EFFECT OF THE TIME TAKEN ON THE PERFORMANCE OF FABRICATED GAS–SOLID FLUIDIZED BED COLUMN FOR THE SEPARATION OF RARE EARTH ELEMENTS

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Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering and Natural Resources

Faculty of Chemical & Natural Resources Engineering
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JULY 2014

SUPERVISOR’S DECLARATION

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I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Specially dedicated to my beloved family, and those who have guided and inspired me throughout of learning

ACKNOWLEDGEMENT

Alhamdulillah, thank to Allah the almighty for his blessing to complete the project by given duration.

I am grateful and would like to extend the heartfelt gratitude to my supervisor Dr. Wan Hanisah bt Wan Ibrahim and also to my co-supervisor Dr. Anwaruddin Bin Hisyam for their germinal ideas, invaluable guidance, continuous encouragement and constant support in making this research successfully. They inspired me greatly to work in this project. Their willingness to motivate me contributed tremendously to my project.

My appreciation also goes to my Colleagues University Malaysia Pahang and all FKKSA laboratory industry for their advice and sometimes helped me with the problems that occurred in laboratory during the project.

ABSTRACT

A gas–solid fluidized bed separator used for the separation of rare earth elements (REEs) from printed circuit board was design and fabricated. In this work, the effect of time taken on the performance of the fabricated gas-solid fluidized bed column for the separation of rare earth elements was study. Waste from electronic devices was grinded to powder forms with the particles sized less than 1.0 µm were used in this study. Beds column with different heights of sampling port were analyzed at 10 cm, 20 cm, and 30 cm while the air pressure was 15 psi, 20 psi and 25 psi were tried in this investigation. The rare earth elements were subsequently analyzed by float–sink testing. The results showed the weight percent of the fine powder increases with increasing the time taken for collecting the sample. However, the separation density decreased with increasing the sampling port height of the column.

Key words: Gas- solid fluidized bed column, Rare Earth Elements (REEs), Printed Circuit Board (PCB), Separation, Recycle.

ABSTRAK

Pemisah fluidized gas- pepejal digunakan untuk pemisahan unsur-unsur nadir bumi (nadir) dari papan litar bercetak adalah reka bentuk dan dibina. Dalam karya ini , kesan masa yang diambil terhadap prestasi ruangan katil direka gas- pepejal fluidized untuk pemisahan unsur-unsur nadir bumi adalah kajian . Sisa dari peranti elektronik telah dikisar kepada bentuk serbuk dengan zarah bersaiz kurang daripada 1.0 µm telah digunakan dalam kajian ini. Katil tiang dengan ketinggian yang berbeza pelabuhan pensampelan telah dianalisis pada 10 cm, 20 cm, dan 30 cm manakala tekanan udara adalah 15 psi, 20 dan 25 psi psi orang dibicarakan dalam penyiasatan ini. Elemen-elemen nadir bumi kemudiannya dianalisis dengan ujian apungan - tenggelam. Hasil kajian menunjukkan peratus berat serbuk bertambah baik dengan meningkatkan masa yang diambil untuk mengumpul sampel. Walaubagaimanapun , ketumpatan pemisahan menurun dengan peningkatan ketinggian pelabuhan pensampelan tiang.

Kata kunci: Gas pepejal ruangan cecair, Elemen-elemen bumi(nadir bumi), Litar Bercetak Lembaga (PCB), Perpisahan, Kitar semula

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LIST OF ABBREVIATIONS

Ce	*Cerium*
°C	*Degree Celsius*
Dy	*Dysprosium*
e.g.	*exempli gratia = for example*
Er	*Erbium*
etc	*et cetera*
Eu	*Europium*
Gd	*Gadolinium*
HCl	*Hydrochloric acid*
HREE	*Heavy Rare Earth Element*
HTS	*High temperature superconductor*
Ho	*Holmium*
ICP-MS	*The Interdisciplinary Center for Plasma Mass Spectrometry*
K	*Potassium*
kW	*Kilowatt*
La	*Lanthanum*
LAP	*Lanthanum phosphate*
LCD	*Liquid crystal display*
LED	*Light Emitting Diode*
LREE	*Light Rare Earth Element*
Lu	*Lutetium*
Mg	*Milligram*
m	*Meter*
Nd	*Neodymium*
NH_3	*Ammonia*
Ni	*Nickel*
Pr	*Praseodymium*
Pm	*Promethium*
R&D	*Research and development*
Ra	*Radium*

REE	*Rare earth element*
Sc	*Scandium*
Sm	*Samatium*
Tb	*Terbium*
Tm	*Thulium*
U_3O_8	*Uranium (V, VI) oxide*
TV	*Television*
U	*Uranium*
WEEE	*Waste Electrical and Electronic Equipment*
Y	*Yttrium*
Yb	*Ytterbium*

1 INTRODUCTION

Rare earth elements (REEs) are the 17 elements in the periodic table, 15 surrounded by the chemical group called lanthanides including yttrium and scandium. The lanthanides consist of the following elements: lanthanum, cerium, praseodymium, neodymium, promethium, samarium, europium, gadolinium, terbium, dysprosium, holmium, erbium, thulium, ytterbium, and lutetium as shown in Table 1.1. Rare earths are abounding in the earth's crust and some even more lavish than copper, lead, gold, platinum, and many other minerals. However, REEs are not concentrated enough to make them easily exploitable economically (Xiaoyue *et al.*, 2011).

Humphries (2012) stated that the REEs are typically split into two sub-group, the sub group of light rare earth elements (LREEs)—lanthanum through europium (atomic numbers 57- 63) and the heavier rare earth elements (HREEs)—gadolinium through lutetium (atomic numbers 64-71). Yttrium is also classified as a heavy element.

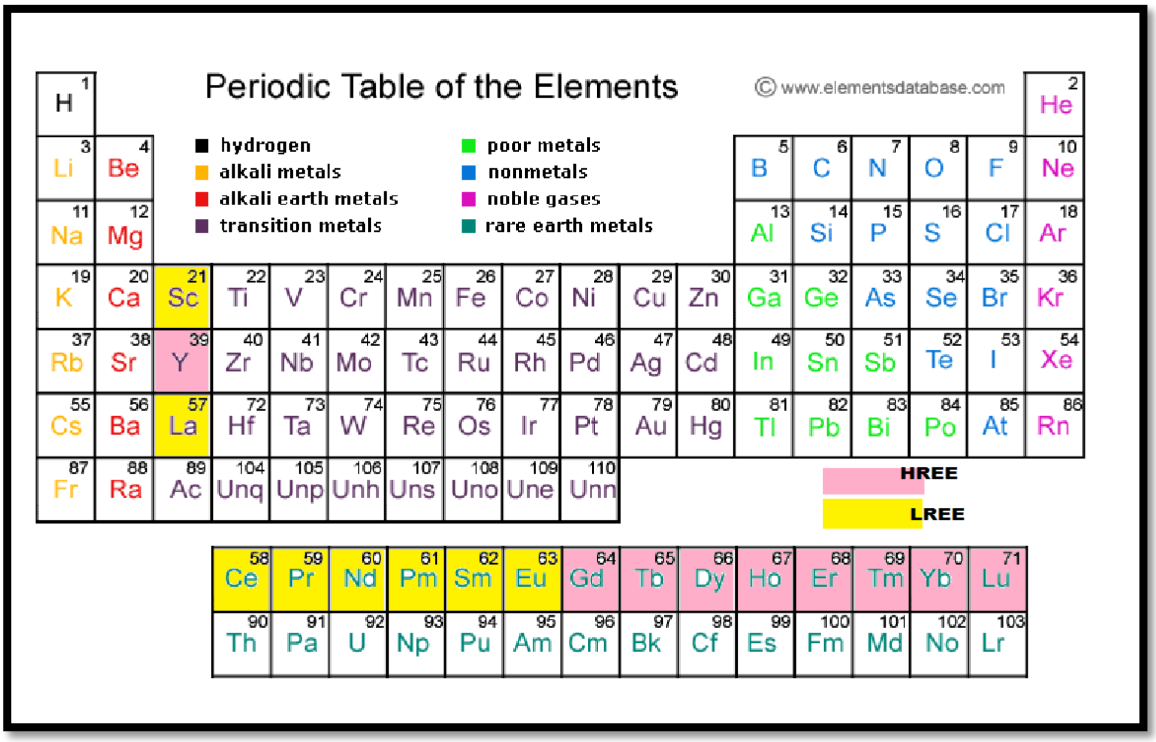


Figure 1.1: Periodic table of rare earth elements

1.1 Motivation and statement of problem

The raw material REEs is become more critical issues due to political tensions or shortages. In fact, REEs is really demanding for many important developing technologies regional industries. The rare earth group raw material as critical when after having carefully evaluated the political and economic situation of the producing countries, the level of supply concentration, the potential for substitution and the recycling rate is not enough. The situation it gives big impacts on the economy of each country due to the high dependency rate of REEs (Massari & Ruberti., 2012). Therefore, to defeat this issue, the recycle is the best ways because of the rapid development of electronic and information technologies has resulted in the arrival of low cost and enhanced the electronic products in the market resulting in the generation of the huge quantities of end-of-life electrical and electronic equipments (EOL-EEEs) due to their replacement (Lee *et al*., 2007).

Chen, (2011) highlighted that the most critical situation seems of rare earth groups to be due to the high import dependency rate, low substitution and low recycling rate. In fact, although their industrial demand is relatively small scale, they are noteworthy for many expanding high technology applications. Currently the global exploitation of rare earth elements has registered a steady and steeply increasing, but the supply of REEs has drastically diminished or decline.

The substitution for limited REE has shown that there are quite uncommon REE compounds by simple substitution of other compounds. In most cases replacements requires a completely new product design. The identified options for replacement in the case of major green applications are summarized below (Doris *et al*., 2011):

- Rare earth is currently used in approximately 14% of newly installed wind turbines with less gear design and technical advantages in terms of reliability. A shortage supply of rare earth will lead to a shift to alternative types of turbines. Further research on higher accuracy of traditional techniques with gear will support this substitution.

- Rare earth is commonly used in hybrid electric vehicles and also electric vehicles. Substitutions based on other electric motor design are predominantly available. However, R & D is needed for higher achievement of different types of existing electric motors and for the awareness of new motor concepts.
- Majority the new energy efficient lighting is containing rare earth element (compact fluorescent lamps, LED, plasma displays, LCD displays).The substitution is limited, especially for compact fluorescent lamps. R & D is required for alternative phosphors with high efficiency and high quality of light.
- Automotive catalysts contain cerium while catalyst for petroleum cracking and other industrial processes contain lanthanum. The replacements or substitution are really occasional, and R & D is needed for alternative catalysts.

REE distribute a lot of beneficial performance that can be irreplaceable by other material. They are becoming an important in the transition to a green and low-carbon economy. The increasing popularity of hybrid and electric cars, wind turbines and compact fluorescent lamps is causing an increase in the demand and price of REEs hence sparked the global competition for these resources (Binnemans *et al*., 2013). Recovery of rare earth element from liquid as well as solid waste is desirable due the lack or limited resources of rare earth elements. Instead of using pure raw material of rare earth elements we can recycle from the electronic waste.

Usually, REE can be found in electronic waste such as battery, chipboard, computer set, refrigerator and etc. Conventionally, this waste can be separated by using varies of method such as extraction, separation and purification. Therefore, it can illustrate the current situation of international markets, the availability of these strategic resources, the critical points of their supply, the possibility for substitution and recycling, and the environmental problems related to their extraction and the possible solutions to enhance their future supply security for the European countries.

