminate magnetic separator which results in considerable reduction of operating costs in coal preparation plant. Additional classifying cyclone and thickener needs to be installed for solid–liquid separation and recovering dense particles to be reused as the dense medium.

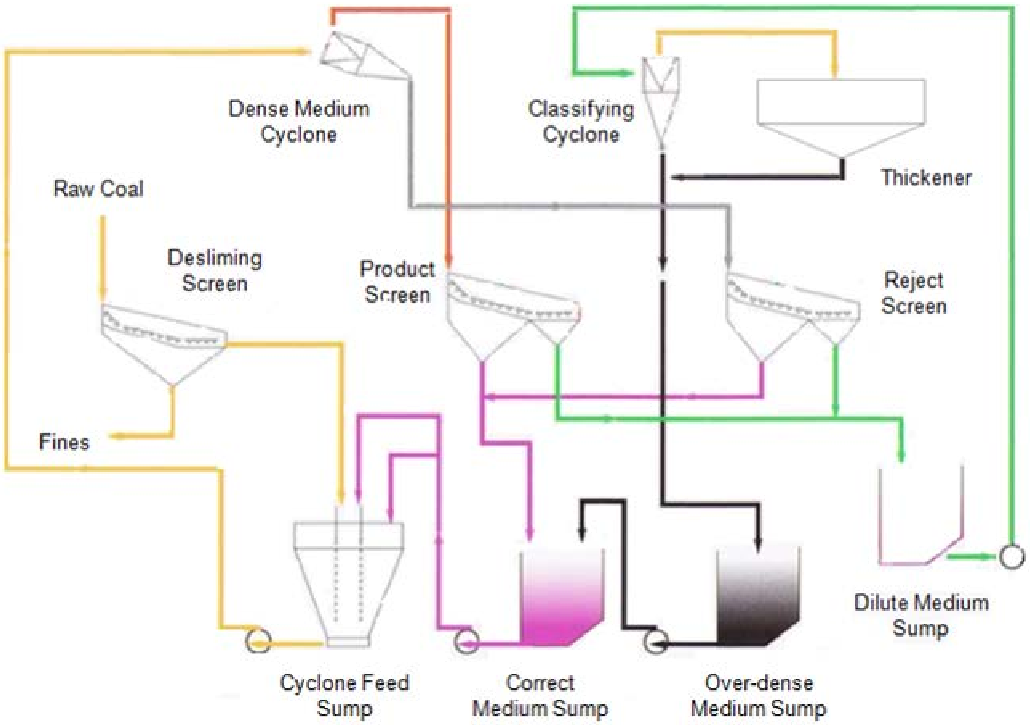

Figure 5.15: A modified dense medium cyclone circuit in the coal preparation plant that produces ultra-clean coal for the silicon market.

6 CONCLUSIONS AND RECOMMENDATIONS

6.1 Summary and Conclusions

Metallurgical grade silicon, defined as at least 96% silicon content, is an important raw material for various markets and applications, such as chemical industries, steel, copper and aluminum markets, and electronic industries. The relatively small portion (<10%) of very highly purified silicon is utilized in the electronic industries as a semiconductor. Due to very low amount of contamination requirements, use of silicon in electronic industries may be the most challenging application.

The presence of the metal contaminations in silicon product results in poor performances and efficiency degradation in devices and materials with basis of silicon. Iron is one of the most abundant elements in nature and is difficult to entirely remove from a silicon production line. Consequently, the amount of accidental iron contamination in the final silicon product is higher than of other metallic impurities. Due to its electrical activity, iron has always been considered the main impurity in silicon used in semiconductor industries. The critical level of iron impurity concentration in semiconductor industries is decreasing annually as sensitivity of devices yield to iron impurity is increasing due to the device dimension shrinkage.

Coal preparation technology plays a crucial role in the silicon market by upgrading run–of–mine (ROM) coal, which contains undesirable impurities, to satisfy the size and quality specifications. Modern coal preparation plants consist of a complicated arrangement of solid–solid and solid–liquid separation processes with objective of improvement in the purity of coal by reducing detrimental impurities; and the control of final product lump size to meet the designated product requirement.

Dense medium cyclone is one of the most efficient concentrators for cleaning ROM coal with particles coarser than 1–mm. The DMCs utilize a dense medium consisting of a suspension of ultra–fine magnetite to produce low ash clean coal which is applied commercially in silicon smelting process. The magnetite that remains on the surface of the clean coal after cleaning in a dense medium process is a main contamination in silicon production that considerably decreases the purity of final silicon product.

Given that silicates are the main raw materials in silicon production, silica–based materials were evaluated as a replacement for magnetite in the establishment of a dense medium. Fine silicon and ultra–fine silica fume (micro–silica) was obtained from the silicon production process while fine silica sand from an outside commercial source was purchased for evaluation.

The test program began with an analysis of the static and dynamic medium stability that the alternative materials provided over a range of medium density values. The target medium density values were between 1.20 and 1.55 based on the current operating practice of the coal preparation plant that generates the clean coal for silicon production. The fine silicon material generated a stable medium over a range of inlet pressures (3 to 7 psi) and the target medium relative density values between 1.20 and 1.45. The medium formed by the silica sand material was completely unstable over a wide range of medium density and feed inlet pressure while the ultra–fine silica fume material by itself creates a stable medium within target range but medium is very viscous and would create separation efficiency issues. To improve the stability of silica sand suspension, different ratios of silica fume was blended with the sand to generate dense mediums with bimodal composition.

