

THE PREPARATION OF ACTIVATED CARBON FROM SOUTH AFRICAN COAL FOR USE IN PGM EXTRACTION

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Dissertation submitted in fulfilment of the requirements for the degree Magister in Engineering at the School of Chemical and Minerals Engineering at the North-West University, Potchefstroom Campus

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Potchefstroom

SOLEMN DECLARATION

I declare herewith that the thesis entitled:

The preparation of activated carbon from South African coal for use in PGM extraction

which I herewith submit to the North-West University in completion of the requirements set for the degree Magister in Engineering is my own work and has not already been submitted to any other university. All quoted are indicated and acknowledged by means of a comprehensive list of references.

Signed at Potchefstroom on this 8th day of April 2008.

DJ Kruger

ABSTRACT

Activated carbons used in the Platinum Group Metals extraction industry are characterised by large internal surface areas and a great affinity for platinum, palladium and ruthenium. It is therefore necessary in this study to develop a method to produce an activated carbon that is suitable and yet cost effective, for use in the extraction of PGM's. The quality of the coal-based activated carbon may not prove to be as good as activated carbon produced from other traditional sources, but the production costs involved may make South African coal a feasible alternative feedstock.

The purpose of this research is to prepare activated carbon from a South African based bituminous coal by physical activation. The activated carbon produced are characterised by BET surface area, activated carbon pH and phenol adsorption studies. The results of the different characterisation methods for the prepared activated carbons are compared to the results of a commercially available activated carbon, Norit RO 0.8 (control sample).

Bituminous coals from various sources including Witbank Seam 4 and New Vaal are used. The preparation method chosen is raw material activation by means of physical activation with superheated steam. The effects of process variables such as activation time (1 – 3 hr) and temperature (600 – 800°C) are studied in order to optimise those preparation parameters.

Activated carbon surface area is characterized by means of nitrogen adsorption isotherms at 77K. BET surface area analysis showed that Witbank Seam 4 coal activated at a temperature of 800°C and activation time of 3 hours, resulted in a surface area of 340m²/g.

Quality control of each sample was performed by measuring the pH of a known amount of the prepared activated carbon in distilled water over time. Results showed that the pH of some of the prepared activated carbons reached a value of 11.

Phenol adsorption results for the different activated carbons prepared corresponded well to the results obtained for the Norit RO 0.8 activated carbon sample.

Keywords: South Africa, activated carbon, PGM

OPSOMMING

Die geaktiveerde koolstof wat in die Platinum ekstraksie industrie gebruik word, word gekarakteriseer deur groot interne oppervlak areas en 'n groot affiniteit vir platinum, palladium en rutenium. In hierdie studie is 'n metode ontwikkel om 'n geaktiveerde koolstof te produseer wat geskik en koste-effektief is, vir gebruik in die ekstraksie van die Platinum Groep Metale. Daar word verwag dat die kwaliteit van die steenkool gebaseerde geaktiveerde koolstof nie gaan vergelyk met geaktiveerde koolstof berei uit tradisionele bronne nie, maar 'n laer produksiekoste kan Suid Afrikaanse steenkool 'n ekonomies vatbare alternatiewe bron maak.

Die doel van hierdie studie is om geaktiveerde koolstof te produseer deur van Suid Afrikaanse steenkool gebruik te maak as rou materiaal. Die geaktiveerde koolstof produk is gekarakteriseer deur middel van die volgende analises:

- BET oppervlak area analises
- Bepaling van die pH van die gevormde geaktiveerde koolstof
- Fenol adsorpsiestudies

Resultate van bogenoemde analises op die geaktiveerde koolstof is dan vergelyk met 'n kommersieel beskikbare geaktiveerde koolstof, Norit RO 0.8.

Geaktiveerde koolstof van bituminieuse steenkool is berei deur fisiese aktivering met superverhitte stoom. Die afhanklikheid van prosesveranderlikes soos aktiveringstyd (1 – 3 ure) en temperatuur (600 – 800°C) is bestudeer.