1.2 Objectives

The aims of this work is to study the effect of the time taken on the performance of fabricated gas–solid fluidized bed column for the separation of rare earth elements.

1.3 Scope of this research

Separation of rare earth elements has been conducted by using fabricated fluidized bed column. The sample used is chipboard from electronic waste. The sample firstly grinded by physically crush using a crusher until it becomes to fine powder. The concentration of the powder is analyzed by using Sequential X-ray Fluorescence Spectrometer (Model No.XRF1700, Shimadzu, Japan) (XRF). Once the element concentration is known, the sample was injected into the fluidized bed column. The sample distributed according to their properties and density. This process separated the particles of sample which was collected at different sampling ports of the column. After collecting the sample at different sampling port, each sample will be analyzed for rare earth element concentration by using inductively coupled plasma-mass spectrometry. Consequently, a comprehensive kinetic study is on the effect of the time taken on the performance of fabricated gas–solid fluidized bed column for the separation of rare earth elements by varying the pressure of air flow rate and height of sampling port.

1.4 The recycling of rare earth elements

According to the research made by Doris *et al*., (2007) the recycling of rare earth elements could be stated as a very phenomenal issue until today. The recycling of rare earth metals has been undertaken most notably in Japan and there are number of extraction process but none of them has been developed commercially due to drawbacks on yield and cost. Principally, the recycling processes for the rare earth are quite complex and extensive if reuse is not possible and a physical and chemical treatment is necessary. Most of the recycling procedures are energy-intensive process. The recycling of rare earth from motors, hard disc and other electronic components which is the main post-consumer activities will require intensive dismantling.

Moreover, the potential shortages and the steep increase in price of rare earth are providing for the first time the opportunity to address the problem of today's rare earth supply in more depth and to seriously build up a recycling economy. The advantage of rare earth recycling includes the exploitation European resources, independence from foreign resources and also for environmental benefits also (Doris *et al*., 2011).

The recycling of rare earth has some advantages compared to the used of primary resource (Doris *et al*., 2011):

- The processing of secondary rare earth elements are free from radioactive impurities, the mining and further processing of primary rare earth is correspond with nuclear radiation coming from radioactive elements of natural deposits are commonly produces radioactive waste.
- The recycling requires some energy carriers and chemical but at the same time it saves significant amount of energy, chemical and emissions in primary processing chain. It is expected that most recycling will have a lot of benefit which is concerning air emissions, groundwater protection, acidification, eutrophication and climate protection.
- Dependence on foreign resources will be decreasing by supplying the European market with secondary rare earth materials.
- Europe is one of the globally large consumers of rare earths. Increasing amounts of waste from final products containing rare earths are arising in Europe.
- Apart from a few specialised industries and applications, the know-how in rare earth processing is quite low in Europe. The building up of know-how in recycling will widen the competency of enterprises and scientific institutions in Europe concerning rare earth processing.

1.5 Organisation of this thesis

This thesis will be divided into six chapters. First chapter is the introduction to the thesis and briefing about the project's idea such as the information about the rare earth elements and its advantages. It also included the application in various industries. There will also include problem statement, objectives and the scopes of the project.

Chapter 2 is for literature review which provides a description of the applications and general design feature in recycling of rare earth elements. A general review on the characteristics of the separation by using fluidized bed column processes for recycling rare earth elements. This chapter also provides a brief discussion of the advanced experimental techniques of recovery of rare earth elements. A summary of the previous experimental work on separation technique and its application also presented.

Chapter 3 gives a review of the fluidized bed column approach applied for the separation of the rare earth elements. This chapter is all about the method and procedures used in the experiment process. This chapter can be divided into five partitions which is design of fabricated fluidized bed column, The Interdisciplinary Centre for Plasma Mass Spectrometry (ICP-MS), sample preparation, parameter of study and the consumable procurements. The detail of the procedure was discussed in this chapter.

Chapter 4 is for the design of cylindrical fluidized bed column. The detailed explanation and characteristic is briefly described the fluidized bed column in this chapter. This will helps readers to understand the fluidized bed column thoroughly and completely.

Chapter 5 is for result and discussion of the data collection and analysis the results of the experiments. The collected data will be discussed and the results will be shown in the table. This will helps readers to understand the result thoroughly and completely. This chapter also included the discussion of the result that obtains from the experiments and factors that affecting the result.

Chapter 6 draws together conclusion and recommendation of this study. Hence, a summary of the thesis and outlines the future work which might be derived from the model developed in this work.

2 LITERATURE REVIEW

2.1 Overview

This research presents the experimental studies of gas-solid fluidized bed column which is dry separation technique in order to separate particles of rare earth elements. Research on recovery of rare earth elements is quite rare because the recycling processes for the rare earth elements are quite complex and extensive if reuse is not possible and physical and chemical treatment is necessary. However, potential supply shortage and the steep increase in price of rare earth are the opportunity to address problem of today's rare earth supply in more depth and jump to seriously build up recycling economy.

2.2 Introduction

The concentration of rare earth elements (REEs) production in China raises the notable issue of supply vulnerability. REEs are used for many commercial applications including new energy technologies, electronic devices, automobiles, and national security applications. The examination of REEs for new energy technologies reveals a concentrated and complex global supply chain and numerous end-use applications. Therefore, placing the REE supply chain in the global context is unavoidable and becoming important resources (Humphries, 2012). In order to alter this issue, the rare earths elements can be recovered by recycle the waste from electrical and electronic equipment (WEEE).

The separation process of mineral particles using fluidization technique is described in this study. When a mixture of particles with sufficiently different physical properties is articulately fluidized, under some operating conditions the mixture separates and displays a behaviour called layer inversion (Rasul *et al*., 2000). This study supports: (1) a comprehensive criterion for particles of any type (size and density variant, only size variant or only density variant) to separate or mix and invert; and (2) mixing or separation regime map in terms of size ratio and density ratio of the particles for a given fluidizing. Therefore, knowing the physical properties of mineral particles, appropriate fluidization conditions can be chosen in order to separate them. Separations which use differences in specific gravity are one of the most commonly used, because of their effectiveness, low cost and operational simplicity. Recovery of rare earth material from electronic waste device provides multipurpose target which is control the hazardous materials, protect the environment against pollution, conserve the mineral resources and recover pure chemical compounds needed in the market. Moreover, the applied method is environmentally friendly with no hazardous by products and the cost of recovered products is competitive with the market price for the same chemicals prepared from primary resources.

2.3 *Previous work on recovery of rare earth elements*

The technology development of the last decades in the various sectors of the electronic industry has stimulated the replacement of obsolete gadgets. As a result, there is a growing disposal of obsolete computers and other electronic equipments into landfill sites throughout the world. To overcome this problem, the recovery of rare earth elements is one of the alternative ways.

According to the journal studied by Morais & Resende (2010) was studied recycle the rare earth elements from the tubes of colour TV set and computer monitor. Tubes of colour TV set and computer monitor present as coating, a powder containing a mixture of oxides and sulphides containing RE, mainly europium and yttrium. Despite the low amount recovered, the high commercial value europium makes it worthwhile. Therefore, study on the recycling of rare earth elements from the electronic waste device is valuable and desirable because it is currently incredibly commercial resource. The recovery of REE from electronic scraps and other metals is extremely important as the economic and environmental issues are concerned and also it leads to the recovery of other metals, e.g. lead, zinc, strontium, zirconium and indium, which are also present in computer monitor and TV screen coating powder.

Kumar V. *et al* (2013) was recommended to do research on physical separation process for eco-friendly recycling of rare and valuable metals from end-of-life DVD-PCBs because nowadays the end-of-life of printed circuit board was abundantly in any citizen. Even so, the PCBs made of from a lot of valuable elements and rare earth elements. Therefore, the PCBs is the most suitable component to recycle all the valuable elements. According to the journal from Kumar *et al.,* (2013), the separation of material is based on distribution of metallic and non-metal constituents in different size fractions. This study showed the enrichment of metals in coarser particles and non-metals in the finer particles by following pneumatic separation and froth flotation process. The result showed a grade of 88% with 75%recovery was achieved by froth flotation, but lower grade of 75% with 65% recovery were obtained by pneumatic separation with. Experimental results include metallic concentrate containing 91%, 82% and 95%metals in specific sizes such as -1500 + 1000 µm, -1000 + 850 µm and -850 + 500 µm size ranges correspondingly under optimized condition by pneumatic separation, while encouraging result in overall grade and recovery accomplished by froth flotation.

The liberation characteristic and physical separation of printed circuit board (PCB) was taken from the journal made by Wang *et al.,* (2011). The elaboration is highlighted of recycling of printed circuit board (PCB), beneficial because we can do both treatment of waste as well as recovery of valuable material. By using physical and mechanical method, a good liberation is the premise to further separation. In this study, two-step crushing process is in use, and standard sieve is applied to screen crushed material to different size fractions. Moreover, the liberation situation and particles shape in different size are observed. Then metal of the PCB is separated by physical methods, including pneumatic separation, electrostatic separation and magnetic separation.

2.4 Analysis methods of separation by using fluidized bed column

The fluidization technique has been widely used in industry. In the field of mineral separation industry, the fluidization technique is always used for the separation of minerals according to the difference of physical properties such as size, density and grade (Yanfeng *et al*., 2013). Fluidized beds is provide a method for particle separation based on density difference and are an established technology for particulate processing. The principles and techniques for effective particle separations by using the fluidized bed process is going to study and research in order to determine and investigate the behaviour of rare earth elements with suitable operating conditions

Rasul *et al*., (2000) highlighted that the mixture of particles with different nature characterized by size, density, shape and surface property is fluidize, a driving force for particle separation is established. This driving force depends on particle property differences and the operating conditions. Although material separation in mineral dressing represents a complex separation process, important insights can be obtained through experimental observations and theoretical analysis of mixing or segregation behaviour of binary mixtures in fluidized beds. A fluidized mixture of particles segregates and displays a behaviour called layer inversion under certain operational conditions.

Besides that, the present invention relates to methods and apparatus using fluidized bed principles for separating mixtures of solid particles of different densities, and more particularly to such methods and apparatus as are relevant to the grading of agricultural products or the separation of agricultural products from connected waste material (Albert *et al*., 1995). The use of density variation as a means of separating mixtures of particles is widespread. In agriculture, the separation and organization of produce on this basis is accomplished using both wet and dry methods (Albert *et al*., 1995).

According to Albert *et al* (1995) journal, the wet methods use a liquid as a medium with which to separate denser particles, which sink in the given liquid, from the lighter ones that will float at the top of fluidized bed column. Because of the use of fluids, however, these techniques have drawbacks which limit their application with agriculture products. Some liquids employed are expensive or present fire and social hazards when used in huge quantities. In addition, some agriculture commodities require pre-wetting in order to remove air bubbles and thereby permit their effective sorting in fluids. Other products are not suitable to processing in any liquid because the absorption of liquid usually affects the properties of the product. Finally, the liquids involved frequently become contaminated with foreign materials during the sorting process, affecting their density and requiring periodic changing or filtering.

Dry methods of sorting or cleaning of agriculture products are not impaired by the above described disadvantages. Some dry methods of sorting employ a form of compressed separation based on a combination of differing densities and differing aerodynamic properties associated with the components to be sorted. In such separation techniques, a gas, such as air, is forced upwardly through a moving bed of the mixture to be separated. This gas flow through the gaps of the particles of the mixture tends to disengage the particles from each other, permitting the gas flow to support at least some of the weight thereof. As a result, the bed of the mixture resembles a liquid of high viscosity, and the particles of the mixture are freed to a degree to migrate within the bed under the influence of physical forces that might tend to induce separation among the constituent components. However, Oshitani *et al* (2008) state in their paper that a wide range of apparent densities are possible by changing the air velocity for fluidization and by using a mixture of two types of particle with different densities for the fluidized medium.

