DEVELOPMENT OF A BATCH REACTOR FOR THE PRODUCTION OF A ACTIVATED CARBON FROM COCONUT SHELLS

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ABSTRACT

Nigeria currently imports large quantities of Activated Carbon (AC), for its domestic usage, despite abundance of agricultural wastes (coconut shells, palm kernel shells, etc), that can be used as raw materials. Short-circuiting of heating elements and absence of inert gases inlet have been identified as major challenges associated with AC production in Nigeria. This work was designed to develop a batch reactor incorporating functional materials for production of AC using locally sourced materials.

Design parameters (refractory and insulator thicknesses, thermal stress and strain, heat loss and heating rate) for 800g batch reactor with 32-1200 ^{o}C temperature range were obtained using standard heat conduction equations. Materials were selected for reactor shell, refractory lining, thermal insulation and heating elements. Heating elements were fully embedded in refractories to avoid short–circuiting due to activation fumes. Tuyere was incorporated into the charging inlet for passage of inert gas (Nitrogen) into the reactor. Reactor was constructed and temperature profiles were obtained at different loading conditions (0, 25, 50, 85 and 100%) by charging it with prepared dried samples of powdered coconut shell, carbonised to 1000 ^{o}C. Validation of produced AC was by chemical activation using 200 g of coconut shell, crushed, milled into powder, oven dried for 48 hours at 110 ^{o}C and impregnated with Potassium hydroxide at (KOH : sample) ratio 3:2, as recommended. Impregnated samples were heated in flowing stream of Nitrogen (15 l/min) till 450 ^{o}C activation temperature. Samples (AC), were held at this temperature for 1-hour and 3-hour activation times. Experimental results were characterised by X-ray diffraction and scanning electron microscopy and compared with descriptive statistics and chi-squared required standard values.

Refractory lining and insulator thicknesses were 0.3 and 0.2 m respectively. Thermal stress and strain were 166.9 MN/m^{2}, and 5.6, respectively. Heat loss through wall of reactor was 912.92 W/K and heating rate was 10 ^{o}C/min. Materials for shell, refractory lining, thermal insulation and heating element were galvanised steel plate, aluminosilicate bricks, fibre glass and nichrome respectively. Temperature drop across aluminosilicate bricks and fibre glass were 168 and 299 K, respectively. Peak temperatures were attained in 9.0, 35.8, 108.6, 174.6 and 218.3 minutes at 0, 25.0, 50.0, 85.0 and 100.0% loading respectively. Production time and efficiency were 5 hours and 80% respectively. X-ray diffraction pattern showed that the major part of the AC produced were amorphous with small amount of crystalline matter. Scanning electron microscope at 1 hour activation showed a few micropores with not well- developed pores of sizes 0.58 nm, while well-formed elliptical micropores of sizes 1.8 nm were observed at 3 hours activation time. There is no significant difference ($\alpha_{0.05}$) between experimental pore sizes of 1.8 and standards for 2 nm.

A batch reactor, suitable for the production of Activated Carbon from coconut shell was developed.

Keywords: Batch reactor, activated carbon, coconut shell, reactor efficiency.
Word count: 451

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CERTIFICATION

I certify that this work was carried out by Mr. J. O. Otulana in the Department of Mechanical Engineering, University of Ibadan.

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TABLE CONTENTS

LIST OF TABLES

LIST OF FIGURES

CHAPTER ONE
INTRODUCTION

1.1 Background

Recently, carbon has been one of the magnificent elements which have revolutionized material science. From carbon we obtain the best porous absorber (activated carbon) with excellent properties for large spectrum of industrial applications. Activated Carbon (AC) is the common term used for a group of absorbing substances of crystalline form, having a large internal pore structures that make the carbon more absorbent (Strand, 2001). These properties are obtained when a char is subjected to controlled gasification by oxidizing gases, or when a raw material impregnated with dehydrating agents is subjected to carbonization. Generally, the raw materials for the production of AC are those with high carbon but low inorganic contents such as wood, lignite, peat and coal (Lua and Guo, 2000). Besides that, lot of agricultural waste and by product have been successfully converted to AC for examples macadamia nutshell (Ahmadpour and Do, 1996), olive stones (Khalili, et al., 2001), peach stones (Arriagada, et al., 1997) coconut shell (Kirubakaran et al., 1991), pistachio shell (Abe et al., 1990), saw dust (Xiongzun et al., 1986), walnut shell (Khan et al., 1985) and tropical wood (Maniatis and Nurmala, 1992).

Activated carbon has a wide range of application such as in water treatment, removal of oil from effluent in petroleum refining, removal of odour and colour in food and beverage industry, adsorption of dyes in textile industry, decolourization agent in the sugar industry, adsorption of gold and silver in the mining industry and in removal of impurities the chemical and pharmaceutical industry (Marsh et al. 1997). To meet her demand, Nigeria, relies on importation of AC as shown in Table 1.1. Between the year 2006 and 2014, the amount spent on importation of activated carbon and its product increased from 6% (₦ 570,774,368 in 2006) to 20% (₦ 1,944,360,008 in 2014) . Between this periods, the amount spent on importation of AC alone, increased from 3% (₦ 865,833,368) to 41% (₦ 1,214,644,908). This further collaborate the high demand of AC in industries in Nigeria.

Table 1: ACTIVATED CARBON IMPORTATION FIGURE

Year	Activated carbon ($)	Activated net mineral Product ($)	Total ($)	Total ₦
2006	273,998	1,532,250	1,806,248	570,774,368
2007	423,420	1,365,605	1,789,025	565,331,900
2008	1,099,173	426,831	1,526,004	482,217,264
2009	523,925	3,600,927	4,124,852	1,303,453,232
2010	289,844	17,524393	17,814,237	5,629,298,892
2011	305,244	2,439,769	2,745,014	867,424,424
2012	1,355,959	3,602,659	4,958,618	1,566,923,288
2013	1,179,997	6,108,854	7,288,851	2,303,276,916
2014	3,843,813	2,309,221	6,153,038	1,944,360,008

(Source: United Nations Commodity Trade Statistics Database 2017)

Table 1.2 Annual Coconut Production Quantities in Nigeria

Year	Quantity (Tons)	Year	Quantity (Tons)	Year	Quantity (Tons)
1995	149,000	2002	171,000	2009	234,000
1996	151,000	2003	182,000	2010	194,350
1997	152,000	2004	195,000	2011	211,050
1998	152,000	2005	209,000	2012	212,000
1999	158,000	2006	225,000	2013	266,045
2000	160,000	2007	225,200	2014	267,520
2001	212,000	2008	225,500		

(Source: United Nations Commodity Trade Statistics Database 2017)

There are abundant potential raw materials resources for the production of the activated carbon, in Nigeria (Tables 1.2 and 1.3). The mean annual production quantities of coconut increased from 4.3% (between 1995 and 2005), to 6% (2006 and 2014), the annual production quantities of coconut increased from 4% in 1995 to 6% in 2014 . Between the years 1995 and 2014, the net coconut production quantities are 3,951,665 tons. This statistics indicates the abundance of coconut shell as precursor for AC production.

Table 1.3 Annual Palm Kernel Production Quantities in Nigeria

Year	Quantity (Tons)	Year	Quantity (Tons)	Year	Quantity (Tons)
1995	7,800,000	2002	8,500,000	2009	8,500,000
1996	7,750,000	2003	8,700,000	2010	8,000,350
1997	7,750,000	2004	8,600,000	2011	8,000,050
1998	7,800,000	2005	8,500,000	2012	8,100,000
1999	8,000,000	2006	8,500,000	2013	8,000,000
2000	8,000,000	2007	8,300,000	2014	7,976,213
2001	8,200,000	2008	8,500,00		

(Source: United Nations Commodity Trade Statistics Database 2017)

A wide variety of furnaces and kilns are used for production of activated carbon (carbonization and activation), including rotary kilns fired directly or indirectly, multiple-hearth furnaces, fluidized-bed reactors, and vertical retorts (Marsh et al, 1997). Although several types of furnaces are described in the literature, three are most commonly used by producers of activated carbon; rotary kilns, multiple hearth furnaces and fluidized bed furnaces

Rotary kilns are the most popular among the producers because in general terms, they produce activated carbon with a more controlled porous structure. The residence time is greater than other furnaces, and because their length is large, several burners and gas supply lines (natural gas, propone or the gases from the carbonization unit) are distributed along the kiln in order to improve the control of the temperature and the temperature profiles (Rodnguez-Reinoso et al 1997). Multiple hearth furnaces are used as activating furnaces. There is a central vertical axis moving the rotary arms of the stationary floor on each stage, and the material is transported downward failing alternatively near to the axis on one stage and near to the wall at the next stages. This type of furnace is also used for regeneration of spent activated carbon (Rodnguez-Reinoso et al., 1997). Fluidized bed furnaces are characterized by a good heat and mass transfer, the activating gases being in excellent contact with the carbon particles. This means that the residence time is the shortest of all activating furnaces. Industry uses fluidized, furnaces that are operated continually. The main problem with this type of furnace is that reaction is extensive and there is much external burning of the particles with the end result of activated carbon of relatively poor quality, mainly used for powdered activated carbon (Rodnguez-Reinoso et al., 1997). The indirectly fired rotary kiln, for example, is relatively large and expensive, but avoids a large discharge of inert gases to the afterburner. The fluidized-bed reactor is extremely compact, but is generally used only for the activation of abrasion-resistant raw materials because of the attrition that occurs during fluidization. Carbonization and activation used to be carried out separately,

In Nigeria, annealing furnaces have been adapted for the production of activated carbon owning to the non availability of any of the three furnaces above. The heating element of this adapted furnace are damaged due to short- circuiting when they come in contact with oxidizing fumes in the furnace heating chamber during activation process. The output from this annealing furnace is low due to the absence of inert gas inlet which allows oxidation of some of carbonised materials into ash.

In this research a reactor / furnace would be designed and fabricated specifically for the production of activated carbon from coconut shell.

1.2 Problem Statement

Annealing furnaces have been adapted in Nigeria for the production of activated carbon. This has caused damage of heating elements due to reaction with activation fumes in the furnace. The absence of inert gas tuyere in annealing furnaces has equally affected the efficiency of the furnace in producing AC. In view of these, there is urgent demand for developing a reactor specifically for AC production while preventing damage of heating elements and incorporation of inert gas inlet, through efficient, effective and careful selection/development reactors' materials. It is therefore pertinent to critically address the following:

i. Short-circuiting of heating elements and absence of inert gases inlet have been identified as major challenges associated with reactors used in AC production in Nigeria.
ii. Determine the optimal production condition for activated carbon production.
iii. The production time is prolonged because the carbonization and activation processes are carried out in separate units.
iv. Nigeria spends a huge amount annually in importing activated carbon (Table 1.1)
v. The waste material of coconut shells, that cause environmental effect, can be used as raw material to produce activated carbon.

1.3 Objectives

The main aim of this study is to develop a batch reactor that eliminates the problem of short circuiting and produce activated carbon at reduced time. Specifically the following objectives will be pursed:

i. To develop and fabricate a reactor for the production of activated carbon using locally sourced materials.
ii. To eliminate the damage of heating element resulting from short circuiting caused by activating fumes.
iii. To evaluate the performance of the reactor
    a) Quality of activated carbon
    b) AC production time

1.4 Scope of the Study

The study is limited to the:

i. Development a pilot scale reactor;
    a. with 32-1200 °C temperature range
    b. with a throughput of 800 g
ii. Determination of relevant design parameters (refractory and insulator thicknesses, thermal stress and strain, heat loss and heating rate).
iii. Production of activated carbon from coconut shells.
iv. Validation of reactor by chemical activation using potassium hydroxide.
v. Characterisation of the AC (using scanning electron micrograph and x-ray diffraction)

1.5 Justification

i. The enormous financial loss in importing AC and environmental treat poised by unused coconut shells are strong motivations for this research.
ii. The waste material of coconut shells, that cause environmental effect, can be used as raw material to produce activated carbon, which has found application in many industrial fields such as water purification and treatment.

iii. The strong market position held by AC relates to their unique properties and low cost compared with that of possible competitive inorganic adsorbents like zeolites.

iv. Liquid phase applications represent the largest outlet for AC. In these applications, AC is used in the purification of a variety of liquid streams, such as those used in water treatment and the processing of food, beverages and pharmaceuticals.

v. This demand can be satisfied considering the large number of raw material available for the production of AC, the variety of activation processes described, and the available forms of AC. Thus, the continuous research has to be implemented to develop the high quality of AC for specific uses.

CHAPTER TWO
LITERATURE REVIEW

2.1 Activated Carbon

Activated carbon is a microcrystalline form of carbon with very high porosity and surface area. It is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions. (Odesola and Daramola, 2009). Benaddi (2000) also stated that AC is predominantly an amorphous solid with a large internal surface area and pore volume. While according to Norlia Baharun (1999) AC is an organic material that has an essentially graphitic structure. Marsh (1989) defined AC as a porous carbon material, usually chars, which have been subjected to reaction with gases during or after carbonization in order to increase porosity. AC is distinguished from elemental carbon by the removal of all non-carbon impurities and the oxidation of the carbon surface (Benaddi, 2000).

From all the definition, it can be summarized that AC is black, amorphous solid containing major portion of fixed carbon content and other materials such as ash, water vapour and volatile matters in smaller percentage. The main features common to all AC are; graphite like planes which show varying degrees of disorientation and the resulting spaces between these planes which constitute porosity, and the unit built of condensed aromatic rings are referred to as Basic Structure Units (BSU). AC contain physical characteristic such as internal surface area and pore volume. The large surface area results in a high capacity for absorbing chemicals from gases or liquids. The adsorptive property stems from the extensive internal pore structure that develops during the activation process.

Activated carbons with highly developed surface area are widely used in a variety of industries for applications which include separation/purification of liquids and gases, removal of toxic substances, as catalysts and catalyst support (Moon and Shim, 2006; Fuente et al., 2001). With the advancement in technology, the applications of activated carbons keep expanding, with newer applications such as super-capacitors, electrodes, gas storage, and so on (Yuan and Zhang, 2006; Oda and Nakagawa, 2003; Biloé et al., 2002). Because activated carbons have well-developed pore structures and high internal surface area, they have been employed in a wide number of applications on an industrial scale, including technologies for the purification of gases; the removal of organic pollutants from water (i.e., purification of drinking water and wastewater); used as a catalyst or a catalyst support in the catalytic processes and used as electrode materials in electrochemical devices and processes over the last few decades (Gurrath et al., 2000; Henning and Schafer, 1993; Mazyck and Cannon, 2000;Walker and Weatherley, 2000). Thus activated carbon has played important roles in the chemical, pharmaceutical and food industries.

2.1.1 Use of different precursors

Activated carbons can be prepared from a variety of precursors with high carbon content and low levels of inorganic compounds. Various carbonaceous materials such as coal, lignite, nutshells, wood, and peat are used in the production of commercial activated carbon. However, abundance and availability of agricultural by-products make them good sources of raw materials for activated carbons. Harvesting and processing of various agricultural crops result in considerable quantities of agricultural by-products. Such agricultural by-products are usually inexpensive, for which the effective utilization has been desired. Many researchers have made efforts in preparing activated carbons from agricultural by-products such as apricot stones, sugarcane bagasse, nutshells, forest residues and tobacco stems coconut shell, oil-palm shell, (Cigdem et al., 2006 ; Ioannidou and Zabaniotou, 2007; Li et al., 2008; Zhang *et* al., 2004 Odesola and Daramola 2009).

The commercial activated carbons on the market today are the result of continuous and intensive research and development toward optimization of application. The economics and availability of parent materials are as important as extents of available internal pore volumes (surface areas) associated with the right kind of porosity and surface chemistry. The availability of activated carbon for industrial use has much to do with accessing resources, renewing resources and processing to rigid specifications to control specific industrial applications. Only a handful of resources are used for activated carbon production, including coals of several rank, peat (not quite a coal) as well as woods, fruit stones and nutshells, as with coconut shells, as well as some synthetic organic polymers. A key element is the reliability and constancy of the resource. Manufacturing processes are so finely tuned that variations in the quality of the resource are unacceptable. Of the other resources which frequentiy come up for consideration, are banana skins, straw, woodcuttings, casings from coffee beans and many other organic waste materials. Collection from separated areas, transportation, bulk availability and seasonal variations in quality and availability are reasons why these resources are not used.

Gonzalez et al. (1995a) investigated the behaviour of six different lignocellulosic materials and compared and contrasted differences in porosities of the resulting activated carbons. The precursors selected were coconut shell, almond shell and peach, plum, olive and cherry stones. Carbonizations were carried out in nitrogen (80 cm^3 min^{-1}) at 600 °C for 1 h, followed by activation in steam at 800 °C for various times to produce various degrees of activation. The results for the 600 °C chars (Table 2.1) indicate that the coconut shell, in terms of density, mesopore and macropore volumes ($V_{Hg}(cm^3g^{-1})$) stands out as being very different from the other five precursors. Similarly, the micropore volumes, as calculated from the adsorption data, indicate the highest micropore volumes for the coconut shell, values around 25% lower being found for plum and peach stones. Much larger differences were found for mesoporosity and macroporosity, ranging from 0.1 to 0.6 cm^3 g^{-1} for coconut shell and olive stones respectively.

Table 2.1 Characteristics of chars obtained at 600 °C from different lignocellulosic precursors

Raw material	**Mercury density (gcm^{-3})**	V_{Hg} (cm^3g^{-1})	**Activation rate ($mgg^{-1}h^{-1}$)**
Coconut	1.16	0.07	11
Almond	0.99	0.19	29
Peach	0.99	0.18	12
Plum	0.89	0.29	19
Olive	0.91	0.24	11
Cherry	0.86	0.37	10

Source : Gonzalez *et al*. (1995)

2.1.2 Characteristics and properties of activated carbon

The raw material for an activated carbon plays a major part in determining the ability of the final product to adsorb certain molecular species. Activated carbons produced from coconut shells exhibit a predominance of micropores, while coal based carbons have a wider range of transitional pores. The development of an extensive macropore structure is found when either peat or wood is used as the raw material (Odesola and Daramola 2009). Guo and Lua (2003) mentioned that the characteristics of activated carbon depend on the physical and chemical properties of the raw materials as well as activation method used. Physical properties of AC, such as ash content and moisture content can affect the use of a granular AC and render them either suitable or unsuitable for specific applications. While the specific surface area of activated carbon and surface chemistry is classified as chemical properties. Furthermore, the porous structure of activated carbon also can be characterized by various techniques such as adsorption of gases (N_2, Ar, Kr, CO_2) or vapours (benzene, water), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM).

2.1.3 Pore structure

A pore is a class of void which is connected to the external surface of a solid and will allow the passage of fluids into, out of, or through a material. Figure 2.1 shows the type of pores. Marsh (1989) claim that, in the scientific literature on porous solids the terms open pore and closed pore are used, the former a pore which is not so connected. Transport pores are those pores in which a concentration gradient exists during steady state or time-independent fluid flow through the material. Blind pores are connected to transport pores by a single opening so that in them concentration gradients and hence fluid flow only occur during unsteady state or time independent flow. Differences in pore sizes affect the capacity for molecules of different shapes and sizes, and this is one of the criteria by which carbons are selected for a specific application.

Porosity is classified by International Union of Pure and Applied Chemistry (IUPAC) into three different groups of pore sizes (Guo and Lua, 2003); micropores- width less than 2 nm, mesopores- width between 2 and 50 nm and macropores- width greater than 50 nm.

Understanding how the porosity is developed will make possible the control and prediction of the nature and extent of porosity in activated carbons and consequently the control and prediction of their performance during application, (Wei- Li et al., 2008). Activated carbon, samples derived from carbonized chars obtained at high carbonization temperature had higher micro pore volume as compared to those prepared from carbonized chars obtained at low carbonization temperature, (Daud et al., 2000).

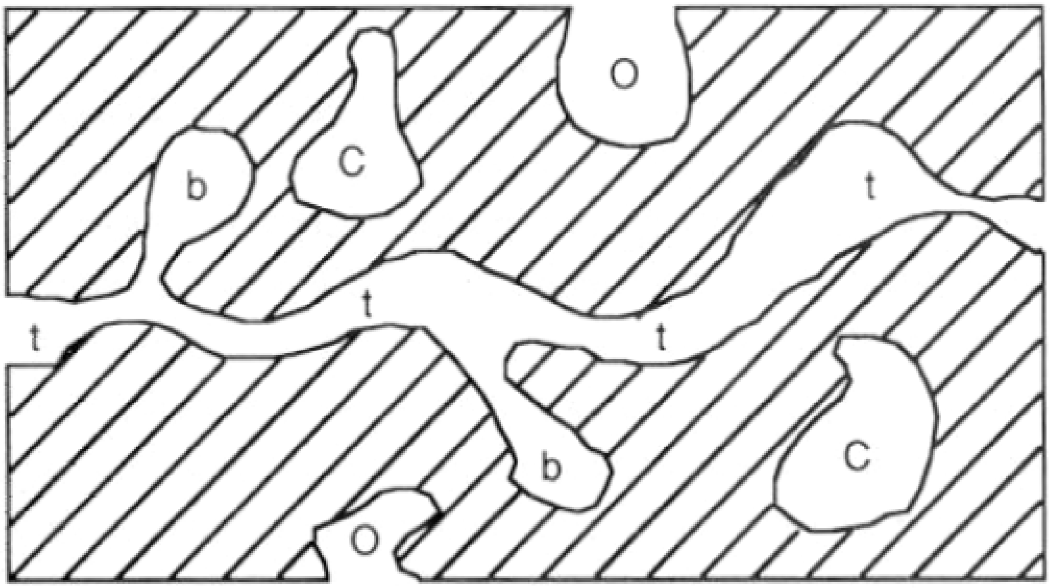

Figure 2.1 Different Types of Porosity in a Porous Solid

O-open pores; C- closed pores; t-transport pores; b-blind pores

Source: Marsh, (1989)

2.1.4 The microstructure of activated carbon

Activated carbon shows a very disordered microcrystalline structure in which graphitic micro crystals are randomly oriented (Gomez-Serrano et al., 2005). The term turbostratic has been used to describe a graphite -like structure with random translation of layer planes along the axis and rotation of layer planes about the c-axis. The interlayer spacing in a turbostratic structure is 0.344 nm, which is larger than the spacing in a graphite single crystal, 0.3354 nm. Various microporous models have been proposed based on High-Resolution Transmission Electron Microscopy (HRTEM) studies (McEnaney , 2002). One of the examples of microporous structure for activated carbon is shown in Figure 2.2. An essential feature of all of the models is a twisted network of carbon layer planes, cross linked by an extended network of aliphatic carbon. The layer planes are defective, for example, they may contain vacancies and hetero-elements. The latter are bound to the edges of the layer planes as simple functional groups. e.g., -OH, C-O -C--O -C-. There is a continuum of interlayer spaces ranging from the turbostratic value up to sizes that can form micropores. Thus, micropores are an inherent feature of the microstructure of activated carbon. Another feature of the microstructure is constrictions in the microporous network that control access to much of the micropore space (McEnaney, 2002). Also entrances to micropores may be blocked by functional groups attached to the edges of layer planes and by carbon deposits formed by thermal cracking of volatiles released during carbonization. Thus, the micropores in activated carbons are incorporated into an aperture cavity network. Constrictions and blockages in the microporous network cause activated diffusion effects at low adsorption temperatures when the adsorptive has insufficient kinetic energy to penetrate the pore space completely (McEnaney, 2002).

Figure2.2 Microstructure of a Microporous Carbon

Source: McEnaney, (2002)

2.2 Processing of Activated Carbon

In principle, the methods for preparing an activated carbon can be divided into two categories: physical activation and chemical activation (Wei-Li et al., 2008). As would be expected, chemical methods use chemicals for activation, whereas physical processes use gases (usually carbon dioxide and air), vapours (steam), or mixtures of gases and vapours.

Physical or thermal activation is a two step process: carbonization followed by activation. Carbonization in an essentially inert atmosphere eliminates the bulk of the volatile matter, enriches carbon content, and leads to some increase in porosity. The carbonization steps serves to modify the pore structure of the precursor. The activation stage is a controlled gasification process in the presence of activating agents such as steam, CO_2, or their mixture. The activation process serves to develop further pore structure and increase surface area.

2.2.1 Chemical activation

The wet-chemical process is generally employed to convert uncarbonized cellulosic material, primarily wood, into activated carbon. This process is about impregnation with chemical (such as phosphoric acid or potassium hydroxide, sodium hydroxide or zinc chloride, sodium carbonate, sodium and calcium hydroxide, and the chloride salts of magnesium, calcium, ferric iron, and aluminium), followed by carbonization at temperature in the range 450-900 °C. (McDougall, 1991) In the chemical activation process the two steps i.e. carbonization and activation, are carried out simultaneously, with the precursor being mixed with chemical activating agents, as dehydrating agents and oxidants (McDougall, 1991). The most popular activating agent is phosphoric acid. The process using sawdust as the starting material involves mixing of the raw material and the dehydrating agent into a paste. The paste is then dried and carbonized in some type of kiln, usually a rotary kiln, at between 200 and 650 °C (McDougall, 1991). Upon carbonization, the impregnated chemicals dehydrate the raw material, resulting in charring and aromatization of the carbon skeleton, with the concomitant creation of a porous structure and extended surface area. This procedure yields a powder product. If a granular product is required, granular materials are simply impregnated with the activating agent, and the same general procedure as that described above is followed. However, these granular products are generally soft (unless they are manufactured from the powder after suitable pelletization), which limits their application (McDougall, 1991). After carbonization of the paste, the activating agents are usually recovered and recycled for economic reasons. All the commonly used chemical activators impose certain constraints on the overall activation process and the equipment employed, or on the quality of the product. Mixtures of sulphuric acid and wood cannot be heated above 200 °C, and the product, after the sulphuric acid has been recovered by leaching, has adsorptive properties only when wet. Activation with phosphoric acid requires temperatures in the range 375 to 500 °C, and the reagent is readily recovered by leaching with water. However, corrosion of the equipment is a major problem. Activation with zinc chloride occurs in the temperature range 550 to 650 °C and, although most of the zinc chloride is recovered by leaching with dilute hydrochloric acid, problems may arise in the application of the product because it retains traces of zinc salts. The activated carbon produced with phosphoric acid and zinc chloride is dried with little, if any, loss of adsorptive capacity. The activity of the product can be controlled by alteration of the proportion of raw material to chemical reagent. For phosphoric acid, the proportion is usually between 1:0, 5 and 1:4. The activity, as measured by the number of pores created, increases as the proportion of the chemical activator increases, and is also affected by the temperature and residence time in the kiln (McDougall, 1991).

Chemical activation offers several advantages since it is carried out in a single step, combining carbonization and activation, performed at lower temperatures and therefore resulting in the development of a better porous structure. (Ioannidou and Zabaniotu, 2007)

2.2.2 Physical activation

Physical thermal activation is a two step process: carbonization followed by activation. Carbonization in an essentially inert atmosphere eliminates the bulk of the volatile matter, enriches carbon content, and leads to some increase in porosity. The carbonization steps serves to modify the pore structure of the precursor. It involves carbonization of a carbonaceous material followed by the activation of the resulting char at elevated temperature in the presence of suitable oxidizing gases such as carbon dioxide, steam or air. The main purpose of carbonization is to reduce the volatile content of the source material (a fixed carbon content of 80% or higher is desirable) to convert it to a suitable form for activation (Balci et al., 1992). Rearrangement of the carbon atoms into graphitic-like structures also occurs during carbonization. Carbonization temperature range between 400 and 850 °C, and sometimes reaches 1000 °C, and activation temperature range between 600 and 900 °C. (Wei et al., 2008) The first step in the production of activated carbon by the thermal route is carbonization, which is the formation of a char from the source material. Carbonization is generally accomplished by heating of the source material-as lumps or pre-sized material, or in molded form, e.g. as briquettes in an inert atmosphere such as flue gas to a temperature that must not exceed 700 °C so that dehydration and devolatilization of many of the carbon atoms can occur in a controlled manner. The activation stage is a controlled gasification process in the presence of activating agents such as steam, CO_2, or their mixture. The activation process serves to develop further pore structure and increase surface area. Physical activation is carried out most frequently by burning off some of the raw carbon in an oxidizing environment to create mesopores.

Activation is the process by which the carbonized product develops an extended surface area and a porous structure of molecular dimensions. This step is generally conducted at temperatures between 800 and 1100 °C in the presence of suitable oxidizing agent such as steam, air, carbon dioxide, or any mixture of these gases. The activation gas is usually CO_2, since it is clean, easy to handle and it facilitates control of the activation process due to the slow reaction rate at temperatures around 800 °C.

The active oxygen in the activating agent burns away the more reactive components of the carbon skeleton as carbon-monoxide and carbon dioxide, depending on the oxidizing agent employed (McDougall, 1991).

$$C + O_2 \rightarrow CO_2 \qquad (1)$$

$$CO_2 + C \rightarrow 2CO \qquad (2)$$

2.2.3 Method of carbonization

a. PIT METHOD

The shells are burned in a limited supply of air so that they do not burn away to ash but are only carbonized. The shells are often burned in pits in the ground although large steel and brickwork kilns do exist (coconut board.nich.in/charcoal.htm 2013).

b. DESTRUCTIVE DISTILLATION

When coconut shells are heated in a retort to a sufficiently high temperature, they are broken down in the absence of air into a number of products. Child (1939, as cited in Brian and Ashman, 1975) used laboratory and semi commercial methods and found that 454 kg of shells yielded 153 kg of charcoal, 171 kg of pyroligenuous acid, 27 kg of settle tar and 86 kg of incondensable gases. The yield and indeed the composition of the products are likely to vary considerably with the maturity of the coconut from which the shells are derived (Brian and Ashman, 1975).

c. DRUM METHOD

Drum kiln is used for carbonization of shells. The drum consists of three sets of six 1" diameter holes provided at its bottom, middle and upper layers and a lid. A detachable chimney is provided which is installed on the lid after closing the drum (coconut board.nich.in/charcoal. html 2013).

2.2.4 Activation methods.

According to Smisek and Cernyl, (1970), gasification of the carbonaceous material by steam and carbon dioxide occurs in accordance with the endothermic reactions shown in equations (3) and (4):

$$C + H_2O \rightleftharpoons CO + H_2 \; (M_f = 29 \text{ kcaVmol}) \qquad (3)$$

$$C + CO_2 \rightleftharpoons 2CO \; (M_f = 39 \text{ kcaVmol}) \qquad (4)$$

Because the reactions of carbon with steam and carbon dioxide are endothermic, the activation process lends itself to accurate control of the conditions in the kiln. External heating is required to drive reactions (3) and (4), and to maintain the reaction temperatures.

The reaction of water vapour with carbon is accompanied by the secondary reaction of water-gas formation, which is catalysed by the carbon surface as shown in equation (5):

$$CO + H_2O. \rightleftharpoons CO_2 + H_2 \; (M_f = -10 \text{ kcaVmol}) \qquad (5)$$

However, as shown in equation (6), the reaction of carbon with air (oxygen) is extremely exothermic:

$$C + O_2 \rightleftharpoons CO_2 \quad (M_f = -97 \text{ kcaVmol} \qquad (6)$$

This reaction is difficult to control, and excessive burnoff of the external carbon surface can easily occur, resulting in a decrease in average particle size, which obviously reduces the yield of final product. The reaction of steam with carbon is catalysed by certain chemicals (e.g. the oxides and carbonates of alkali metals, iron, copper, and other metals), and some commercial operations use these chemicals as catalysts. Therefore, as combustion proceeds, preferential etching occurs, which results in the development of a large internal surface area and the creation of a pore structure (McDougall, 1991).

The term burn-off is used to denote the degree of activation, which is the loss of char (in percentage by mass) that is allowed to occur. The burn-off (B) and activation yield (Y) are related as indicated in equation (7):

$$B = 100 - Y \qquad (7)$$

2.2.5 One-step and two-step activations

Rodriguez-Reinoso et al. (1984) prepared activated carbons from almond shells and olive stones using (i) the conventional carbonization (850 °C) followed by activation at 825 °C in carbon dioxide and (ii) a single (direct) activation step in carbon dioxide from room temperature to 825 or 850 °C. Both types of activation produce a very similar yield, this means that the direct reaction of the raw material with CO_2 during the heating from room temperature to reaction temperature is not important and does not imply a noticeable activation; in other words, its effect is similar to carbonization under nitrogen. The volume of micropores and the surface area of the resulting activated carbons (for a common yield) are relatively similar, but slightly larger for carbons prepared by direct activation. Similar results were found for other lignocellulosic materials. This result shows that it is possible to produce activated carbon without previous carbonization of the precursor, although the two-stage process is more common in industrial manufacture of activated carbon.

2.3 Applications of Activated Carbon

It is impossible, to describe adequately the large number of applications allocated to AC in a single section. The following sections indicate the wide-ranging scenarios for AC.

2.3.1 Gas-phase applications

AC has a large number of gas- phase applications such as; personal protection, cigarette filters, industrial gas masks, effluent gas purification, industrial off-gas purification, removal of SO_2, H_2S, CS_2, etc, petroleum refineries, sewage and geothermal plants, Vinyl Chloride Monomer (VCM) plants and solvent recovery in general, separation of gas mixtures using carbon molecular sieves (CMS), organic and inorganic process catalysis, both as a support material and as a catalyst, adsorption of radionuclides, natural gas storage and purification, automobile/gasoline recovery, and odour control.