Oppervlak area is gekarakteriseer deur stikstof adsorpsie isoterme by 77K. Die resultate het aangedui dat die geaktiveerde steenkool (Witbank Soom 4, berei by 800°C en 3 ure) 'n oppervlak area van tot so veel as 340m²/g gelever het.

pH metings van die bereide geaktiveerde koolstof monsters is as kwaliteitsbeheer gebruik. Die metode behels om die pH van die geaktiveerde koolstof in gedistilleerde water oor 'n sekere tydperk te meet. Resultate het getoon dat die pH van sommige van die bereide koolstofmonsters waardes van 11 gelever het.

Fenol adsorpsie resultate vir die verskillende geaktiveerde koolstofmonsters het goed vergelyk met die adsorpsie resultate wat vir Norit RO 0.8 verkry is.

Sleutelwoorde: Suid Afrika, geaktiveerde koolstof, PGM

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LIST OF SYMBOLS

Symbol	Description	Units
C	Solution concentration	ppm
T	Temperature	°C
W	Change in mass	kg/hr
t	Time	min
ΔC	Change in solution concentration	ppm/min
ΔpH	Change in solution pH	-
Δt	Change in time	min

NOMENCLATURE

Abbreviation	Description
AC	Activated carbon
ACF	Activated carbon fibre
ASTM	American Society for Testing and Materials
BET	Brunauer Emmett Teller
DB	Dry basis
DAFB	Dry ash free basis
HGI	Hardgrove grindability index
ISO	International Standards Organization
MSDS	Material safety data sheet
PGM	Platinum Group Metals
PSA	Pressure swing adsorption
SABS	South African Bureau of Standards
UV-vis	Ultra-violet visible spectroscopy
WS4	Witbank Seam no. 4
NV	New Vaal

CHAPTER 1

INTRODUCTION AND MOTIVATION

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1.1 INTRODUCTION

This chapter will provide a brief introduction to the insights relevant to the motivation and scope of this project. The project objectives will also be stated, as well as a short description of the information conveyed in each section of this study.

1.2 PROBLEM STATEMENT – PGM TAILINGS TREATMENT PROCESS

Current PGM concentrator plants are unable retrieve all of the available platinum and huge quantities of this precious metal are dumped on tailing dams. This proposed process will try to meet this demand by further purifying this tailing stream, resulting in a more concentrated platinum stream recycled into the process.

1.3 PURPOSE OF PROJECT

Use South African coal as raw material for the preparation of activated carbon for use in PGM extraction. The activated carbon should have similar adsorptive properties as the Norit RO 0.8 activated carbon that is currently used in separate studies.

Thus the purpose of this project is to follow and optimise a novel method for the preparation of activated carbon and characterise it by means of BET surface area, pH measurements and phenol adsorption studies. The possibility do exist that this activated carbon can be imported, but it may be viable activating South African coal to produce activated carbon, with properties similar to the Norit RO 0.8 activated carbon.

1.4 SCOPE OF INVESTIGATION

The scope of the project is to produce an activated carbon which will be able to adsorb platinum ions from a dilute chloride solution. Thus far it has been proven by other studies (Mbaya and Kasaini, 2006) that Norit RO 0.8 is a suitable adsorbent for this adsorption process, but this raw material has to be imported. It has been shown that South African coal can be used as raw material for the manufacturing of activated carbon, but studies have not been conducted to explore further applications of activated carbon.

An activated carbon will be prepared using South African coal which must meet certain criteria specifically concerning surface area and adsorption characteristics. The successful activated carbon will be used in further adsorption studies to characterise the success of the choice of raw material and preparation method.

A range of South African coals will be selected as precursor materials for the preparation of activated carbon. Experiments will then be conducted to determine the optimum parameters such as activation time and temperature. BET surface area analysis, pH measurements and phenol adsorption will be used to quantify if the prepared activated carbon is of a suitable quality.

1.5 PROPOSED PROCESS

The proposed process to recover Platinum from dilute solution streams is described as follows:

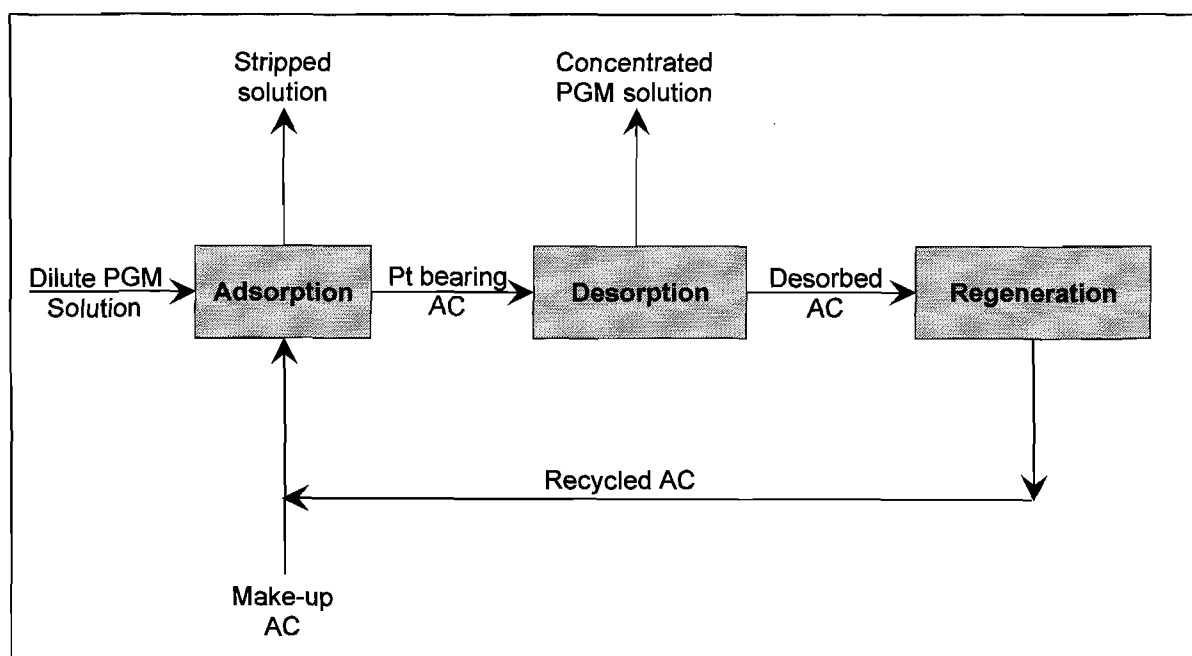


Figure 1.5.1: Proposed process for platinum stripping

A dilute platinum (Pt) solution enters an adsorption column where it is brought into contact with a bed of activated carbon (AC). At the optimum conditions, the platinum is adsorbed onto the activated carbon. The stream that leaves the adsorption column is a stripped solution and a platinum-bearing activated carbon. The platinum is then stripped off from the activated carbon in a desorption column. A platinum concentrated solution leaves the desorption column and the desorbed activated carbon is then regenerated by means of heating under vacuum. The regenerated activated carbon is recycled back into the process.

1.6 PROJECT OBJECTIVES

The objectives of this project will be to produce an activated carbon, using South African coal as precursor, which must have acceptable adsorption area and adsorption characteristics if compared to a commercial activated carbon. These objectives can be categorised as follows:

- Conduct a literature study to define the boundaries for this project
- Select a suitable precursor for the manufacture of activated carbon
- Choose a suitable activation method to convert the raw material into activated carbon
- Characterise the formed activated carbon
- Compare results to an activated carbon commercially available

1.7 DISCUSSION OF CHAPTERS

1.7.1 Chapter 2: Literature survey

Chapter 2 contains the literature survey which was conducted to define the boundaries of the project. It gives an insight into the building blocks of activated carbon including aspects such as activated carbon structure and adsorption characteristics. The potential success of the activated carbon, using South African coal, is also discussed.

1.7.2 Chapter 3: Experimental

Chapter 3 discusses the experimental apparatus and procedures which were followed to produce an activated carbon with suitable characteristics. It also discusses the activated carbon analyses (surface area, activated carbon pH and phenol adsorption) which were used in this study.

1.7.3 Chapter 4: Results and discussion

Chapter 4 presents the results and gives a discussion and interpretation of these results. The observations during the experiments are also mentioned. It relates to Chapter 5 which will conclude on the success of this project.

1.7.4 Chapter 5: Conclusions and recommendations

Chapter 5 concludes this study and makes recommendations regarding further studies.

1.7.5 Appendices

Appendix A: Relevant literature

Gives information regarding relevant literature which was not included in Chapter 2: Literature study.

Appendix B: Equipment information

Describes the different equipment used in this study.