Furthermore, according to the journal Kwant *et al* (1995), the separation occurs when a mixture to be separated is itself fluidized is not one that results exclusively due to differing density among the components of the mixture. Instead, the aerodynamic properties of the particles of the mixture also have a considerable impact upon the rate and quality of the separation that result (Kwant *et al*., 1995). The upward flow of gas through the mixture will tend to draw with it the less compact particles of the mixture, regardless of their density. Typically, the fluidization of such a mixture is effected as it passes down an inclined trough. At the discharge end of the trough the mixture of the materials has become somewhat stratified according to the combined density and aerodynamic property of the component particles. Nevertheless, such devices have several inherent drawbacks which render them less than optimally desirable in relation to the broad range of circumstances in which agriculture separators of the dry variety are nevertheless desirable (Kwant *et al*., 1995).

Piumsomboon *et al* (2013) pointed out that the fluidization regime also plays an important role in particle mixing process which is the vigorous particle movement yields a better mixed bed. In the case of binary, it may be impossible point to the fluidization regime for the entire bed. Each particle type may be fluidized according to a different regime. For binary fluidized system with a strong segregation tendency, it is reported that complete solids mixing is quite difficult to achieve. One can also imagine that at a moderately high gas velocity the flotsam particles are subject to conditions normally encountered with turbulent fluidization while the jetsam are fluidized in the bubbling regime (Piumsomboon *et al*., 2013).

2.5 Application of rare earth elements

Nowadays, there are demanding on the resources of rare earth elements due to the application by applying the rare earth element. The recycling is the best ways due to the rapid development of electronic and information technologies has resulted in the coming of low cost and enhanced the electronic products in the market increases the quantities of end-of-life electrical and electronic equipments (EOL-EEEs) due to their replacement (Lee *et al*., 2007).

Some of the major end uses for rare earth elements consist of use in automotive catalytic converters, fluid cracking catalysts in petroleum refining, phosphors in colours television and flat panel displays (cell phones, portable DVDs, and laptops), permanent magnets and rechargeable batteries for hybrid and electric vehicles, and generators for wind turbines, and various medical devices. There are important protection applications, such as jet fighter engines, missile guidance systems, antimissile defence, and space-based satellites and also communication systems.

Briefly, according to the journal studied by Ortegon *et al* (2010), rare earth element is the main material to create the wind turbines. Wind turbines are an important driver for the Nd-magnet demand. There are three different technologies for wind turbines and only one of them uses the Nd-magnets. All three systems are accessible on the market. These wind turbine work without gear, which makes them tough and a good candidate for off-shore applications. Furthermore, a new technology based on high temperature superconductor (HTS) rotors is under research and development.

The Figure 2.1 shows that the most applicable fields of application economically are magnets and phosphors. For phosphors, expensive REE such as europium and terbium are used. For magnets, mainly neodymium and praseodymium (medium price) and dysprosium and terbium (high prices) are used. The applications glass, polishing, ceramics and catalysts are relevant in terms of their volume but less relevant in terms of their value. The main reason for this is that the cheaper REE cerium and lanthanum are used very frequently for these applications Kingsnorth., (2010).

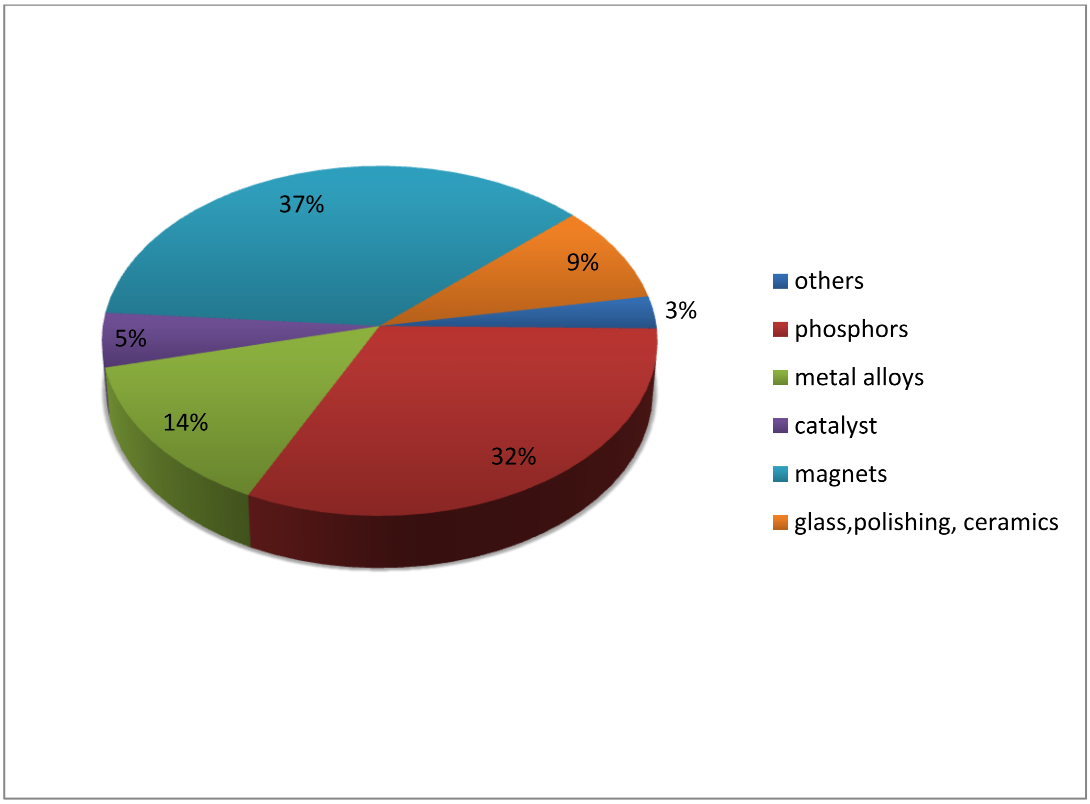


Figure 2.1: Global demand of rare earths in terms of economic value in 2008 according to Kingsnorth, (2010).

Furthermore, REEs have their own unique characteristics that are applicable and important in the production of worldwide industry. For example, erbium-doped super fluorescent fiber sources (SFSs) have been generally studied for the fiber-optic gyroscope (FOG) applications due to their high efficiency, broad bandwidth and low shot-noise limit (Lloyd *et al.*, 2010) and (Zatta *et al.*, 2002). Most of the studies as to improve SFSs' output performance and wavelength stability have been reported. This previous study recommended on improving the SFS's output efficiency such as optimizing the SFS's configurations with double-pass configuration or bidirectional pumping structure (Tsai *et al.*, 2003) and (Huang *et al.*, 2007) . Also the photonics crystal fibers (PCFs) or the ytterbium–erbium-codoped fibers are suggested as the gain medium to produce higher power (Wang *et al.*, 2009).

Besides that, the purity of the steels has been improved quite a lot since there are some rich resources of REE ores in China and also due to the China is well known as the biggest steel producer. It is to be compulsory to carry out the research on the application of RE in the clean steels and to develop REE micro-alloyed steels for example, RE-weather resistance steel, RE-heavy rails steel, REE-low alloyed steels and etc (Wang *et al.*, 2006).

2.6 Summary

This paper presents a recycling the rare earth elements by using fabricated fluidized bed column. Many researchers have been done on separation of rare earth elements. The methods mainly used by these researches are physical separation and mechanical separation.

Rare earth elements are very important for green technology. Some of the application of rare earth elements are automotive catalytic converters, fluid cracking catalysts in petroleum refining, phosphors in colours television and flat panel displays (cell phones, portable DVDs, and laptops), permanent magnets and rechargeable batteries for hybrid and electric vehicles, and generators for wind turbines, and various medical devices.

3 MATERIALS AND METHODS

3.1 Overview

This paper presents a separation of rare earth elements by using fluidized bed column. The separation of rare earth elements also considered the effect of the pressure and velocity of the fluidized bed column. In principle, the predictions were compared to the experimental measurement adopted from literature, the lighter density of rare earth elements will be at the top of fluidized bed column while the heavier will be at the bottom of the fluidized bed column. Although the results seem to suggest that further improvement of the fluidized bed column is necessary. Furthermore, the dry separation method is certainly less expensive and safe to handle.

3.2 Introduction

This chapter presents a methodology of recovery by recycling the rare earth elements by using fabricated fluidized bed column form printed circuit board (PCB) end of life.

3.3 Design of fluidized bed column

The fabricated fluidized bed column consists of a cylindrical glass fluidized bed equipped with an air regulator connected to a compressor. At bottom of the column, there will be an air inlet. The height of the column is 96 cm and 2.54 cm (1 inches) respectively. This equipment consists of 7 sampling ports attach. The function of this sampling ports is to collect the sample obtain. There are three different variables to control which is volumetric flow rate, pressure in the fluidized bed column and the size of particles for the sample to be collected at the sampling port. The principle of this equipment is the lighter density of element will be collected at the top of the column while the heavier density of element will be collected at bottom of the column.

3.4 The Interdisciplinary Centre for Plasma Mass Spectrometry (ICP-MS)

The concentration of each component will be determined using inductively coupled plasma-mass spectrometry (ICP-MS) at central lab (Rooney and Tyrone, 2011). Apparently, (ICP-MS) is dedicated to precise and accurate determinations of inorganic trace element and isotope abundances in geological, biological, agricultural, nuclear, environmental and engineering materials aimed at understanding the composition, rates and pathways of chemical transport and evolution in natural and laboratory systems.

3.5 Sample preparation

Computer motherboards and PCBs that is weight of 1.5 kg, which are from various brands and provided by a waste recycling company of Sigmaplus Capital Sdn. Bhd. in Kuantan, Pahang region, are selected as experimental material. The reusable parts and toxic components have to disband before crushing. In order to analyze the PCBs, the samples need to cut into pieces before grinding. Afterwards, the sample will be grinding until it turns to fine powder by using 6” bench grinder. The analysis of fine powder elements concentration percent by weight is carried out by using Sequential X-ray Fluorescence Spectrometer (Model No.XRF1700, Shimadzu, Japan).

3.6 Parameter of study

The sample is separate by using fabricated fluidized bed column. Three different parameter / variables:

- Time taken to collect the sample
- Pressure of air inlet
- The height of sampling port

The fine powder is distributed evenly to different sampling port of the column while the experiment is running. The sample from each sampling port is collected and analyzed the concentration of rare earth elements by using inductively coupled plasma-mass spectrometry (ICP-MS).

3.7 Consumable procurement

Air regulator, compressor, electronic device chipboard, fabricated fluidized bed column, 10 ml glass beaker, plastic packing polyethylene zip lock, safety glove, safety goggle and stopwatch.

3.8 Experimental

The methodology procedure of separation of rare earth elements by fabricated fluidized bed column. In order to get enriched metallic concentrate, several techniques are requires such as milling and pneumatic separation have been studied.

For enrichment of metallic concentrate from electrical waste PCBs beneficiation equipments were deployed, as illustrated in methodology. In order to liberate the metallic components, the PCBs were cut into 10 cm x 8 cm size using cutting tools and then shredded in as cutter crusher (Hoysung,S.No.996938005,Korea) to smaller than 10 mm with the help of spalling and crushing blades working on single shaft simultaneously. The shredded mass was milled using 6’’ Bench blender. Further down size screen aperture may lead to generation of excessive fines posing processing constraints and loss of metals and rare earth elements during separation.