Rodriguez-Reinoso and Sepulveda-Escribano (2001) and Rodriguez-Reinoso (2002) reviewed the applications of porous carbons in adsorptions and catalysis. For gas-phase applications, carbon adsorbents, generally, are used in the form of hard granules, hard pellets, fibers, cloths and monoliths, because these avoid an excessive pressure drop when the gas to be treated passes through the adsorbent bed. They usually have a well- developed microporosity to provide a high adsorptive capacity and selectivity for gases and organic vapours. Their surface areas range between 1000 and 2000 m^2 and have a high adsorptive capacity per unit volume, high retention capacity, high preferential adsorption of gases in the presence of moisture, low resistance to gas flow and complete release of adsorbates at increasing temperatures and decreasing pressures.

The humidity of the gas to be treated is an important parameter in gas-phase applications of AC. Although the interaction between the polar water molecule and the relatively non-polar carbon surface is very weak, AC can adsorb appreciable amounts of water when the relative humidity of the gas stream is high. The organic molecules that are usually to be retained interact more strongly than water with the carbon surface and thus can easily displace the water molecules from the surface.

2.3.1.1 Gas purification

AC in different forms (granular, extruded, fibre or cloth) is used for production of pure gases in the chemical industry, to reduce pollutant gases to very low concentrations in a single stage, in protection against poison gases, in air conditioning, for removal of oil from compressed air, etc. The actual processes, using AC, include hydrogen sulphide removal from sour natural gas, removal of sulphur dioxide and nitrous oxides from flue gas, gasoline vapour recovery in gasoline loading facilities, chlorofluorocarbon recovery in foam blowing, removal of mercury vapour from air, hydrogen and other gas streams, cigarette filters, military uses, nuclear uses and automotive evaporation control systems.

The use of carbonaceous adsorbents for gas purification, by H_2S removal, benefits from two important properties of these materials: i) AC is an effective catalyst for the direct oxidation of H_2S with air and ii) It is a powerful adsorbent for sulphur oxides.

The combination of these two properties facilitates further steps in the industrial process, such as the regeneration of the adsorbent and the extraction of the sulphur compounds.

Carbons impregnated with caustic materials such as KOH and NaOH are widely used to control odours of H_2S and organic substance in sewage treatment plants. However, there is a risk of bed self-ignition, likely due to the high heats of reaction. Alternatively, the adsorption capacity for H_2S can be improved by impregnating the AC with compounds such as KI or $KMn0_4$, which promote the oxidation to elemental sulphur. In both cases, the impregnation process decreases the adsorption capacity, as the microporosity may be blocked.

Bandosz (1999) reported that the combination of a suitable Pore Size Diameter (PSD) and the presence of polar groups on the carbon surface (containing oxygen and phosphorous) contributes to the process of H_2S retention. These results have been explained on the basis of the adsorption of water on surface groups, which enhances the oxidation of H_2S to sulphur or even to sulphuric acid. Boudou et al. (2003) introduced nitrogen groups into a viscose-based Activated Carbon Cloth(ACC) by reaction with ammonia/air at 300 °C and by reaction with anmionia/steam at 800 °C. Extensive surface characterisations were carried out. Ammonia/steam treatment was more effective for the adsorption of H_2S or SO_2 by enhancing the microporosity and by modifying the distribution of the surface oxygen complexes.

The first process for the simultaneous dry removal of SO_2 and nitrogen oxides (NO_2) was developed in the 1960s by the Bergbau Forschung Co. (now DMT) in Germany, by using a two-stage moving bed with activated coke (Knoblauch et al., 1981). The flue gas is denitrified in the first bed by reduction of NO_2 with ammonia Selective Catalytic Reduction (SCR) and then de-sulphurized in the second bed. Sulphur dioxide is retained as H_2SO_4 on the activated coke, and it is recovered as SO_2 by heating at 400-550 °C. Simultaneously, the activated coke is regenerated and recycled to the first bed.

Mochida et al. (2000) reviewed the applicability of Activated Carbon Fibres (ACFs), for the removal of SO_2 and NO_2. The highest activity for SO_2 removal in the presence of water, at 25°C, was achieved with pitch- based ACFs and this activity could also be improved by heat treatment of the ACFs in nitrogen at temperatures ranging from 600 to 900 °C. For NO_2 removal by reaction with ammonia, it was shown that the activity at room temperature of the ACFs was markedly increased if the ACFs were previously oxidized to form surface oxygen complexes.

Volatile organic compounds (VOCs) are a group of low-boiling-point compounds, with different chemical properties and are generally toxic, which are produced in the petrochemical industry, food processing, wastewater treatment and electronic industries, etc. Benzene, toluene, xylenes, hexane, cyclohexene, thiophene, diethylamine, acetone, acetaldehyde and methyl ethyl ketone are examples of VOCs. They can be removed by catalytic combustion, but the low concentrations in which they are present in the gas streams to be treated make it necessary to add energy to keep the combustion at the operating temperature. To overcome this, a process by which the VOCs are concentrated by adsorption, and then desorbed and burnt by catalytic combustion, has been proposed (Meeyoo et al., 1998).

VOCs can also be removed by adsorption processes (vapour recovery). In this case, ACCs are used in preference over granular activated carbon (GAC) because they are more easily contained, have faster adsorption kinetics and higher adsorption capacities, and can be regenerated, in situ, by electro-thermal methods (resistive heating). ACCs have been chemically modified by treatment with ammonia (to introduce basic nitrogen complexes), chlorine (to introduce polar — CI groups) and nitric acid (to introduce acidic oxygen complexes). In this way, the water vapour adsorption capacity can be tailored to obtain ACCs with enhanced adsorption of individual VOCs in the presence of humidity.

Some VOCs, as well as other air pollutants, are emitted from automotive vehicles. They can occur in the vehicle exhaust emissions, but also can be produced by evaporative emission from the fuel system. These evaporative emissions are fuel vapours, generated and released from the vehicle's fuel system, which depend on the interactions of the specific fuel in use, the fuel system characteristics and environmental factors.

The AC required for use in automotive applications must adsorb gasoline vapours efficiently, and also must be able to release them during the regeneration cycle. The regeneration conditions are mild, and so the adsorbent-adsorbate interaction must not be too strong. The most effective pores for this purpose lie in the mesopore range. Some of the automotive grade AC produced by Westvaco are WV-A 900 and WV-A 1100 (granular) and BAX 950, BAX 1100 and BAX 1500 (pelleted). They have Brunauer- Emmett -Teller (BET) surface areas from 1400 to 2000 m^2/g and apparent densities of 0.2-0.35 gcm^{-3}.

The fuel vapours evaporated from the carburettor or from the fuel tank are adsorbed on to AC (0.5-2.0 L) contained in a canister. Recovery of the fuel is carried out by drawing an air stream through the canister. The fuel desorbed is thus taken to the engine and mixed with air, to be burnt during normal operation. Other systems have been designed to capture fuel vapours emitted during vehicle refueling and when tank trucks are filled and drained.

Lillo-Rodenas et al. (2005), studied the adsorption of benzene and toluene of VOC on 10 AC, 2 commercial from wood, 1 from bituminous coal using steam activation, 5 from anthracite by chemical activation using NaOH and KOH and 2 from bituminous coal by chemical activation using NaOH and KOH. It is concluded that the most important property controlling adsorbate uptake is the narrow microporosity, <0.7 nm. Removal of oxygen surface functionality enhances the adsorption of the benzene and toluene which can reach as high 34 and 64 gg^{-1} for benzene and toluene, respectively, from low concentrations.

2.3.1.2. Separation of gas mixtures

Carbon Molecular Sieves (CMS), as their name suggests, are designed to separate gases in mixtures by sieving, a process controlled by PSD. CMS are widely used for the separation of air into nitrogen and oxygen. CMS can be prepared from a number of carbonaceous precursors and by several preparation methods, although those to be used industrially for the separation of air are generally prepared from AC, with a post-treatment (chemical vapour deposition) to narrow their porosity. Taking into account that the molecules to be separated have very similar sizes (kinetic diameters 0.346 nm for O_2 and 0.364 nm for N_2), the PSD in these materials has to be precisely tailored in the 0.3-0.4 nm range to within 0.02 nm.

Whereas zeolites adsorb nitrogen preferentially and produce an O_2-rich stream at the exit of the separation device, CMSs separate the components of air on a kinetic basis. Although the adsorption equilibrium isotherms for the two gases are nearly the same, oxygen is adsorbed faster than nitrogen.

Methane-carbon dioxide mixtures can also be separated using AC (Kyotani, 2000). There are three main applications where this process is of interest, including treatment of landfill gas, which contains about 50% CO_2 as an impurity, purification of natural gas , which can contain up to 12% CO_2, and tertiary oil recovery, where the effluent gas contains variable CO_2/CH_4 ratios. The industrial practice of Pressure Swing Adsorption (PSA) has become one of the most applied techniques for the separation of gas mixtures in general, and separation of nitrogen from oxygen in particular (Yang, 1987). The process for obtaining clean natural gas from landfill includes a first purification treatment with AC, followed by separation with CMS in a PSA unit. As for air, separation of methane from carbon dioxide is based on adsorption kinetics, the smaller CO_2 molecule (0.33 nm) being adsorbed faster than the slightly larger CH_4 molecule (0.40 nm).

2.3.1.3 Methane storage

The instability in oil markets and the increase in environmental concerns have stimulated the research for alternative fuels for transportation. Among them, natural gas has become a promising choice from both the economic and environmental points of view. On the one hand, it is a fuel of considerable natural abundance and therefore, commercially attractive. On the other hand, it consists mainly of methane (85-95%), higher hydrocarbons, nitrogen and carbon dioxide, and thus it has inherent clean burning characteristics and a less adverse effect on the environment, with the emission of lower levels of pollutants (hydrocarbons, sulphur and nitrogen oxides, etc.), per unit of energy provided. The main problem arising when natural gas is used as a fuel in mobile vehicles is its storage. Although it has higher hydrogen-to-carbon ratio than other fuels and consequently a greater energy per unit mass, it cannot be stored at the same density (Cook et al., 1999). In a first approach, natural gas can be conveniently stored on a large scale in liquid form (Liquefied Natural Gas (LNG)) at its normal boiling point (112 K) at atmospheric pressure. Under these conditions, the system will deliver 600 volumes of gas per volume of storage (transportation V/V). Although the delivery is very high, the energy density (defined as the heat of combustion per unit volume) is 23 MJ L^{-1} which is low compared to the values for diesel fuel (37 MJ L^{-1} or gasoline (32 MJ L^{-1}). Additionally, the cost of liquefaction, the special insulated vessels required and the potential fire hazard are such as to make it unsuitable for use on a small scale.

A second approach is the use of Compressed Natural Gas (CNG). There is a possible market worldwide for vehicles fueled by CNG at pressures up to 20 MPa and room temperature. The delivery is 220 V/V (220 volume units of gas at atmospheric pressure are compressed to one volume of storage container) (Parkyns et al., 1995) and has an energy density of less than 10 MJL^{-1} . For the same driving range, a CNG vehicle using these pressures requires a storage vessel at least three times the volume of a gasoline tank. Higher pressures would help to increase the energy density, but technical difficulties concerning the storage container make this approach infeasible. The size and cost of the cylinders needed for storage are considerable deterrents to the use of these systems on a wide scale, and the cost of providing the high-pressure facilities is also relatively high. Natural gas can also be stored if adsorbed on a suitable material such as AC, at relatively low pressure (Cook et al., 1999). Adsorbed Natural Gas (ANG) is stored at pressures ranging from 3.5 to 4.0 MPa, with the help of an adsorbent. The density of the natural gas in the adsorbed phase is much higher than in the gaseous phase, and this compensates for the space in the storage container occupied by the solid. If an appropriate adsorbent is used, it is even possible to reach the same delivery (per unit volume of storage) with natural gas adsorbed at relatively low pressures (3.5 MPa) as with natural gas compressed at much higher pressures (20 MPa). It is evident that the cost of the storage system and the potential risks of use will be considerably reduced, thus making more competitive the use of natural gas for transportation. Efforts have therefore been addressed to the search for a suitable porous material to further improve ANG storage volumetric energy density. The requirements for an adsorbent to have optimal storage capacity are well established, the most important being high microporosity and high density (Menon and Komameni, 1998). In this application methane, the main component of natural gas, is adsorbed at room temperature, higher than its critical temperature (191 K).

Adsorption of supercritical gases takes place predominantly in pores that are less than two or three molecular diameters in width (Chen et al., 1997). On increasing the pore width, the forces responsible for the adsorption decrease rapidly. Thus, pores larger than 2 nm (meso- and macropores) are not useful for the enhancement of methane storage, although they may be necessary for transport into and out of the micropores. To enhance the adsorption storage of methane, the fraction of micropores should be maximized, with no voids or macropore volume. However, this contribution has to come from pores with a width of around 0.76 nm (larger than the thickness of two methane molecules), in order to maximize the deliverability at ambient pressures. Macropore volume and void volume in a storage system (adsorbent-packed storage vessel) should be minimized. There are many adsorbents that could fit these requirements (high microporosity, high density and minimum inter particle space). AC, by virtue of their very high micropore volumes, have shown the greatest promise. Furthermore, they have other properties that could enhance methane storage. AC have low heats of adsorption that prevent excessive heating or cooling of the storage vessel during the adsorption-desorption cycles. Also, they are mainly hydrophobic, so the competition between water and methane for the adsorption sites is minimized. Moreover, the costs of highly AC are of the same order as those of the cheapest zeolites.

2.3.1.4 Solvent recovery

The main industrial method of recovering solvent vapours from air streams is adsorption by AC. This method has the advantage over water scrubbing that it can be applied to water-insoluble solvents; it has the advantage over condensation that the solvent content of the air can be reduced as much as desired. AC recovery systems are used in the paint, petrochemical, printing, rubber, synthetic fibre, paper, adhesives, metal fabrication, solvent manufacturing and coatings industries. Typical solvents recovered by AC are acetone, benzene, ethanol, ethyl ether, pentane, methylene chloride, tetrahydrofuran, toluene, xylene, chlorinated hydrocarbons and aromatics. An important use lies in the recovery of acetone from cellulose acetate spinning and film production. For every kilogram of acetate or triacetate produced, 3 or 4 kg each of acetic acid and solvent must be recovered to make the process economically viable.

2.3.2 Liquid-phase applications

The following list indicates the wide-ranging scenarios of liquid-phase applications allocated to for AC.

i. Drinking water availability, to improve taste, smell and colour including removal of chlorinated compounds and other VOCs.
ii. Improvements to ground water purity, contaminants coming from disused sites of heavy industries.
iii. Treatments of both industrial and municipal wastewater.
iv. Mining operations require feed water treatment, metallic ion adsorption (gold and other metals), adsorption of excess flotation reagents and adsorption of Natural Organic Materials (NOM).
v. Pharmaceutical processes, including purification of process water, use with fermentation broths and purification of many products.
vi. The food, beverage and oil industries for removal of small, colour and unacceptable tastes.
vii. The dry-cleaning industries require purification of solvents.
viii. The electroplating industries require purification of wastewaters containing Pb, Cr, etc.
ix. Household water purification, cleaning of aquaria and use in oven-extract hoods.
x. The sugar and sweetener industries need decolourization agents for the production of white sugar, etc.

Both GAC and powdered AC are used in liquid-phase applications, the former being used in continuous processes because they are capable of regeneration, whereas the latter are generally used in batch processes (after completion, the carbon is separated from the liquid and discarded or eluted). Liquid-phase applications require AC with a larger pore size than gas-phase ones, because of the need for rapid diffusion of the liquid to the interior of the carbon particles and because of the large size of many dissolved molecules to be retained. It is usual for AC for Liquid-phase applications to be prepared by chemical activation of wood, peat, lignite, etc. There are two types of liquid-phase applications for AC: odour, colour, or taste removal from a solution, and concentration or recovery of a solute from solution including water purification and sugar and sweetener decolourization as principal applications.

2.3.2.1 Water treatment

The application of AC in water treatment is mainly centered in the removal of pollutant organic compounds. These compounds can be classified in three different categories:NOM, synthetic organic compounds and by- products of chemical water treatment. NOM is mainly composed of residues of the metabolism of living things. These compounds produce bad tastes and odours, and also may constitute a source of infection. Among the synthetic organic compounds that can be present in water, one can find oil, benzene and toluene, phenols and chlorophenols, trichloromethane and carbon tetrachloride, detergents, pesticides, dyes and so on. Finally, trihalomethanes are the most important group of compounds that are to be found in water as a product of chemical treatments to disinfect water with chlorine: $CHCI_3$, $CHBrCl_2$, $CHBr_2Cl$ and $CHBr_4$. They are very strongly adsorbed on AC, and this is the reason for the increasing number of potable-water plants using AC as the polishing step.

The removal of water contaminants by active carbon is the major market for liquid-phase applications. AC is used both as a primary treatment, to render the effluent more amenable to other purification processes, and as the final tertiary stage in the purification of the effluent. Of the total water treatment market, about 50% is in drinking water, 40% in wastewater and the rest in ground water markets. Both powdered and GAC are used in water treatment. The tendency being toward use of the granular type because of its regeneration capability (Rodriguez-Reinoso, 2002).

When the powdered form of AC is used, it is added to the water as slurry with automatic feeders. After a suitable contact time, it is removed by clarification or filtration. Dosage rates of AC in taste and odour control depend on the type of carbon and the level of impurities in the water, but in general terms the dosage is low, and the carbon can last for up to one year. As a result, it is not usually economic to regenerate the carbon, and spent carbon is generally discarded. GAC is preferred when there is a persistent problem with taste and odour control, and it is also used in special filters and disposable cartridges in industrial, commercial and residential installations. GAC is used in gravity columns, through which water flows continuously for a set contact time. Contacting systems can be of the up-flow or down-flow type, the former adsorbing organic compounds, whereas the latter filters suspended solids in addition. In an up-flow system, replacement of the spent carbon is carried out from the bottom of the column, with addition of new carbon at the top, while the unit remains in operation. In a down-flow system that does not have pre-filtration, suspended solids may accumulate at the top of the bed, requiring periodic back-washing of the bed to relieve the pressure drop caused by the accumulated solids. This type of bed is operated in series or in parallel. As the carbon will be exhausted first at the top of the bed, it is necessary to remove the entire bed in order to replace the carbon.

2.3.2.2 Waste water treatment

Wastewater is treated in four stages.

1. The first stage, or pre-treatment, is carried out when the wastewater contains toxic or non-biodegradable compounds that can affect the subsequent biological treatments. Redox reactions, followed by precipitation and filtration are used to separate the metals contained in the wastewater. Ozone treatment followed by adsorption into GAC is used to eliminate high molecular weight organics, whereas light organics and ammonia are eliminated by air-stripping.
2. The secondary treatment involves the removal of suspended solids, non-soluble oil and floating material by treatment with lime or other chemicals, followed by nitration, as well as neutralization by addition of acids or bases.
3. Dissolved and colloidal organic compounds like proteins, sugars, starches and phenols are removed in the secondary treatment by biological oxidation. These two processes remove about 85% of the suspended solids and of the Biological Oxygen Demand (BOD) of wastewater. For many purposes, however, further purification is needed to satisfy stringent effluent regulations.
4. This treatment involves the removal of inorganic and organic compounds by adsorption. It results in an extremely high-purity effluent, where the BOD can be reduced by over 99%, to 1 mgL^{-1} (Marsh et al., 1997).

There are three possible locations in a wastewater treatment plant for treatments with AC. This can be used as an adsorbent after primary and secondary biological processes, it can be used as an independent physico-chemical treatment, or it can be added to biological aeration tanks and used as part of the secondary biological treatment. This last choice has been used effectively to obtain a high-quality effluent. The selection of an appropriate treatment depends on the nature and contaminant loading of the wastewater, the scale of operation, specific requirements for effluent purity and the cost of carbon regeneration compared with alternative available treatments. The use of tertiary AC processes on wastewater that has already undergone conventional secondary biological treatment results in a very high-purity effluent. These processes consist of packed beds of granular material arranged either for down-flow in series, for down flow in parallel, for up-flow in series, or in moving beds. The efficiency of a tertiary treatment depends on the consistent and efficient operation of the previous treatment. Changes in wastewater composition, large variations in flow and the presence of toxic material can all disrupt the biological oxidation process. One method of improving efficiency is to treat the influent waste streams with ozone prior to treatment with AC, as is widely done in Western Europe (Marsh et al., 1997).

AC is also used in the treatment of industrial wastewater to upgrade the water for reuse or to pre-treat effluents prior to discharge into municipal treatment plants, rivers and streams. Adsorption by AC may be used as the only treatment before biological treatment or as a tertiary process after biological treatment. AC is used to purify industrial wastewater, as it removes not only biodegradable organic compounds, but also chemicals that are not responsive to, or are toxic to, conventional biological treatments. These include pesticides, phenols, organic dyes and detergents.

AC is used to treat effluent wastes from chemical factories, rubber tread factories, fabric dyeing, fertilizer plants, pulp and paper mills, etc. AC systems are more flexible than biological ones as they can handle sudden fluctuations in the concentration of impurities, and the water purity can be controlled to meet specific requirements (Marsh et al., 1997).

2.3.2.3 Removal of oil from effluent water in petroleum refining.

Another important use for AC is the removal of oil from effluent water in petroleum refining, petrochemicals, metal extraction, detergent, margarine and soft fat manufacture and mineral extraction. The presence of oil in the effluent inhibits the biological treatment in sewage works. AC is also used to remove oil and organic material from recycled condensate in boilers, because water contaminated with oil would cause foaming or priming even in low-pressure boilers. In high-pressure boilers, where the feed water is de-ionized, the oil causes serious fouling in the ion-exchange resins.

2.3.2.4. Food and beverage processing

AC is used in food and beverage industries to remove colour or odour from products. Applications include the processing of fruit juices, honey, sugar, sweeteners, vegetable oils and fats, alcoholic beverages, soft drinks, yeast, maple, syrup etc (Marsh and Rodriguez-Reino, 2006).

2.3.2.5 Chemicals and pharmaceutical

By removing impurities in chemical processes, AC helps to control product quality; it is also used for the removal of toxic chemicals. AC is used to extract pharmaceuticals in processes involving fermentation. Antibiotics, vitamins and steroids are adsorbed into the AC and recovered by solvent extraction followed by distillation. Other uses include filters for the dialysis of poisons and drugs, felts for wounds, etc.

2.3.2.6 Adsorption of dyes

It is estimated that about 40,000 tonnes of dyes are not used but are discharged (lost) into wastewaters. This is out of a total production of about 450,000 tonnes. A large variety of dyestuffs is available with such names as acid, basic, reactive, direct, disperse, sulphur and metallic dyes. Losses are at a minimum (5%) in the dyeing of acrylic fibers but can be as high as 50% for the use of reactive dyes with cottons. Pereira et al. (2003) review much of the earlier literature of adsorption of dyes by AC. This appears to have been confined to studies of adsorption behaviour in terms of surface areas and pore sizes. As a result, little progress was made in understanding of adsorption behaviour by dyes. Pereira et al. (2003), being aware of the importance of surface functionality of carbons in adsorptions from solution (e.g. phenols from aqueous systems), as well as in catalysis on carbon surfaces, explored this aspect using dyes and AC with surfaces modified by oxidation and reduction procedures.

2.3.2.7 Other applications

AC has many smaller-scale applications such as industrial dry cleaning, as well as in coin operated dry-cleaning machines, cleaning of electroplating solutions, public and private aquariums, decaffeination, etc. Medical applications include oral ingestion into the stomach to remove poisons or toxic materials.

2.3.3 Liquid-phase adsorptions

AC is dominantly used for purposes of adsorption, a task for which it is well designed. Essentially, adsorption is restricted to working in one of two phases, from the gas/vapour phase (usually air) or from the Liquid phase (usually water). Fortunately, there is only one gas /vapour phase, but for liquids, two distinct phases need to be considered, namely adsorption from aqueous systems and adsorption from non-aqueous systems (Marsh and Rodriguez-Reino, 2006).

2.3.3.1 Adsorption of inorganic solutes from aqueous solution

In wastewater treatments, special attention is given to removal of dangerous inorganic material such as the heavy metals, including mercury, chromium, molybdenum, cobalt, nickel, copper, cadmium, lead, uranium, gold, arsenic, barium, iron and vanadium. Whereas the forces of adsorption from the gas phase are well understood, adsorption from solution of inorganic species is not so well understood.

2.3.3.2 Adsorption of gold and silver species

A major use of AC in the metallurgical industries is the extraction of gold and silver from low-grade ores. Three processes have been used to extract these metals. In the first, the Carbon-In Pulp (CIP) method, the prepared ore is conditioned with air and lime and mixed with an oxygenated solution of sodium cyanide plus caustic soda and then contacted with AC (coarser in size) which retains the gold-cyanide complex, the carbon being then screened from the system. The Carbon-In-Leach (CIL) method is a variant of the CIP method and involves making a pile of crushed ore together with lime or sodium hydroxide. This pile is sprayed with oxygenated alkaline sodium cyanide solution, the emerging solution then being passed through a carbon adsorption circuit, the carbon adsorbing up to 3 wt% of metal. The Carbon-In-Tower (CIT) method involves contacting the solution of silver and gold in vertical columns in which carbon is circulated counter-currently. The gold is recovered electrolytically, by alcohol or glycol stripping or zinc precipitation. The AC is regenerated by heating 600-800 °C in an inert atmosphere or by using superheated steam. Coconut shell is the major source of AC because of its hardness (Marsh and Rodriguez-Reino, 2006).

2.3.3.3 Adsorption of organic solutes from aqueous solution

Adsorption of organic solutes covers a wide spectrum of systems such as drinking water and waste water treatments, and applications in the food, beverage, pharmaceutical and chemical industries. AC adsorption has been cited by the US-EPA as one of the best available environmental control technologies. Adsorption of organic solutes is just as important as inorganic solutes, from the point of view of environmental control. Here, adsorption of organic compounds and of aromatics in particular, is a complex interplay of electrostatic and dispersive interactions. This is particularly true for phenolic compounds (Marsh and Rodriguez-Reino, 2006).

2.3.4 Porous carbons in catalytic processes

Catalytic processes account for over 90 percent of the chemical manufacturing processes in use throughout the world, the catalyst increasing the rate of a reaction and controlling its selectivity. Carbon materials have been used for some time in heterogeneous catalysis, acting as direct catalysts or as a catalyst support. The Catalytic Reaction Guide, (CRG) by Johnson Matthey (1988), one of the world's leading suppliers of catalysts, includes a list of 69 organic reactions catalyzed by precious metals, nine of which use carbon as the only support material. The number of industrial processes using carbon as catalyst is however, very small, although there is a clear and increasing interest in this area, as denoted by the rapid growth in the literature. It is now recognized that carbon as a catalyst support, as well as a catalyst in its own right, offers unparalleled flexibility in tailoring catalyst properties to specific needs. However, the future growth of the use of carbon materials in catalysis depends on the better understanding (and subsequent control) of the chemistry of carbon surfaces (Marsh and Rodriguez-Reino, 2006).

2.3.5 AC: Worldwide

The roles of AC extend, ultimately, to the entire atmosphere of this planet as well as to its rivers, seas and ocean, Derbyshire et al. (2001). The role also extends to the individual person, who, by bad luck, is blessed with odorous feet. The shoe insole, fitted with an AC, comes to the rescue.

2.3.6 The Outlook for AC

The AC industry, in a competitive market, is subject to changes which call for the use of both less AC and for more AC. Alternative systems of adsorption may be more cost-effective in some environmentally driven applications than those using AC. Continued emphasis by the EPA and industry toward the prevention of generated waste, rather than "end-of-pipe" solutions, is one limiting concern.

2.4 What is a furnace?

A furnace is an apparatus in which heat is generated and transferred directly or indirectly to a molten or solid mass for the purpose of effecting a physical, chemical or metallurgical change in the mass (Atanda et al., 2014) . It is a refractory lined equipment used to melt metals for casting or to heat materials to change their shape (e.g. rolling, forging) or properties (heat treatment).

Since flue gases from the fuel come in direct contact with the materials, the type of fuel chosen is important. For example, some materials will not tolerate sulphur in the fuel. Solid fuels generate particulate matter, which will interfere with the materials placed inside the furnace. For this reason, most furnaces use liquid fuel, gaseous fuel or electricity as energy input while induction and arc furnaces use electricity to melt steel and cast iron. Melting furnaces for nonferrous materials use fuel oil while oil-fired furnaces mostly use furnace oil, especially for reheating and heat treatment of materials and Light Diesel Oil (LDO) is used in furnaces where sulphur is undesirable. Furnace ideally should heat as much of material as possible to a uniform temperature with the least possible fuel and labour. The key to efficient furnace operation lies in complete combustion of fuel with minimum excess air. Furnaces operate with relatively low efficiencies (as low as 7%) compared to other combustion equipment such as the boiler (with efficiencies higher than 90 percent. This is caused by the high operating temperatures in the furnace. For example, a furnace heating materials to 1200 ^{o}C will emit exhaust gases at 1200 ^{o}C or more, which results in significant heat losses through the chimney (UNEP, 2006).

All furnaces have the components shown in Figure 2.3 (Carbon Trust, 1993): refractory chamber constructed of insulating materials to retain heat at high operating temperatures, hearth to support or carry the steel, which consists of refractory materials supported by a steel structure, part of which is water-cooled, burners that use liquid or gaseous fuels to raise and maintain the temperature in the chamber (coal or electricity can be used in reheating furnaces), chimney to remove combustion exhaust gases from the chamber and charging and discharging doors through which the chamber is loaded and unloaded.

Loading and unloading equipment include roller tables, conveyors, charging machines and furnace pushers.

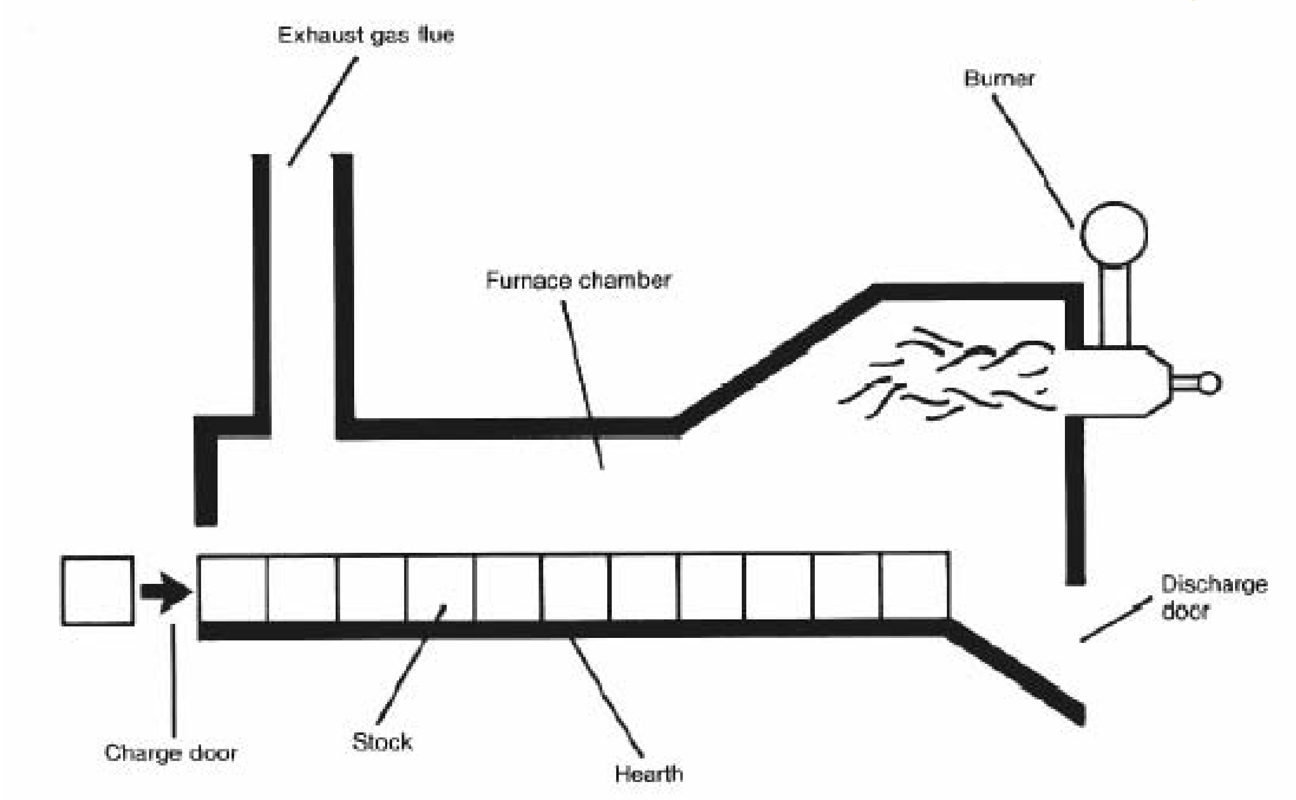

Figure 2.3 Typical Fuel Fired Batch Furnace Component

Source: The Carbon Trust, (1993)

2.4.1 Types of furnaces

The furnaces used in the heat treating process can be classified in several different ways. The most popular classification method is shown in Figure 2.4

2.4.2 Classification based on heating method

Based upon the heating or energy input method, furnaces can be divided into:

i. combustion based heating and
ii. electric heating methods.

2.4.2.1 Combustion-based (fuel-based) process heating

Heat is generated by the combustion of solid, liquid, or gaseous fuels, and transferred either directly or indirectly to the material. Common fuel types are fossil fuels (e.g. oil, natural gas, coal) The combustion gases can be either in contact with the material (direct heating), or be confined and thus be separated from the material (indirect heating; e.g., radiant burner tube, radiant panel, muffle). Combustion furnaces can be classified in several bases as shown in Table 2.2; type of fuel used, mode of charging the materials, mode of heat transfer and mode of waste heat recovery.