Appendix C: Experimental information

This appendix presents all the detailed procedures that were followed to obtain an activated carbon from the raw material supplied.

Appendix D: Tabulated measured data

Presents the measured data for pH measurements and phenol adsorption studies

Appendix E: Sample calculations

This appendix shows sample calculations which were applied to the measured data.

Appendix F: Tabulated calculated data

Presents the calculated data, derived from Appendix D using the calculations given in Appendix E.

Appendix G: Graphs

Shows the pH and phenol adsorption graphs which did not form part of Chapter 4.

CHAPTER 2

LITERATURE SURVEY

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2.1 INTRODUCTION

Activated carbon is a widely used material in adsorption processes involving liquid-solid extraction or gas-solid extraction. Adsorption is the process where a specific adsorbate attaches to the surface of a specific adsorbent. By combining these two subjects, an activated carbon with sufficient adsorption capacity can be manufactured and used in the PGM extraction industry.

This literature survey will focus on the origin of the precursor, methods available for the preparation of activated carbon and its different applications, specifically in the mineral industry.

2.2 ACTIVATED CARBON

2.2.1 Defining activated carbon

Activated carbon (also activated charcoal, activated coal), an amorphous, non-graphitic form of carbon, are characterised by a large specific area of 300 - 2500m²/g which allows for the physical adsorption of gases and vapours from gases, and dissolved or dispersed substances from liquids (Snell *et al*, 1974: 139), (Kirk and Othmer, 1956: 561). Activated carbon is a generic term for a family of highly porous carbonaceous materials, none of which can be characterised by a structural formula or by a chemical analysis. The large internal surface area and greater number of pores of the activated carbon are mainly dependent on the choice of raw material and method of manufacture (Snell *et al*, 1974: 139). Commercial grades of activated carbons are designated into two classes; gas-phase and liquid-phase carbons.

Gas-phase or vapour adsorbent carbons are hard granules or hard, generally dust-free pellets, while *liquid-phase or decolorising carbons* are generally powdered or granular in form (Kirk and Othmer, 1956: 561). The major difference between these two types lies in the pore size distribution. *Gas-phase carbons* have predominantly fine pores, less than 100 Å in diameter, whereas the pore diameters for *liquid-phase carbons* have a broad range of 5 - 100000 Å. The class of activated carbon produced is dependent primarily upon the raw material and to a lesser extent upon the process of manufacture (Snell *et al*, 1974: 139).

Activated carbon can be manufactured from any cellulosic or ligno-cellulosic material (Almansa *et al*, 2004). Bituminous coal is one of these types of materials which can be used to manufacture activated carbon because of its inherent microstructure (Lorenc-Grabowska *et al*, 2004: 194). Other precursors that have been proved successful are agricultural wastes, coconut shells (Bandosz, 1999: 483), pecan nutshells (Dastgheib and Rockstraw, 2001: 1849; Dastgheib and Rockstraw, 2002a: 1843) and even broiler manure (Lima and Marshall, 2005: 699).

2.2.2 Structure of activated carbon

In the structure of an activated carbon particle, four different groups of pores can be distinguished (IGCL, 2007):

- Sub-micropores (radius $< 0.4\text{nm}$)
- Micropores ($0.4\text{nm} < \text{radius} < 1\text{nm}$)
- Mesopores ($1\text{nm} < \text{radius} < 25\text{nm}$)
- Macropores (radius $> 25\text{nm}$)

Figure 2.2.1 shows the pore distribution of a typical activated carbon particle.