After that, the samples were used for pneumatic separation. The ground PCB particles of <1500 µm were separated into light and heavy fractions containing various of element by gravity separation using a multiplex laboratory. The separator consists of different height of sampling port, depends on the weight of sample, the lighter weight of elements are goes up to higher height of sampling port. The volume of air flow and respective feed rate in the fluidized bed as adopted in the experiment for varied particle sizes, are as follows. Air infused from the bottom triggers buoyancy in providing upward thrust to the low density materials while the high density metal particles report to the sink.

Technically, the sparger used and be attached at the bottom of fluidized bed are beneficial in order to make sure the air flow is equipped to brings all the elements upwards. Hence, it well suited to separation of liberated metal from mix of ground element, since the up flow of air configuration of the pneumatic channels factually aid generating clean metallic concentrates with minimal loss of fines metals. Beds column with different heights of sampling port were analyzed at 10 cm, 20 cm, and 30 cm while the air pressure was 15 psi, 20 psi and 25 psi were tried in this investigation.

Last step in this study is analytical procedure. In order to analyze the PCBs, a representative sample was collected by using a beaker put end of each sampling port. The analysis of samples containing various type of metals element was carried out using Sequential X-ray Fluorescence Spectrometer (Model No. XRF1700, Shimadzu, Japan) and, the result showed the abundance of Cu, Si and Fe in the comminute PCBs sample and a very few concentrated of rare earth element which is found only two types of rare earth element, Erbium and Ytterbium.

3.9 Summary

This chapter presents a methodology and procedure of the experiment which tend to separate the rare earth elements from the electronic waste by using fluidized bed column.

4 DESIGN OF FABRICATED FLUIDIZED BED COLUMN

4.1 Overview

This paper presents a separation of rare earth elements by using fluidized bed column. The separation of rare earth elements also considered the effect of the pressure and velocity of the fluidized bed column. In principle, the predictions were compared to the experimental measurement adopted from literature, the lighter density of rare earth elements will be at the top of fluidized bed column while the heavier will be at the bottom of the fluidized bed column. There are seven sampling port attached to the fluidized bed column and the pump is connected to the fluidized bed column to let the air flow, thus the particles will fabricate according to their density.

4.2 Introduction

An apparatus of the experimental setup used in this study is drawn in Figure 4.1 and the material used is listed in table 4.1. Experiments were carried out in a cylindrical fluidized bed column of 4.0 cm inner diameter and 96 cm height with an air regulator connected to a compressor. This cylindrical chamber of 4.0 cm inner diameter and 96 cm height consist consists of with 7 sampling ports attach. The function of this sampling ports is to collect the sample obtain. A piece of fabric, as a distributer, was mounted between the main column and the chamber in order to feed the air uniformly into the fluidized bed. An air filter was installed at the top of the column to capture outgoing fine particles. Airflow pressure was controlled by a valve and measured using a compressor meter (TESTO-6441 Compressed Air Counter) with an accuracy of 3% of measured. A filter was placed on the entering airflow in order to collect dust, moisture and probable oil droplets. Fluidizing gas was provided by a compressed air supply including a compressor.

Materials	**Quantity**
Cylindrical PVC pipe 96 cm long	1
Stand	1
Air Compressor	3
Tube 1 m long	4
White fabric	1
Valve ¼ inch	8
Marble	30

Table 4-1: List of the material

- **The valve ¼ inch**

The particles will comes out from the fluidized bed column through the sampling port valve due to the air pressure in the fluidized bed column is higher than the outside of the column. Therefore, this valve functional is to open and close of the sampling port during the experiment.

- **White fabric as a sparger**

The white fabric will wrap the filter standard steel, this have to do so because the particle is in powder form. Therefore, while the experiment is run the particle is filtered before the air exit.

- **Marbles**

The marbles is placed at the bottom of the fluidized bed column. It is to ensure uniform distribution of air.

4.3 Schematic diagram of fluidized bed column

Figure 4.1 shows the schematic of fluidized bed column while Figure 4.2 shows the fabricated fluidized bed column.

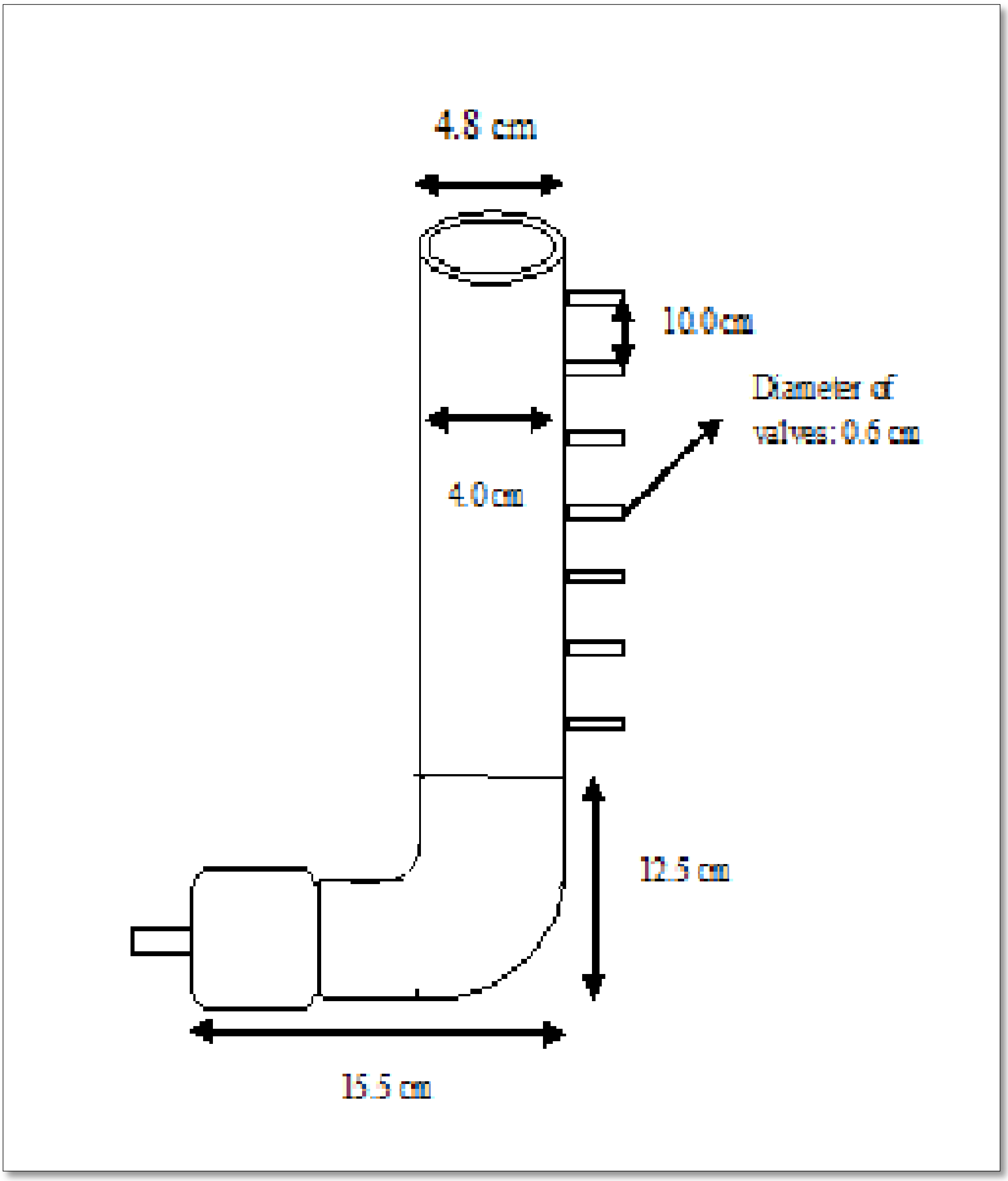


Figure 4.1: Schematic diagram of fluidized bed column

Figure 4.2: The fabricated gas –solid fluidized bed column

4.4 *Sequence of fabricated fluidized bed column*

The sequence of fabricated fluidized bed column is shown in figure 4.3.