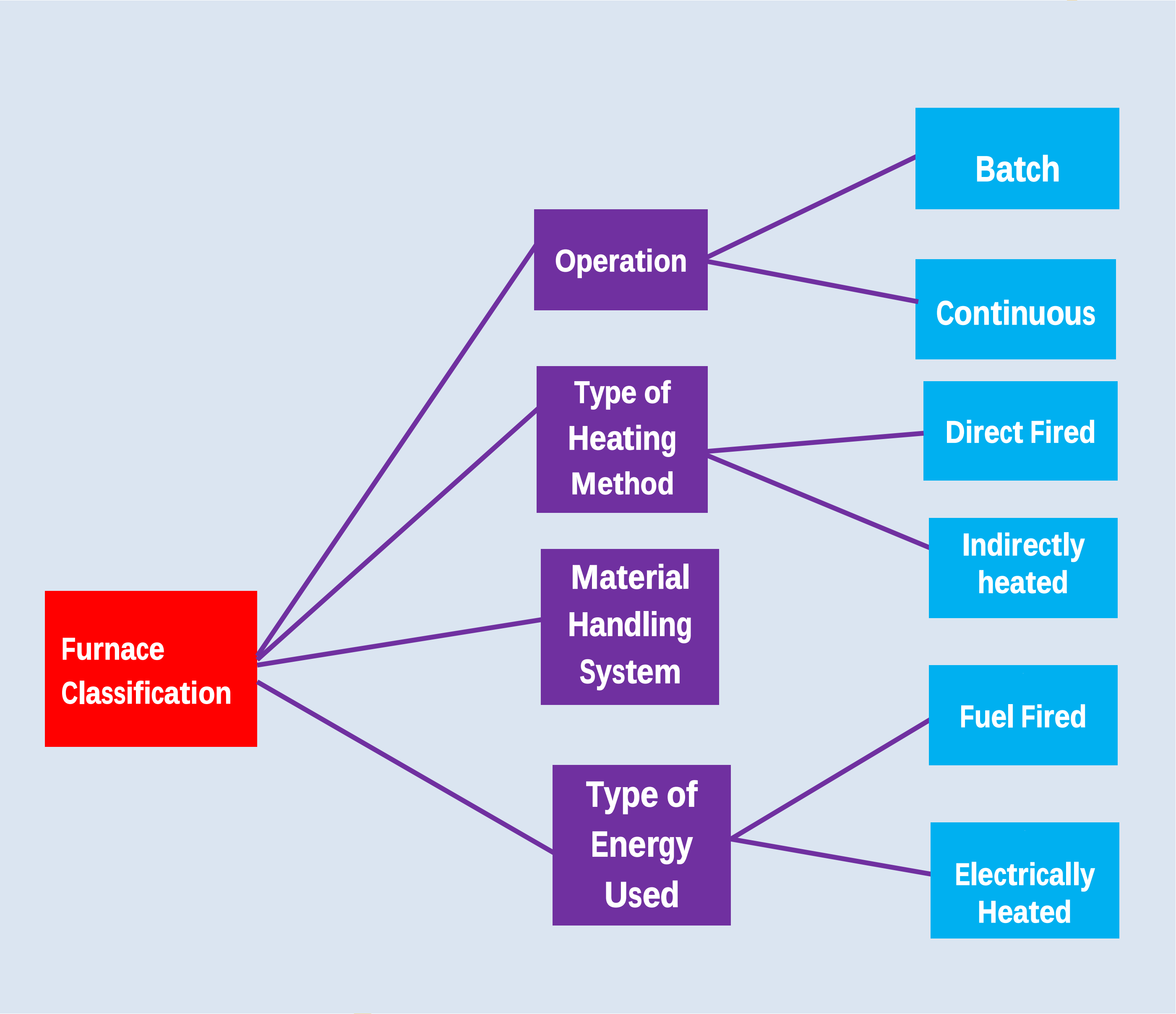

Figure 2.4 Classification of Furnace

Source: Purushothaman, (2008)

Table 2. 2 Classification of Furnaces

Classification method	**Types and examples**
Type of fuel used	Oil-fired Gas-fired Coal-fired
Mode of charging material	Batch/intermittent Periodical a. Forging b. Re-rolling (batch/pusher) c. Pot Continuous a. Pusher b. Walking beam c. Walking hearth d. Continuous recirculating bogie furnaces e. Rotary hearth furnaces
Mode of heat transfer	Radiation (open fire place) Convection (heated through medium)

Source: United Nations Environment Programme, (2006)

2.4.2.2 Electric process heating

Electric currents or electromagnetic fields are used to heat the material. Direct heating methods generate heat within the work piece, by either passing an electrical current through the material, or inducing an electrical current ("eddy current") into the material, by exciting atoms/molecules within the material with electromagnetic radiation (e.g. microwave). Indirect heating methods use one of these three methods to heat a heating element or and transfer the heat either by conduction, convection, radiation or combination of these to the work piece.

2.4.3 Classification based on mode of operation

Another classification of the furnaces is based on the mode of operation. The classification is either a batch type operation or a continuous operation.

2.4.3.1 Batch furnaces

The basic batch furnace normally consists of an insulated chamber with an external reinforced steel shell, a heating system for the chamber, and one or more access doors to the heated chamber. Standard batch furnaces such as box, bell, elevator, car bottom, and pit types are most commonly used when a wide variety of heat-hold-cool temperature cycles are required. Batch furnaces are normally used to heat treat low volumes of parts (in terms of weight per hour). Batch furnaces are also used to carburize parts that require heavy case depths and long cycle times. These furnaces are either electrically heated or gas/oil fired.

The gas/oil fired furnaces can further be classified as direct fired and in-direct fired (radiant tube burners)

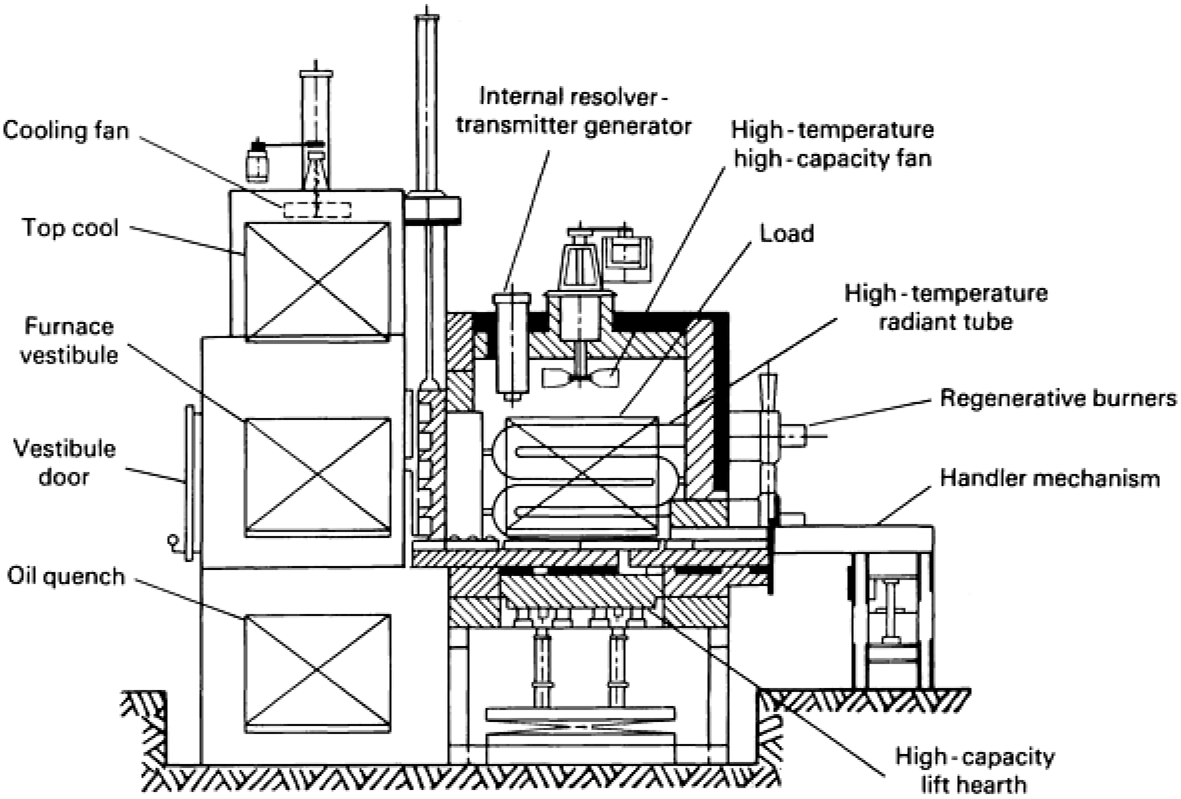

Figure 2.5 A schematic of Batch Integral Furnace

Source: Chapman, (1989)

2.4.3.2 Vacuum furnaces (type of batch furnace)

In most heat-treating processes, when materials are heated, they react with atmospheric gases. If this reaction is undesirable, the work must be heated in the presence of some gas or gas mixture other than normal air. This is done in normal atmosphere furnace processing. The gas or gas mixture may be varied to cause desirable reactions with the material being processed or it may be adjusted so that no reactions occur. At different temperatures, different reactions may occur with the work and furnace atmosphere. In most atmosphere furnaces it is not possible to change the atmosphere composition rapidly enough for optimum reactions or to control the atmosphere composition with the degree of precision required for some heat-treating processes. Vacuum furnaces allow gas changes to be made quite rapidly because they contain gases of low weight. Vacuum furnace technology removes most of the components associated with normal atmospheric air before and during the heating of the work.

2.4.3.3 Continuous furnaces

Continuous furnaces consist of the same basic components as batch furnaces: an insulated chamber, heating system, and access doors. In continuous furnaces, however, the furnaces operate in uninterrupted cycles as the work pieces move through them. Consequently, continuous furnaces are readily adaptable to automation and thus are generally used for high-volume work. Another advantage of continuous furnaces is the precise repetition of time-temperature cycles, which are a function of the rate of travel through the various furnace zones. A multi chamber pusher-type continuous carburizing furnace system is widely used in industry where the heating, carburizing, and diffusion portions of the cycle are separated.

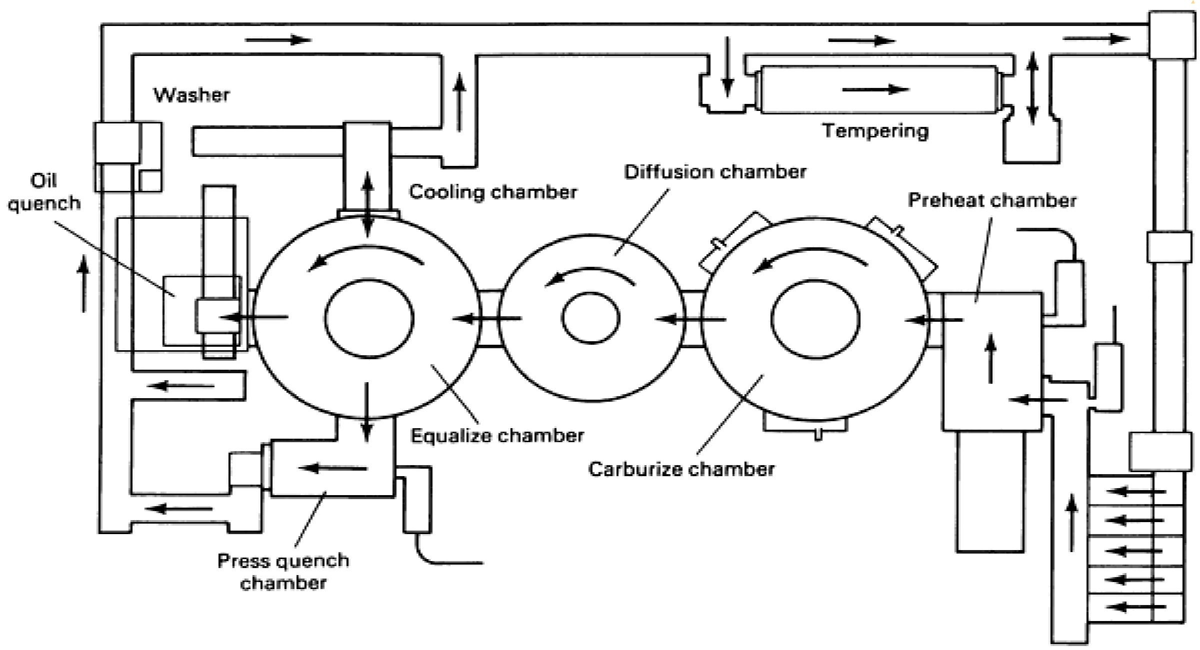

Figure 2.6 - Pusher-type and rotary-hearth-heat-treat system

Source: Chapman , (1989)

2.4.4 Classification based on material handling system

The selection of the material handling system depends on the properties of the material, the heating method employed, the preferred mode of operation (continuous, batch) and the type of energy used. An important characteristic of process heating equipment is how the load is moved in, handled, and moved out of the system. Several important types of material handling systems are; conveyor belts, buckets, roller, rotary hearth furnaces, walking beam furnace pusher type furnaces, car bottom furnaces and continuous strip furnaces

2. 4.5.1 Forging furnace

The forging furnace is used for preheating billets and ingots to attain a 'forge' temperature. The furnace temperature is maintained at around 1200 to 1250 ^{o} C. Forging furnaces use an open fireplace system and most of the heat is transmitted by radiation. The typical load is 5 to 6 ton with the furnace operating for 16 to 18 hours daily. The total operating cycle can be divided into heat-up time, soaking time and forging time. Specific fuel consumption depends upon the type of material and number of 'reheats' required.

2.4.5.2 Re-rolling mill furnace

a) Batch type

A box type furnace is used as a batch type re-rolling mill. This furnace is mainly used for heating up scrap, small ingots and billets weighing 2 to 20 kg for re-rolling. Materials are manually charged and discharged and the final products are rods, strips etc. The operating temperature is about 1200 ^{o} C. The total cycle time can be further categorized into heat-up time and re-rolling time. During heat-up time the material gets heated up-to the required temperature and is removed manually for re-rolling. The average output from these furnaces varies from 10 to 15 tons / day and the specific fuel consumption varies from 180 to 280 kg of coal / ton of heated material.

b) Continuous pusher type

The process flow and operating cycles of a continuous pusher type is the same as that of the batch furnace. The operating temperature is about 1250 ^{o} C. Generally, these furnaces operate 8 to 10 hours with an output of 20 to 25 ton per day. The material or stock recovers a part of the heat in flue gases as it moves down the length of the furnace. Heat absorption by the material in the furnace is slow, steady and uniform throughout the cross-section compared with batch type.

2.4.5.3 Continuous reheating furnace

In continuous reheating, the steel stock forms a continuous flow of material and is heated to the desired temperature as it travels through the furnace. The temperature of a piece of steel is typically raised to between 900 and 1250 ^{o}C, until it is soft enough to be pressed or rolled into the desired size or shape. The furnace must also meet specific stock heating rates for metallurgical and productivity reasons.

To ensure that the energy loss is kept to a minimum, the inlet and outlet doors should be minimal in size and designed to avoid air infiltration. Continuous reheating furnaces can be categorized by the two methods of transporting stock through the furnace; Stock is kept together to form a stream of material that is pushed through the furnace. Such furnaces are called pusher type furnaces (Figure 2.8) and Stock is placed on a moving hearth or supporting structure which transports the steel through the furnace. The furnaces include walking beam furnace, (Figure 2.9), walking hearth furnace, (Figure2.10), continuous re-circulating bogie furnaces, (Figure 2.8) and rotary hearth furnaces (Figure 2.7) (UNEP, 2006).

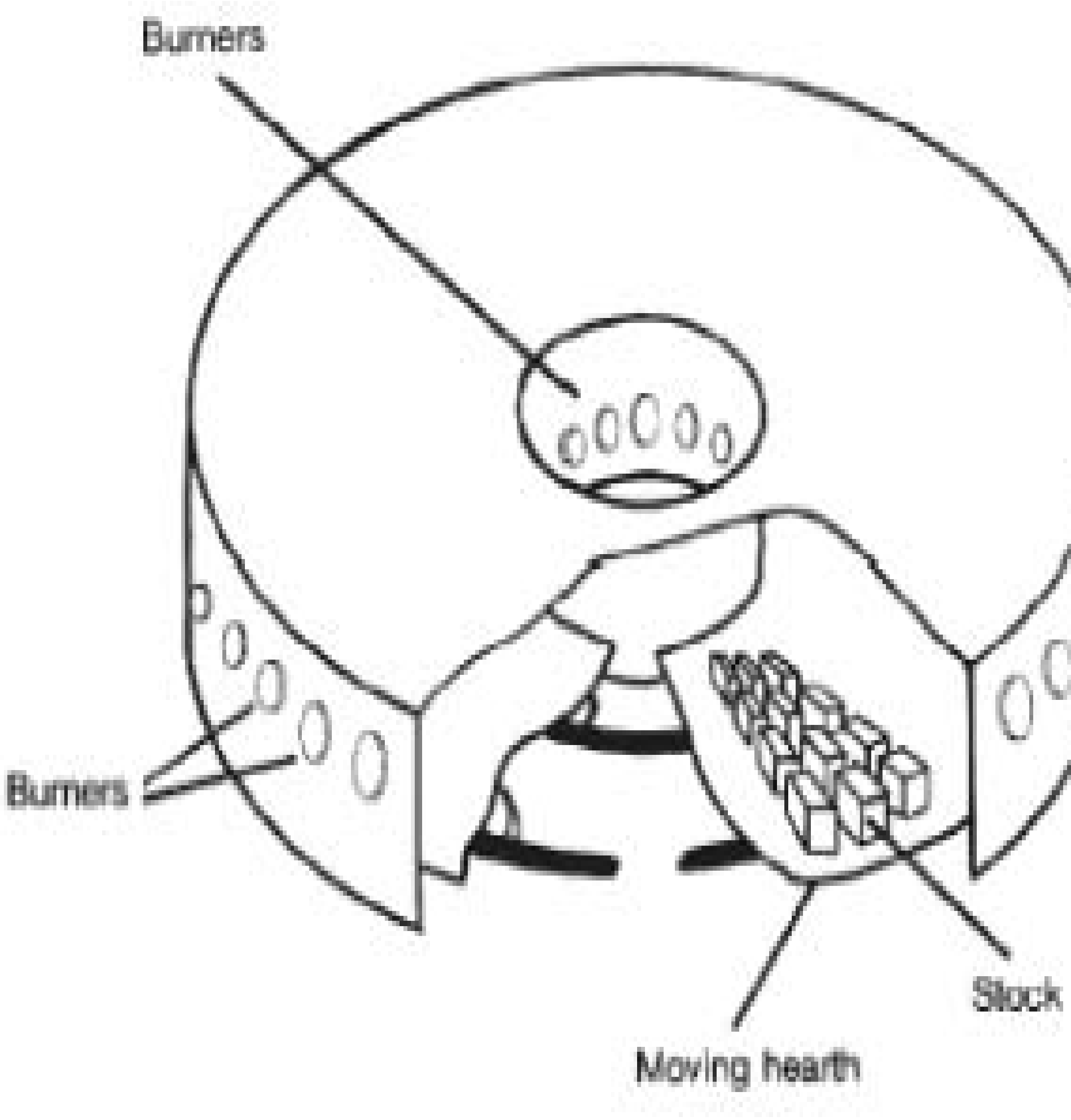

Figure 2.7 Rotary Hearth Furnace

Source: The Carbon Trust, (1993)

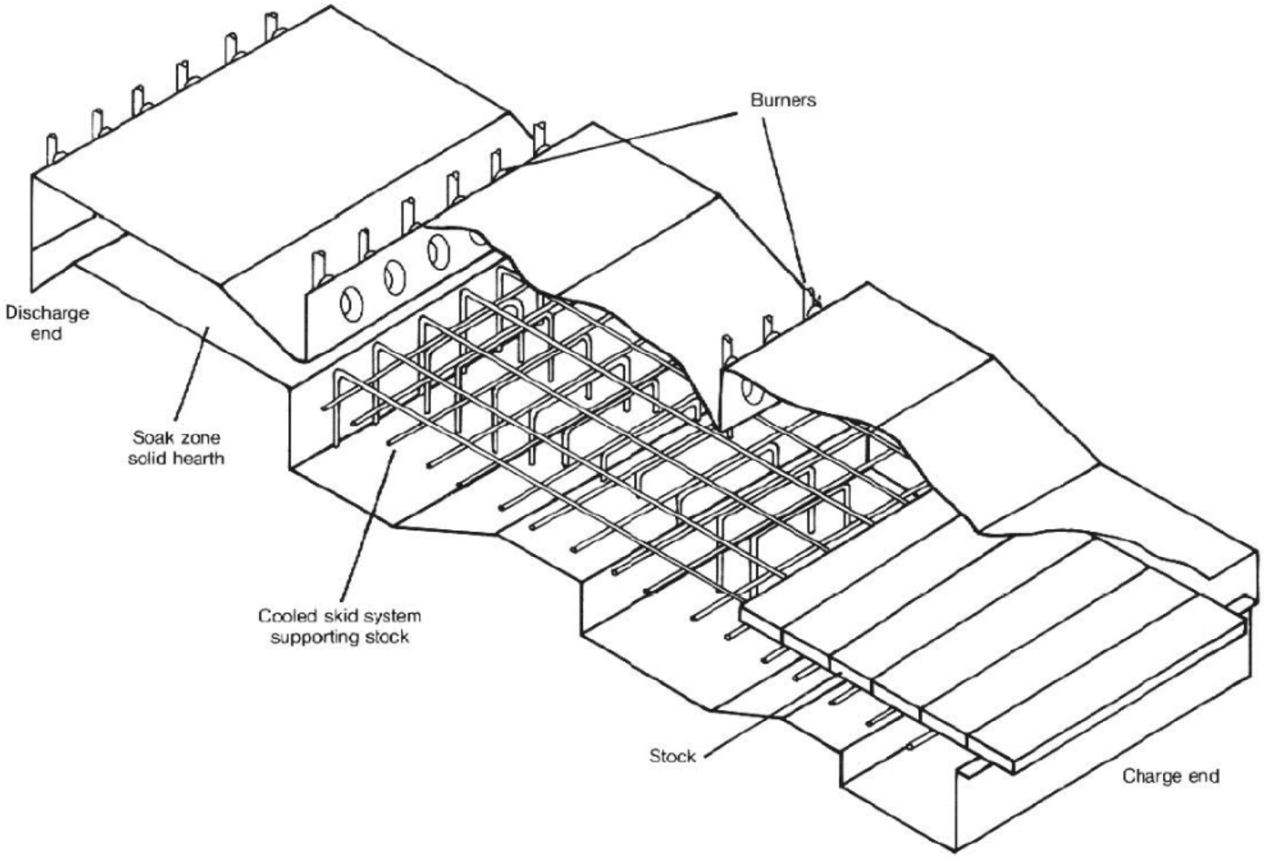

Figure 2.8 Pusher Furnace

Source: The Carbon Trust, (1993)

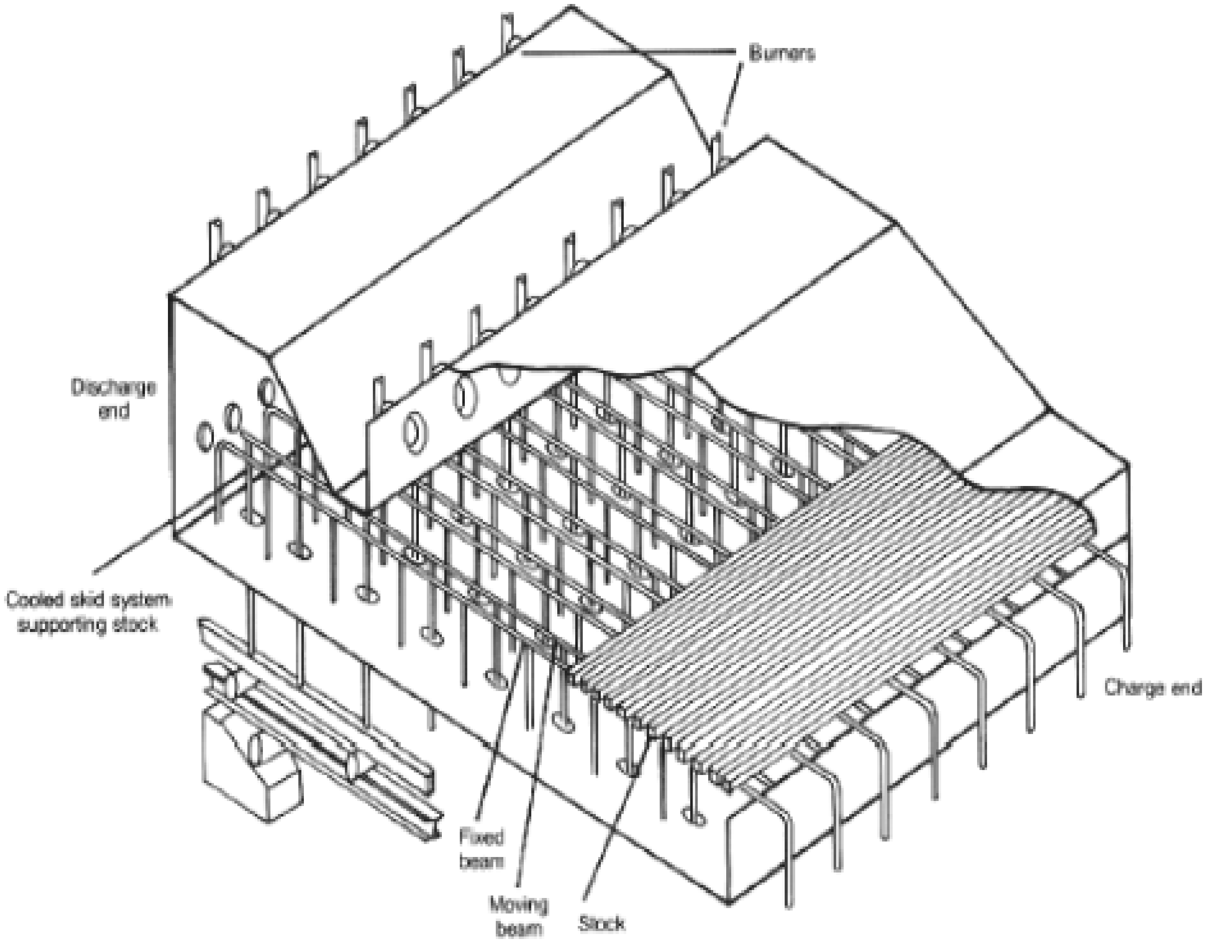

Figure 2.9 Walking Beam Furnace

Source : The Carbon Trust, (1993)

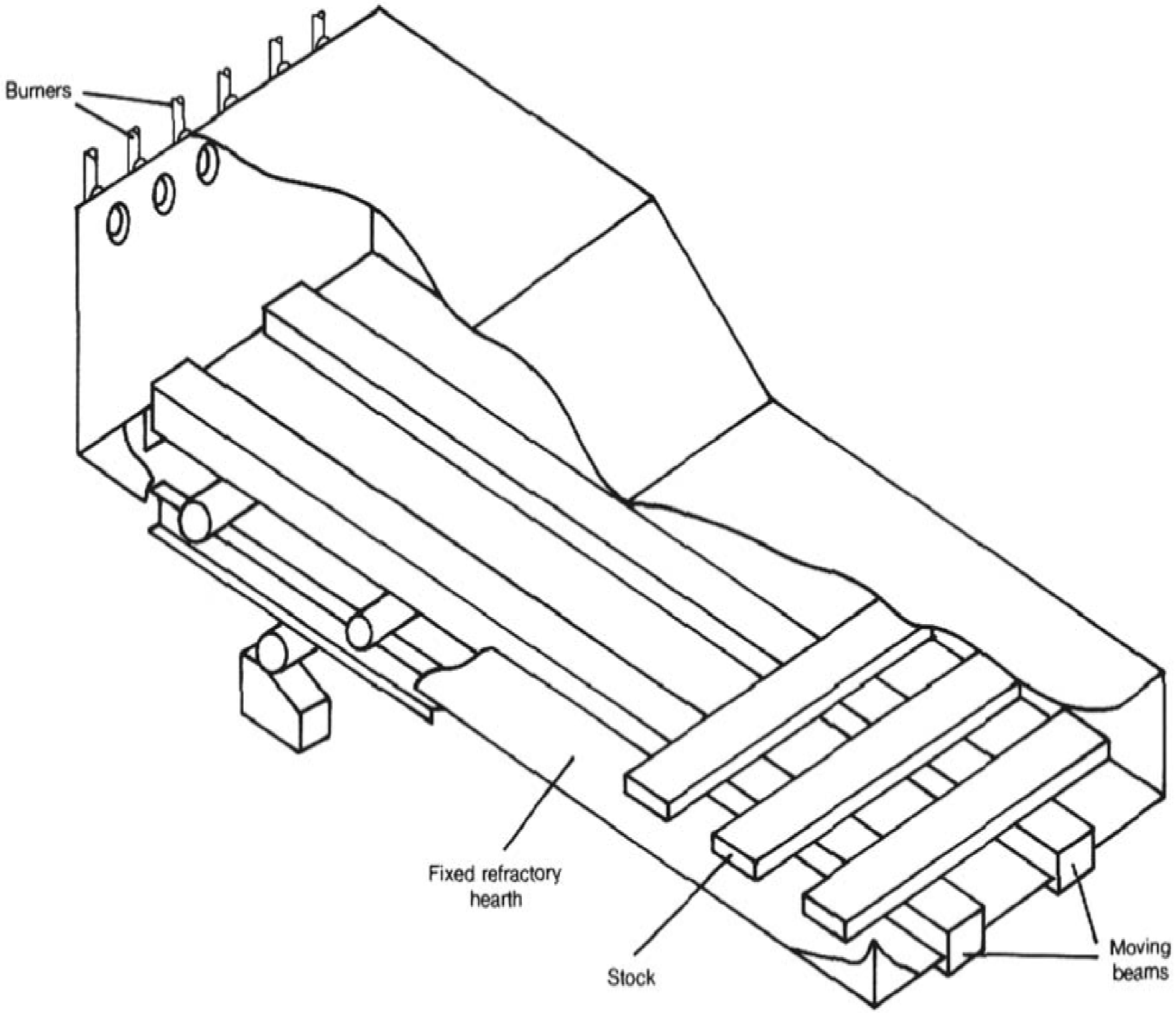

Figure 2.10 Walking Hearth Furnace

Source: The Carbon Trust, (1993)

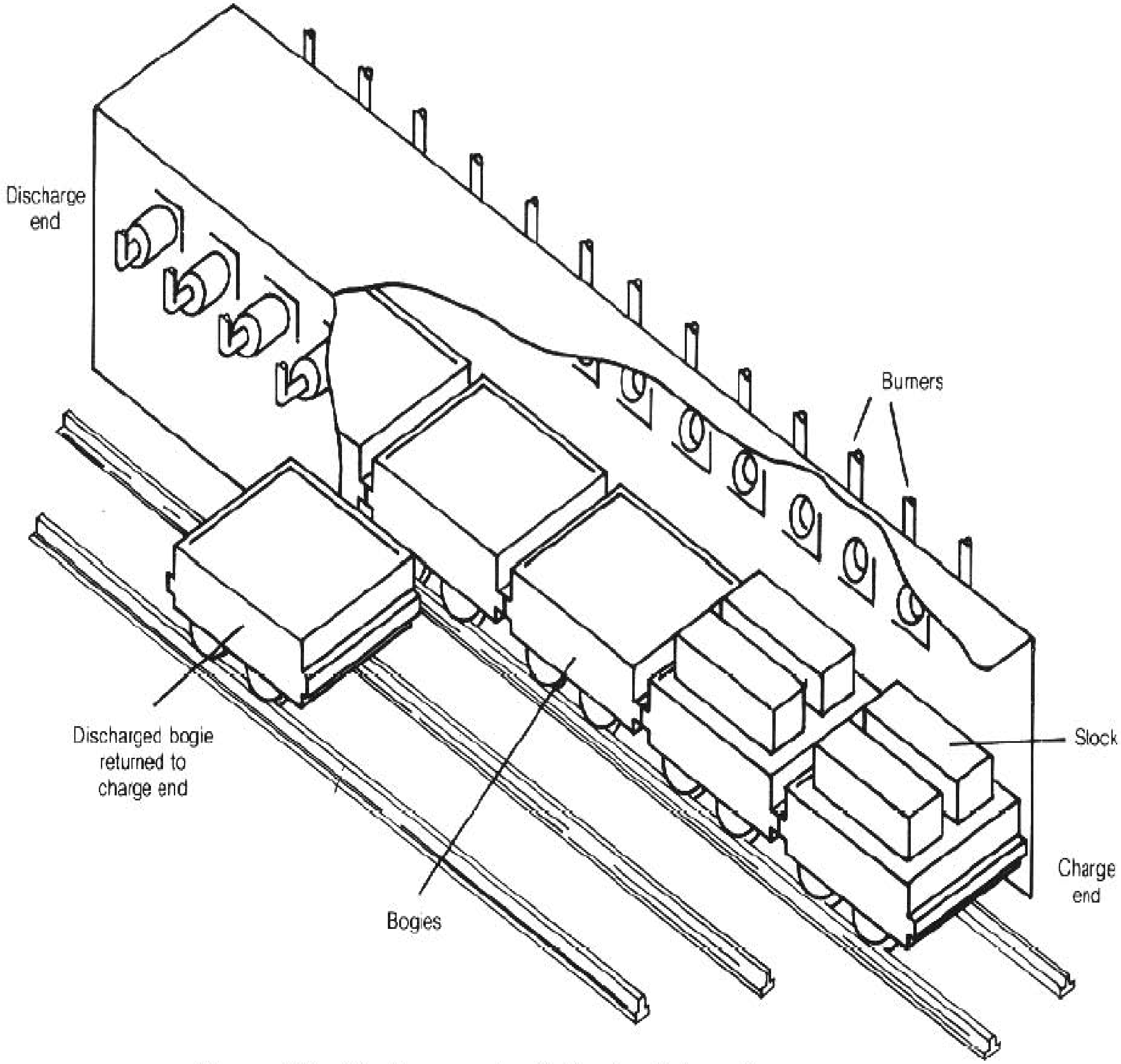

Figure2.11 Continuous Re-circulating Bogie Furnace

Source : The Carbon Trust, (1993)

2.5 Furnaces Used in Activation Carbon Production

A wide variety of furnaces and kilns are used for carbonization and activation, including rotary kilns fired directly or indirectly, multiple-hearth furnaces, fluidized-bed reactors, and vertical retorts, each of which has advantages for a particular purpose.