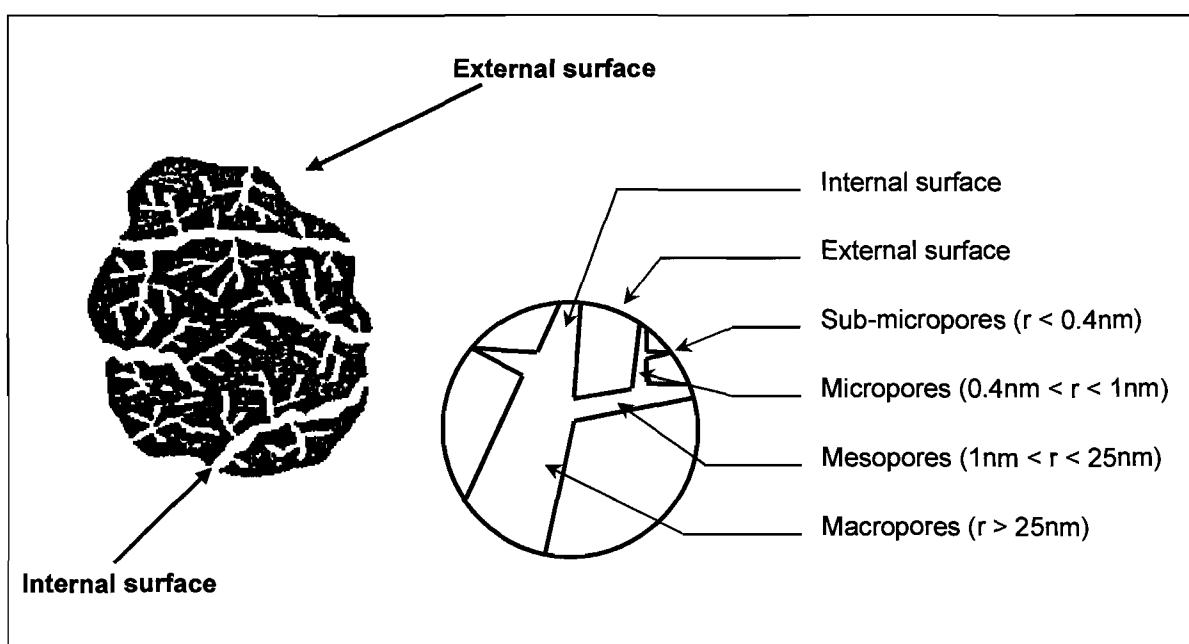


Figure 2.2.1: Activated carbon pore size distribution (IGCL, 2007)

Small diameter micropore and medium diameter mesopore regions contribute to a larger extent to the surface area than sub-micro and macropores. Micropores have been found to be the most effective in trapping small molecules in gas and liquid phase applications. Mesopore regions are most suitable for adsorbing large molecular species (such as colour molecules). Coal based activated carbons have a wider range of mesopores, while precursors such as coconut shells exhibit a predominance of micropores. The development of an extensive macropore structure is found when either peat or wood is used as the raw material.

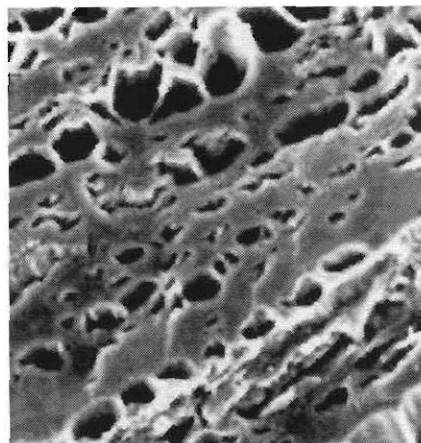


Figure 2.2.2: Pore structure of activated carbon (IGCL, 2007)

2.2.3 Industrial applications

The application of activated carbon in nearly every extraction/purification industry has been proven successful. Some of these applications include gas purification, metal extraction, environmental, medical, food and beverage, and sewage treatment. Some of these applications are further described below.

Gas purification

In the gas purification industry, activated carbon has found many uses:

- Flue Gas Treatment
- Gas Masks
- PSA (Pressure Swing Adsorption)
- Solvent Recovery
- Vapour Recovery
- Air/Gas Purification
- Desulphurisation
- Demercurisation

Metal extraction

Numerous articles have been published with regards to the extraction of metal utilising activated carbon. A few of these research articles are summarised below:

- Riaz Qadeer (2006: 353) has performed extensive research on the adsorption of ruthenium ions on activated carbon, where the removal of this ion is important for purification, waste treatment and trace metal analysis.
- Yalcin and Ihsan Arol (2002: 201) reported that due to the ever expanding gold industry, historical use of coconut shells as raw material for the production of activated carbon suitable for gold production by cyanide leaching, one has to look at exploitation of other raw materials. They investigated the use of hazelnut shells, apricot and peach stones and studied the gold loading capacity and adsorption kinetics.
- Zhang *et al* (2004a: 291) studied the adsorption of the gold thiourea complex onto activated carbon and found that equilibrium gold loading decreased with an increase in thiourea concentration, solution pH and temperature.
- Fraga *et al* (2002: 355) investigated the role of carbon surface sites on the properties of carbon supported platinum catalysts. They pre-treated a commercially available activated carbon (Norit ROW 0.8s) and did oxidative treatments on the carbon to create surface acidic sites and destroy surface basic sites.
- Fu *et al* (1998: 19) studied the adsorption and reduction of Pt(IV) on activated carbon fibre (ACF). They investigated the relationship between the adsorption-reduction of the metal and the activated carbon preparation conditions as well as adsorption conditions (solid/liquid ratio, Pt(IV) concentration, reaction temperature and solution pH). They concluded that Pt(IV) adsorption-reduction capacity onto normal activated carbon fibres is not proportional to the specific surface area.

- Jia *et al* (1998: 1299) investigated the adsorption characteristics of gold and silver cyanide anionic species on a suite of activated carbons derived from coal, coconut shell and polyacrylonitrile.
- Kasaini *et al* (2005: 1) investigated the selective adsorption of platinum from mixed chloride solutions containing base metals using chemically modified activated carbons.

Environmental applications

Carbon adsorption has numerous applications removing pollutants from air and water effluents streams such as (Kirk and Othmer, 1956: 567):

- Drinking water filtration
- Spill cleanup
- Groundwater remediation
- Air purification
- Volatile organic compounds capture

As mentioned above, the primary use for activated carbon is the treatment of water, including potable water (24% of all use), wastewater (21%) and groundwater remediation (4%). These are indirectly related to organic production, because disinfected water used filtered through activated carbon is a common food ingredient.

2.2.4 Parameters affecting activated carbon performance

The amount and distribution of pores play key roles in determining how well contaminants are adsorbed. The best adsorption occurs when pores are barely large enough to admit the contaminant molecule.

Adsorption is also affected by the chemical nature of the adsorbing surface because the surface interacts to a certain extent with organic molecules. Electrical forces between the surface and some organic molecules may also results in adsorption or ion exchange. These adsorption mechanisms are largely dependent on the activation process.

Large organic molecules are most effectively adsorbed by activated carbon. A general rule of thumb is that similar materials tend to associate. Organic molecules and activated carbon are similar materials; therefore, there is a stronger tendency for most organic chemicals to associate with the activated carbon in the filter rather than staying dissolved in a dissimilar material like water. Generally, the least soluble organic molecules are most strongly adsorbed. Often, the smaller organic molecules are held the tightest, because they fit into the smaller pores.

Concentration of organic contaminants can affect the adsorption process. A given activated carbon may be more effective than another type of activated carbon at low contaminant concentrations, but may be less effective than the other carbons at high concentrations.

Adsorption usually increases as pH and temperature decrease. Chemical reactions and forms of chemicals are closely related to pH and temperature. When pH and temperature are lowered, many organic chemicals are in a more absorbable form.

The process of adsorption is also influenced by the length of time that the activated carbon is in contact with the contaminant in the water. Increasing contact time allows greater amounts of contaminant to be removed from the water. Contact is improved by increasing the amount of activated carbon in the filter and reducing the flow rate of water through the filter.

2.2.5 Activated carbon preparation methods

The core idea of the production of carbonaceous adsorbents is the selective removal of functional groups of a suitable carbon material without decreasing the particle size. These removed components must be substituted with well-defined micropores. When preparing activated carbon, there are certain factors that need to be considered when selecting a suitable precursor material. Ash content and mineral constituents are some of the important factors (Kirk and Othmer, 1956: 562). Since South African coal was used, the quality (ash content) of the raw material is not that good if compared to raw materials (coconut shells, apricot and peach stones) used in the manufacturing of commercially available activated carbon.

2.2.5.1 Preparation of raw material prior to activation

If the coal has a low ash content, preparation of the precursor is not necessary. In the case of most South African coals, which contain high percentages of ash, preparation prior to use is necessary since the ash content of the coal has a definitive influence on the final quality of the activated carbon product. Preparation of coal includes washing the coal a very low concentration HCl-solution to remove any impurities that may have been introduced during the prior preparation steps.

2.2.5.2 Activation methods

The activation of the raw material in order to prepare activated carbon can be followed via two ways: chemical and physical activation (Cameron, 2007).