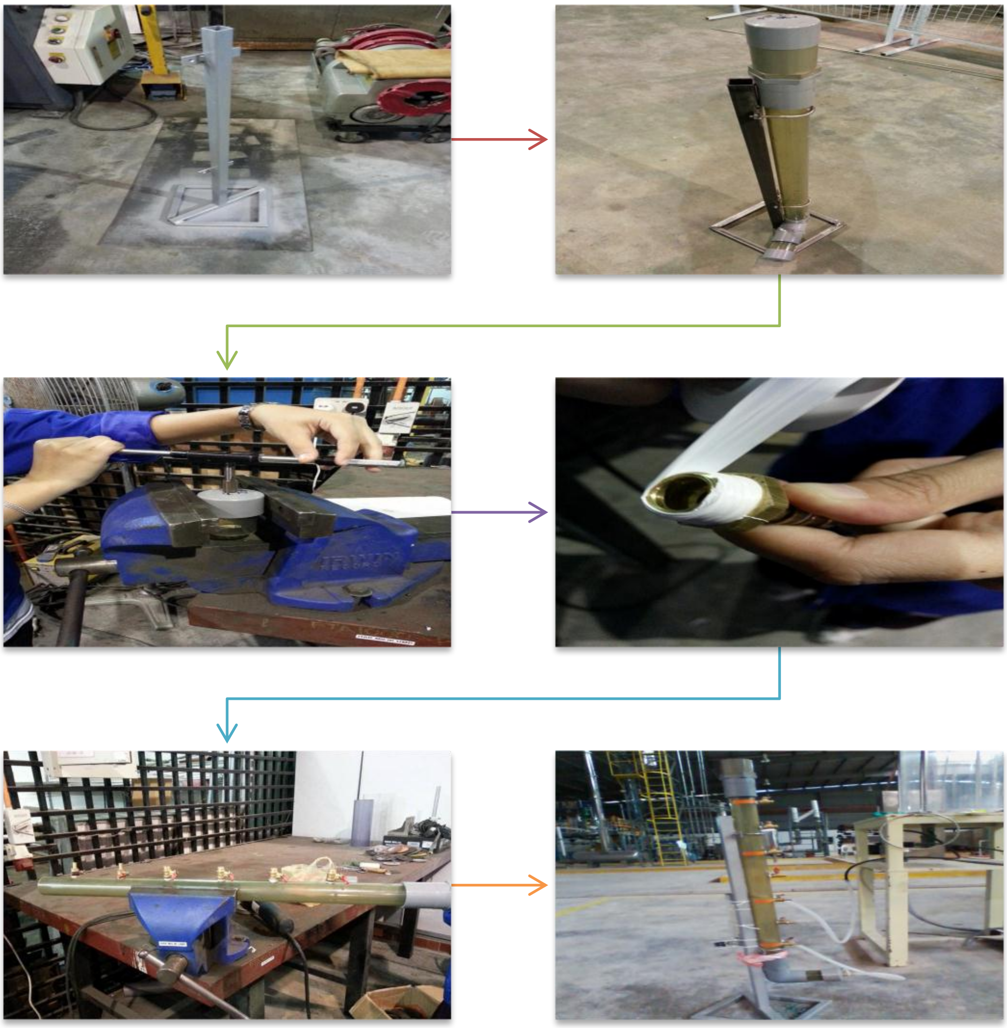


Figure 4.3: Sequence of fabricated fluidized bed column

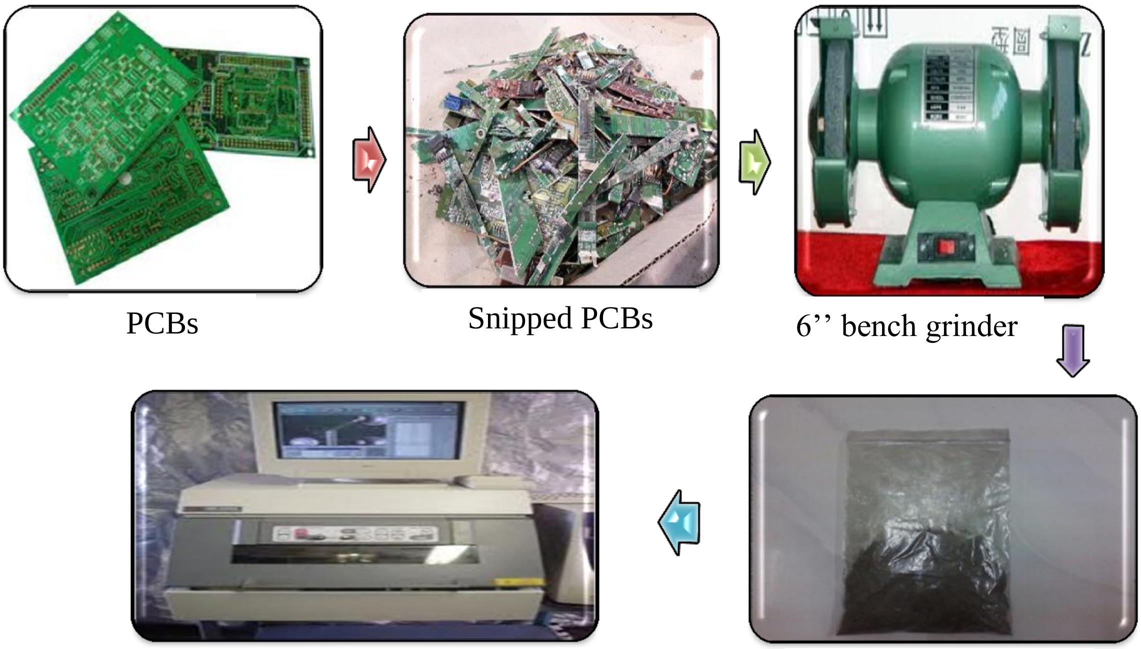
4.5 *Summary*

This chapter presents the sequence and schematic design of fluidized bed column. The fluidized bed column is ready for experiment analysis.

5 RESULT AND DISCUSSION

5.1 Sample analysis

Computer motherboards and PCBs that is weight of 1.5 kg, which are from various brands and provided by a waste recycling company of Sigmaplus Capital Sdn Bhd in Kuantan, Pahang region, are selected as experimental material. The reusable parts and toxic components have to disband before crushing. In order to analyze the PCBs, the samples have to cut into pieces before grinding. Afterwards, the sample then will be grinding until it turns to fine powder by using 6” bench grinder. The analysis of fine powder elements concentration percent by weight is carried out by using Sequential X-ray Fluorescence Spectrometer (Model No.XRF1700, Shimadzu, Japan). Figure 5.1 shows the describe process.



PCBs

Snipped PCBs

6’’ bench grinder

Sequential X-ray Fluorescence Spectrometer (XRF)

Sample 130 gram

Figure 5.1: Sequence of techniques for analysis PCBs powder elements

The XRF analysis shown in table 5.1, there are only a small amount of rare earth elements from lanthanide group in the printed circuit board which consists of erbium (Er) and Ytterbium (Yb) only. However, it is worthwhile because it can be able to investigate the behaviour of rare earth elements in the gas-solid separation by using fabricated fluidized bed column.

No.	Parameter	Result	Unit	Test method
1.	Silicon (Si)	16.06	%	Quantexpress (Best Detection)
2.	Copper (Cu)	15.62	%	Quantexpress (Best Detection)
3.	Iron (Fe)	10.10	%	Quantexpress (Best Detection)
4.	Calcium (Ca)	8.65	%	Quantexpress (Best Detection)
5.	Bromine (Br)	7.22	%	Quantexpress (Best Detection)
6.	Aluminium (Al)	6.02	%	Quantexpress (Best Detection)
7.	Tin (Sn)	4.58	%	Quantexpress (Best Detection)
8.	Lead (Pb)	2.29	%	Quantexpress (Best Detection)
9.	Manganese (Mn)	1.66	%	Quantexpress (Best Detection)
10.	Zinc (Zn)	1.15	%	Quantexpress (Best Detection)
11.	Barium (Ba)	0.55	%	Quantexpress (Best Detection)
12.	Titanium (Ti)	0.47	%	Quantexpress (Best Detection)
13.	Potassium (K)	0.35	%	Quantexpress (Best Detection)
14.	Magnesium (Mg)	0.25	%	Quantexpress (Best Detection)
15.	Nickel (Ni)	0.21	%	Quantexpress (Best Detection)
16.	Sulphur (S)	0.41	%	Quantexpress (Best Detection)
17.	Antimony (Sb)	0.14	%	Quantexpress (Best Detection)
18.	Chlorine (Cl)	0.13	%	Quantexpress (Best Detection)
19.	Erbium (Er)	0.10	%	Quantexpress (Best Detection)
20.	Zirconium (Zr)	0.10	%	Quantexpress (Best Detection)
21.	Tungsten (W)	0.07	%	Quantexpress (Best Detection)
22.	Strontium (Sr)	0.07	%	Quantexpress (Best Detection)
23.	Phosphorus (P)	0.05	%	Quantexpress (Best Detection)
24.	Silver (Ag)	0.04	%	Quantexpress (Best Detection)
25.	Bismuth (Bi)	0.04	%	Quantexpress (Best Detection)
26.	Chromium (Cr)	0.03	%	Quantexpress (Best Detection)
27.	Ytterbium (Yb)	97	Ppm	Quantexpress (Best Detection)
28.	Scandium (Sc)	57	Ppm	Quantexpress (Best Detection)

Table 5.1: The elements in the Printed Circuit Board (PCB) from XRF analysis

However, to predict the particle distribution in the fluidized bed column is of great importance in industrial application and in the designing of fluidized bed column with good performance. The following sections was analyzed various transition pressure of air inlet in fluidized bed system in order to characterize the influence of height sampling port and pressure of air inlet by effect of time taken on the fluidization state.

5.2 Experimental analysis

In view of the up-flow of air plus a continuous feed of fine solids to a vertical tube, if the air distribution is go well with, then all the solids will be carried up the tube as separate particles extensively dispersed in the gas. In this current study, the experiment carried up into three parts which is first experiment have pressure of air inlet 15 psi and the time taken is 25 seconds, second experiment have 20 psi and 30 seconds and third experiment have 25 psi and 35 seconds.

Research work undertaken previously mainly focuses on the behaviour of elements or substance of non rare earth, it is commonly used parameters like pressure drop, fluctuation ratio, expansion ratio, mixing characteristic, etc on fluidization quality, particularly in the lower velocity range. But the present work makes an attempt to study the effect of time taken on fluidization quality. Besides, the novelty of present work lies in the fact that this work has studied the performance of fabricated fluidized bed column on the separation of rare earth elements.

Particle was distributed according to their particle density and characteristic. Therefore, the results from the ICP-MS analysis are shown below:

For the first experiment, the pressure of air inlet was 15 psi and the time taken to collect the sample was 25 seconds. The result shown as table below:

Sampling port	Weight (gram)	Height of sampling port	Rare earth element	Concentration of REEs by ICP-MS analysis	Unit
1	0.00	11.5 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb
2	6.01	32 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb
3	2.67	52.5 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb

Table 5.2: First experiment analysis

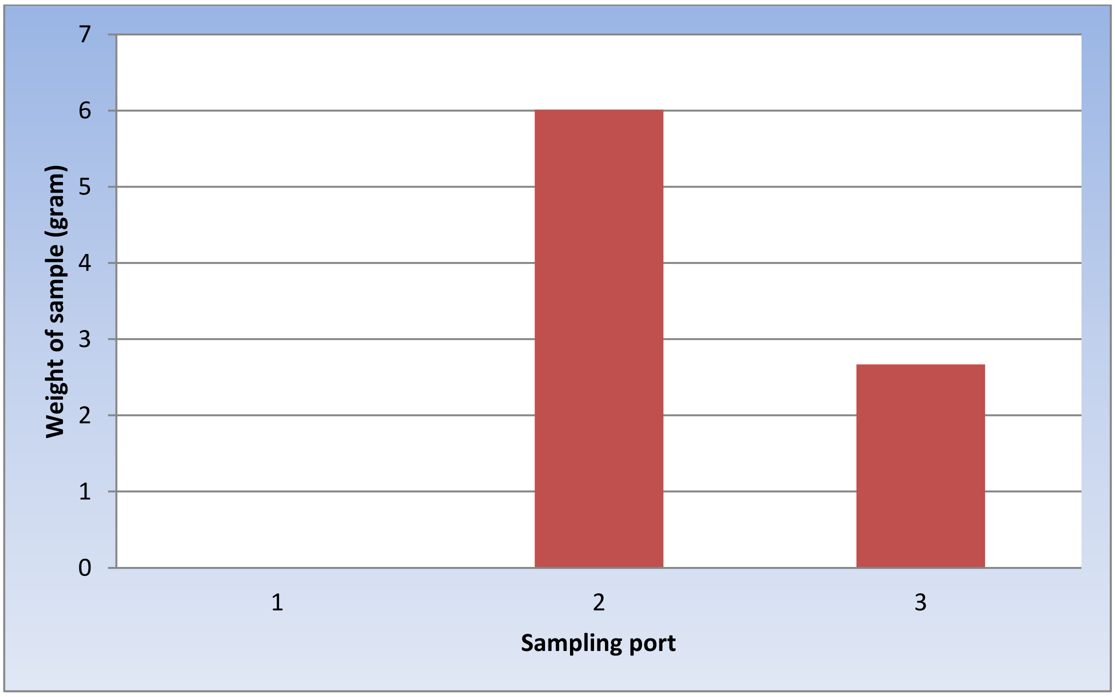


Figure 5.2: Graph of first experiment

Second experiment, the pressure of air inlet was 20 psi and the time taken was 30 seconds. The result shown as table below:

Sampling port	Weight (gram)	Height of sampling port	Rare earth element	Concentration of REEs by ICP-MS analysis	Unit(gram)
1	0.1	11.5 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb
2	1.79	32 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb
3	8.02	52.5 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb

Table 5.3: Second experiment analysis

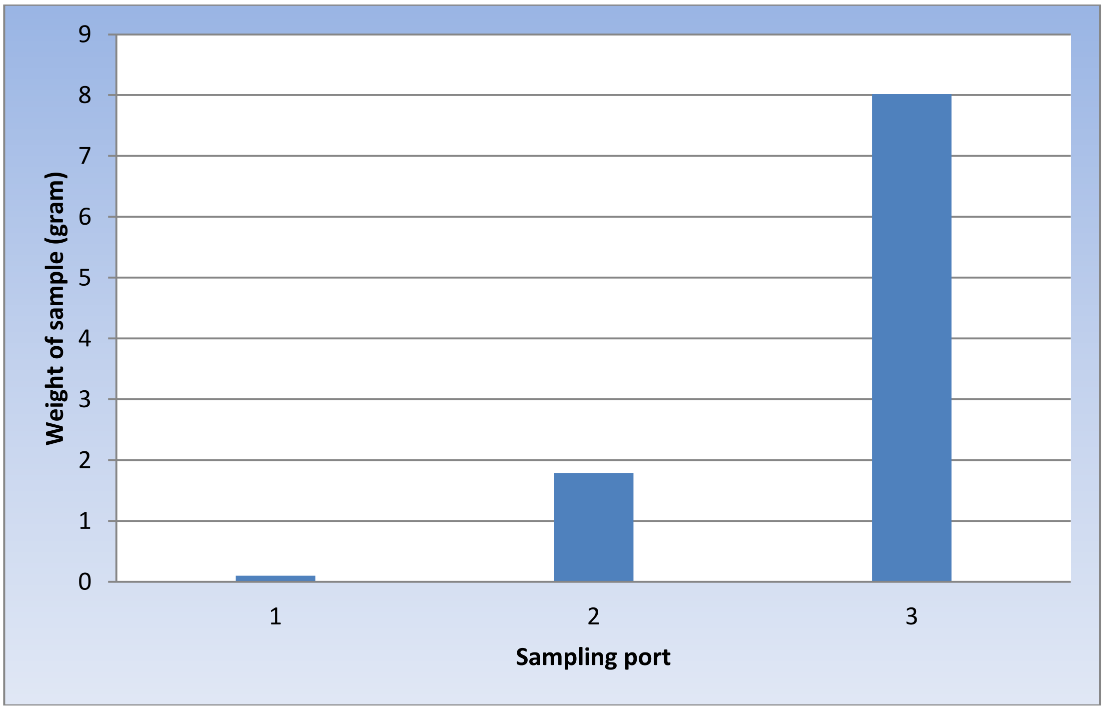


Figure 5.3: Graph of second experiment

Third experiment, the pressure of air inlet was 25 psi and the time taken was 35 seconds. The result shown as table below:

Sampling port	Weight (gram)	Height of sampling port	Rare earth element	Concentration of REEs by ICP-MS analysis	Unit
1	6.1	11.5 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb
2	1.08	32 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb
3	0.39	52.5 cm	Erbium (Er) Ytterbium (Yb)	Less than 0.00022 Less than 0.000018	ppb ppb

Table 5.4: Third experiment analysis

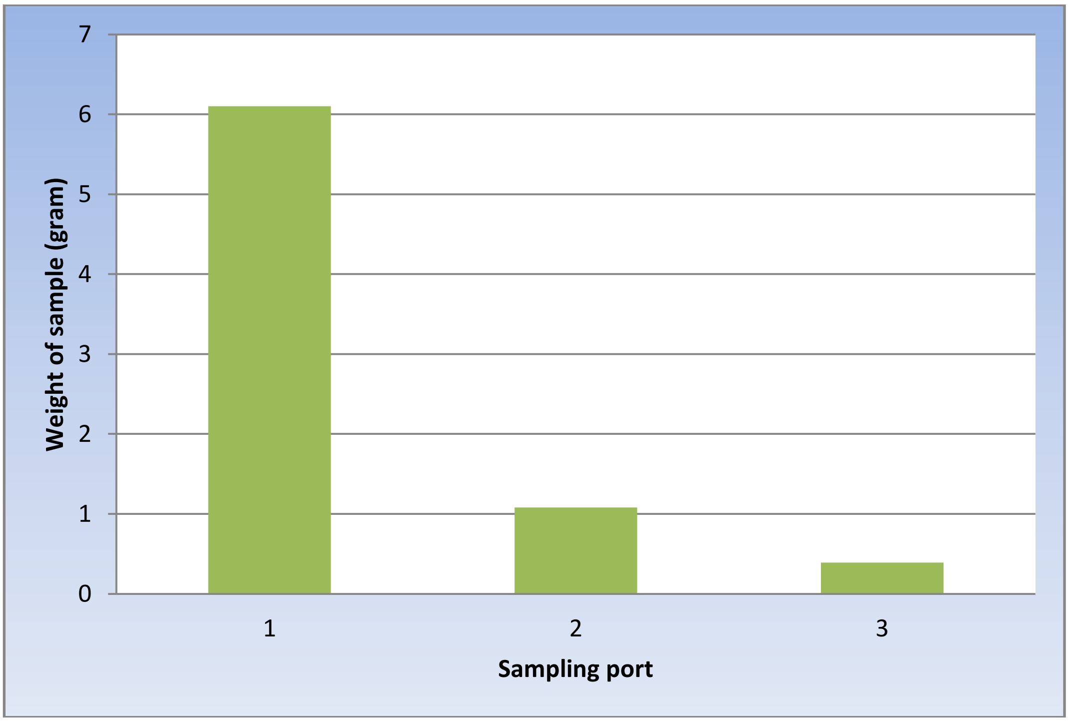


Figure 5.4: Graph of third experiment

By using ICP-MS analysis, the rare earth elements was the only were analyzed, therefore, erbium and ytterbium concentration were known as the three tables above and the result shows the concentration of erbium and ytterbium has a very small number of amount. This amount of concentration is not in range or is approximately to zero. This kind of result were obtained is because of the some errors occur during the experiment where some of the parameter and characteristic are significantly should be considered. The parameters and characteristics have to be considered are the particles characteristics, the air distributor of fluidized bed column and the inner diameter of bed column.

Nevertheless, the remaining at the bottom of the fluidized bed column was analyzed by using XRF and the result shows in Table 5.5 below. This result shows the concentration of erbium and ytterbium is not have much changes, which means the concentration for both elements is remain at the bottom during the experiment. There are several reason of this results obtained were happened in this study. These reasons were analyzed and discussed in the following sub-topic.

No.	Parameter	Result	Unit	Test method
1.	Iron (Fe)	18.05	%	Quantexpress (Best Detection)
2.	Silicon (Si)	14.94	%	Quantexpress (Best Detection)
3.	Copper (Cu)	11.22	%	Quantexpress (Best Detection)
4.	Aluminium (Al)	7.63	%	Quantexpress (Best Detection)
5.	Calcium (Ca)	5.02	%	Quantexpress (Best Detection)
6.	Tin (Sn)	4.62	%	Quantexpress (Best Detection)
7.	Bromine (Br)	3.52	%	Quantexpress (Best Detection)
8.	Manganese (Mn)	3.32	%	Quantexpress (Best Detection)
9.	Lead (Pb)	3.25	%	Quantexpress (Best Detection)
10.	Zinc (Zn)	2.23	%	Quantexpress (Best Detection)
11.	Barium (Ba)	0.63	%	Quantexpress (Best Detection)
12.	Titanium (Ti)	0.60	%	Quantexpress (Best Detection)
13.	Potassium (K)	0.49	%	Quantexpress (Best Detection)
14.	Nickel (Ni)	0.30	%	Quantexpress (Best Detection)
15.	Magnesium (Mg)	0.28	%	Quantexpress (Best Detection)
16.	Zirconium (Zr)	0.19	%	Quantexpress (Best Detection)

17.	Erbium (Er)	0.14	%	Quantexpress (Best Detection)
18.	Chlorine (Cl)	0.13	%	Quantexpress (Best Detection)
19.	Sulphur (S)	0.12	%	Quantexpress (Best Detection)
20.	Antimony (Sb)	0.11	%	Quantexpress (Best Detection)
21.	Bismuth (Bi)	0.05	%	Quantexpress (Best Detection)
22.	Tungsten (W)	0.05	%	Quantexpress (Best Detection)
23.	Silver (Ag)	0.05	%	Quantexpress (Best Detection)
24.	Strantium (Sr)	0.05	%	Quantexpress (Best Detection)
25.	Phosphorus (P)	0.05	%	Quantexpress (Best Detection)
26.	Chromium (Cr)	0.03	%	Quantexpress (Best Detection)
27.	Sodium (Na)	0.03	%	Quantexpress (Best Detection)
28.	Scandium (Sc)	56	Ppm	Quantexpress (Best Detection)
29.	Ytterbium (Yb)	37	Ppm	Quantexpress (Best Detection)

Table 5.5: Remaining sample analysis

5.3 Effect of the time taken on the performance of the separation

Experiment 1, 2 and 3 was carried out at different pressure and different sampling time, taken at different sampling port. Details of the parameters used and the results are shown in Table 5.6.

Experiment	Column height	Pressure (psi)	Sampling time (s)	Weight (g)
Exp 1	11.5 cm	15	25	0.00
			30	0.00
			35	0.00
Exp 2	32 cm	20	25	1.79
			30	1.85
			35	2.10
Exp 3	52.5 cm	25	25	2.08
			30	1.50
			35	0.39

Table 5.6: Effect of the sampling time

From the results, it shows the optimum separation condition is at 20 pressure of air inlet. Besides that, from the observation, it is the most stable condition during the experiment. There are a number pattern of graph obtain, therefore it may conclude here that it is quite difficult to recommend an appropriate correlation, due to the fact that it is not only depends on the physical properties of the solids but also on the bed configuration.

5.4 Particle characteristic

The characteristic of particles is one of the main parts need to be considered in this separation because it may give the big affect to the result outcome. The important properties for fluidization are particle size, particle density and sphericity of particle (Mohanty *et al*., 2009). Fluidized bed design procedures involve an understanding of particle properties. The solid particles used in a fluidized bed are not identical in size and hence follow a particle size distribution. An average particle diameter is generally used for the design. It has been found that it is necessary to give relatively more stress to the lowest of the particle size distribution (Mohanty *et al*., 2009). Therefore, the fines powder sample has been used to investigate the behaviour of separation in this experiment.

However, Mastellone & Arena (1999) studied the effect of particle size and density on solid distribution along the riser of a circulating fluidized bed and concluded that an increase in particle density from 1800 to 2600 kg/m3 led to a higher solid concentration at the bottom. According to Geldart & Abrahamsen (1973) journal they have classified powders depending on their fluidizability using air at the ambient temperature. Hence, this previous study highlighted there are four types category of particles. Firstly, group B particles are those, which have an average particle size exceeding about 100μm. Thick particles (e.g., glass, sand, ore, etc) are likely to be in group B. Many of the gas-solid reactions are operated industrially with this size group of particles. Second category is group A which the particles are smaller and lighter than group B particles. Most manufactured catalysts are in this category, with particle sizes ranging from about 10 to 130 μm. These particles are cohesive due to inter-particle forces and when gas velocity is increased beyond Umf, the bed continues to expand smoothly without the formation of bubbles.

Third category is group C particles are smaller and lighter than group A particles and are cohesive. They are typically less than 30μm in average particle diameter. The large external surface area and low mass of these particles produce large attractive forces. The particles do not flow well in pipes and are difficult to fluidize. When fluid bed measurements are performed on group C systems at low gas velocities, a low pressure drop is observed since the gas is flowing in a channel without encountering most of the particles. Regularly group C particles can be fluidized by using a high gas velocity to overcome the cohesive forces between the particles. Group D particles are large, of the order of 1.0 or more millimeters (1000μm) in average particle size. Group D particles behave exactly like the group B particles. High gas velocities are required to fluidize group D particles. However, it is often more economical to process them in spouted beds, where lower gas flow rates are applicable. For example of group D, dried grains and peas, roasted coffee beans and metal ores, these kind of solids and are normally processed in shallow beds or in spouted beds. Therefore, in this study the particles used were in group C.

However, Figure 5.5 shows are applicable for binary phase gas-solid system of fine powders with different density of particles. From the observation, we can analyse the particle with lighter density will go off after the segregation process. For incline phase fluidization, with higher bed invalid age, the bed materials at higher gas velocity are expected to go out of the bed and so the pressure of air inlet have to controlled properly. Therefore, we can conclude that the lighter density of elements will go off after the segregation will the denser density will remain at the bottom.