Although several types of furnaces are described in the literature, three are most commonly used by producers of activated carbon;rotary kilns, multiple hearth furnaces and fluidized bed furnaces It is to be understood that carbonization can be carried out in any of these furnaces (Marsh and Rodriguez-Reino, 2016).

2.5.1 Rotary kilns

These are the most popular among the producers because in general terms, they produce activated carbon with a more controlled porous structure. The residence time is greater than other furnaces, and because their length is large, several burners and gas supply lines (natural gas, propone or the gases from the carbonization unit) are distributed along the kiln in order to improve the control of the temperature and the temperature profiles. The lifters placed along the interior of the kiln improve the mixing of the material thus providing a more homogenous gasification. The rate of production is defined by the rotation speed, the angle of the kiln, the steam injection and the rate of carbon dosage and withdrawal. The indirectly fired rotary kiln, for example, is relatively large and expensive, but avoids a large discharge of inert gases to the afterburner. It is generally admitted that more micro porous carbons with lower proportion of mesopores can be manufacture using rotary kilns (Marsh and Rodriguez-Reino, 2016).

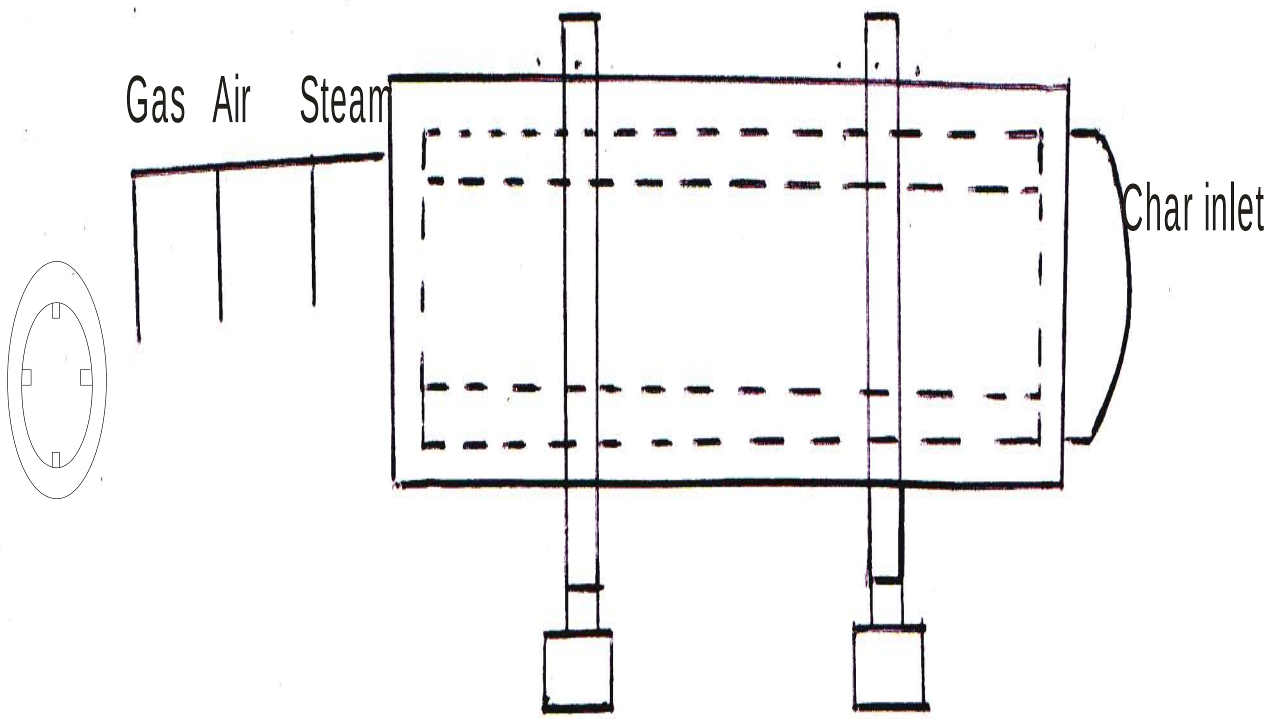

Figure 2.12 Schematic Representation of a Rotary Klin
Source : Rodriguez – Reinoso, (1997)

2.5.2 Multiple hearth furnaces

Multiple hearth furnaces are used as activating furnaces. There is a central vertical axis moving the rotary arms of the stationary floor on each stage, and the material is transported downward failing alternatively near to the axis on one stage and near to the wall at the next stages. As there are several supplies of heat and steam at different levels (in each stage in some furnaces) The temperature regulation is relatively simple, the relatively low carbon bed height makes the residence time shorter than in rotary kilns and the quality of the carbon may by slightly lower, with more development of mesoporosity. This type of furnace is also used for regeneration of spent activated carbon (Marsh and Rodriguez-Reino, 2016).

2.5.3 Fluidized bed furnaces

Fluidized bed furnaces are characterized by a good heat and mass transfer, the activating gases being in excellent contact with the carbon particles. This means that the residence time is the shortest of all activating furnaces. Industry uses fluidized, furnaces that are operated continually. The main problem with this type of furnace is that reaction is extensive and there is much external burning of the particles with the end result of activated carbon of relatively poor quality, mainly used for powdered activated carbon

However, there have been some recent developments in the design of fluidized bed furnaces, which are now more commonly used by producers

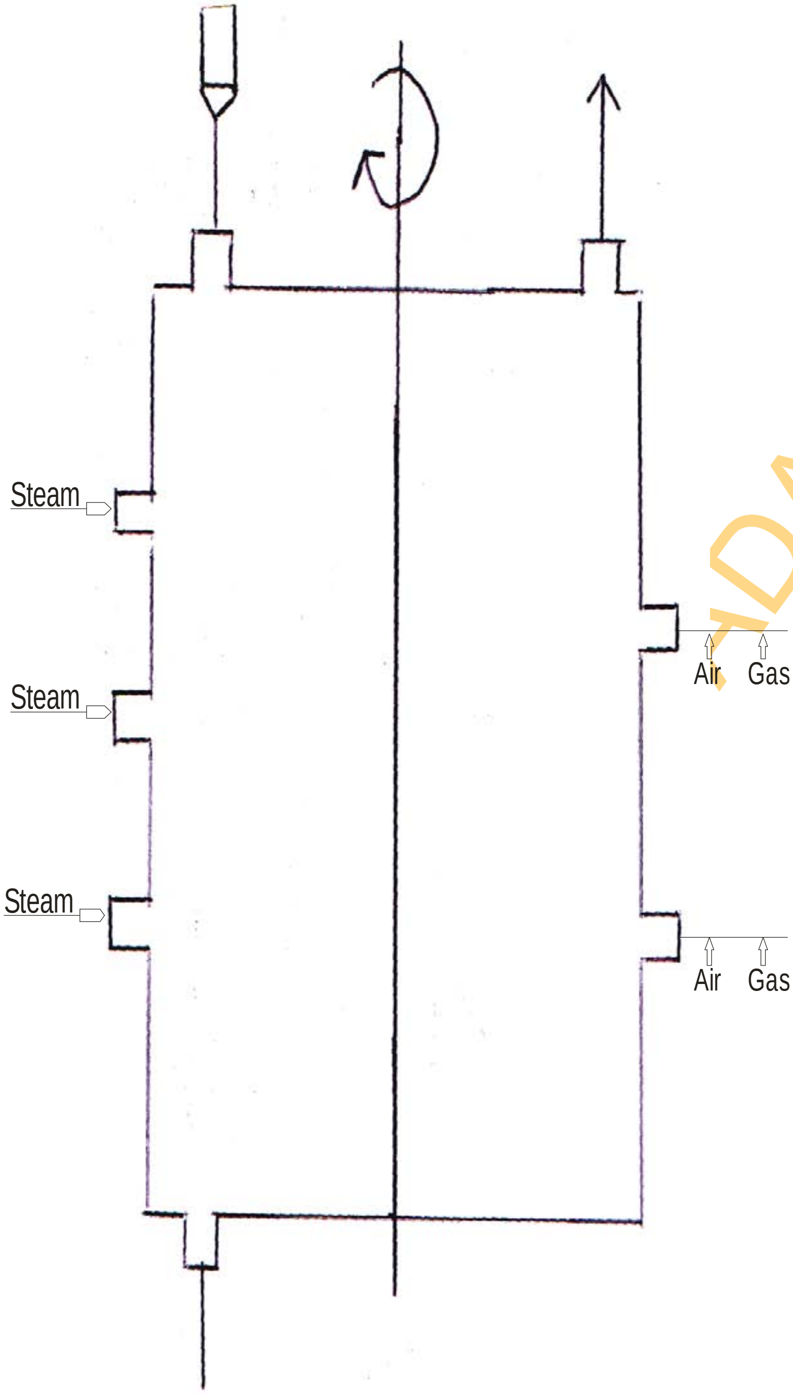

Figure 2.13 Schematic representation of a Multiple –Hearth Furnace
Source : Rodriguez – Reinoso, (1997)

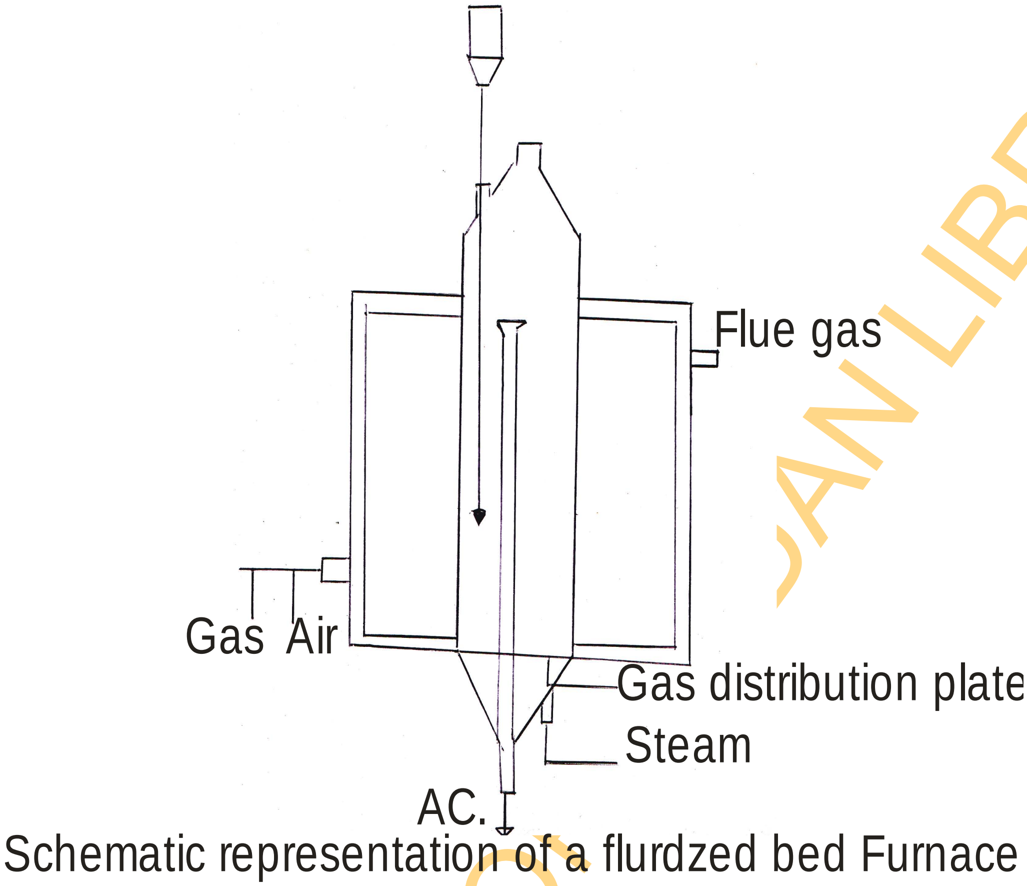

Figure 2.14 Schematic Representation of a Fluidized Bed Furnace

Source: Rodriguez – Reinoso, (1997)

The indirectly fired rotary kiln, for example, is relatively large and expensive, but avoids a large discharge of inert gases to the afterburner. The fluidized-bed reactor is extremely compact, but is generally used only for the activation of abrasion-resistant raw materials because of the attrition that occurs during fluidization. Carbonization and activation used to be carried out separately, but there is currently an increasing tendency for the two processes to be conducted in the same furnace (Marsh and Rodriguez- Reino, 2016).

2.6 Refractory

According to Choulet, (1997) refractory are heat resistant materials used in almost all processes involving high temperatures and/or corrosive environment. They are typically used to insulate and protect industrial furnaces and vessels due to their excellent resistance to heat, chemical attack and mechanical damage. Any failure of refractory could result in a great loss of production time, equipment, and sometimes the product itself. The various types of refractories also influence the safe operation, energy consumption and product quality; therefore, obtaining refractories best suited to each application is of supreme importance.

2.6.1 Forms of refractory

a) Classification based on physical form

According to Bhatia (2011), refractories are classified according to their physical form. These are the shaped and unshaped refractories. The former is commonly known as refractory bricks and the latter as “monolithic” refractories.

i) Shaped refractories:

Shaped refractories are those which have fixed shape when delivered to the user. These are what we call bricks. Brick shapes maybe divided into two: standard shapes and special shapes. Standards shapes have dimension that are conformed to by most refractory manufacturers and are generally applicable to kilns and furnaces of the same type. Special shapes are specifically made for particular kilns and furnaces. This may not be applicable to another furnaces or kiln of the same type.

Shaped refractories are almost always machine-pressed, thus, high uniformity in properties are expected. Special shapes are most often hand-moulded and are expected to exhibit slight variations in properties.

ii)Unshaped refractories: Unshaped refractories are without definite form and are only given shape upon application. It forms joint less lining and are better known as monolithic refractories. These are categorized as plastic refractories, ramming mixes, castables, gunning mixes, fettling mixes and mortars.

Table 2.3 Classification of Refractory Based on Physical Form

Shaped	**Unshaped**
1. Alumino Silicates a. High Alumina b. Low Alumina	1. Mortars
2. Silica	2. Castables a. LCC b. ULCC c. NCC d. SFC
3. Basic a. Magnesite b. Mag-Chrome c. Mag-Carbon d. Forsterite e. Dolomite	3. Gunning Mass
4. Carbon	4. Ramming Mass
5. Zirconia • Zircon (Acidic) • Zirconia (Neutral)	5. Filling Mass
	7. Spraying Compound
	8. Patching/Coating Mass

Source: Bhatia , (2011)

b) Classification based on chemical composition

Refractories are typically classified on the basis of their chemical behaviour, i.e. their reaction to the type of slags. Accordingly the refractory materials are of three classes - acid, basic and neutral (Bhatia, 2011; Kathiat, 2016).

i) Acid refractories**:** Acid refractories are those which are attacked by alkalis (basic slags). These are used in areas where slag and atmosphere are acidic. Examples of acid refractories are: silica (SiO_2) and zirconia (ZrO_2).

ii) Neutral Refractories**:** Neutral Refractories are chemically stable to both acids and bases and are used in areas where slag and atmosphere are either acidic or basic. The common examples of these materials are: carbon graphite (most inert), chromites (Cr_2O_3) and alumina. Graphite is the least reactive and is extensively used in metallurgical furnaces where the process of oxidation can be controlled.

iii) Basic refractories**:** Basic refractories are those which are attacked by acid slags but stable to alkaline slags, dusts and fumes at elevated temperatures. Since they do not react with alkaline slags, these refractories are of considerable importance for furnace linings where the environment is alkaline; for example non-ferrous metallurgical operations. The most important basic raw materials are: magnesia (MgO) - caustic, sintered and fused magnesia, dolomite (CaO*MgO) - sintered and fused dolomite and chromite -main part of chrome ore (Bhatia, 2011; Kathiat, 2016).

Chemical characteristics of the furnace process usually determine the type of refractory required. Theoretically, acid refractories should not be used in contact with basic slags, gases and fumes whereas basic refractories can be best used in alkaline environment (Bhatia, 2011; Kathiat, 2016).

c) Classification based on method of manufacture

The refractories can be manufactured in either of the following methods; dry press process , fused Cast, hand molded , formed (normal, fired or chemical bonded) and unformed (monolithic – plastics, ramming mass, gunning, castable, spraying) (Bhatia, 2011; Kathiat, 2016).

2.6.2 Refractory raw material

The principal raw materials used in the production of refractories are: the oxides of silicon, aluminium, magnesium, calcium and zirconium and some non-oxide refractories like carbides, nitrides, borides, silicates and graphite. The main types include fire-clay bricks, castables, ceramic fiber and insulating bricks that are made in varying combinations and shapes for diverse applications. The value of refractories is judged not merely by the cost of material itself, but by the nature of the job and/or its performance in a particular situation. Atmosphere, temperature, and the materials in contact are some of the operating factors that determine the composition of refractory materials. (Bhatia, 2011; Kathiat, 2016)

2.6.3 Properties of refractories

Important properties of refractories are: chemical composition, bulk density, apparent porosity, apparent specific gravity and strength at atmospheric temperatures. These properties are often among those which are used as 'control points' in the manufacturing and quality control process. The chemical composition serves as a basic for classification of refractories and the density, porosity and strength is influenced by many other factors. Among these are type and quality of the raw materials, the size and fit of the particles, moisture content at the time of pressing, pressure at mould, temperature, duration of firing and the rate of cooling. (Bhatia, 2011; Kathiat, 2016)

Some of the important characteristics of refractories are:

1) Melting Point**:** Melting temperatures (melting points) specify the ability of materials to withstand high temperatures without chemical change and physical destruction. The melting point of few elements that constitute refractory composition in the pure state varies from 3000–6270 °C as indicated in the Table 2.4. The melting point serves as a sufficient basis for considering the thermal stability of refractory mixtures and is an important characteristic indicating the maximum temperature of use, (Bhatia 2011).

Table 2.4 Melting Point of Pure Samples Refractory Element

REFRACTORY ELEMENT	MELTING TEMPERATURES (° C)
Graphite C Pure	6268
Thoria, ThO_2 Pure Sintered	5398
Magnesia, MgO, Pure Sintered	5048
Zirconia, ZrO_2 , Pure Sintered	4858
Lime, CaO	4628
Beryllia, BeO, Pure Sintered	4588
Silicon Carbide, SiC, Pure	4048
Magnesia, 90-95%	3948
Chromite, $FeO\text{-}Cr_2O_3$	3928
Chromium Oxide	3788
Alumina, Al_2O_3, Pure Sintered	3688
Chromite, 38%, Cr_2O_3	3548
Alumina Fused Bauxite	3368
Silicon Carbide, 80-90%	3368
Fireclay	3368
Titania, TiO_2	3328
Kaolin, Al_2O_3 -, SiO_2	3268
Silica, SiO_2	3088

Source: Bhatia, (2011)

2) Size and dimensional stability: The size and shape of the refractories is an important feature in design since it affects the stability of any structure. Dimensional accuracy and size is extremely important to enable proper fitting of the refractory shape and to minimize the thickness and joints in construction.

3) Porosity: Porosity is a measure of the effective open pore space in the refractory into which the molten metal, slag, fluxes, vapours etc can penetrate and thereby contribute to eventual degradation of the structure. The porosity of refractory is expressed as the average percentage of open pore space in the overall refractory volume. High porosity materials tend to be highly insulating as a result of high volume of air they trap, because air is a very poor thermal conductor. As a result, low porosity materials are generally used in hotter zones, while the more porous materials are usually used for thermal backup. Such materials, however, do not work with higher temperatures and direct flame impingement, and are likely to shrink when subjected to such conditions. Refractory materials with high porosity are usually not chosen when they will be in contact with molten slag because they cannot be penetrated easily.

4) Bulk density: The bulk density is generally considered in conjunction with apparent porosity. It is a measure of the weight of a given volume of the refractory. For many refractories, the bulk density provides a general indication of the product quality; it is considered that the refractory with higher bulk density (low porosity) will be better in quality. An increase in bulk density increases the volume stability, the heat capacity, as well as the resistance to abrasion and slag penetration.

5) Thermal Conductivity: Thermal conductivity is defined as the quantity of heat that will flow through a unit area in direction normal to the surface area in a defined time with a known temperature gradient under steady state conditions. It indicates general heat flow characteristics of the refractory and depends upon the chemical and mineralogical compositions as well as the application temperature. High thermal conductivity refractories are required for some applications where good heat transfer is essential such as coke oven walls, regenerators, muffles and water cooled furnace walls. However, refractories with lower thermal conductivity are preferred in industrial applications, as they help in conserving heat energy.

Porosity is a significant factor in heat flow through refractories. The thermal conductivity of a refractory decreases on increasing its porosity. Although it is one of the least important properties as far as service performance is concerned, it evidently determines the thickness of brick work. Others include : cold crushing strength, Pyrometric Cone Equivalent (PCE), refractoriness under load, creep at high temperature, volume stability, expansion and shrinkage at high temperature and reversible thermal expansion:

2.6.4 Requirements of right refractory

According to Bahtia (2011), the general requirements of a refractory material can be summed up as its ability to:

1) Withstand high temperatures and trap heat within a limited area like a furnace;
2) Withstand action of molten metal, hot gasses and slag erosion etc;
3) Withstand load at service conditions;
4) Resist contamination of the material with which it comes into contact;
5) Maintain sufficient dimensional stability at high temperatures and after/during repeated thermal cycling;
6) Conserve heat.

2.6.5 Insulating refractories

According to Pichor and Janiec (2009), high-temperature processes require a considerable amount of energy. Often the energy consumption for high-temperature processes is used only partially for the actual technical process and 30 to 40% energy escapes through the walls into the atmosphere. To optimize the energy use and to prevent its escape into the ambience, special materials called insulating refractories are necessary. The function of insulating refractory is to reduce the rate of heat flow (heat loss) through the walls of furnaces. Insulation is effected by providing a layer of material having low heat conductivity, which means heat does not readily pass through them (Subramanian et al., 1996).

2.6.5.1 Characteristics of good insulating refractory

According to Fragoulis et al. (2004), the desirable feature of insulating bricks is the low thermal conductivity, which usually results from a high degree of porosity. Structure of air insulating material consists of minute pores filled with air which have in them very low thermal conductivity. The air spaces inside the brick prevent the heat from being conducted but the solid particles of which the brick is made conduct the heat. So, in order to have required insulation property in a brick a balance has to be struck between the proportion of its solid particles and air spaces. The thermal conductivity is lower if the volume of air space is larger. Importantly, the thermal conductivity of a brick does not so much depend on the size of pores as on the uniformity of size and even distribution of these pores. Hence, uniformly small sized pores distributed evenly in the whole body of the insulating brick are preferred.

The high porosity of the brick is created during manufacturing by adding a fine organic material to the mix, such as sawdust. During firing, the organic addition burns out, creating internal pores (Kogel *et al.*, 1996). Other ways to accomplish high porosity involves: a) by using materials which expand and open up on heating; b) by using volatile compounds like naphthalene; c) using aluminium (Al) powder in combination with NaOH solution (called chemical bloating); d) by using substances which by themselves have open texture e.g. insulating brick grog, vermiculite, ex-foliated mica, raw diatomite etc; e) using foaming agents to slip; f) aeration etc

Because of their high porosity, insulating bricks inherently have lower thermal conductivity and lower heat capacity than other refractory materials (Ghanbarnezhad et al., 2014).

2.6.5.2 Types of insulating refractories

Insulating materials can be classified with respect to application temperature: i) heat resistant insulating materials for application temperatures up to 2000 °F: calcium silicate materials; products from siliceous earth, perlite or vermiculite; silica based micro porous heat insulators; alumino-silicate fibres; ii) refractory insulating materials for application temperatures up to 2500°F: lightweight chamotte and kaolin bricks; lightweight castables; mixed fibres and aluminium oxide fibres; iii) high refractory insulating materials for application temperatures up to 3100°F: lightweight mullite and alumina bricks; lightweight hollow sphere corundum castables and bricks; special high refractory fibres; iv)Ultra-high refractory insulating materials for application temperatures up to 3600 °F: zirconia lightweight bricks and fibers; non-oxide compounds. Other types of insulating refractories include; castables, granular insulation, and ceramic fibre insulation, which is light weight. Extremely lightweight materials have a porosity of 75 to 85% and ultra-lightweight, high-temperature insulating materials have a total porosity greater than 85% (Ghanbarnezhad et al., 2014).

2.6.5.3 Applications and advantages

These are widely used in the crowns of glass furnaces and tunnel kilns. They can also be used as linings of furnaces where abrasion and wear by aggressive slag and molten metal are not a concern. These offer several distinct advantages; decreased heat losses through the furnace lining and less heat loss to the refractory leads to savings in fuel cost, the insulating effect causes a more rapid heat-up of the lining and lowers heat capacity of the insulating refractory, thinner furnace wall construction to obtain a desired thermal profile and less furnace mass due to the lower mass of the insulating refractory (Subramanian *et al.*, 1996; Ghanbarnezhad et al., 2014)

2.6.5.4 Some drawbacks of insulating bricks

The porosity in insulating refractories creates a large amount of free surface area. Although porosity decreases thermal conductivity and density of the brick, it also degrades the mechanical strength of the brick as compared to a dense refractory firebrick. The poor strength of insulating bricks due to their high porosity can pose structural design problems.

Porosity in insulating refractory leads to poor chemical resistance as compared to dense refractories of similar compositions. Gases, fumes, liquids such as slags, molten glass etc. at high temperatures can penetrate porous bricks easily, making insulating fire bricks unsuitable for direct contact with such liquids or gases.

Insulating fire bricks often suffer from thermal spalling problems, particularly in an environment of rapidly changing temperature. Since these bricks are good insulators, a substantial temperature gradient will occur between the hot and the cold face of each brick. The hot face will expand more than the cold face. The thermal gradient thus, gives rise to a mechanical stress in the body of the brick (Bhatia, 2011).

2.6.5.5 Ceramic fibres

Ceramic fibres are a family of insulating refractory products that is fluffy, white cotton like fiber and can be spun and fabricated into textiles, blankets, felts, boards, blocks, etc. These products have low thermal conductivity, very low heat storage, extremely light weight, immunity to thermal shocks and are chemically stable. The lightweight construction ensures that the required temperature in high-temperature plants is reached more rapidly, as only a small proportion of the temperature released into the processing vessel/furnace is used for the heating of the walls.

Ceramic fibres composed of 52 Al_2O_3 - 48 SiO_2 combinations can be applied as a hot face insulation material up to ~2600°F, whereas a 62 Al_2O_3 - 38 SiO_2 combinations impart greater refractoriness to fibre. 42% Al_2O_3, 52% SiO_2 and 6% ZrO_2 produces extra long staple fibre of 10 inches and are used for manufacturing ceramic fibre textiles and ropes (Bhatia, 2011).

The advantages of light weight ceramic fibre are; better fuel economy (savings as high as 60% are feasible in the case of certain intermittent furnaces), higher productivity capacity of furnaces, due to reduced heat storage capacity, higher service life of the furnace and reduced maintenance costs due to longer refractory life and ease of installation.

They are used with great success in metal treating furnaces, ceramic kilns, and numerous other periodic operations whose atmosphere do not negate their revolutionary thermal and lightweight qualities. Fibre mats also continue to be used in expansion joints and door seals, and in tunnel kilns and other exposed - brick structures as either original or retrofit layers on the outside or cold-face surface. (Bhatia, 2011)

2.6.5.6 Limitations

The chief limitation of ceramic fibre is shrinkage at high temperatures. A high quality ceramic fibre blanket rated for continuous use at 2400 °F will have 5% shrinkage after 24-hr exposure at 2400 °F. Shrinkage will not continue past this level in normal operating conditions, but this shrinkage must be carefully considered in designing a furnace lining. Others include ;

Mechanical strength of ceramic fibres is poor. These are not really structural materials. Proper support must be given to all refractory fibre products, fibre tend to sag at high temperature due to softening of fibres if improperly supported.

They are not suitable in severe hostile environments. These are handy repositories of dusts, fogs, and combustible fumes; not to mention for process liquids like slags and metals.

They are more expensive than conventional refractories however; installation labour savings and energy savings offset the high initial costs (Bhatia, 2011)

2.7 Heat Transfer in a Furnace

A furnace is an apparatus in which heat is generated and transferred directly or indirectly to a molten or solid mass for the purpose of effecting a physical, chemical or metallurgical change in the mass (Atanda et al., 2014). Furnace is an equipment isolated from the surrounding by an insulated wall and is used to transfer heat to the material to be melted or heat treated within the furnace.

Ideally, all heat added to the furnaces should be used to heat the charge, load or stock. In practice, however, a lot of heat is lost in several ways. The losses include energy conversion losses, furnace wall losses, furnace opening losses and the likes. In order to prevent these losses, refractory materials are used in lining the furnace, (Atanda et al., 2014) .

Generally, there are three methods of heat transfer: conduction, convection and radiation.

2.7.1 Conduction

This is when energy is transferred from one body to another as a result of a temperature gradient existing between them. When a furnace is operating, there is a gradual flow of energy from the inner surface to the outer thereby causing an increase in the outer surface temperature. Heat transfer rate per unit area according to Fourier's law of heat conduction for one-dimensional heat conduction as shown in equation 8, is proportional to the normal temperature gradient across the wall surface.

For a constant area along the heat flow path and constant thermal conductivity of the material, the heat flow can be written at steady state as

$$Q_{cond} = -kA\frac{dT}{dx} \quad (8)$$

$$Q = \frac{-KA}{\Delta x}(T_2 - T_1)$$

Here K is assumed to be constant,

T_1 and T_2 are surface temperatures.

Furnaces are usually multi- layered,

Fourier‘s law of conduction for steady conduction through composite wall is shown in equations 9 and 10

T_i =temperature of interfaces

Xi = wall thickness

I = 1, 2, 3

Layer A $$Q_A = -K_A A\frac{(T_2 - T_1)}{X_A} \quad (9)$$

Layer B $$Q_A = -K_B A\frac{(T_3 - T_2)}{X_B} \quad (10)$$

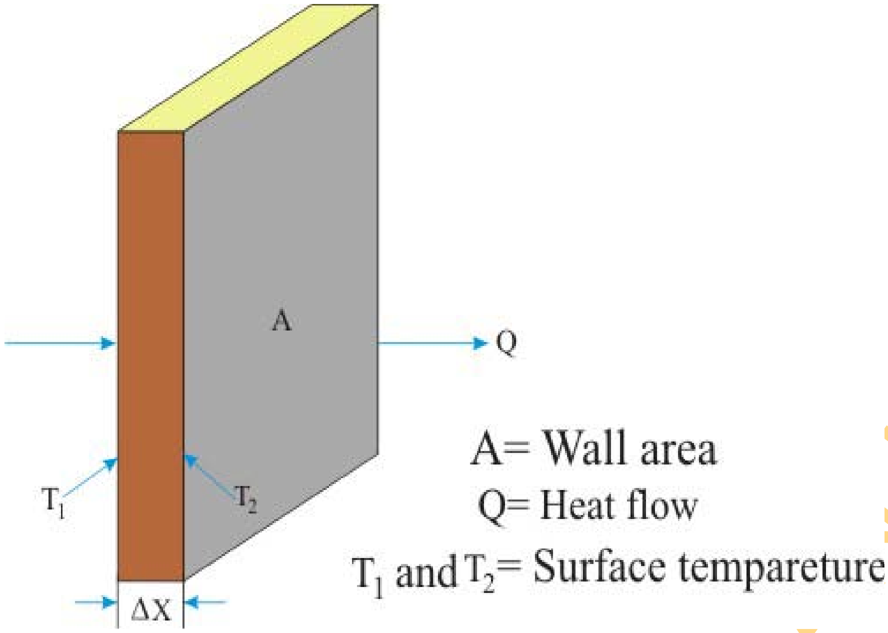

Figure 2.15 Heat Flow Through a Flat wall

Source: Holman, (2002)

Since heat flow in constant

$$Q_A = -K_A A \frac{(T_2 - T_1)}{X_A} \quad = \quad Q_A = -K_B A \frac{(T_3 - T_2)}{X_B}$$

$$-T_1 = \frac{Q_A X_A}{-K_A \ A} - T_2 \tag{11}$$

$$-T_2 = \frac{Q_A X_B}{-K_B \ A} - T_3 \tag{12}$$

Substitute equation 12 into equation 11

$$-T_1 = \frac{Q_A X_A}{-K_A \ A} + \frac{Q_A X_B}{-K_B \ A} - T_3$$

$$T_1 - T_3 = Q_A \left(\frac{X_A}{K_A \ A} + \frac{X_B}{K_B \ A} \right)$$

$$T_1 - T_3 = \frac{Q}{A} \left(\frac{X_A}{K_A} + \frac{X_B}{K_B} \right) \tag{13}$$

$$\frac{T_1 - T_3}{\frac{X_A}{K_A \ A} + \frac{X_B}{K_B \ A}} = Q \tag{14}$$

$$T_1 - T_3 \times \frac{A}{Q} = \frac{X_A}{K_A} + \frac{X_B}{K_B} \tag{15}$$

$\frac{X_A}{K_A} + \frac{X_B}{K_B}$ is the conductive heat transfer resistance.