Based on the results of the medium stability study, the factors controlling the stability properties of dense medium can be summarized as following:

1. Particle size distribution: the stability of dense medium using ultra–fine silica fume was higher than fine silicon medium. On the other hand, the silica sand suspension was completely unstable at the medium density fraction between 1.20 and 1.50. In a word, finer particles provide more stable medium.
2. Particle shape: in the fine silicon medium, high population of irregularly shaped particles caused the elevation of turbulent drag forces, thereby achieving highly stable medium even at relatively low specific gravities. On the contrary, the

instability property of silica sand medium was corresponded to the high presence of relatively spherical particles, with the fast–settling rate, in the suspension. It can be concluded that spherical or smooth particles produce lower stability values compared to angular or rough shape particles.

3. Medium density: As the medium relative density increased volumetric solid concentration raised, which hindered particle settling rate thereby providing a medium with higher stability values.
4. Solid density: to achieve a given medium density, lower degrees of solid concentration were required when higher density particles were used to generate a dense medium.
5. Medium composition: The bimodal dense medium of coarse and fine narrow size fractions may provide high medium stability properties. Dense medium formed by the blend of sand and fume became more stabilized with the elevation of ultra–fine silica fume particles in the suspension.

Using the fine silicon medium, a detailed experimental program was conducted to determine the separation performance and efficiency achievable when treating 9×1 mm Blue Gem coal using a dense medium cyclone. The test program involved a range of medium densities from 1.25 to 1.45 and different sizes of apex diameter (40, 45, and 50 mm). The parametric test results revealed that the production of clean coal containing around 1.00% was possible over a range of selected medium density. Moreover, the maximum organic efficiency of 99% and the minimum probable error value of 0.005 were achievable. There was no measurable bypass of high density particles to the product stream or low–density particles in the reject stream. The results of the dense medium cyclone performance, which were obtained from the statistically designed test program, based on the 2–Factorial model, were used to develop empirical model that characterize the overall process. The empirical model describes the response variables (i.e. organic efficiency, probable error, and low–density bypass) as a function of operating parameter values (i.e., apex diameter and medium relative density).

Utilizing the optimum blend of fine silica sand and ultra–fine silica fume, the test program based on a Box–Behnken statistical experiment design involving three individual parameters (i.e., medium density, percentage of silica fume content, and apex diameter) was performed. Dense medium cyclone tests for the treatment of the Blue Gem coal resulted in production of clean coal containing less than 1.5% ash with organic efficiency values ranging from 1.03% to 99.67%; Also a wide range of low–density bypass (between zero and 99.24%) and probable error (from 0.018 to 3.25) values were realized.

The conclusions derived from the parametric studies are:

1. In the DMC separation using silica–based dense medium, the separation efficiency was closely related to the medium density. At lower medium density values the effect of medium stability played a major role. With a fine silicon dense medium, high separation efficiency obtained since the medium was relatively stable; while poor separation efficiency was achieved due to instability property of the silica sand–fume suspension at low medium density fractions. By the elevation of medium density, medium stability properties was enhanced which resulted in better separation efficiency.
2. The apex diameter strongly affects the achieved separation efficiency and low–density bypass values when medium is unstable. At lower medium density values in the presence of low concentration of silica fume, the apex enlargement resulted in separation efficiency assessed. When a low volumetric amount of medium reports to the overflow stream, as a result of apex enlargement, coarse low–density particles are unable to exit; thus low–density bypass takes place. Moreover, with an increase in the apex diameter, medium–to–coal ratio in the product stream decreases thereby significantly increases the amount of floatable materials that reports into the reject stream.
3. The achieved separation efficiency with bimodal silica–based suspensions is strongly affected by the concentration of ultra–fine particles in dense medium. Increasing the proportion of the ultra–fine silica fume in the dense medium resulted in a continuous improvement in the medium stability, thereby enhancing efficiency

of the process. However, if the silica fume concentration exceed the specific limitation, detrimental rheological effects may dramatically reduce the separation efficiency.

6.2 Recommendations for Future Works

The author of this thesis recommends the following items for continued study:

1- Investigate other sources of silica–based materials. The main objective of this investigation was to evaluate the potential of utilizing silica–based materials in dense medium processes. The alternative materials included fine silicon, silica sand and silica fume. Considering availability, cost, stability and rheological properties of the dense media, other sources of silica–based materials, such as ultra–fine silica sand and ultra–fine waste materials in the thickener underflow stream, is recommended.

2- Determination of the DMC separation performance as a function of dense medium rheological properties. The efficiency of dense medium cyclone separation as a function of stability properties of the silica–based mediums was quantified. Further study to determine the Casson Yield Stress and Casson Viscosity values may produce more logical explanation for the variation in the efficiency of dense medium processes.

3- Medium recovery circuit design. The initial design of the dense medium cyclone circuit was presented. More detailed study, which considers not only the performance efficiency but also economic aspect of design is recommended. Evaluation of dense medium cyclone circuit can be based on the operating cost of dense medium cyclone circuit using magnetite as a dense media.

4- Further studies for ultra–clean coal production from the fine and ultra–fine circuits. Although the new technologies in fine and ultra–fine coal cleaning processes may be capable of producing a high quality coal, the final coal product below 1 mm is not saleable to the silicon market. Conducting research for the Coagulation and flocculation of fine and ultra–fine clean coal particles is strongly recommended.

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