In this current research, the sample of printed circuit board was grinded until it turns to fine powder, according from the discussion above the separation must used at high pressure value due to the size of particles in group C. Subsequently, the segregation of particles in fluidized bed column during the experiment was going smoothly. Therefore, it does distribute the particles to the sampling ports efficiently.

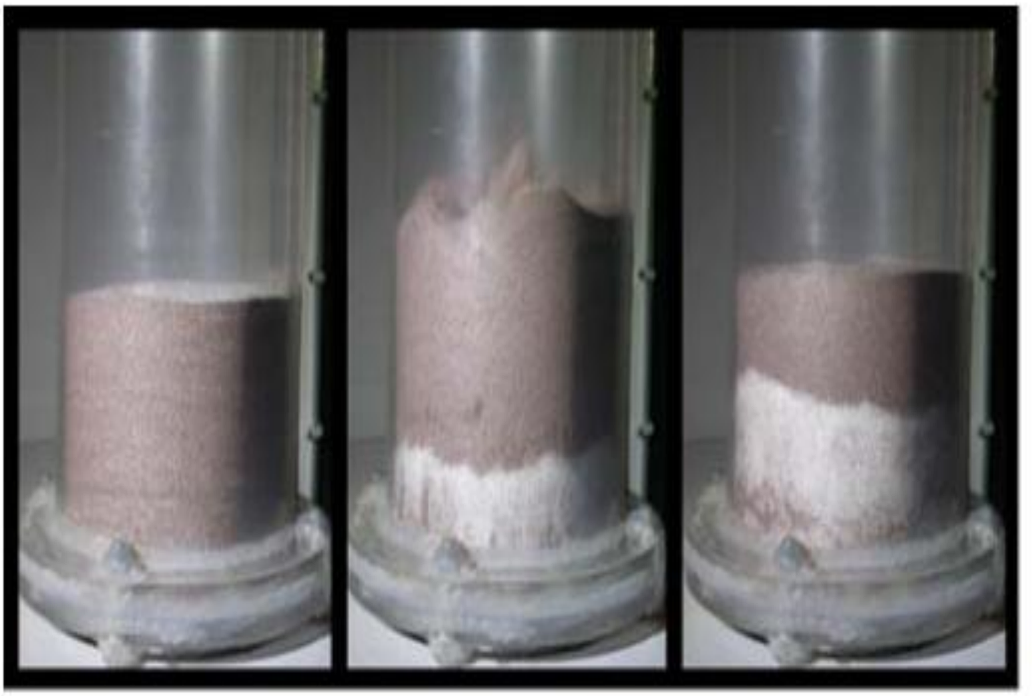


Figure 5.5: Example of fine powders in fluidized bed before, during and after the segregation process. (Hassan B. T. *et al*., 2013).

From the observation in current study it is clear that a larger and denser particle will tend to sink at the bottom and a smaller and less dense particle will be at the bed surface. More interesting is the case in which the particle is able with contrasting properties with respect to the background suspension, i.e. larger but less dense or the reverse. However, at the beginning solution to the problem will be required in the general case. Quite obviously, the investigate particle will be at automatic equilibrium, which means it will remain still, if the flowing gas is able to support exactly its weight (Alberto *et al*., 2012). Correspondingly, if the hydrodynamic interaction prevails over gravitational force the particle will float on top of the suspension, even as if its weight is larger than the action of the gas then the particle will sink towards the bottom of the column (Alberto *et al*., 2012).

The density of erbium and ytterbium is quite heavier compared to others element. The list of density of all elements is shown in Table 5.7:

No.	ELEMENT	DENSITY (g/cm^3)
1	Potassium (K)	0.89
2	Sodium (Na)	0.97
3	Calcium (Ca)	1.378
4	Magnesium (Mg)	1.738
5	Phosphorus (P)	1.82
6	Sulphur (S)	2.07
7	Silicon (Si)	2.3296
8	Strontium (Sr)	2.375
9	Aluminium (Al)	2.70
10	Scandium (Sc)	2.989
11	Bromine (Br)	3.11
12	Chlorine (Cl)	3.214
13	Barium (Ba)	3.51
14	Titanium (Ti)	4.505
15	Zirconium (Zr)	5.8
16	Chromium (Cr)	6.68
17	Antimony (Sb)	6.697
18	Ytterbium (Yb)	6.99
19	Zinc (Zn)	7.14
20	Manganese (Mn)	7.21
21	Tin (Sn)	7.287
22	Nickel (Ni)	7.81
23	Iron (Fe)	7.874
24	Copper (Cu)	8.94
25	Erbium (Er)	9.066
26	Silver (Ag)	9.78
27	Bismuth (Bi)	9.78
28	Lead (Pb)	11.342

29	Tungsten (W)	19.3

Table 5.7 : The list of density of PCBs elements

5.5 Air distributor

Mostly experiments were carried in cylindrical beds equipped with conical or rectangular bottom sections with varying cross sectional area. In spite of these expansions, the formation of dead zones was reported at the intersection. This results in reduced mixing.

Briefly, the selection of other operating parameters such as inlet flow rate, bed aspect ratio, spout dimension and physical properties of the particles plays an imperative role in discharge formation. Small changes in these parameters give big impact in remarkable changes in emit properties and bed behavior. Hence, spouted beds have rarely been used in applications involving large changes in physical or chemical properties.

By considering this, Chatterjee (1970) proposed a work of fiction gas–solid contactor, compromising unified features of both fluidized and spouted beds, known as spout fluidized bed or spout-fluid bed. Even tough, both names are used in literature, from now on we will refer to these apparatus as spout fluidized beds. In these beds an additional background gas (also known as auxiliary or fluidizing gas) is supplied through the bottom as compared to the conventional spouted bed as shown in Figure 5.3 below.

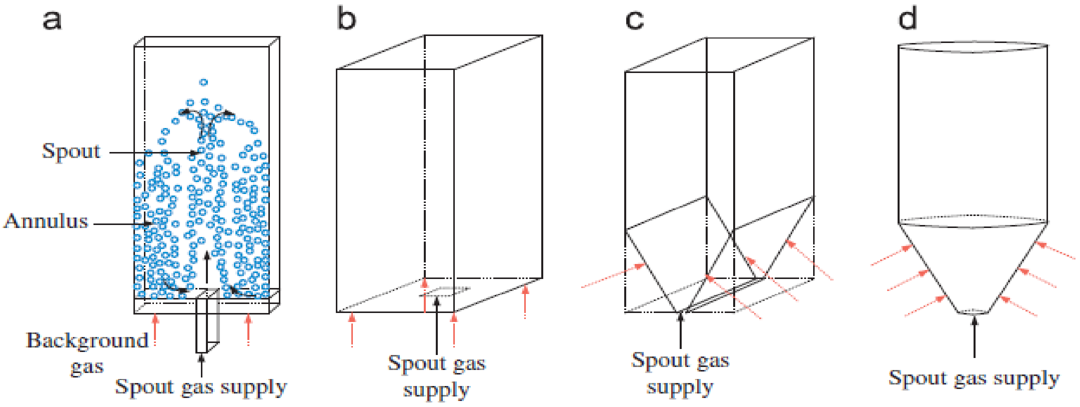


Figure 5.3 : Schematic representation of different spout fluidized bed contactors: (a)pseudo2D (b)rectangular (c)slotted rectangular and (d)cylindrical spout fluidized beds. The dotted arrows indicate the positions and angles of the background gas supply. (Niels *et al.*, 2013)

The design in terms of ratio of bed diameter to spout diameter, bed aspect ratio, spout and background flow rate and geometrical configurations plays a very important role in the particle dynamics and hence in the specific application. Based on the critical analysis of existing literature the following important design related information has been summarized:

Some of the distinguishing features of spout fluidized beds are:

1. Additional background air flow leads to higher circulation and mixing rates, due to the bubble generation in the annulus, leading enhanced particle movement in vertical and radial directions (Niels et al., 2013). Since, this current study, the fabricated fluidized bed column do not have additional background study, this may lead to the inconsistently distribution of air and thus the particles were not segregate well.

2. The total flow rate required to fluidize particles is lower in comparison to fluidized and spouted beds. This is principally qualified to cross flow of additional background gas supply. This statement was supported by Chatterjee

(1970) who performed experiments using sand particles (of average diameter equal to1.08 mm) in a cylindrical bed.

3. Moreover, in this current investigation, the uneven distribution of the gas at a certain distance from the distributor plate gives an arrow operational range. Besides that, it has been observed for fluidized and spouted beds, which often suffer from slugging behavior, whereas in spout fluidized beds wider flow rates can be used with a lower tendency to slugging. This provides a wider operational range, specifically for particles with varying density.

In order to improve the quality of fluidization in this study, changing the distributor design is one of the important parts in fluidization. Besides, a number of investigations have stressed the use of distributors is important to considered while improve fluidization quality and to increase the range of applicability of gas-solid fluidized beds. Ghosh & Saha (1987) showed that the quality of bubble formation (slugging) is strongly influenced by the type of distributor. Moreover, Saxena et al. (1979) studied the effect of distributors on a gas-solid fluidized bed.

Luo *et al.* (2004) studied the effect of gas distributor on performance of dense phase high density fluidized bed for separation. Their study has found that the higher the pressure drop of gas distributor is, the better the fluidizing performances of the fluidized bed is. Though, the fluidized bed with uniform and stable density for mineral separation can be formed when pressure drop of the gas distributor is higher than its critical value.

Based on the Zhong *et al.* (2006) journal, they found that the total bed pressure drop appears a spouted bed characteristic when increasing the spouting gas velocity and keeping the fluidizing gas velocity constant, while it appears a fluidized bed characteristic when increasing the fluidizing gas velocity and keeping the spouting gas velocity constant. Mohanty *et al.* (2007) have highlighted in their literature, a distributor plate having 10% open area of cross-section of the column cross-section gives a better result in terms of bed pressure drop, fluctuation and expansion as compared to 6%, 8% and 12% open areas of cross section.

Sasic *et al*. (2006) highlighted the effect of air distributor on pressure drop and found that there is a strong interaction between the fluidized bed and the air supply system in the form of pressure waves. Therefore the formation of dead zones as shown in Figure 5.4 was reported at the intersection and results in reduced mixing. Moreover, for low air distributor pressure drop a clear interaction was found, whereas with a high air distributor pressure drop, pressure fluctuations in the plenum were not related to the fluctuations in the bed.