The reciprocal of the heat transfer resistance is the conductive heat transfer coefficient as shown in equation 16

$$\frac{1}{\frac{X_A}{K_A} + \frac{X_B}{K_B}} = \frac{K_A K_B}{X_A K_A + X_B K_B} \tag{16}$$

2.7.1.1 Heat conduction through cylindrical bodies

The rate of conductive heat flow in a cylindrical furnace is given by equation 17

$$Q_k = 2\pi l K\,(T_f - T_s)\ \ln\left(\frac{r_2}{r_1}\right) \tag{17}$$

Where $Q_{K=}$ heat flow by conduction

l = length of furnace

T_f = furnace temperature

T_s = Temperature of the outside shell of the furnace

$r_1 = radius\ of\ furnace\ tube$

$r_2 = radius\ of\ furnace\ shell$

2.7.2 Convection

Heat exchange between the solid and fluid is said to occur due to convection whenever there is a difference in temperature between two phases and there is motion in the fluid. If the motion of the fluid is caused by the buoyancy forces which arise due to variation in the density of the fluid with temperature, the process is called free or natural convection. If the motion of the fluid is caused by external means such as blower, wind, fan or pump, the process is refered to as forced convection. Heat exchange due to convection may be described in terms of a heat transfer coefficient ‘h’ by Newton’s law of cooling as:

$$Q = Ah(T_s - T_f) \tag{18}$$

A is the area of the solid-fluid interface,

T_s is the solid temperature

T_f is the fluid temperatures .

2.7.3 Radiation

Heat transfer through radiation in a furnace is always small and assumed negligible.

2.8 Furnace Efficiency

There are two methods of measuring furnace efficiency:

i) Input-Output method

$$\eta_{te} = \frac{Q_{stock}}{Q_{in}} \times 100 \qquad (19)$$

ii) heat loss method

$$\eta_{te} = \frac{Q_{in} - Q_{loss}}{Q_{in}} \times 100 \qquad (20)$$

Where η_{te} represent thermal efficiency

Q_{stock} Represent Weight the quantity of output stock

Q_{in} Represent Weight the quantity of input stock

Q_{loss} Represent the total heat losses

2.8.1 Factors determining furnace efficiency

The factors affecting furnace efficiency are excess air, air infiltration, stack loss and Combustion losses (Blasiak and Weihong, 2006).

CHAPTER THREE
MATERIALS AND METHODS

3.1 Materials

The materials used in the development of the reactor are listed in Table 3.1

Table 3.1 Materials used in the development of reactor

Maerials	**Materials**
Heating element	Gas regulator
Heat sensor(36-1200^{0}C)	Gas hose
Refractory binder	Electric wires
Nitrogen gas	Refractory bricks
Galvanized steel plate	Fired bricks plate
50mm diameter galvanized pipe	
Coconut shells	
Potassium hydroxide	

3.2 Methodology

This project was executed in four stages as follows:

I. Collection and preparation of feedstock: In this stage the coconut shells, were sourced for, collected, broken into smaller sizes and dried to reduce moisture.

II. Design and fabrication of reactor : In this stage, the reactor was designed and fabricated using locally sourced materials.

III. Installation and testing of machines: The designed machines was installed and tested.

IV. Production of activated carbons: In this phase activated carbon was produced from coconut shells. The products were characterized.

3.2.1 Collection and preparation of feedstock

Coconut shells, (Figure 3.1) were sourced from the local markets along Badagry expressway in Lagos state, Nigeria. These shells were first cleaned and broken into smaller sizes manually. These smaller shells were air dried to reduce moisture. The dried shells were further grinded into finer sizes (Figure 3.2) in a milling machine. The grinded samples were sieved to obtain 0.425 mm to 2.00 mm particle size. Coarse particles (larger than 2.00 mm) were recycled back to the mill to produce more feedstock.

Figure 3.1 shows samples of coconut shells, after removing the nuts, the shells were collected and exposed to sunlight to remove the moisture present.

Figure 3.1 Coconut Shells

Figure 3.2 Coconut Grinding Machine

Figure 3.2 shows the coconut grinding machine. This machine is domicile in Kuto market Abeokuta

Figure 3.3 Grinded Coconut shells

Figure 3.3 shows the grinded coconut shells, these were obtained from the dried coconut shells. in Figure 3.1 above by first crushing the dried coconut shells into smaller sizes, these crushed samples were exposed to the sun to further remove any moisture still present in the samples before milling into finer samples in a milling machine (Figure 3.2).

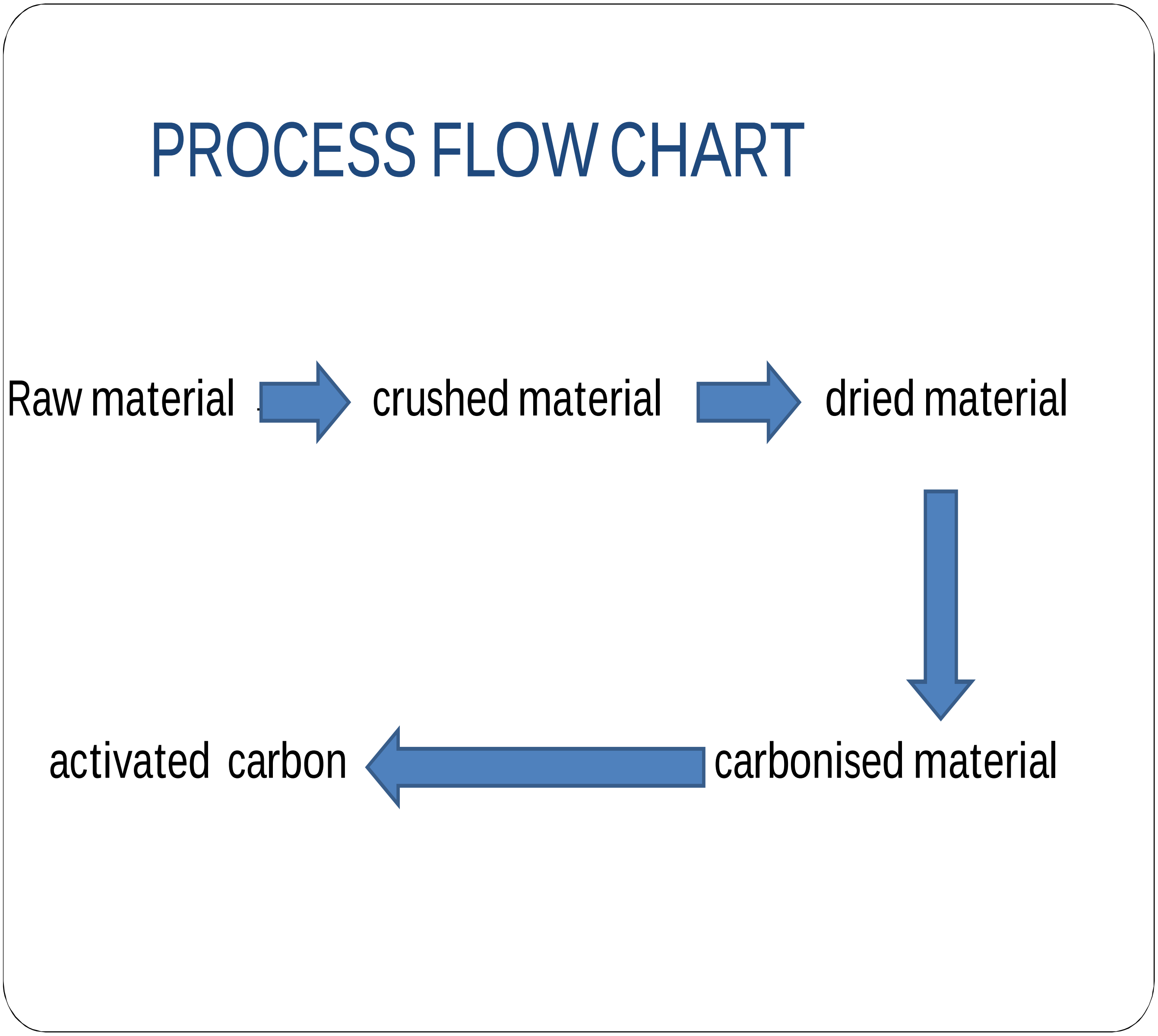

Figure 3.4 Flow Diagram for activated carbon Production

Figure 3.4 shows the flow diagram for the production of activated carbon. Sourcing for the raw materials (coconut shells) is the first step, this involves sourcing the coconut shell from the fields or market, and this is followed by crushing. Crushing involves breaking the shells into smaller and finer sizes. There are two steps involved, first is breaking the shells into smaller sizes in a crusher, which is followed by grinding the crushed samples into finer samples in a milling machine. These finer samples were dried in the sun to remove all moisture present.

The carbonisation and activation stages would be carried out in the reactor and it involves drying

Figure 3.5 Laboratory Electric oven

Figure 3.5 shows laboratory electric oven used on drying the samples, in order to remove moisture. This machine is domicile in Pharmaceutical Department Moshood Abiola Polytechnic Abeokuta. The oven has a maximum temperature of 500°C.

3.2.2 Heat transfer in the furnace

The equation for conductive heat transfer through flat furnace wall with multiple layers is given by equation 14

$$Q = \frac{T_1 - T_3}{\frac{X_A}{K_A \ A} + \frac{X_B}{K_B \ A}} \qquad (14)$$

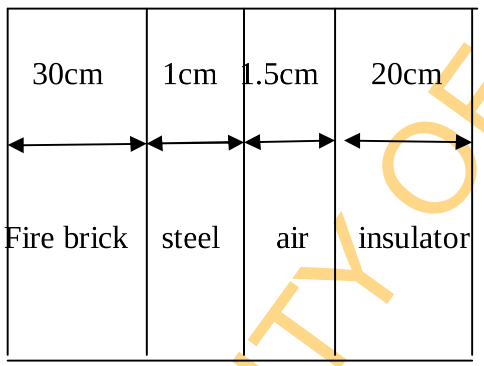

Figure 3.6 Schematic Diagram of Furnace wall

The schematic diagram of the furnace wall is shown in Figure 3.6, the composite furnace wall is made of firebricks, steel, air gap and insulator. The firebricks have thickness of 30 cm, the steel has a thickness of 1 cm, the air gap occupied 1.5 cm^3 and the fibre insulator has a thickness of 20 cm.

Table 3.2 Thermal Conductivity of Refractory Materials

Materials	Thermal Conductivity (W/m ^{o}C)
Arcylic	0.20
Air	0.024
Aluminium	250.00
Copper	401.00
Carbon Steel	54.00
Concrete	1.050
Glass	1.050
Gold	310.00
Nickel	91.00
Paper	0.050
PTFE(Teflon)	0.250
PVC	0.190
Silver	429.00
Steel	46.00
Water	0.580
Wood	0.130

Source : Somers, (2012)

Table 3.3 Thermal Condutivity of Cold Insulating Materials

Material	**Thermal Conductivity (W/m ^{o}C)**
Mineral or Glass Fibre Blanket	0.039
Cellular Glass	0.058
Cork Board	0.043
Glass Fibre	0.0457
Expanded Polystyrene (smooth) thermocole	0.029
Expanded Polystyrene (cut cell) thermocole	0.036
Expanded Ployuethane	0.017
Phenotherm (trade name)	0.018
Loose Fill	
Paper or WoodPulp	0.039
Sawdust or Shavings	0.065
Mineral Wool(Rock,Glass,Slag)	0.039
Wood Fibre Soft	0.43

Source: Bureau of Energy Efficiency, (2005)

The amount of heat transmitted through composite wall of the furnace per unit area is

$$\frac{Q}{A} = \frac{T_1 - T_3}{\frac{X_A}{K_A} + \frac{X_B}{K_B}} \qquad (19)$$

T_1 represent inner/ initial temperature of furnace

T_3 represent outer/ final temperature of furnace

X_A and X_B represents furnace wall thicknesses

K_A and K_B represents thermal conductivities of furnace refractory materials

$$\frac{Q}{A} = \frac{1473-303}{\frac{0.3}{1.63}+\frac{0.01}{16.9}+\frac{0.015}{0.0457}+\frac{0.2}{0.26}}$$

$$\frac{Q}{A} = \frac{1170}{0.184+0.0006+0.328+0.769}$$

$$\frac{Q}{A} = \frac{1170}{1.2816}$$

$$\frac{Q}{A} = 912.92\text{watt/K.m}^2$$

Therefore,

The heat loss through the walls of the furnace is 912.92watt/K.m^2

Temperature drop at the interface $= \frac{temperature\ drop\ over\ A}{total\ temperature\ drop} = \frac{resistance\ of\ A}{total\ resistance}$

$$= \frac{\Delta t_1}{\Delta T} = \frac{\mathrm{r_A}}{R_T}$$

Δt_1 represent temperature drop across fire bricks and steel interface

ΔT represent total temperature drop across furnace wall

$\mathrm{r_A}$ represent resistance of firebricks and steel

R_T represent total resistance across furnace wall

Temperature drop at fire bricks and steel interface

$$\Delta T = T_1 - T_3$$

$$= 1473 - 303 = 1170 \text{ K}$$

$$\mathrm{r_A} = \frac{X_{firebrick}}{K_{firebrick}} + \frac{X_{steel}}{K_{steel}}$$

$$= \frac{0.3}{1.63} + \frac{0.01}{16.9} = 0.1846 \text{ m}$$

$$\mathrm{R_T} = \frac{X_{firebrick}}{K_{firebrick}} + \frac{X_{steel}}{K_{steel}} + \frac{X_{air}}{K_{air}} + \frac{X_{fibre\,glass}}{K_{fibre\,glass}}$$

$$= \frac{0.3}{1.63} + \frac{0.01}{16.9} + \frac{0.015}{0.0457} + \frac{0.2}{0.26}$$

$$= 1.2816 \text{ m}$$

Temperature drop at fire bricks and steel interface

$$= \frac{\Delta t_1}{1170} = \frac{0.1846}{1.2816}$$

$$\Delta t_1 = \frac{1170 \times 0.1846}{1.2816}$$

$= \Delta t_1 = 168.5\ K$

Temperature drop at fire bricks and steel interface $= 1473 - 168.5$

$=1304.5$ K

$\cong 1305\ K$

Temperature drop at air /fibreglass interface

$$\frac{\Delta t_2}{\Delta T} = \frac{\mathrm{r_B}}{R_T}$$

Δt_2 represent temperature drop across air and fibre glass interface

$\mathrm{r_B}$ represent resistance of air and fibre glass

$$r_B = \frac{X_{air}}{K_{air}} + \frac{X_{fibre\,glass}}{K_{fibre\,glass}}$$

$$= \frac{0.015}{0.0457} + \frac{0.2}{0.26} = 0.328\ m$$

Temperature drop at air /fibreglass interface

$$\frac{\Delta t_2}{1170} = \frac{0.328}{1.2816}$$

$$\Delta t_2 = \frac{1170 \times 0.328}{1.2816}$$

$= \Delta t_2 = 299.4\ K$

Temperature at air /insulator interface $= 1305 - 299.4$

$=1005.6$ K

$\cong 1006\ K$

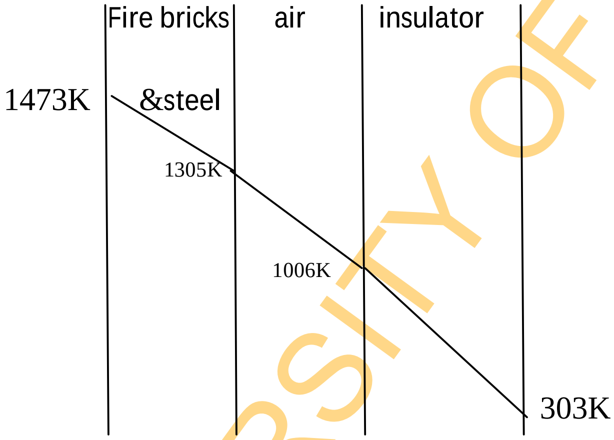

Figure 3.7 Furnace Wall temperature Profile

Temperature gradient across the furnace wall, is shown in Figure 3.7, from an initial temperature of 1473 K, inside the furnace, the temperature fell to 1305 K across the firebricks and steel and to 1006 K across the air gap before attaining 303 K at the outer shell of the furnace across the insulator.

3.2.3 Design consideration for sizing

The furnace has a volumetric capacity for 800 g of coconut shell but consideration was made for higher volume. For this reason (350 x 350 cm) was used. This dimension is adequate since larger internal volume would require more energy input to raise the furnace temperatures. The outer dimension of the furnace is (520 x 550 x 550 cm) because of the allowance for the refractory bricks.

3.2.4 Design for inert furnace environment

Nitrogen gas was continuously passed into the furnace through an orifice of 2 mm at the inlet of the furnace to prevent oxidation of the samples to ash

3.2.5 Selection of Refractory Materials

The furnace lining was designed in two layers; the inner working lining and the outer insulating lining. The inner working lining was made 300 mm thick and the insulating lining was made 200 mm thick. These linings were interspersed with air to reduce the heat loss in the furnace.

3.2.5.1 Aluminosilicate Ceramic Properties

Aluminosilicates is one of the most cost effective and widely used materials in the family of engineering ceramics. The raw materials from which this high performance technical grade ceramic is made are readily available and reasonably priced, resulting in good value for the cost in fabricated aluminosilicate refractory. Aluminosilcates has a very wide range of applications such as good thermal conductivity, hard wear resistance and excellence dielectric properties.

Table 3.4 Mechanical Properties of Aluminosilicate Refractory

Mechanical	**Units of Measure**	**SI/Metric**	**(Imperial)**
Density	gm/cc (lb/ft^3)	3.89	(242.8)
Porosity	% (%)	0	(0)
Colour	—	ivory	—
Flexural Strength	MPa (lb/in^{2}x10^3)	379	(55)
Elastic Modulus	GPa (lb/in^{2}x10^6)	375	(54.4)
Shear Modulus	GPa (lb/in^{2}x10^6)	152	(22)
Bulk Modulus	GPa (lb/in^{2}x10^6)	228	(33)
Poisson's Ratio	—	0.22	(0.22)
Compressive Strength	MPa (lb/in^{2}x10^3)	2600	(377)
Hardness	Kg/mm^2	1440	—
Fracture Toughness K_{IC}	MPa•m$^{1/2}$	4	—
Maximum Use Temperature (no load)	°C (°F)	1750	(3180)
Thermal			
Thermal Conductivity	W/m°K (BTU•in/ft^2•hr•°F)	35	(243)
Coefficient of Thermal Expansion	10^{-6}/°C (10^{-6}/°F)	8.4	(4.7)
Specific Heat	J/Kg•°K (Btu/lb•°F)	880	(0.21)
Electrical			
Dielectric Strength	ac-kv/mm (volts/mil)	16.9	(420)
Dielectric Constant	@ 1 MHz	9.8	(9.8)
Dissipation Factor	@ 1 kHz	0.0002	(0.0002)
Loss Tangent	@ 1 kHz	—	—
Volume Resistivity	ohm•cm	>10^{14}	—

Source: Perti (1996)

Table 3.5 Thermal Conductivity of Hot Insulation

Mean temperature °C	Calcium Silicate	Resin Bonded Mineral Wool	Ceramic Fiber Blankets
100	-	0.04	-
200	0.07	0.06	0.06
300	0.08	0.08	0.07
400	0.08	0.11	0.09
700	-	-	0.17
1000	-	-	0.26
Specific heat (kJ/kg °C)	0.96 (at 40 °C)	0.921 (at 20 °C)	1.07 (at 980 °C)
Service temp(° C)	950	700	1425
Density kg/m^3	260	48 to144	64 to 128

Source: Bureau of Energy Efficiency, (2005)

Owing to the mechanical properties of aluminosilicate (Tables 3.4 and Table 3.2), aluminosilicates bricks were selected for the inner lining while fibre glass was selected as the outer insulating lining because of its low thermal conductivity value and mechanical properties (Table 3.3).

The aluminosilicates were cast into 6 slabs of dimension: 31cm x 21cm x 3cm, (for the furnace base, roof, sides and the inlet)

$$surface\ area\ of\ furnace = 0.31\,m \times 0.21\,m$$

$$surface\ area\ of\ furnace = 6.5\,m^2$$

$$height\ of\ furnace = 0.21\,m$$

$$Therefore, volume\ of\ furnace = surface\ area \times height$$

$$volume\ of\ furnace = 6.5m^2 \times 0.21\,m$$

$$volume\ of\ furnace = 13.65\,m^3$$

3.2.6 Principle of Design:

For a constant temperature of the furnace, the electric power must equal the rate of heat losses to the surroundings by convection and radiation. Heat flows by conduction from inside wall to the outer wall through the refractory thickness. From the outside wall, heat is lost partly by radiation heat balance gives the followings:

$$Q_k = \frac{T_f - T_s}{\frac{X_A}{K_A\ A} + \frac{X_B}{K_B\ A}} \tag{20}$$

$$Q_c = Q_k = Ah\left(T_s - T_f\right) + \sigma A\left(T_s^4 - T_o^4\right) \tag{21}$$

$$Q_k = Q_c = \mathrm{VI} \tag{22}$$

Where Q_k = heat flow by conduction

Q_c = heat flow by convection and radiation

T_f = furnace inner temperature

T_s = Temperature of the outside wall of the furnace

T_o = ambient temperature

K = thermal conductivity of the refractory

h = heat transfer coefficient

σ = Stefan Boltzmann constant

A = Area

V = voltage

I = current

3.2.7 Reactor /Furnace Construction Procedure

The galvanized plates was marked out after which the marked portion were cut out .These were welded and fitted together, to form the furnace frame/body. The plates were welded into a rectangular box with opening at both ends; this is the heating chamber of the reactor. The rectangular box was welded to the frame. Aluminosilicates were cast into six firebricks of dimension: 3 1cm x 21 cm x 3 cm, (6pieces ; for the furnace base, roof, rear, 2 sides and the inlet). The refractory firebricks (Figure 3.8) were marked at 10 mm on the inside and outer parts of the bricks. These were sawn, making grooves on the bricks, for placement of nichrome heating elements. The grooved firebricks were arranged in the heating chamber of the furnace frame. The heating elements were stretched and fixed in the grooves of the firebricks, (Figure 3.10), the remaining firebricks (Figure 3.8) were fitted as appropriate. Fibre Glass (Figure 3.9) was stuffed between the outer furnace shell and the fired bricks. Steel plates thickness 1 cm was used to enclose air between the firebircks and the fibre glass.

Figure 3.8 Fibre bricks

Figure 3.9 Fibre Glass insulator

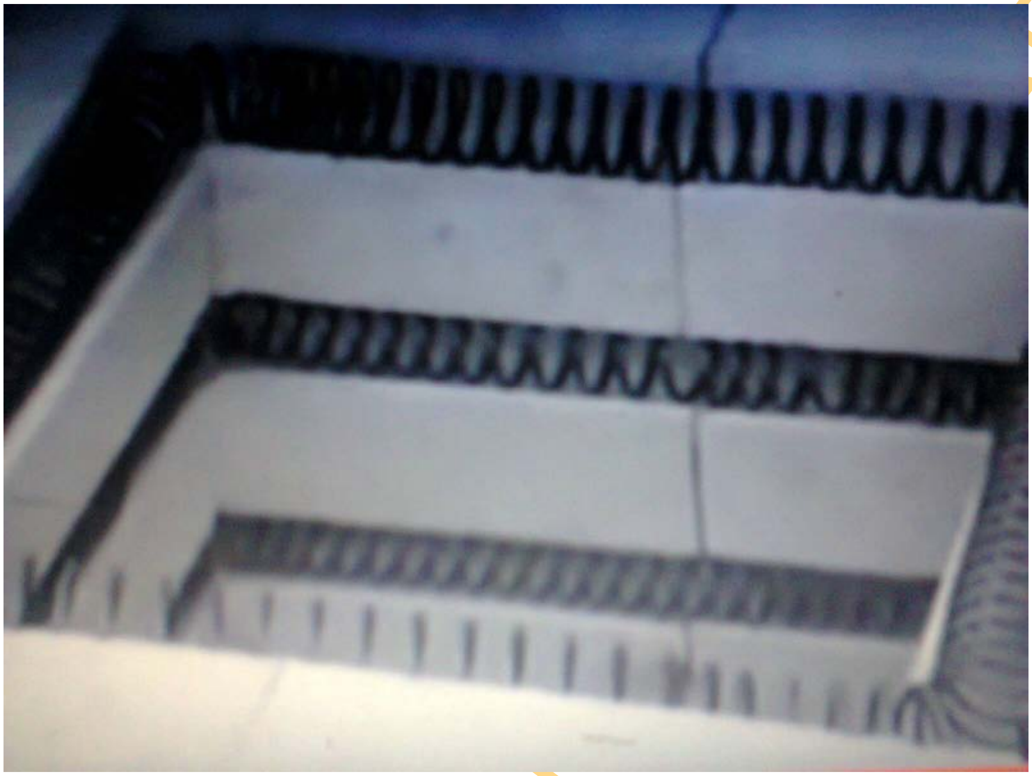

Figure 3.10 Heating elements fitted in fire bricks

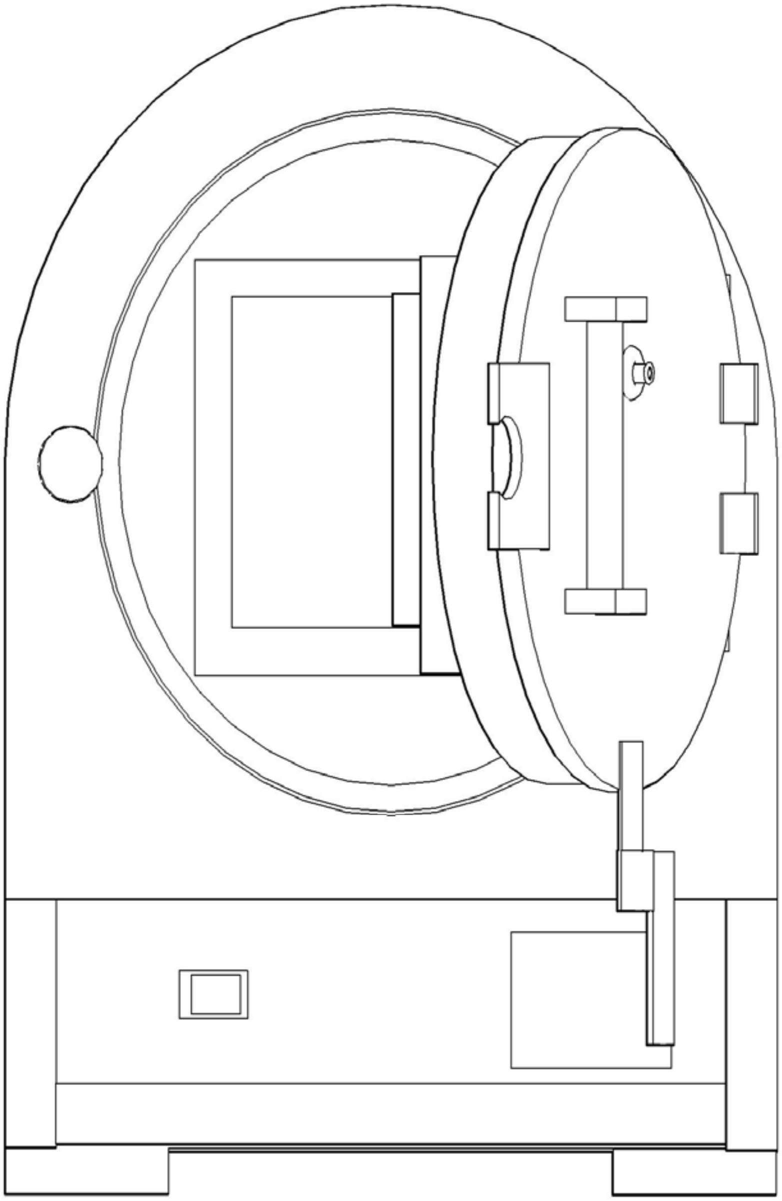

Figure 3.11 Furnace Front View

Figure 3.11 shows, the front view of the furnace reactor, the door of the reactor located in front , the control section is located at the base , it houses the control panel, thermocouple gives readings to the control panel at the bottom . The power switch is located at the base near the control panel.

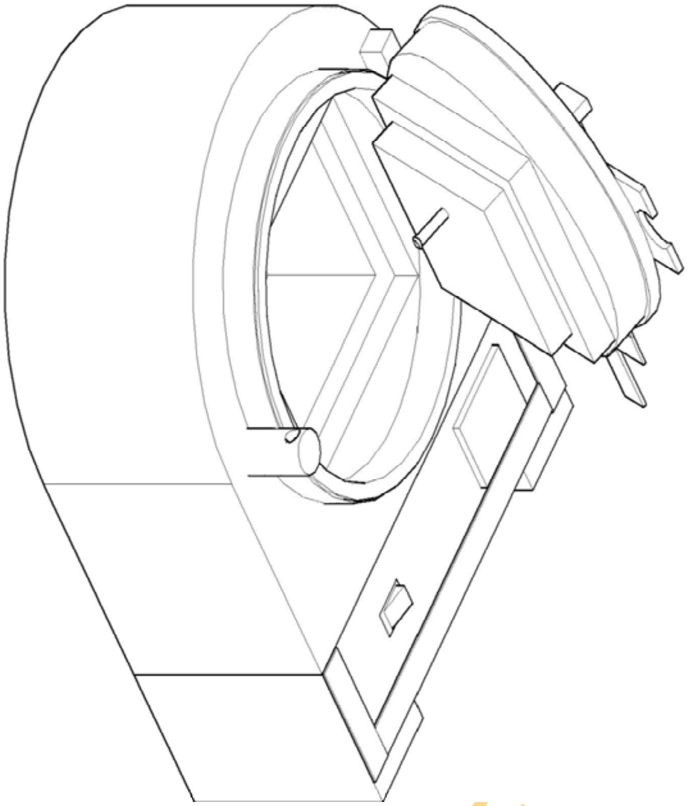

Figure 3.12 Furnace Side View

Figure 3.12 shows the side view of the furnace, the fire bricks were fitted to the door of the furnace. Protruding from the door is a pipe of 2 mm diameter bored through the door passing through the refractory attached to the door into the heating chamber. It serves as nitrogen gas inlet. The door closes with the furnace by means of a latch attached to it. The power switch is shown at the base of the furnace.

3.2.8 Design of heating unit

The heating unit consist of two nichrome heating elements (Figure 3.14) connected in series a type K thermocouple, contactor, thermo regulator and temperature indicator. The heating element were designed to be placed at the sides of the furnace imbedded inside the inner refractory lining. The sensors (connected to the thermocouple) measures and transmit the temperature to the regulator which trips off the heating elements when above the preset temperature.

Figure 3.13 Heating element

Figure 3.14 Furnace Refractory

Figure 3.14 shows the heating chamber of the furnace with the firebricks refractory of dimension 31x 21x 3 cm, arranged in rectangular form. A thermocouple is attached at the rear to sense the temperature in the heating chamber. The thermocouple acts as a sensor. As it senses the heat that is hot enough, it will turn that heat into an electric signal and sends it over to the contactor in the control panel.