Chemical activation is where the raw material is treated with any dehydrating agent which dissolves any cellulosic components.

Physical activation is when the carbonaceous material is first devolatilised for an extended period of time (1 – 48 hours) and then this formed char is treated with any oxidising agent such as carbon dioxide or steam. The oxidising agent reacts with the carbon to form gaseous products which results in pores and channels being created.

Chemical Activation

The raw material used in chemical activation is usually sawdust and the most popular activating agent is phosphoric acid (Smisek and Cerny, 1970: 23), although zinc chloride (McKetta, 1976: 112) and sulphuric acid are well documented. Others used in the past include calcium hydroxide, calcium chloride, manganese chloride and sodium hydroxide, all of which are dehydrating agents.

The starting raw material is mixed with a concentrated solution of the dehydrating reagent to form a plastic mass which is kneaded at $\pm 90^{\circ}\text{C}$ for several hours to promote chemical decomposition. The mass is then extruded to produce granules which are dried and carbonised in a furnace at 600°C (McKetta, 1976: 112). When phosphoric acid is the activating agent the carbonised product is further heated at $800 - 1000^{\circ}\text{C}$ during which stage the carbon is oxidised by the acid. The acid is largely recovered after the activation stage and converted back to the correct strength for reuse. The activated product is washed with water and dried.

Activity can be controlled by altering the proportion of raw material to activating chemical agent, between the limits of 1:05 to 1:40. By increasing the concentration of the activating agent, the activity increases although control of furnace temperature and residence time can achieve the same objective (Cameron, 2007).

Physical Activation

Physical activation can be used for any precursor (wood, peat, brown coal, bituminous coal, anthracite, etc.). The activation components which are mostly used include steam and carbon dioxide (McKetta, 1976: 112). The physical thermal activation process usually involves two steps namely carbonisation and thermal activation. This will be discussed briefly below.

Carbonisation (Also called pyrolysis, destructive distillation or devolatilisation)

During this step, the carbonaceous material is heated in the absence of air (under vacuum or in an inert atmosphere) and this results in the evolution of gases and liquids (volatilisation of the component elements such as hydrogen, oxygen and other low temperature pyrolysis products). Other complex reactions such as dehydration with the formation of double bonds, polymerisation and condensation also take place (Habashi, 1986: 185). This results in a solid porous carbon residue with a high carbon and very low volatile content. The temperature used for this carbonisation step is dependent on the starting raw material

During carbonisation, the volatiles of the coal are removed usually as a yellow-brown gas. This gas usually consists of:

- Flammable gases for example hydrogen (H_2), carbon monoxide (CO), methane (CH_4) and higher carbons
- Non-flammable gases such as carbon dioxide, water and ammonia
- Tar fumes

The liquid products are condensed into tars, which represent the products of repolymerisation of free radicals formed during the thermal decomposition of the coal. Should the tars be catalytically treated in the presence of hydrogen, the tar molecules are hydrogenated to simpler aromatic molecules, and this provides an important source of benzene, toluene and xylene (Howard-Smith and Werner, 1976: viii). The relative yields of liquid and gaseous pyrolysis products depend on the proportions of volatile matter present in the coal, the reaction temperature and residence time of the coal particles within the reaction zone. A significant increase in the yield of volatiles can be obtained by the technique of "flash" with consequent very short residence times (of the order of seconds or fractions of a second) to inhibit the further decomposition or polymerisation of the products of pyrolysis. To optimise tar yields, the time spent within the reaction zone must be limited to prevent the thermal decomposition of the tars to gaseous products. Very high temperatures must be employed to produce an optimum gas yield under flash pyrolysis conditions.

According to Habashi (1986: 185), two types of carbonisation exist namely high temperature and low temperature carbonisation. High temperature carbonisation is

conducted at temperatures above 900°C and low temperature carbonisation is conducted at temperatures between 400°C and 700°C.

Thermal Activation

The use of steam for activation can be applied to virtually all raw materials. A variety of methods have been developed but all of these share the same basic principle of initial carbonisation followed by activation with steam or carbon dioxide.