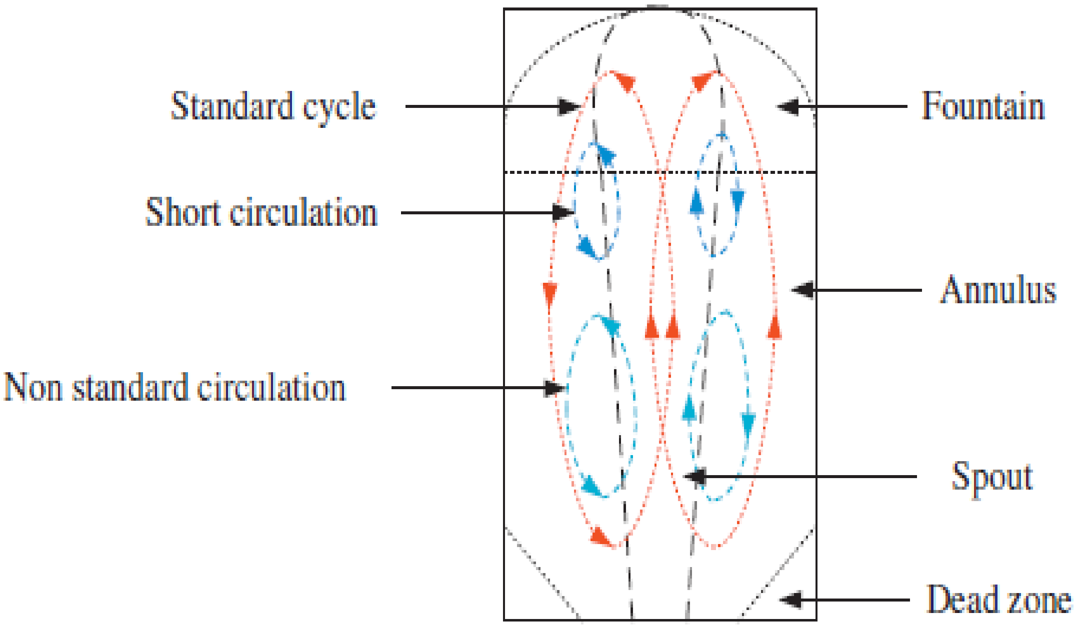


Figure 5.4: Representation of circulation pattern in a spout fluidized bed (dimensions W x D x H = 0.1 x 0.03 x 0.3 m^3) with 2.8mm polymer particles of 900kg/ m^3density (Zhong W. *et al*., 2010).

Furthermore, the figure 5.4 above, we can observe that the eddy circulating (short circulation) was form at the certain pressure of air supplied. The same phenomenon was occurred in this research study, during the experiment the eddy circulating was appearing either huge circulating or small circulating. From the result were obtained, at low pressure of air inlet will distributed high amount of sample (g) at the sampling port 26.3 cm height. While, when given at high pressure of air inlet the particles was distributed high amount of sample at the lowest height of sampling port. Therefore, the analysis is given, the higher the pressure of air inlet, the abundant amount of sample was collected at the lowest height of sampling port.

Outstanding to the effect of the air distributor, the area of fabricated fluidized bed column was too large to distribute the sample particles which have only 130 gram of fine powder. Accordingly, the higher the pressure of air inlet, the eddy circulating will getting faster and smaller compare to low pressure of air inlet. Therefore, the sample powder will distributed high amount of particle at the lowest high of sampling port at high pressure of air inlet.

5.6 Pressure of air inlet

Various regimes of fluidization as defined by Mohanty *et al*. (2009) when a bed of solid materials resting on a distributor plate is contacted with an upward flow of gas in a fluidizer, the bed gradually transforms from packed to fluidized (bubbling) bed, then to slugging bed (if fluidizer diameter is small and L/D_C is high), followed by turbulent and fast fluidized bed (FFB) and finally to pneumatic conveying as shown in Figure 5.5. As the gas flow rate is increased, the pressure drop across the bed increases and a point is reached when the pressure drop across the bed is just sufficient to support the weight of the particles in that section. This point is termed as just beginning fluidization and the corresponding velocity is known as incipient or minimum fluidization velocity, (U_{mf}).

With more rise in gas velocity, the bubbling fluidization regime appears as shown in Figure 5.5. The bubbles originate near the distributor, unite and rise to the surface and burst periodically with irregular pressure fluctuation of appreciable amplitude.

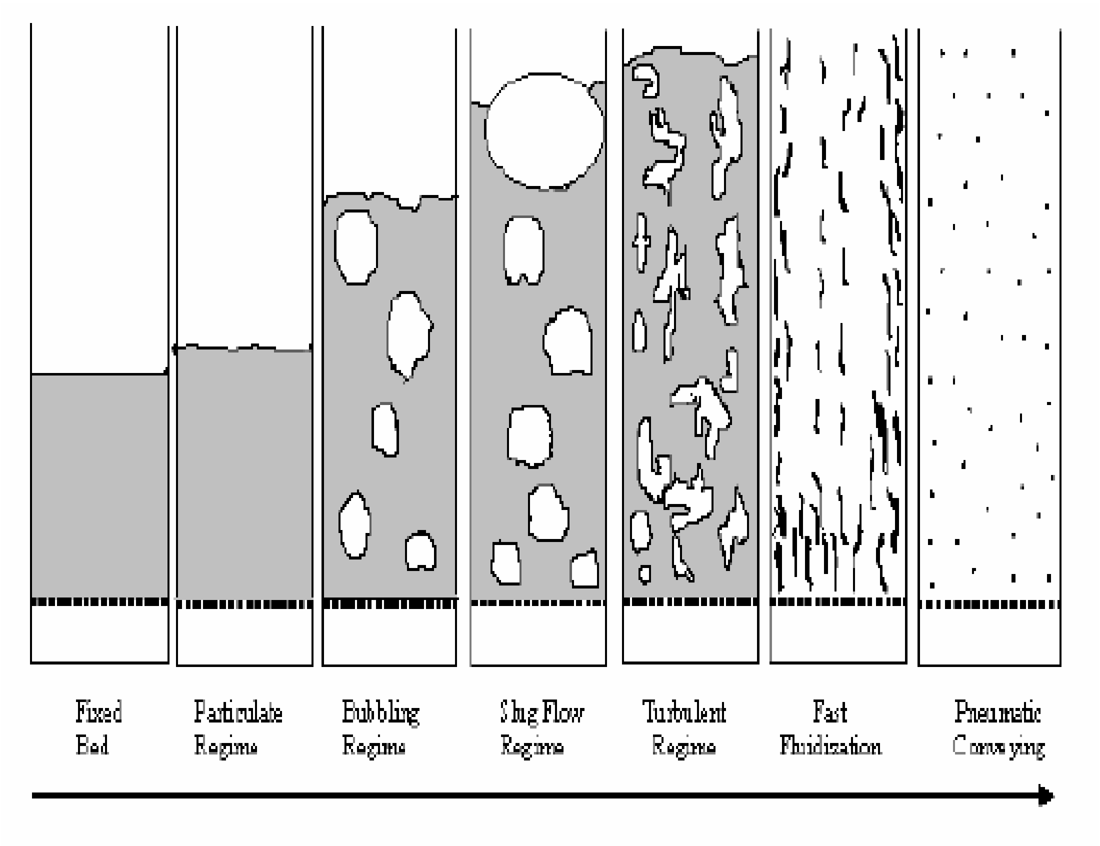


Figure 5.5: Changes in Fluidization Regimes with Increasing Pressure of Air Inlet

A variety of characteristic velocities should be consider during the experiment to achieved optimizing and accurateness such as minimum fluidization velocity (U_{mf}), minimum bubbling velocity (U_{mb}), minimum slugging velocity (U_{ms}), onset of turbulent fluidization (U_{k}) etc. are important in defining different regimes of fluidization(Mohanty *et al*. 2009) . However, visual observation has two drawbacks:

a) Non- intrusive visual access to the spout channel is usually difficult and sometimes unfeasible.
b) Visual observations are difficult to capture in a quantitative measure.

Zhang & Tang (2004) stated that their regime mapping on spectral analysis of pressure drop fluctuations. This technique only requires access to the bed with a pressure sensor, which is usually much easier the visual access and produces quantitative results. Tsuji *et al*. (1993) modelled encounters between particles with a soft sphere approach while Hoomans *et al*. (1996) used a hard sphere approach. The soft sphere approach was most suited fluidized bed column in which defluidized zones can overcome. But, the hard sphere approach was more suited in vigorously fluidized systems, spout-fluid beds because a steep velocity gradient in the gas phase is present near the spout region and the particle Reynolds numbers, mainly near the spout mouth, were much higher than those encountered in regular (bubbling) fluidized beds. Apart from that, Link *et al.* (2005) give recommended to combine experimental and simulation study on the various regimes, which could be encountered during spout-fluid bed operation. For most regimes, the developed model was able to predict the appropriate regime.

6 CONCLUSION

6.1 Conclusion

This project focuses on both the design of the fluidized bed column and the experimental study on dry separation. The recycling of rare earth element technique is depending on the knowledge of separation, purification, extraction and etc.

The minimum spout and spout fluidization velocity increases with increasing bed height, particle and spout orifice diameter. The quantification of minimum spouting velocity plays an important role in the selection of inlet gas flowrate, hence it is necessary to have a unified correlation for the calculation of the minimum spouting velocity for various geometrical configurations. Here, it is difficult to recommend an appropriate correlation, due to the fact that it not only depends on the physical properties of the solids but also on the bed configuration. A small change in the configuration dramatically changes the minimum spout velocity. Also, it is not obvious to directly apply a correlation developed for one particular geometrical configuration to another configuration.

The pressure drop in a spout fluidized bed increases with increasing bed height, spout diameter and density of the particles, whereas it decreases with increasing particle diameter and spout gas velocity. Moreover, quantification of pressure drop can help in the selection of operating parameters, as well to provide sufficient information (after suitable analysis) about the generation and collapse of the bubbles. All this information can be used to gain insight in the flow behavior and its transitions.

Mixing in spout fluidized beds depends on the particle density, spout and background gas velocities. It is mainly attributed to diffusion, convective transport and circulation of particles due to formation and collapse of bubbles. As the spout gas velocity increase, particle circulation and gas mixing also increase, whereas increase in density results in lowering the mean cycle time. Furthermore, increase in background velocity results in lowering the mean cycle time leading to uniform residence time distribution and increase gas mixing at lower bed height. So, selection of background velocity is of greatest important to achieve uniform mixing conditions.

6.2 Recommendation

- It is apparent that the quality of fluidization can be improved by lowering the bed height, particle size, and using a distributor of optimum cross-sectional area. But a 10% distributor plate, in particular, offers the best fluidization quality as evident from the experimental findings.
- Use glass cylindrical bed column instead pipe cylindrical because the particles will move smoother.
- Use more suitable grinder with proper collector. To avoid the particle flutter to surrounding.
- The fluidized bed column strainer should be used more appropriate strainer instead using fabric.
- Fluctuation ratio is small for columns having larger diameters in the lower velocity range.
- Fluctuation ratio has also a small value for columns having smaller diameters in the upper velocity range.
- Fluctuation ratio bears a direct relation with bed height in the lower velocity range.
- Fluctuation ratio varies directly with bed height in the higher velocity range.
- Expansion ratio is in indirect proportion with column diameters and bed heights.
- Expansion ratio is a direct function of mass velocity in all the ranges of mass velocities.
- The introduction of rod and disc types of promoters in the bed improves the bubble behaviour by breaking the bubbles of larger sizes into a number of bubbles of smaller sizes, for almost the complete regime of fluidization, except in the neighbourhood of the minimum fluidization conditions, where the bed dynamics

are of transitional in nature. The use of the rod type promoter in gas solid fluidized bed has been found to be more effective in reducing the bed fluctuation and increasing bed expansion in higher mass velocity ranges, which in turn helps in reducing the overall size of a fluidizer.

6.3 Future work

The following areas may be studied in future for separation distribution of rare earth element:

- Effect of distributor areas on various column diameters
- Effect of combined promoter and simultaneous primary and secondary air on fluctuation and expansion ratios
- Effect of combined promoter and simultaneous primary and secondary air on mixing
- Use of tertiary mixture with different compositions instead of binary ones
- Study of effect of regular particles on pressure drop, fluctuation ratio, expansion ratio and mixing

However, the research carried in this project is gas-solid separation is currently being expanded to develop model separation behaviour of rare earth elements from electronic waste by using fabricated fluidized bed column.

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