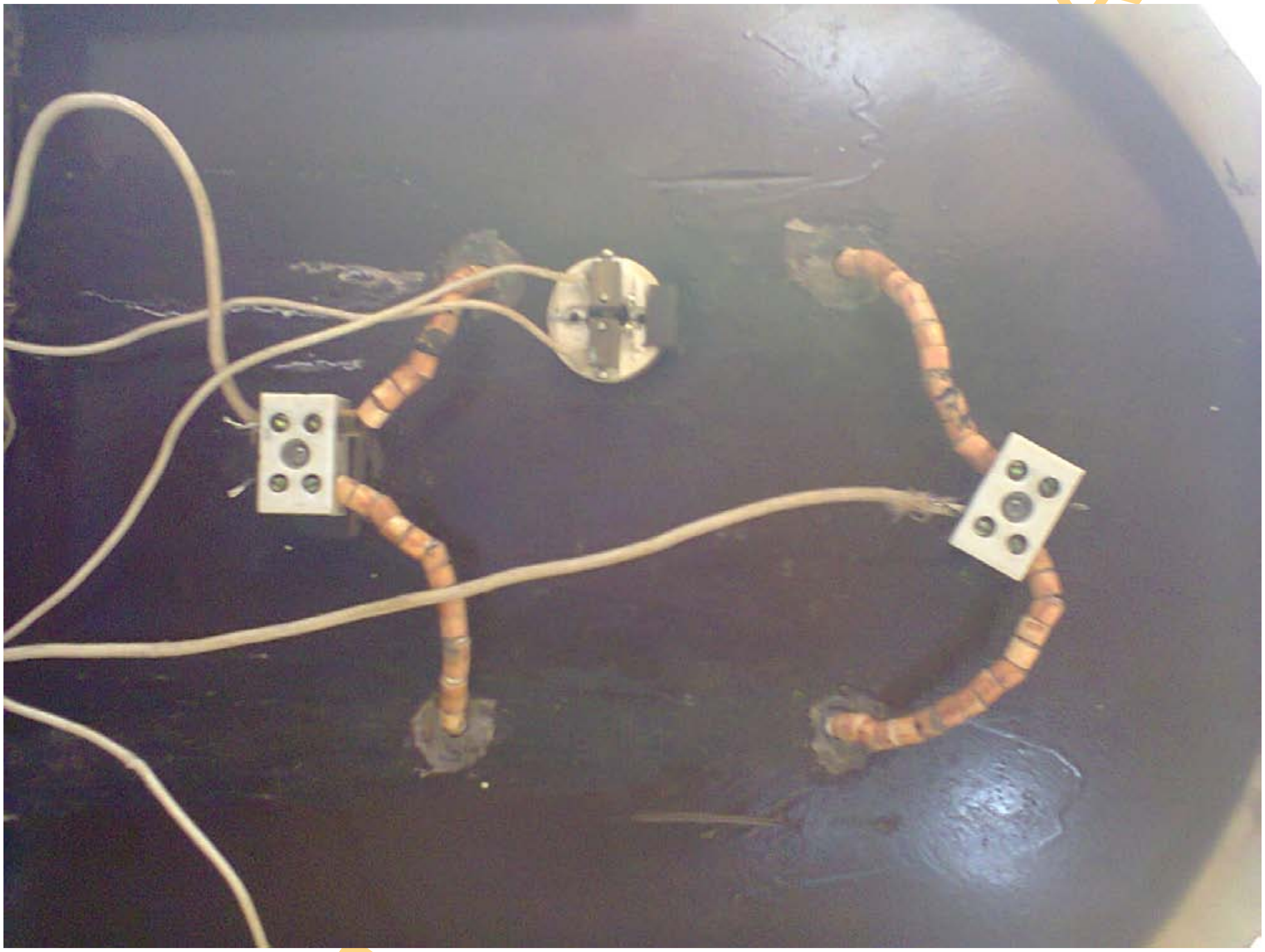

Figure 3.15 Furnace Rear View

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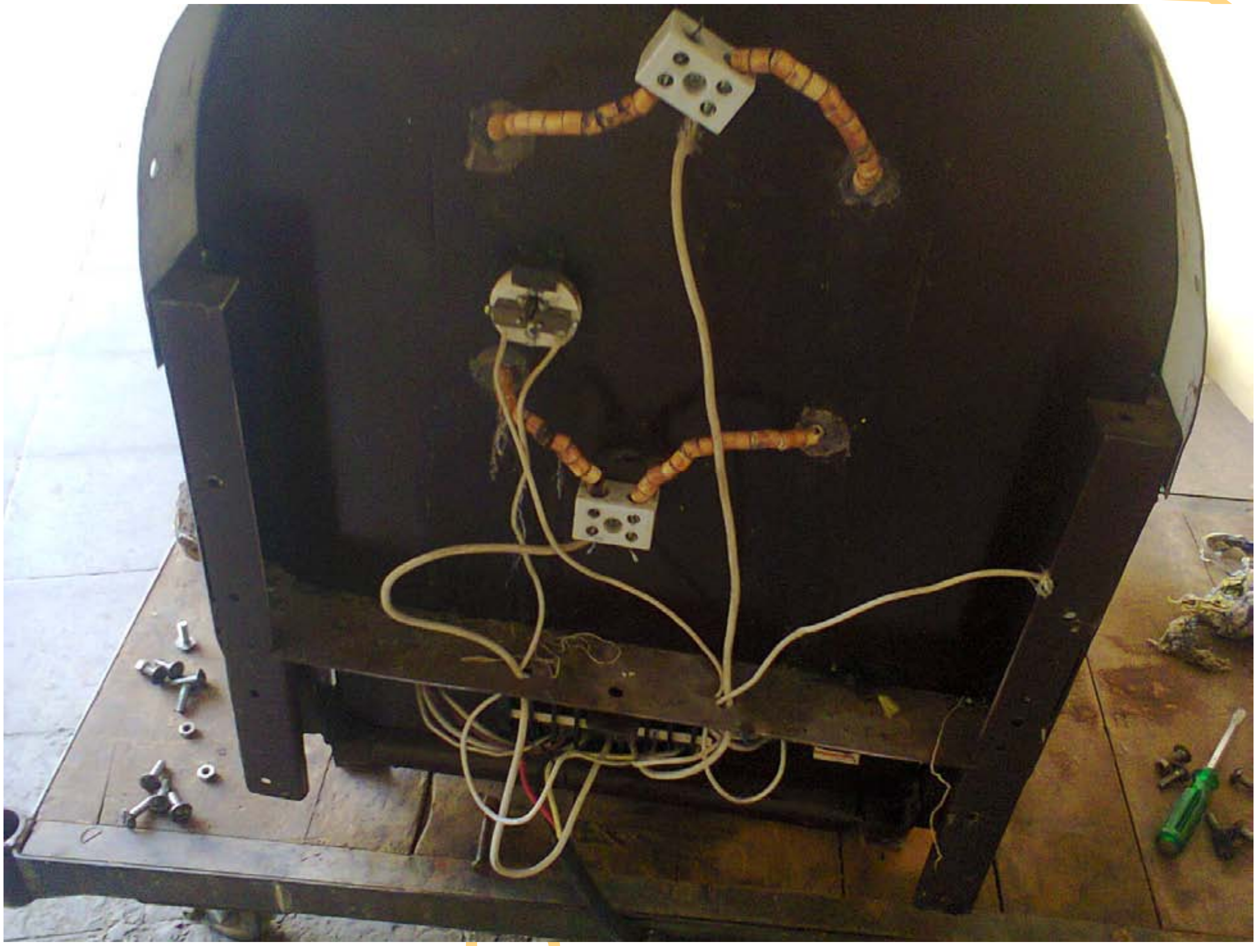

Figure 3.16 Furnace Rear View

Figure 3.16 shows how heating elements are connected in series while Figure 3.17 shows the connection between the terminals of the heating elements and the control panel.

Figure 3.17 Furnace Control Panel

Furnace temperature Control (Figure 3.17) provides independent zone control. It includes; i) circuit breaker: A circuit breaker is serves to turn electricity ON and OFF and to protect the electrical circuit. ii) Controllers: this consists of a microprocessor based three-mode (Proportional, Integral, and Derivative), programmable control with over temperature protection and appropriate output switching devices to control the furnace. iii) contactor, iv) thermo regulator and v) temperature indicator.

The heating elements were designed to be placed at the sides of the furnace imbedded inside the inner refractory lining. The sensors (connected to the thermocouple) measures and transmit the temperature to the regulator which trips off the heating elements when above the preset temperature.

Figure 3.18 Furnace Crucibles

3.2.9 Furnace crucibles

Figure 3.18 shows the crucibles placed in the furnace, they were made of fired clay such that they could withstand the high temperature in the furnace.

3.3 Testing

The furnace was subjected to different loading conditions; 0, 25, 50, 85 and 100% and the temperature profile were drawn at various loading condition.

3.4 Production

200 grams of the grinded coconut shells sample were dried in an electric oven at 110 ^{o}C for 48 hours in order to remove all moisture present in the sample. These were impregnated with potassium hydroxide at ratio 3: 2 (KOH: Sample). A typical carbonization run began by charging 20 grams of impregnated sample in the reactor and heating it up to the carbonization temperature in flowing stream of nitrogen (Wan Daud and Wan Ali, 2004). The temperature of the reactor was increased at the rate of 10 ^{o}C/min until it reached the final carbonization temperature. Since most literatures reported that optimum activation temperature for most biomass materials generally fall between 400 and 500 ^{o}C (Srinivassakannan and Abubakar, 2004), an average of 450 ^{o}C was chosen as the final carbonization temperature. The carbonized samples were subjected to this temperature for 1hour and 3 hours. The samples were cooled down under nitrogen flow and were washed sequentially with warm water and finally with cold water to remove residual chemicals .The samples were dried at 110 ^{o}C in an electric oven. (Figure 3.5)

Figure 3.19 Production of Activated Carbon

Figure 3.19 shows the production of activated carbon, nitrogen gas was passed through the tubes connected to the cylinder into the heating chamber through the orifice at the door to prevent oxidation of the samples in the reactor to ashes. The furnace has been set to the activation temperature of 450 ^{o}C.

3.5 Characterisation

The samples were characterized using X-ray diffraction (XRD), (Figure 3.20) and Scanning Electron Microscope (SEM), (Figure 3.21).

Figure 3.20 X-Ray Diffraction Machine

Figure 3.20 shows the X-ray Diffraction machine that was used for the characterization of the activated samples. The X-ray machine is domicile in the department of chemistry ,KwaZulu-Natal University, KwaZulu-Natal, South Africa.

Figure 3.21 Scanning Electron Microscope

Figure 3.21 shows the scanning electron Microscope that was used for the characterization of the activated samples. The Scanning electron Microscope is domicile in the Department of Chemistry, Kwazulu -Natal University, KwaZulu- Natal, South Africa.

CHAPTER FOUR
RESULT AND DISCUSSION

The results are presented as follows.

4.1 Furnace Temperature Profile

Table 4.1.Temperature Profile of Furnace at Zero Loading Condition

Temp (°C)	Time (min)	Temp (°C)	Time (min)	Temp (°C)	Time (min)	Temp (°C)	Time (min)
36	0.00	230	2.08	420	3.79	625	5.64
50	0.46	240	2.17	430	3.88	650	5.86
60	0.55	250	2.26	440	3.97	675	6.09
70	0.64	260	2.35	450	4.06	700	6.31
80	0.73	270	2.44	460	4.15	725	6.54
90	0.82	280	2.53	470	4.24	750	6.76
100	0.91	290	2.62	480	4.33	775	6.99
110	1.0	300	2.71	490	4.42	800	7.22
120	1.09	310	2.80	500	4.51	825	7.44
130	1.18	320	2.89	510	4.60	850	7.67
140	1.27	330	2.98	520	4.69	875	7.89
150	1.36	340	3.07	530	4.78	900	8.12
160	1.45	350	3.16	540	4.87	925	8.34
170	1.54	360	3.25	550	4.96	950	8.57
180	1.63	370	3.34	560	5.05	975	8.79
190	1.72	380	3.43	570	5.14	1000	9.02
200	1.81	390	3.52	580	5.23		
210	1.90	400	3.61	590	5.32		
220	1.99	410	3.70	600	5.41		

Table 4.2.Temperature Profile of Furnace at 100% Loading Condition

Temp (^{o} C)	Time (min)	Temp (^{o} C)	Time((min)	Temp (^{o} C)	Time (min)	Temp (^{o} C)	Time (min)
36	0.00	230	45.00	420	87.75	625	133.86
50	4.50	240	47.25	430	90.00	650	139.5
60	6.75	250	49.50	440	92.25	675	145.13
70	9.00	260	51.75	450	94.25	700	150.75
80	11.25	270	54.00	460	96.75	725	156.38
90	13.50	280	56.25	470	99.00	750	162
100	15.75	290	58.50	480	101.25	775	167.63
110	18.00	300	60.75	490	103.50	800	173.25
120	20.25	310	63.00	500	105.75	825	178.88
130	22.50	320	65.25	510	108.00	850	184.5
140	24.75	330	67.50	520	110.25	875	190.13
150	27.00	340	69.75	530	112.50	900	195.75
160	29.25	350	72.00	540	114.75	925	201.38
170	31.50	360	74.25	550	117.00	950	207.00
180	33.75	370	76.50	560	119.25	975	212.63
190	36.00	380	78.75	570	121.50	1000	218.25
200	38.25	390	81.00	580	123.75		
210	40.50	400	83.25	590	126.00		
220	42.75	410	85.50	600	128.25		

Table 4.3.Temperature Profile of Furnace at 85% Loading Condition

Temp (^{o}C)	Time (min)	Temp (^{o}C)	Time (min)	Temp (^{o}C)	Time (min)	Temp (^{o}C)	Time (min)
36	0.00	230	36.00	420	70.20	625	107.1
50	3.60	240	37.80	430	72.00	650	111.6
60	5.40	250	39.60	440	73.80	675	116.1
70	7.20	260	41.40	450	75.60	700	120.6
80	9.00	270	43.20	460	77.40	725	125.1
90	10.80	280	45.00	470	79.20	750	129.6
100	12.60	290	46.80	480	81.00	775	134.1
110	14.40	300	48.60	490	82.80	800	138.6
120	16.20	310	50.40	500	84.60	825	143.1
130	18.00	320	52.20	510	106.20	850	147.6
140	19.80	330	54.00	520	108.00	875	152.1
150	21.60	340	55.80	530	109.80	900	156.6
160	23.40	350	57.60	540	111.60	925	161.1
170	25.20	360	59.40	550	113.40	950	165.6
180	27.00	370	61.20	560	115.20	975	170.1
190	28.80	380	63.00	570	117.00	1000	174.6
200	30.60	390	64.8	580	118.80		
210	32.40	400	66.6	590	120.60		
220	34.20	410	68.40	600	122.40		

.

Table 4.4.Temperature Profile of Furnace at 50% Loading Condition

Temp (^{o}C)	Time (min)	Temp (^{o}C)	Time((min)	Temp (^{o}C)	Time (min)	Temp (^{o}C)	Time (min)
36	0.00	230	22.40	420	43.68	625	66.64
50	2.24	240	23.52	430	44.80	650	69.44
60	3.36	250	24.64	440	45.92	675	72.24
70	4.48	260	25.76	450	47.04	700	75.04
80	5.60	270	26.88	460	48.16	725	77.84
90	6.72	280	28.00	470	49.28	750	80.64
100	7.84	290	29.12	480	50.40	775	83.44
110	8.96	300	30.24	490	51.52	800	86.24
120	10.08	310	31.36	500	52.64	825	89.04
130	11.2	320	32.48	510	53.76	850	91.84
140	12.32	330	33.60	520	54.88	875	94.64
150	13.44	340	34.72	530	56.00	900	97.44
160	14.56	350	35.84	540	57.12	925	100.24
170	15.68	360	36.96	550	58.24	950	103.04
180	16.80	370	38.08	560	59.36	975	105.84
190	17.92	380	39.20	570	60.48	1000	108.64
200	19.04	390	40.32	580	61.00		
210	20.16	400	41.44	590	62.72		
220	21.28	410	42.56	600	63.84		

.

Table 4.5.Temperature Profile of Furnace at 25% Loading Condition

Temp (° C)	Time (min)	Temp (° C)	Time (min)	Temp (° C)	Time (min)	Temp (° C)	Time (min)
36	0.00	230	7.40	420	14.36	625	21.95
50	0.74	240	7.77	430	14.73	650	22.87
60	1.11	250	8.14	440	15.10	675	23.80
70	1.48	260	8.51	450	15.47	700	24.72
80	1.85	270	8.88	460	15.84	725	25.65
90	2.22	280	9.25	470	16.21	750	26.57
100	2.59	290	9.62	480	16.58	775	27.50
110	2.96	300	9.99	490	16.95	800	28.42
120	3.33	310	10.36	500	17.32	825	29.35
130	3.70	320	10.73	510	17.69	850	30.27
140	4.07	330	11.10	520	18.06	875	31.20
150	4.44	340	11.47	530	18.43	900	32.12
160	4.81	350	11.84	540	18.80	925	33.05
170	5.18	360	12.21	550	19.17	950	33.97
180	5.55	370	12.58	560	19.54	975	34.90
190	5.92	380	12.95	570	19.91	1000	35.82
200	6.29	390	13.25	580	20.28		
210	6.66	400	13.62	590	20.65		
220	7.03	410	13.99	600	21.02		

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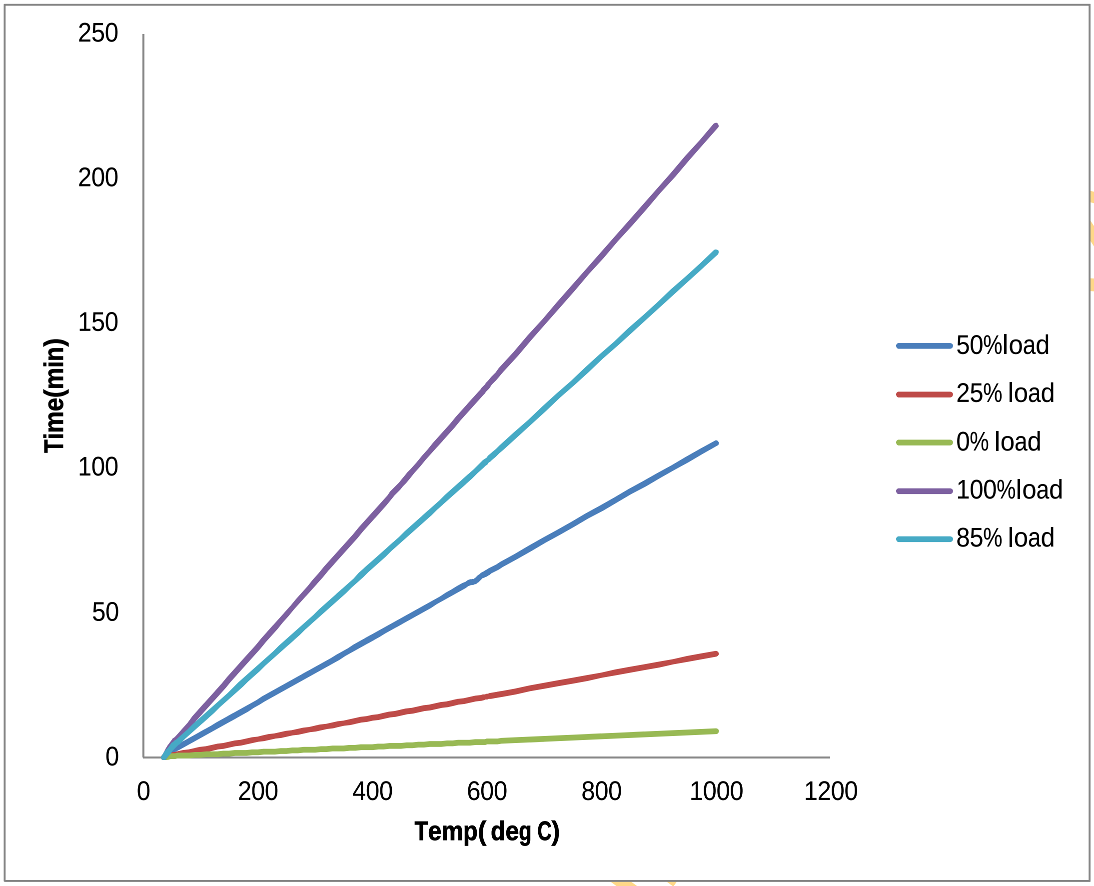

Figure 4.1 Temperature Profile of Furnace at Different loadings

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4.2 Furnace Thermal Efficiency

Table 4.6 Furnace Thermal Efficiency

Voltage (volts)	Resistance (ohms)	Time (min)	Heat input (Q_I) $\frac{V^2}{R \times 4186.8}T$	Heat loss (Q_{loss})	Efficiency $\frac{(Q_{i-}Q_{loss})}{Q_i}\%$
240	20	218.25	9007.7	912.92	79.95

4.3 Thermal Stress and Thermal Strain

Table 4.7.Thermal Stress and Thermal Strain

Young modulus (E) G N/m^2	Coefficient of expansion $(\propto)\ 10^{-6}/^{o}F$	Temperature change $(\Delta T)\ ^{o}F$	Thermal stress $(\sigma = E \propto \Delta T)$ MN/m^2	Thermal Strain $(e = \propto \Delta T)$ 10^{-3}
300	8.1	687	166.9	5.6

Table 4.7 shows the thermal stress and thermal strain of the furnace at optimum production condition.

4.4 Produced Activated Carbon

Figure 4.2 shows samples of activated carbon produced in the furnace.

Figure 4.2 Produced Activated carbon

4.5 Scanning Electron Micrograph of Activated Carbon

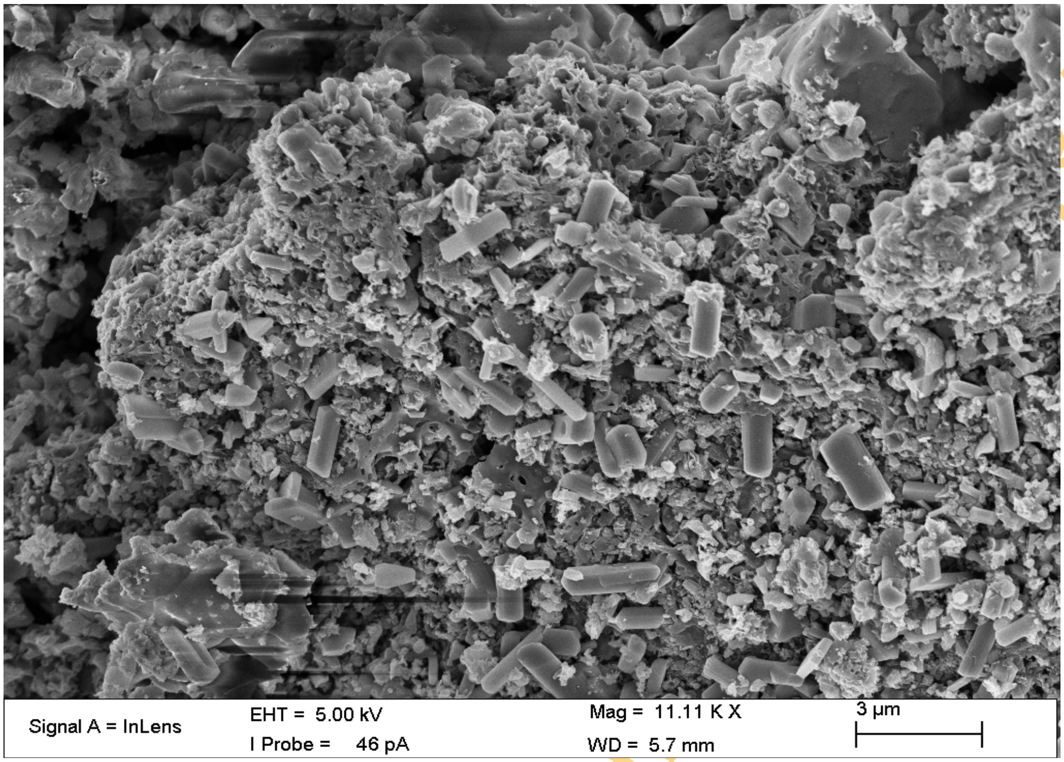

Figure 4.3 Scanning Electron Micrograph of grinded coconut shell sample

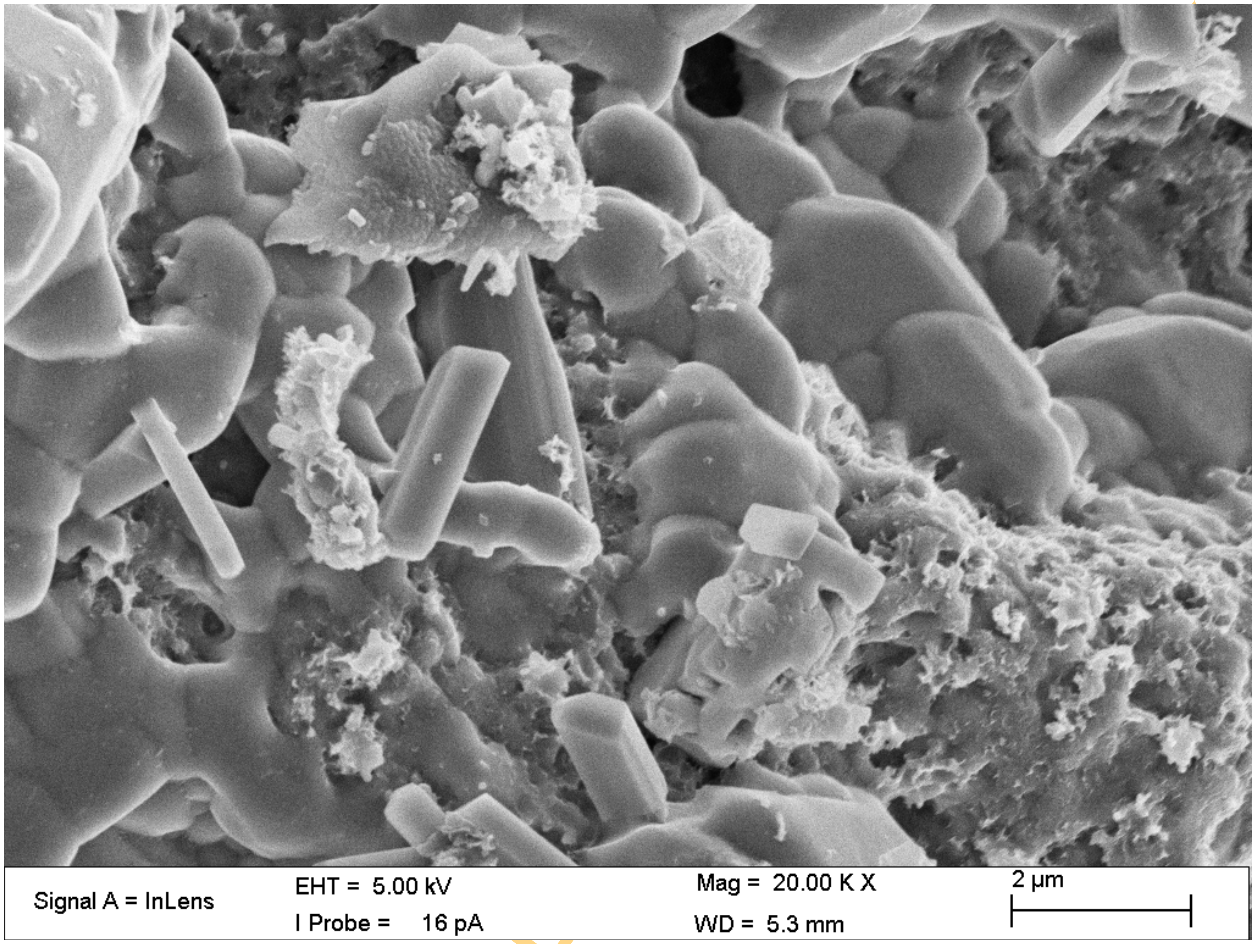

Figure 4.4 Scanning Electron Micrograph of Carbonised sample

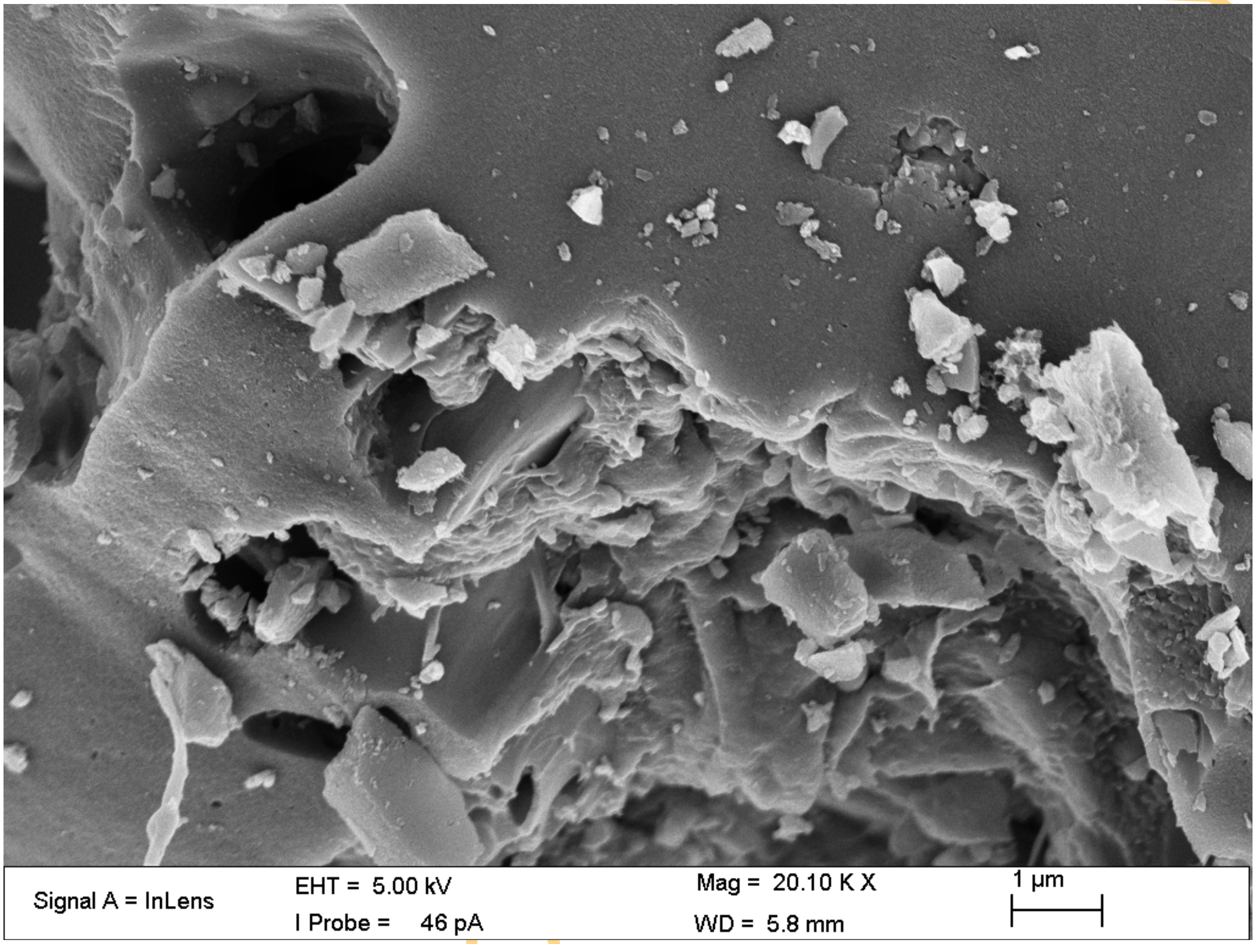

Figure 4.5 Scanning Electron Micrograph of Activated Carbon at One Hour of Activation

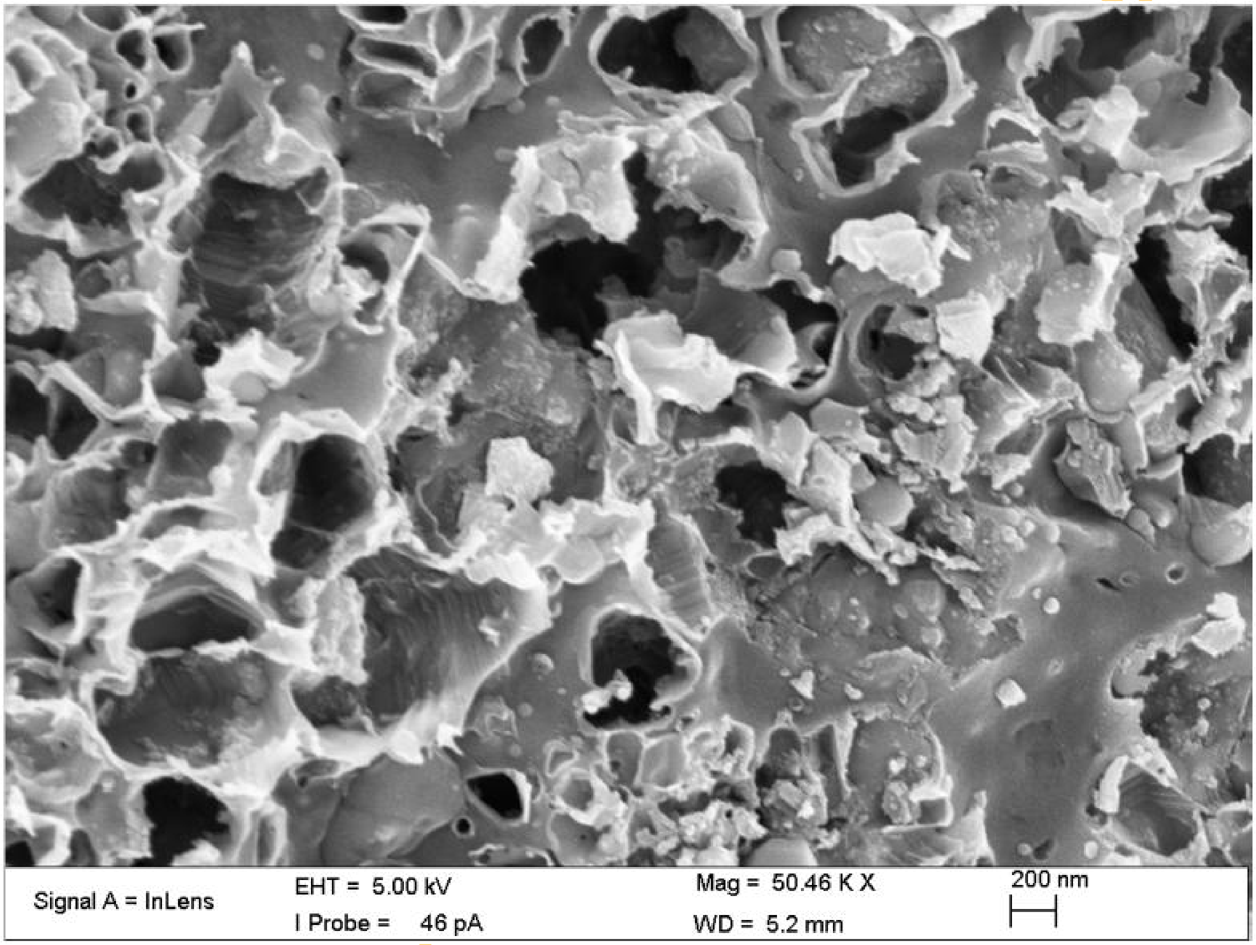

Figure 4.6 Scanning Electron Micrograph of Activated Carbon at Three Hours Activation

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4.6 X- Ray Diffraction

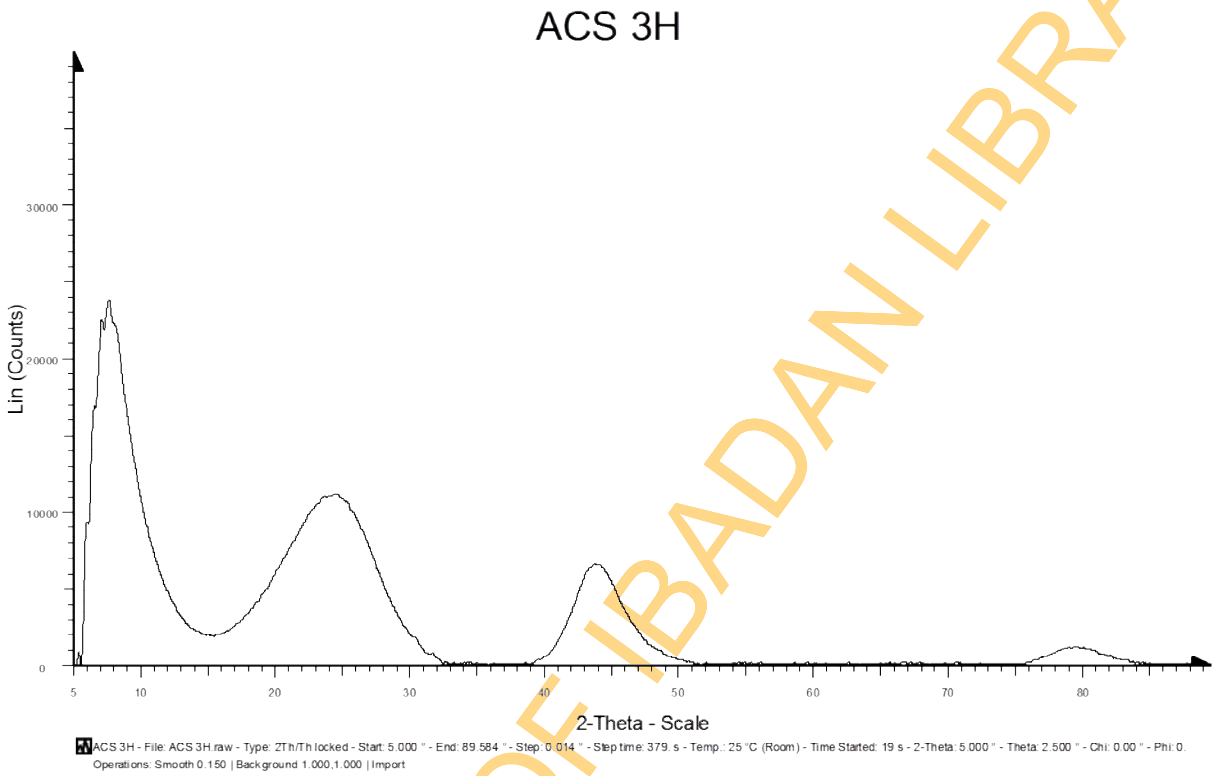

Figure 4.7 X-ray Diffraction Pattern of Activated Carbon

4.7 Discussion

The results presented are discussed as follows.

4.7.1 Reactor linings

The reactor lining was designed in two layers; the inner working lining and the outer insulating lining, this was done to reduce heat loss in the furnace wall. The quantity of heat loss in a single layer furnace is much more than in multilayer furnace. The inner working lining has a thickness of 300 mm while the outer insulating lining has a thickness of 200 mm. These linings were interspersed with air to further reduce the heat loss in the furnace wall. The presence of the air gap between the two linings further helped in reducing the heat loss, air being a good insulator.

Aluminosilicates bricks were selected for the inner lining and fibre glass was used as the outer insulating lining.

4.7.2 Inner lining

Aluminosilicate is preferred to silica bricks as refractory material because the silica will react with the carbonaceous fumes to form glassy silicates which will ingress into the refractories and short circuit the heating elements. In addition, aluminosilicates bricks were selected for the inner lining owing to its ability to

i. withstand high temperatures and trap heat within a limited area like a furnace;
ii. withstand action of molten metal, hot gasses and slag erosion etc;
iii. withstand load at service conditions;
iv. resist contamination of the material with which it comes into contact;
v. maintain sufficient dimensional stability at high temperatures and after/during repeated thermal cycling
vi. conserve heat and because of
vii. its low coefficient of linear expansion and high thermal resistance

(United Nations Environment Programme, 2006)

4.7.3 Outer insulating lining

High-temperature processes require a considerable amount of energy. Often the energy consumption for high-temperature processes is used only partially for the actual technical process and 30 to 40% energy escapes through the walls into the atmosphere. To optimize the energy use and to prevent its escape into the ambience, insulating refractories are necessary. The function of insulating refractory is to reduce the rate of heat flow (heat loss) through the walls of furnaces. Insulation is effected by providing a layer of material with low thermal conductivity and high porosity in order to reduce the rate of heat flow (heat losses) and thus to maximize heat conservation within the furnace.

Insulating fire bricks often suffer from thermal spalling problems, particularly in an environment of rapidly changing temperature, since these bricks are good insulators, a substantial temperature gradient occurs between the hot and the cold face of each brick. The hot face will expand more than the cold face. The thermal gradient thus, gives rise to a mechanical stress in the body of the brick.

Unlike insulated fire bricks, thermal efficiency of a furnace lined with fibre is improved in two ways. First, the low thermal conductivity of fibre allows the lining to be thinner and therefore the furnace can be smaller. Second, the fast response of fibre to temperature changes also allows for more accurate control and uniform temperature distribution within the furnace.

Other advantages offered by fibre are:

i. Lower thermal conductivity; Because of the low thermal conductivity, it is possible to construct thinner linings with the same thermal efficiency as conventional refractories. As a result of thinner lining, the furnace volume is higher.

ii. Light weight; the average density of ceramic fibre is 96 kg/m^3. It is one tenth of the weight of insulating brick and one third of the weight of asbestos / calcium silicate boards.

iii. Lower heat storage; fibre linings absorb less heat because of their lower density. Furnaces can therefore be heated and cooled at faster rates. Typically the heat stored fibre lining system is in the range of 2700 - 4050 cal/m^2 as compared to 54200-493900 kcal/m^2 for conventionally lined systems.

iv. Thermal shock resistant; fiber linings resist thermal shock due to their resilient matrix. This also allows for faster heat up and cools down cycles, thereby improving furnace availability and productivity.

v. Chemical resistance; fibre resist most of the chemical attack and is unaffected by hydrocarbons, water and steam present in flue gases.

vi. Mechanical resilience; The high mechanical resilience of fibre makes it possible to manufacture fibre-lined furnaces off-site, transport them to the site in assembled form without the risk of damage.

vii. Low installation cost; as the application of ceramic fibres is a standardized process, no special skills are required. Fibre linings require no dry out or curing times and there is no risk of cracking or spalling when they are heated after installation.

viii. Ease of maintenance; In case of physical damage, the section of damaged ceramic fibre can be quickly removed and replaced with a new piece.

ix. Ease of handling; all product forms are easily handled and most can be quickly cut with a knife or scissors. (United Nations Environment Programme, 2006)

4.7.4 Heating element

To forestall oxidization of the heating elements during production process, nichrome heating elements were selected for heating the furnace. Nichrome heating elements are corrosion resistant because of the nickel and chromium. The elements are thus able to attain very high temperature (Table 4.8)

Table 4.8 Melting Points of Elements

KEY MATERIALS	MELTING TEMPERATURES (°C)
Iron	2768
Nickel	2618
Copper	1948
Aluminum	1188
Zinc	748
Lead	588
Tin	418

Source: Bhatia, (2011)

4.7.5 Inert nature of furnace

Carbonization and activation processes are carried out inside the reactor in an inert environment, to achieve this inert condition, the inlet of the reactor has an orifice of 2 mm to enable continuous passage of Nitrogen gas into the furnace to prevent oxidation of the samples to ash .

4.7.6 Charging and discharging

The reactor is not a continuous furnace, as such materials are manually charged and discharged intermittently in the front by pulling the handle attached to the door.

4.7.7 Thermal regulator and timer

The heating unit consist of two nichrome heating elements connected in series, a type K thermocouple, contactor, thermo regulator and temperature indicator.

The heating elements were designed to be placed at the sides of the furnace imbedded inside the inner refractory lining. The sensors (connected to the thermocouple) measures and transmit the temperature to the regulator which trips off the heating element when above the preset temperature.

4.8.1. Furnace temperature profile

Temperature profiling is the process of recording and interpreting temperatures of products as they move through either a continuous or batch heating process (typically an oven or furnace). The collected numeric data is converted by temperature analysis software into meaningful information and displayed as a graph – thermal profile. (www.datapaq.com ©2011)

This information tells you what temperatures your product reached, for how long and at what point of the process. Process engineers know what the perfect profile for their product should be and variations from the ideal indicate a potential problem or unacceptable quality. By analyzing the thermal profile, you are able to verify and improve product quality, increase throughput and solve production problems. (www.datapaq.com ©2011)

Understanding what is happening to your product as it travels through an oven or furnace is the first step to controlling and optimizing that heat treatment process. Temperature profiling is a critical tool used by thousands to help obtain that understanding. (www.datapaq.com ©2011)

4.8.2 0% Load

At 0 % load, (Table 4.1), in the 10th percentile, the profile is linear with a gradient of 70.32, in the 20th percentile, the profile in linear with a gradient of 109.89, in the 30th percentile, the profile is linear with a gradient of 109.89, in the 40th percentile, the profile is linear with a gradient of 109.89, in the 50th percentile, the profile is linear with a gradient of 109.89, in the 60th percentile, the profile is linear with a gradient of 109.89, in the 70th percentile, the profile is linear with a gradient of 109.89, in the 80th percentile, the profile is linear with a gradient of 109.89, in the 90th percentile, the profile is linear with a gradient of 109.89, in the 100th percentile, the profile is linear with a gradient 109.89. The low gradient in the 10th percentile is due to the initial heating of the refractory material. On an average, the furnace temperature profile is linear; all through, it attained a peak temperature of 1000 °C at 9.02 minutes with a gradient of 77.7.

4.8.3 25% Load

At 25 % load, (Table 4.2), the furnace temperature profile is linear first quartile, with a gradient of 26.28, in the second quartile, the profile was still linear but with a gradient of 27.23, it maintained the linear profile in the third quartile with a gradient of 27.23, in the final quartile, the gradient is 27.23 with a linear profile. The consistence in the gradient in the second, third and fourth quartiles shows that the furnace has a constant heating rate while the deviation at the first quartile is due to the fact some of the initial energy was used in heating the refractory . In general the furnace loading at 25% loading condition attained a peak temperature of 1000 °C at 35.8 minutes with average gradient of 19.28.

4.8.4 50% Load

At 50% load, (Table 4.3), the furnace temperature profile was linear in the first quartile, with a gradient of 8.69, in the second quartile, the profile was still linear but with a gradient of 8.9, it maintained the linear profile in the third quartile with a gradient of 8.9, in the final quartile, the gradient was 8.9 with a linear profile. The consistence in the gradient in the second, third and fourth quartiles showed that the furnace had a constant heating rate while the deviation at the first quartile is due to the fact, that some of the initial energy was used in heating the refractory. In general the furnace at 50% loading condition displayed a linear temperature profile, and attained a peak temperature of 1000 °C at 108.6 minutes.

4.8.5 85% Load

At 85 % load, (Table 4.4), the furnace temperature profile was in the linear first quartile, with a gradient of 5.4, in the second quartile, the profile was still linear but with a gradient of 5.5, it maintained the linear profile in the third quartile with a gradient of 5.5, in the final quartile, the gradient is 5.5 with a linear profile. The consistence in the gradient in the second, third and fourth quartiles shows that the furnace has a constant heating rate while the deviation at the first quartile is due to the fact some of the initial energy was used in heating the refractory . In general the furnace loading at 85% loading condition attained the furnace temperature profile is linear; attaining a peak temperature of 1000 °C at 174.6 minutes.

4.8.6 100% Load

At 100% load, (Table 4.5), the furnace temperature profile was in the linear first quartile, with a gradient of 4.3, in the second quartile, the profile was still linear but with a gradient of 4.4, it maintained the linear profile in the third quartile with a gradient of 4.4, in the final quartile, the gradient is 4.4 with a linear profile. The consistence in the gradient in the second, third and fourth quartiles shows that the furnace has a constant heating rate while the deviation at the first quartile is due to the fact some of the initial energy was used in heating the refractory . In general the furnace loading at 100% loading condition attained the furnace temperature profile is linear; attaining a peak temperature of 1000 °C at 218.3 minutes.

From Figure 4.1, shows the furnace temperature increment with time at0, 25, 50, 85 and 100% loading conditions. The gradient of furnace at the 0, 25, 50, 85 and 100% are 109.89, 27.23, 8.9, 5.5 and 4.4 respectively. All loading conditions exhibited linear relationship. 100% loading condition exhibited the steepest increment rate while 0% loading exhibited the least.

4.9.1 Heat loss through Furnace wall

The heat loss through the walls of the furnace is 912.92 watt/Km^2, an average for the specific data set used. These calculations were done by using Fourier heat conduction equation for conductive heat transfer through flat furnace wall with multiple layers, shown in equation 14

$$Q = \frac{T_1 - T_3}{\frac{X_A}{K_A\ A} + \frac{X_B}{K_B\ A}} \qquad (14)$$

Steady state one dimension conditions were assumed for these calculations.

4.9.2 Temperature Drop across Furnace Wall

From the schematic diagram of the furnace wall shown in the Figure 3.7, the temperature gradient across the furnace wall, (Figure 3.8), shows that the furnace had an initial inner furnace temperature, of 1473 K, this temperature fell to 1305 K across the firebricks and steel interface. From 1305 K, the temperature dropped to 1006 K across the air gap, on passing through the insulator, the temperature further dropped to 303 K at the outer shell of the furnace.

4.9.3 Thermal Efficiency

From Table 4.6, the thermal efficiency of the furnace is 80%. This indicates that the furnace is highly efficient. There are a number of factors that contributed to this high value;

Heat losses from furnace walls, affect the furnace efficiency substantially. The extent of wall losses depends on emissivity of walls, conductivity of refractories, wall thickness and whether furnace are operated continuously or intermittently.

Different materials have different radiation power (emissivity). From Figure 4.8, the emissivity of walls made of aluminosilicate refractory is shown to be lower than that of ordinary silica bricks.

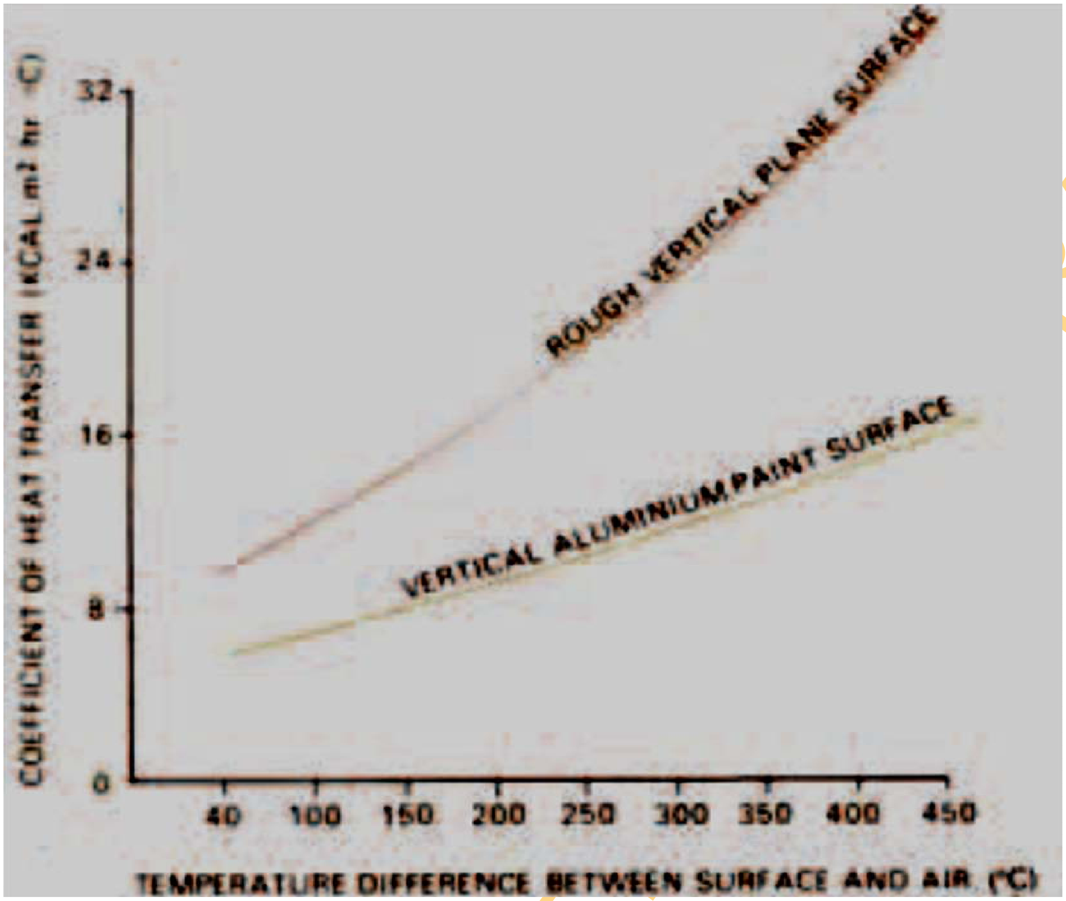

Figure 4.8 Coefficient of Heat Transfer for Different Conditions in Still Air at 40°C

Source: Bureau of Energy Efficiency, (2005)

The variations of thermal conductivity for typical refractory materials (silica brick, fireclay brick and insulation brick) with temperature is depicted in figure 4.8 . Thus at a mean temperature of 600 °C, conductivity of the insulation brick is only 20% of that for fireclay brick.

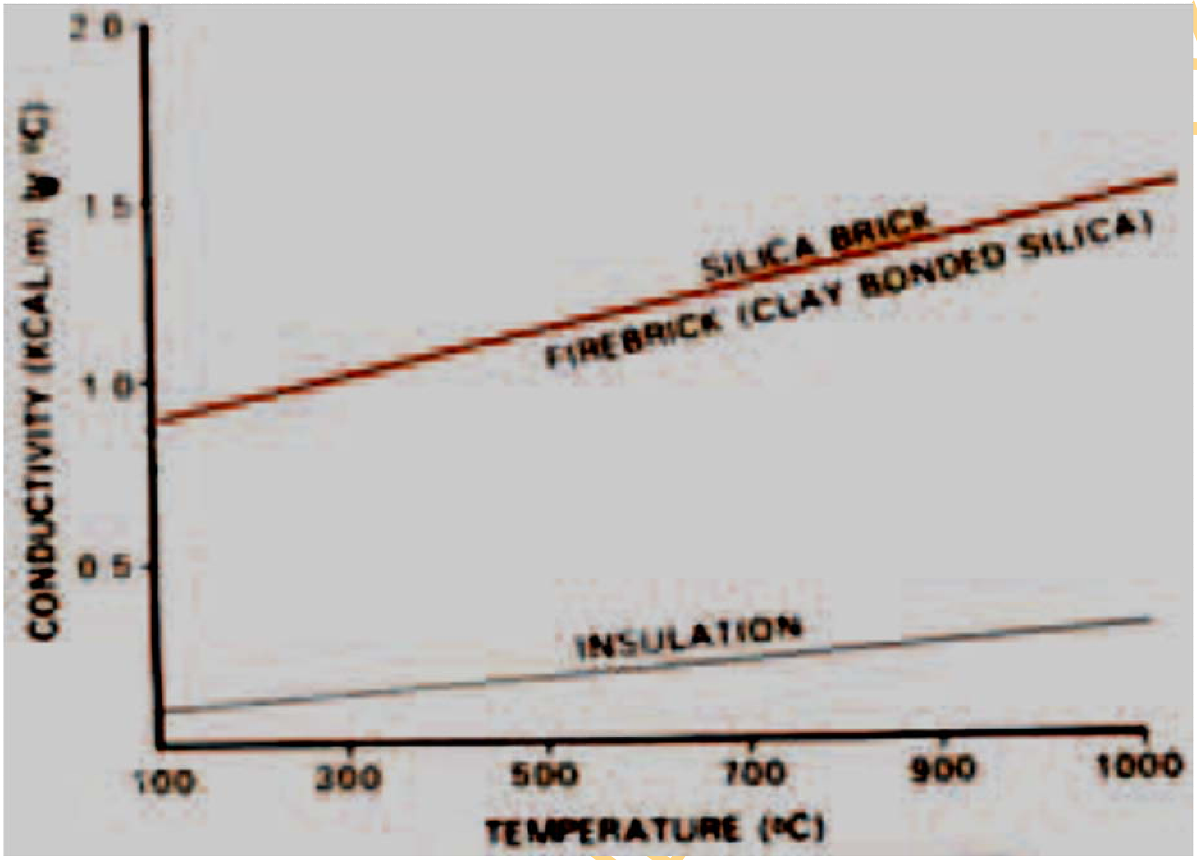

Figure 4.9 Average Conductivity of Refractory Material

Source: Bureau of Energy Efficiency, (2005)

Heat losses were reduced by increasing the wall thickness, and through the application of insulating fibres. Outside wall temperature and heat losses for a composite wall of a certain thickness of firebrick and insulation fibres were much lower due to lesser conductivity of insulating fibre as compared to a refractory brick (Figure 4.9).

In batch furnace, operating periods ('on') alternate with idle periods ('off'). During the off period, the heat stored in the refractories in the on-period is gradually dissipated, mainly through radiation and convection from the cold face. In addition, some heat is obstructed by air flowing through the furnace. Dissipation of stored heat is a loss, because the lost heat is at least in part again imparted to the refractories during the next 'on' period, thus expending power/fuel to generate the heat. If a furnace is operated 24 hr. every third day, practically all of the heat stored in the refractories is lost. But if the furnace is operated 8 hrs per day, not all the heat stored in the refractories is dissipated. For a furnace with firebrick wall (350 mm) it is estimated that 55 per cent of the heat stored in the refractories is dissipated from the cold surface during 166 hours idle period. Furnace walls made of insulating refractories and encased in a shell reduce flow of heat to the surroundings. Inserting a fibre between the refractory and the steel casing can further reduce the loss. The amount of heat loss reduced by application of insulation,depends on the thickness of firebricks and of the insulation and on continuity of furnace operation.

To sum up, the heat losses from the walls depend on inside temperature, outside air temperature outside air velocity and configuration of walls.

The following conclusions can be drawn:

i. As the wall thickness increases, the heat losses reduce.

ii. As thickness of insulation is increased, heat losses reduce.

iii. The effect of insulation in reducing heat losses is more pronounced than the increase of wall thickness. Roughly 1 cm of insulation brick is equivalent to 5 to 8 cm of refractory (firebrick).

iv. In intermittent furnaces, thin walls of insulating refractories are preferable to thick walls of a normal refractory for intermittent operation since less heat is stored in them.

4.9.4 Thermal stress

The thermal stress developed in the wall of the furnace as a result of uniform temperature is 166.9 MN/m^2. In order to accommodate this stress in the furnace wall, aluminosilicate bricks were selected as the inner refractory material because of its high thermal resistance (260 MN/m^2) and strength coupled with its low coefficient of linear expansion. This will prevent cracking and eliminate built in barriers while acting as expansion relief

4.9.5 Thermal strain

The thermal strain in the refractory is 5.6x 10^{-3}, to accommodate this, aluminosilicate bricks were selected because of its low coefficient of thermal expansion.

4.10 Scanning Electron Micrograph (SEM)

Figures 4.3, 4.4, 4.5 and 4.6 show the SEM images of coconut shell based chemically treated activated carbon (ACS) which clearly shows its porous structure and complete opening of cell pores on the surface. As can be seen from the micrograph, there is a formation of pores which may be due to release of some volatiles and activating agent from the sample. The external surface of ACS was full of cavities which are due to the removal of KOH thereby leaving the space which was previously occupied by the activating agent. In this way it provides a larger surface area.

The examination of the initial structure of the raw coconut shell sample (Figure. 4.3) shows the presence of a few micropores of size 0.15 nm. Carbonisation increases the porosity to 0.28 nm (Figure 4.4). After activation, the porosity is more developed. Micropores of sizes 0.58 nm are formed after activation for 1 hour (Figure 4.5). After activation for 3 hours (Figure 4.6), the micrograph is approximately elliptical with a mean equivalent circle pore diameter of about 1.8 nm, so that they are large micropores. They are relics of the cellular structure of the precursor material. This is general characteristic of micropores in active carbon derived from lignocellulosic precursors. The comparison of Figures 4.6 and 4.5, indicate clearly that a regular microporous and homogeneous surface are obtained by activation for 3 hours.

4.11 Chi-squared test for goodness of fit

In order to ascertain the goodness of fit in the porosity of newly produced activated carbon in the batch reactor in comparison to standard values in literature, chi-squared tests (χ^2) was used. The results of the test for the characterized property from the chi-squared distribution table, Table 4.9, showed that there is no significant difference in the characterized property .The P-value obtained is greater than 0.05 and this implies that the difference in porosity is statistically not significant.

Table 4.9 Chi-squared distribution

Degrees of freedom (df)	χ^2 value										
1	0.004	0.02	0.06	0.15	0.46	1.07	1.64	2.71	3.84	6.64	10.83
2	0.10	0.21	0.45	0.71	1.39	2.41	3.22	4.60	5.99	9.21	13.82
3	0.35	0.58	1.01	1.42	2.37	3.66	4.64	6.25	7.82	11.34	16.27
4	0.71	1.06	1.65	2.20	3.36	4.88	5.99	7.78	9.49	13.28	18.47
5	1.14	1.61	2.34	3.00	4.35	6.06	7.29	9.24	11.07	15.09	20.52
6	1.63	2.20	3.07	3.83	5.35	7.23	8.56	10.64	12.59	16.81	22.46
7	2.17	2.83	3.82	4.67	6.35	8.38	9.80	12.02	14.07	18.48	24.32
8	2.73	3.49	4.59	5.53	7.34	9.52	11.03	13.36	15.51	20.09	26.12
9	3.32	4.17	5.38	6.39	8.34	10.66	12.24	14.68	16.92	21.67	27.88
10	3.94	4.87	6.18	7.27	9.34	11.78	13.44	15.99	18.31	23.21	29.59
P value (Probability)	0.95	0.90	0.80	0.70	0.50	0.30	0.20	0.10	0.05	0.01	0.001

The formula for calculating chi – square $\chi^2 = {(observed - expected)^2}/{expected}$

$$\chi^2 = \frac{(1.8-2.0)^2}{2.0}$$

$$\chi^2 = \frac{0.04}{2.0} = 0.02$$

$At\ \chi^2 = 0.02\ (Table\ 4.9),\ p = 0.9$

4.12 X-Ray Diffraction Pattern (XRD)

X-Ray Diffraction (XRD) Studies Figure 4.7 displays the XRD pattern of the activated carbon prepared from coconut shell which was activated at 450 °C using KOH as an activating agent. The activated carbon shows three peaks (2θ = 8°, 25°, and 44°) which corresponds to the peak of graphite (Lua and Yang, 2004). The diffraction pattern does not exhibit a horizontal basic line. This shows that the major part of the matter is amorphous. However, a few diffraction peaks emerge from the basic line, indicating the presence of a small amount of crystalline matter. Further it can be seen that XRD signals of powder sample contains large amount of noises in it. This behaviour confirms the amorphous structure of carbon.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This chapter provides summary of work done. Suggestions made as recommendation for future research works, highlights of contributions to knowledge and potential applications emanated from the completed research activities are provided in this chapter.

This research work was conducted with the aim of developing a batch reactor for the production of activated carbon from coconut shell using locally sourced material while incorporating inlet tuyere for inert gases and avoiding short-circuiting of heating elements from discharge fumes . The summary of conclusions that can be drawn from this research works are provided as follows:

i. Coconut shells served as very good precursor materials in the production of activated carbon. The activated carbon produced, were very stable, high quality and yield with minimal burn-off ratio.
ii. A Batch reactor with 80% efficiency was developed, suitable for producing activated carbon, the reactor has inlet tuyere for inert gases and its heating elements were quite stable and well protected against discharge fumes, thereby eliminating short circuiting.
iii. The batch reactor was used to produce activated carbon from coconut shells. Chemical activation process was employed for the production processes. The temperature profiles of the reactor at different loading conditions were linear.
iv. X-ray diffraction pattern showed the major part of the AC produced were amorphous with small amount of crystalline matter.
v. Activating temperature and holding time had significant role in imparting physical and chemical characteristics of the activated carbon. Scanning electron microscope at 1 hour activation showed a few micropores with not well developed pores of sizes 0.58nm, while well formed elliptical micropores of sizes 1.8 nm were observed at 3 hours activation time

vi. Activating temperature of 450 ^{o}C and holding time for 3 hours proved to be the optimum conditions for producing activated carbon as indicated by the superior physical characteristics.

vii. There is no significant difference ($p > 0.05$) between experimental pore sizes of 1.8 and 2 nm recorded in published standards. The quality of the produced activated carbon shows no disparity from literature.

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5.2 Recommendation

To meet the demand of activated carbon in industries, while reducing the amount spent in importing activated carbon, large scale/ commercial production of the reactor is being suggested, having been able to eliminate the challenges with adapted furnaces (short-circuiting of heating element and absence of inert gas tuyere) used for producing activated carbon in Nigeria. The optimal production condition from this research should be adopted in large scale production of activated carbon.

5.3 Contributions to knowledge

This research was able to develop a reactor specifically suitable for activated carbon production.

The outcomes of this research;

i. Developed a reactor; that eliminated the challenges (short-circuiting of heating element and absence of inert gas tuyere) in adapted furnaces used in production of activated in Nigeria thereby immensely contributing to academic world in the field of activated carbon production and characterisation .

ii. Provided optimal production parameters for activated carbon production from coconut shells thereby expanding and enriching the volume of literature in this field.

iii. Provided insight and opportunities in the exploitation of locally sourced low cost agro-based materials in production of activated carbon by converting waste (coconut shell) to wealth (activated carbon), and thus provide a platform for further research.

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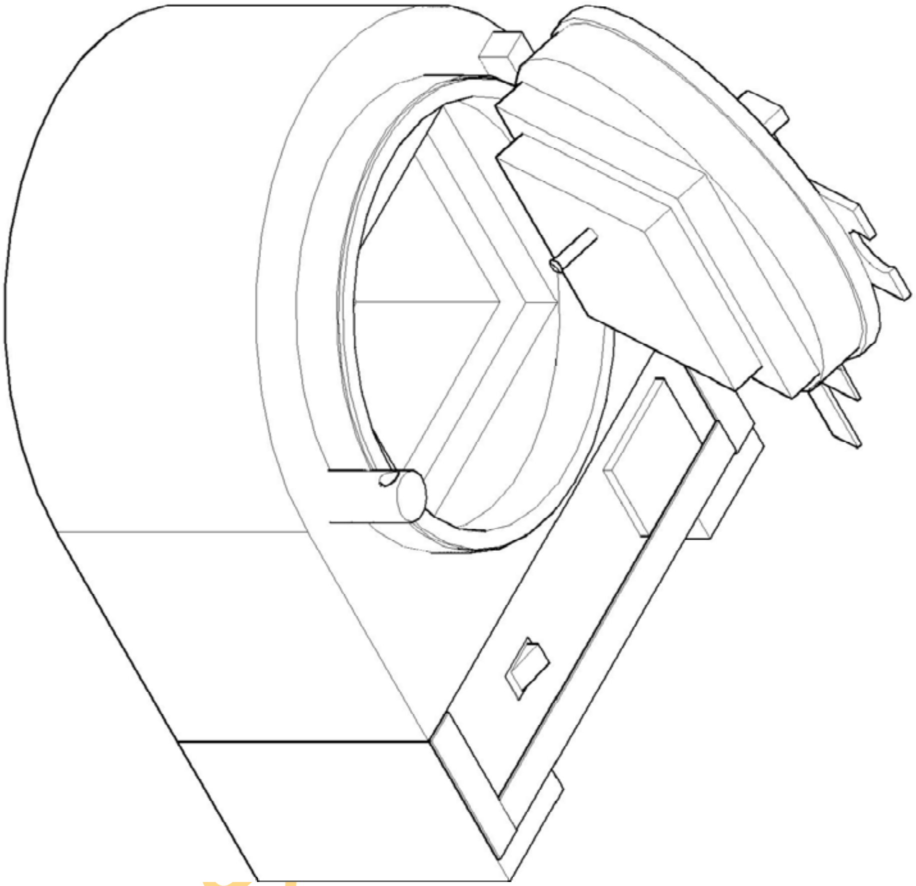
APPENDICES

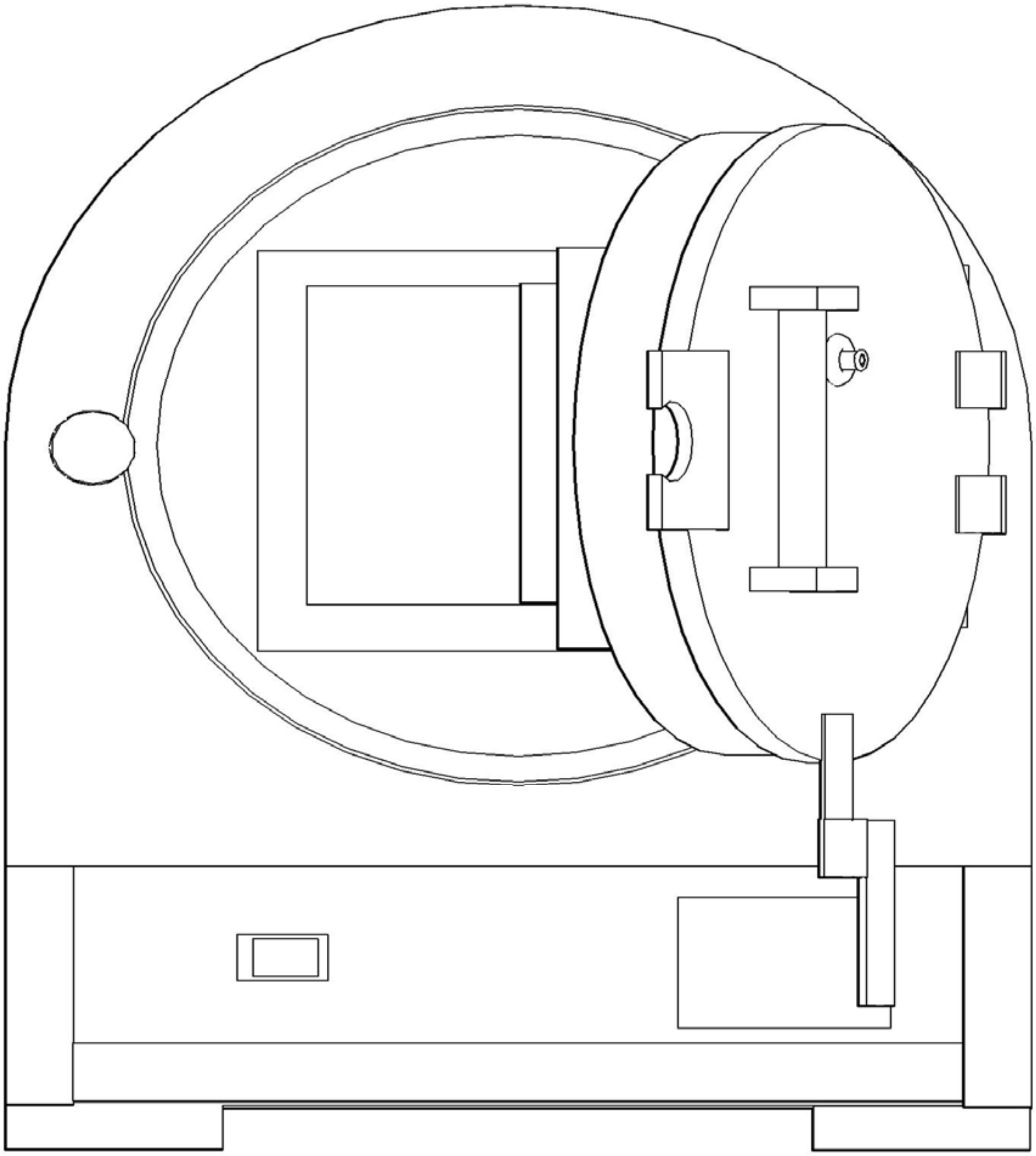
List of Appendices

- A Furnace – isometric
- B Furnace - Front
- C Furnace - Right
- D Furnace – Left
- E Furnace - Top
- F Table of Characteristic IR Absorptions
- G Thermal Conductivity tables
- H Publications

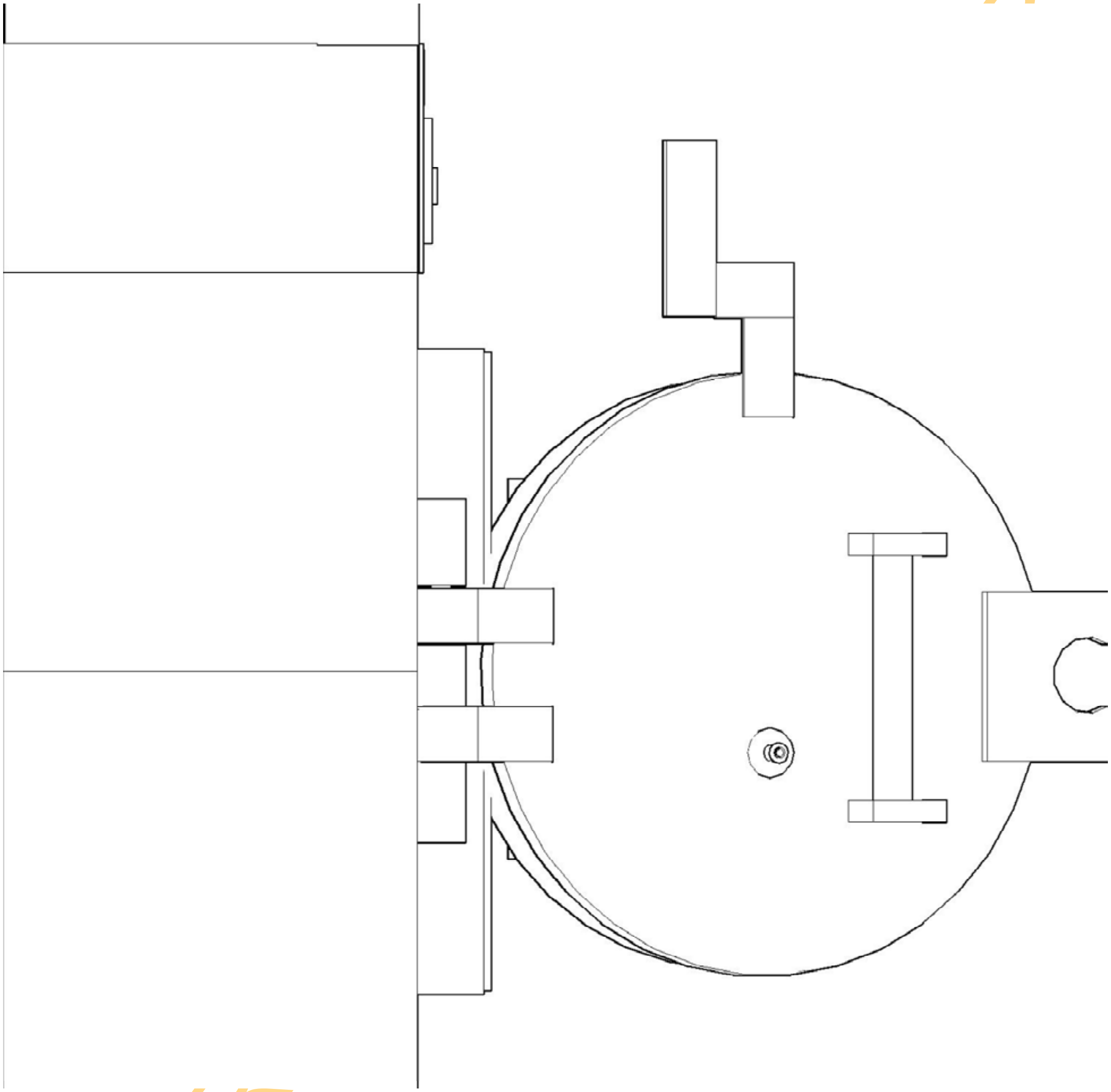
APPENDIX-A Furnace-isometric



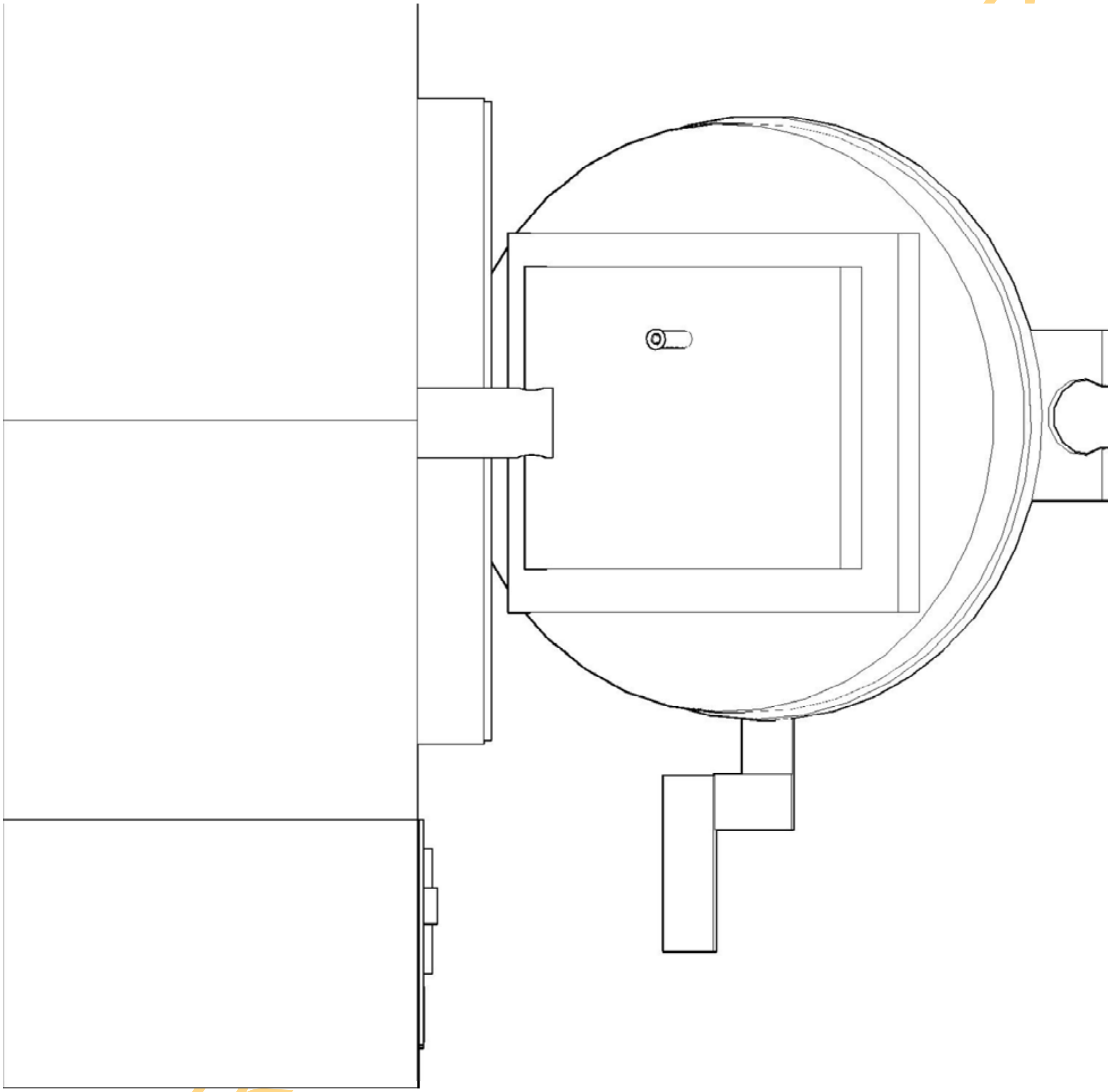
APPENDIX-B FURNACE-FRONT



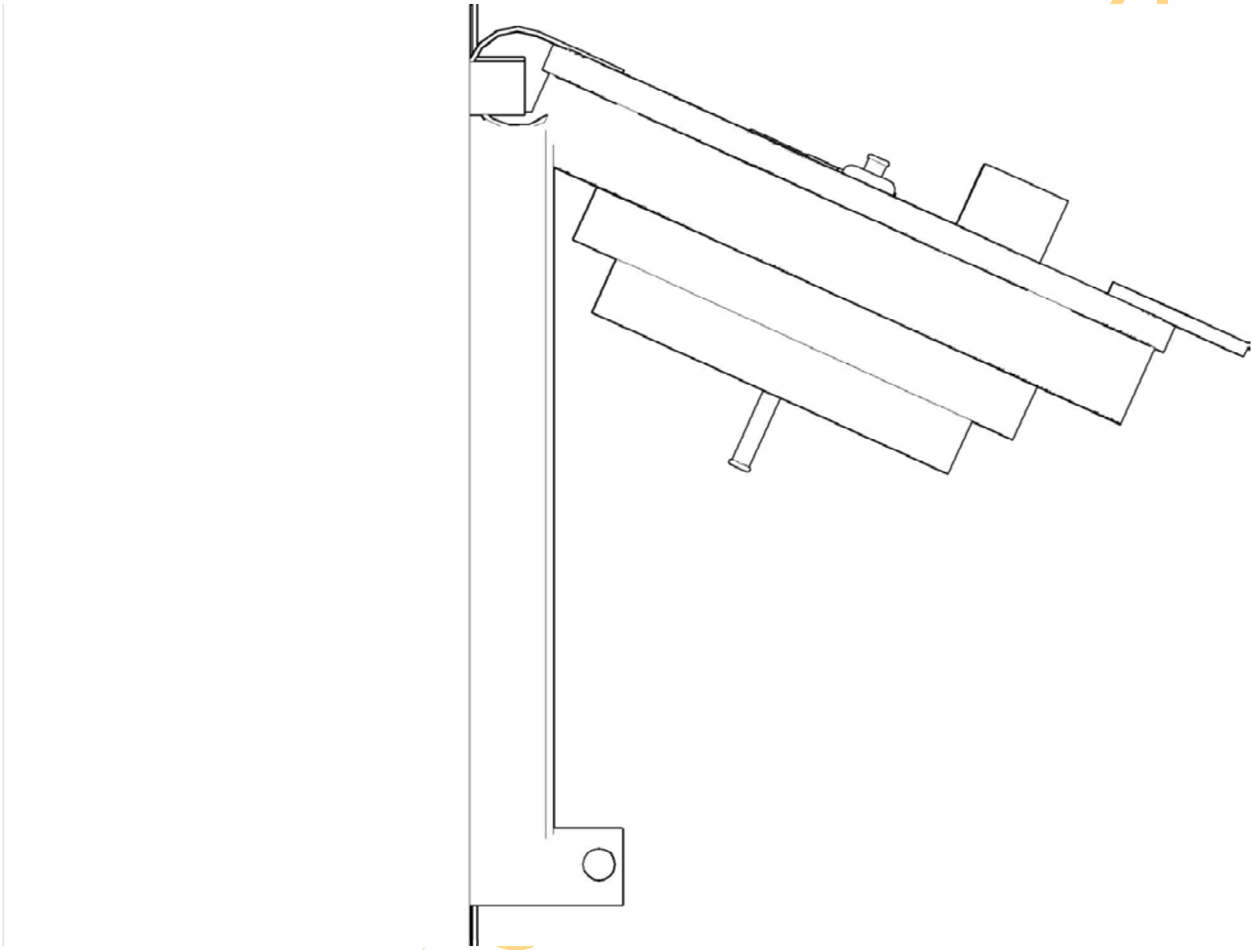
APPENDIX-C   FURNACE-RIGHT



APPENDIX-D FURNACE- LEFT



APPENDIX-E FURNACE- TOP



APPENDIX-F Table of Characteristic IR Absorptions

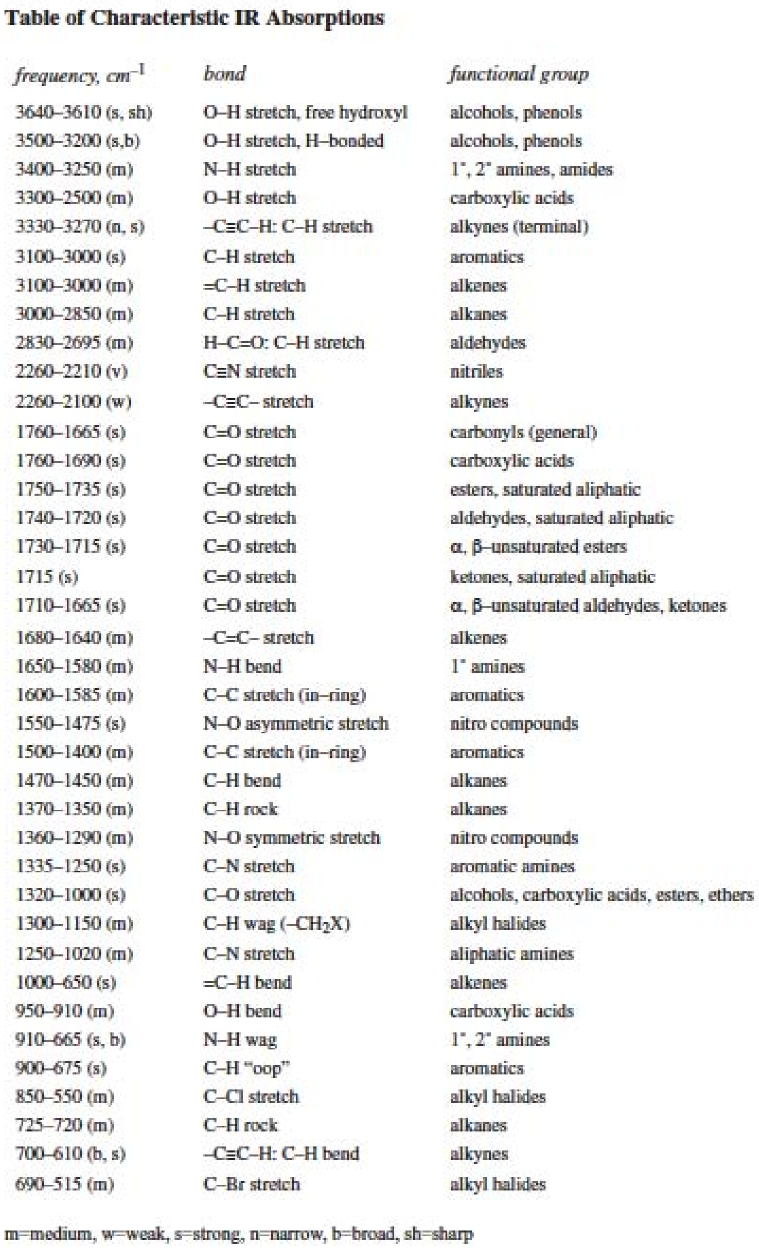


Table of Characteristic IR Absorptions

frequency, cm^{-1}	*bond*	*functional group*
3640–3610 (s, sh)	O–H stretch, free hydroxyl	alcohols, phenols
3500–3200 (s,b)	O–H stretch, H–bonded	alcohols, phenols
3400–3250 (m)	N–H stretch	1°, 2° amines, amides
3300–2500 (m)	O–H stretch	carboxylic acids
3330–3270 (n, s)	–C≡C–H: C–H stretch	alkynes (terminal)
3100–3000 (s)	C–H stretch	aromatics
3100–3000 (m)	=C–H stretch	alkenes
3000–2850 (m)	C–H stretch	alkanes
2830–2695 (m)	H–C=O: C–H stretch	aldehydes
2260–2210 (v)	C≡N stretch	nitriles
2260–2100 (w)	–C≡C– stretch	alkynes
1760–1665 (s)	C=O stretch	carbonyls (general)
1760–1690 (s)	C=O stretch	carboxylic acids
1750–1735 (s)	C=O stretch	esters, saturated aliphatic
1740–1720 (s)	C=O stretch	aldehydes, saturated aliphatic
1730–1715 (s)	C=O stretch	α, β–unsaturated esters
1715 (s)	C=O stretch	ketones, saturated aliphatic
1710–1665 (s)	C=O stretch	α, β–unsaturated aldehydes, ketones
1680–1640 (m)	–C=C– stretch	alkenes
1650–1580 (m)	N–H bend	1° amines
1600–1585 (m)	C–C stretch (in–ring)	aromatics
1550–1475 (s)	N–O asymmetric stretch	nitro compounds
1500–1400 (m)	C–C stretch (in–ring)	aromatics
1470–1450 (m)	C–H bend	alkanes
1370–1350 (m)	C–H rock	alkanes
1360–1290 (m)	N–O symmetric stretch	nitro compounds
1335–1250 (s)	C–N stretch	aromatic amines
1320–1000 (s)	C–O stretch	alcohols, carboxylic acids, esters, ethers
1300–1150 (m)	C–H wag ($-CH_2X$)	alkyl halides
1250–1020 (m)	C–N stretch	aliphatic amines
1000–650 (s)	=C–H bend	alkenes
950–910 (m)	O–H bend	carboxylic acids
910–665 (s, b)	N–H wag	1°, 2° amines
900–675 (s)	C–H "oop"	aromatics
850–550 (m)	C–Cl stretch	alkyl halides
725–720 (m)	C–H rock	alkanes
700–610 (b, s)	–C≡C–H: C–H bend	alkynes
690–515 (m)	C–Br stretch	alkyl halides

m=medium, w=weak, s=strong, n=narrow, b=broad, sh=sharp

Source : http://hemija.pmf.ukim.edu.mk/

APPENDIX-G Thermal conductivity Table

Material	Thermal conductivity [W·m^{-1}·K^{-1}]	Temperature [K]	Electrical conductivity @ 293 K [Ω^{-1}·m^{-1}]	Notes
Acrylic Glass (Plexiglas V045i)	0.17[2]-0.19[2]-0.2[3]	296[2]	7.143E-15[2] - 5.0E-14[2]	
Air	0.024[4][5][6]-0.025[7] 0.0262 (1 bar)[8] 0.0457 (1 bar)[8]	273[4][5]-293[7]-298[6] 300[8] 600[8]	hiAerosols2.95[9]-loAerosols7.83[9]$\times10^{-15}$	(78.03%N$_2$,21%O$_2$,+0.93%Ar,+0.04%CO$_2$) (1 atm)
Alcohols OR Oils	0.1[6][7]-0.110[10]-0.21[6][7]-0.212[10]	293[7]-298[6]-300[10]		
Aluminium nitride	170[13]-175[17]-190[17]	293[17]	1×10^{-11}[17]	
Aluminium oxide, pure	26[18]-30[7]-35[18]-39[13]-40[19]	293[7][18][19]	1×10^{-12}-[18][19]	
Aluminium, pure	204.3[11]-205[4]-220[12]-237[7][13][14][15]-250[6] 214.6[11] 249.3[11]	293[7][11]-298[6][14][15] 366[11] 478[11]	37,450,000[14] - 37,740,000[16]	
Ammonia, saturated	0.507[10]	300[10]		
Argon	0.016[6]-0.01772[15]-0.0179[15][20]	298[6][15]-300[15][20]		

Material	Thermal conductivity [$W \cdot m^{-1} \cdot K^{-1}$]	Temperature [K]	Electrical conductivity @ 293 K [$\Omega^{-1} \cdot m^{-1}$]	Notes
Beryllium oxide	218[13]-260[21]-300[21]	293[21]	1×10^{-12}[21]	
Bismuth	7.97[15]	300[15]		
Brass Cu63%	125[22]	296[22]	15,150,000[22] - 16,130,000[22]	(Cu63%, Zn37 %)
Brass Cu70%	109[4][23] - 121[23]	293[4]-296[23]	12,820,000[23] - 16,130,000[23]	(Cu70%, Zn30 %)
Brick	0.15[4]-0.6[4]-0.69[6]-1.31[6]	293[4]-298[6]		
Bronze	26[12] 42[24]-50[11][24]	293[11]-296[24]	5,882,000[24] - 7,143,000[24]	Sn25%[12] (Cu89%, Sn11 %)[24]
Calcium silicate	0.063[25]	373[25]		
Carbon dioxide	0.0146[6]-0.01465[26]-0.0168[20](sat. liquid 0.087[27])	298[6]-273[26]-300[20](293[27])		
Carbon nanotubes, bulk	2.5 (multiwall)[28] - 35 (single wall, disordered mats)[28]- 200(single wall, aligned mats)[28]	300[28]		"bulk" refers to a group of nanotubes either arranged or disordered, for a single nanotube, see "carbon nanotube,

Material	Thermal conductivity [W·m^{-1}·K^{-1}]	Temperature [K]	Electrical conductivity @ 293 K [Ω^{-1}·m^{-1}]	Notes
				single" .[28]
Concrete	0.8[4] - 1.28[7] - 1.65 [34] - 2.5 [34]	293[7]		~61-67%CaO
Copper, pure	385[4]-386[11][12]-390[7]-401[6][15][35] 368.7[11] 353.1[11]	293[4][6][7][11][15][35] 573[11] 873[11]	59,170,000[35] - 59,590,000[16]	International Annealed Copper Standard (IACS) pure =1.7×10^{-8}Ω•m =58.82×10^{6}Ω^{-1}•m^{-1} For main article, see: Copper in heat exchangers.
Cork	0.04[4] - 0.07[7]	293[7]		
Cotton or PlasticInsulation-foamed	0.03[6][7]	293[7]		
Diamond, impure	1,000[4][36]	273[36] - 293[4]	1×10^{-16}~[37]	Type I (98.1% of Gem Diamonds) (C+0.1%N)
Diamond, isotopically enriched	3,320[38]-41,000[29][39](99.999% ^{12}C calc.200,000[39])	293[38]-104[29][39](~80[39])	(Lateral)10^{-16}[37] - (Ballistic)10^{8}[37]	Type IIa isotopically enriched (>99.9%^{12}C)
Diamond, natural	2,200[38]	293[38]	1×10^{-16}~[37]	Type IIa (99%^{12}C and 1%^{13}C)
Epoxy, thermally conductive	0.682[40] - 1.038 - 1.384[41] - 4.8[42]			

Material	Thermal conductivity [W·m^{-1}·K^{-1}]	Temperature [K]	Electrical conductivity @ 293 K [Ω^{-1}·m^{-1}]	Notes
Expanded polystyrene - EPS	0.03[6]-0.033[4][6][36]((PS Only)0.1[43]-0.13[43])	98[36]-298[6][36](296[43])	1×10^{-14}[43]	(PS+Air+CO_2+C_nH_{2n+x})
Extruded polystyrene - XPS	0.029 - 0.39	98-298		
Fiberglass or Foam-glass	0.045[7]	293[7]		
Gallium arsenide	56[36]	300[36]		
Glass	0.8[4]-0.93[7](SiO_2pure1[13]-$SiO_2$96%1.2[44]-1.4[44])	293[4][7][44]	10^{-14}[45][46]-10^{-12}[44]-10^{-10}[45][46]	<1% Iron oxides
Glycerol	0.285[10]-0.29[7]	300[10]-293[7]		
Gold, pure	314[4]-315[11]-318[12][15][47]	293[11]-298[15][47]	45,170,000[16] - 45,450,000[47]	
Granite	1.73[48] - 3.98[48]			(72%SiO_2+14%Al_2O_3+4%K_2O etc.)
Graphene	(4840±440)[49] - (5300±480)[49]	293[49]	100,000,000[50]	
Graphite, natural	25-470[51]	293[51]	5,000,000-30,000,000[51]	
Helium II	>100000[52]	2.2		liquid Helium in its superfluid state below 2.2 K
Hydrogen	0.1819[53]	290		Hydrogen gas at room temperature.
Ice	1.6[4]-2.1[7]-2.2[36]-2.22[54]	293[4][7] - 273[36][54]		
Indium phosphide	80[36]	300[36]		

Material	Thermal conductivity [W·m^{-1}·K^{-1}]	Temperature [K]	Electrical conductivity @ 293 K [Ω^{-1}·m^{-1}]	Notes
Iron, cast	55[6][12]	298[6]		(Fe+(2-4)%C+(1-3)%Si)
Iron, pure	71.8[12]-72.7[11]-79.5[4]-80[6]-80.2[36]-80.4[15][55] 55.4[11] 34.6[11]	293[4][11]-298[6]-300[15][36][55] 573[11] 1273[11]	9,901,000[55] - 10,410,000[16]	
Lead free solder,Sn/95.6% Ag/3.5% Cu/0.9%,Sn/95.5% Ag/3.8% Cu/0.7%(SAC)	~60[67]			
Lead, pure	34.7[4][11]-35.0[6][12]-35.3[15][56] 29.8[11]	293[4][11]-298[6]-300[15][56] 573[11]	4,808,000[16] - 4,854,000[56]	
Limestone	1.26[48] - 1.33[48]			Mostly $CaCO_3$
Marble	2.07[48]-2.08[6]-2.94[6][48]	298[6]		Mostly $CaCO_3$
Methane	0.030[6]-0.03281[57]	298[6]-273[57]		
Mineral Insulation orWool(Felt/Glass/Rock)	0.04[4][6][7]	293[7]-298[6]		
Nickel	90.9[15]-91[6]	298[6][15]		
Nitrogen, pure	0.0234[4]-0.024[6]-0.02583[15]-0.026[20][36]	293[4]-298[6]-300[15][20][36]		(N_2) (1 atm)
Oxygen, pure (gas)	0.0238[4]-0.024[6]-0.0263[20]-0.02658[15]	293[4]-298[6]-300[15][20]		(O_2) (1 atm)
Paper	0.05[6]	298[6]		
Perlite in partial vacuum	0.00137[6]	298[6]		
Perlite, (1 atm)	0.031[6]	298[6]		

Material	Thermal conductivity [W·m^{-1}·K^{-1}]	Temperature [K]	Electrical conductivity @ 293 K [Ω^{-1}·m^{-1}]	Notes
Plastic, fiber-reinforced	0.23[58] - 0.7[58] - 1.06[7]	293[7] - 296[58]	10^{-15}[58] - 10^{0}[58]	10-40%GF or CF
Polyethylene High Density	0.42[6] - 0.51[6]	298[6]		
Polymer, High-Density	0.33[58] - 0.52[58]	296[58]	10^{-16}[58] - 10^{2}[58]	
Polymer, Low-density	0.04[58] - 0.16[7] - 0.25[7] - 0.33[58]	293[7] - 296[58]	10^{-17}[58] - 10^{0}[58]	
Polyurethane foam	0.02[6] - 0.021[6]	298[6]		
Quartz (single crystal)	12[36] to *c* axis, 6.8[36] to *c* axis	300[36]		
Quartz-Fused or Vitreous Silica or Fused Silica	1.46[59]-3[7] 1.4[36]	293[7][59] 323[36]	1.333E-18[45] - 10^{-16}[59]	
Rice hulls (ash)	0.062[60]			
Rice hulls (whole)	0.0359[60]			
Rubber (92%)	0.16[36]	303[36]	1×10^{-13}~[45]	
Sandstone	1.83[48] - 2.90[48] 2.1[61] - 3.9[61]			~95-71%SiO$_2$ ~98-48%SiO$_2$, ~16-30% Porosity
Silica Aerogel	0.003[36](carbon black9%~0.0042[62])-0.008[62]-0.017[62]-0.02[6]-0.03[36]	98[36] - 298[6][36]		Foamed Glass
Silver, pure	406[4]-407[11]-418[12] 427[13]-429[6][15][36][63]-430[15]	293[4][11] 298[6][15][63]-300[15][36]	61,350,000[63] - 63,010,000[16]	Highest *electrical* conductivity of any metal

Material	Thermal conductivity [W·m^{-1}·K^{-1}]	Temperature [K]	Electrical conductivity @ 293 K [Ω^{-1}·m^{-1}]	Notes
Silver, sterling	361[64]			
Snow, dry	0.05[6]-0.11[4]-0.25[6]	273[6]		
Sodium chloride	35.1 - 6.5 - 4.85[65]	80 - 289 - 400[65]		
Soil, dry w/ organic matter	0.15[7][66]-1.15[66]-2[7]	293[7]		composition may vary
Soil, saturated	0.6[7]-4[7]	293[7]		composition may vary
Solder, Sn/63% Pb/37%	50[67]			
Steel, carbon	36[11][12]-43[6] 50.2[4]-54[6][11][12]	293[4][11]-298[6]		(Fe+(1.5-0.5)%C)
Steel, stainless	16.3[12][68]-16.7[69]-18[70]-24[70]	296[68][69][70]	1,176,000[69] - 1,786,000[70]	(Fe, Cr12.5-25%, Ni0-20%, Mo0-3%, Ti0-trace)
Thermal grease	0.4[71]			
Thermal grease, silver-based	8[71]			
Thermal tape	0.60[72]			
Titanium Alloy	5.8[74]	296[74]	595,200[74]	(Ti+6%Al+4%V)
Titanium, pure	15.6[12]-19.0[11]-21.9[15][73]-22.5[11]	293[11]-300[15][73]	1,852,000[73] - 2,381,000[16]	
Tungsten, Pure	173[37]	293[37]	18,940,000[37]	
Water	0.563[75]-0.596[75]-0.6[4][7]-0.609[10]	273[75]-293[4][7][75]-300[10]	5×Pure10^{-6}[37]-Sweet$10^{-3\pm1}$[37]-Sea1[75]	<4[75]%(NaCl+$MgCl_2$+$CaCl_2$)

Material	**Thermal conductivity $[W\cdot m^{-1}\cdot K^{-1}]$**	**Temperature [K]**	**Electrical conductivity @ 293 K $[\Omega^{-1}\cdot m^{-1}]$**	**Notes**
Water vapor	0.016[6]-0.02479 (101.3 kPa)[76] 0.0471 (1 bar)[8]	293[76]-398[6] 600[8]		
Wood, +>=12% water	0.09091[77]-0.16[36]-0.21[77]-0.4[7]	298[36]-293[7]		Species-Variable[77]
Wood, oven-dry	0.04[4]-0.055[6]-0.07692[77]-0.12[4]-0.17[6][77]	293[4]-298[6]		Balsa[6]-Cedar[77]-Hickory[77]/Oak[6]
Zinc oxide	21[13]			
Zinc, Pure	116[37]	293[37]	16,950,000[37]	
Material	**Thermal conductivity $[W\cdot m^{-1}\cdot K^{-1}]$**	**Temperature [K]**	**Electrical conductivity**	

PUBLICATIONS

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