During the thermal activation step, the previously formed char is treated at temperatures ranging from 400 – 1100°C in the presence of an oxidising gas (carbon dioxide, oxygen (air) or superheated steam) for a limited time. The oxidising gas reacts with the carbon atoms in the interior of the char to form gaseous reaction products and develop the channels and the pores that contribute to high internal surface area (McKetta, 1976: 112). The oxidising gas or activation component is carried during the activation process by a non-flammable carrier gas that is mostly nitrogen.

Chars activated at low temperatures (400°C) are abbreviated L-type activated carbons and those activated at high temperatures (880°C) are abbreviated H-type activated carbons. H-type activated carbons must be cooled in an inert atmosphere; otherwise it will be converted to an L-type carbon (Habashi, 1986: 185). H-type carbons are hydrophobic while L-type carbon is hydrophilic (DeSilva, 2001: 40).

A number of different types of kilns and furnaces can be used for carbonisation/activation and include rotary (fired directly or indirectly), vertical multi-hearth furnaces, fluidised bed reactors and vertical single throat retorts.

In summary two activation methods exists, namely chemical and physical activation. Physical activation consists of two separate steps namely carbonisation and thermal activation where the material is firstly devolatilised and then thermally activated. The manufacturing of activated carbon can make use of only chemical activation or physical activation or a combination of the two activation methods. The procedure described above is graphically illustrated below in Figure 2.2.3.

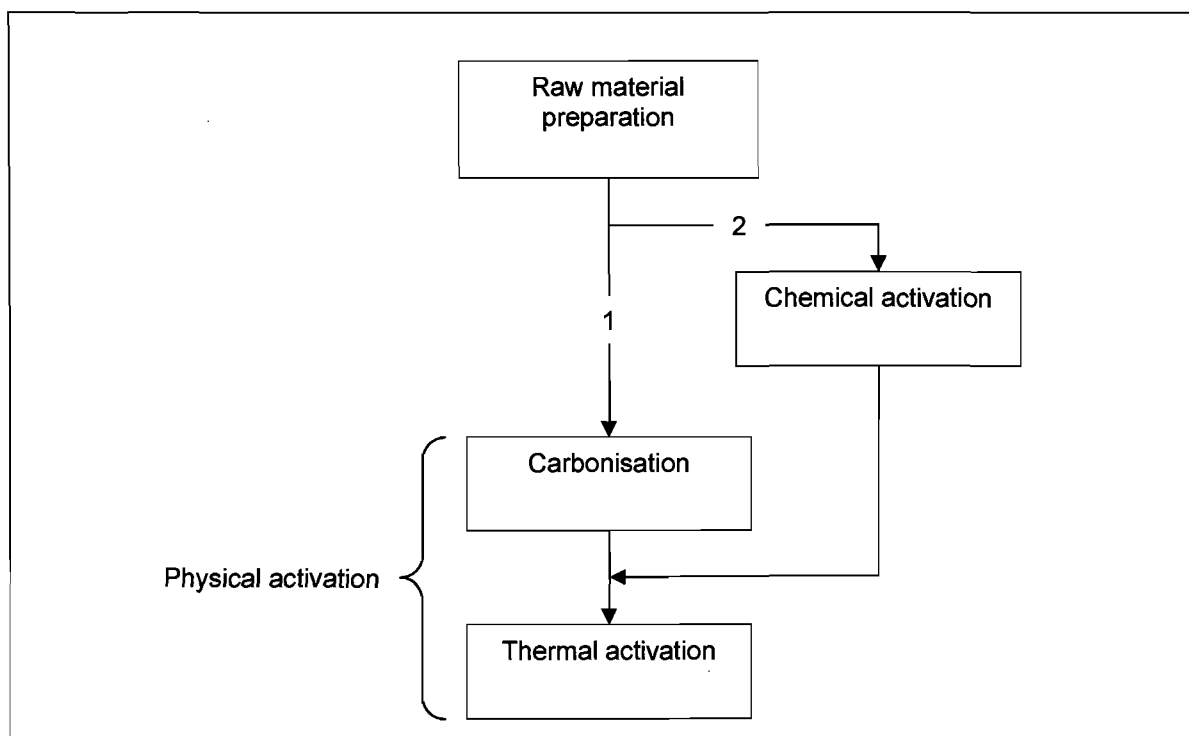


Figure 2.2.3: Graphical presentation of activation methods

2.2.6 Activated carbon reactivation

In many applications (utilising base, non-impregnated carbon) the surface can be regenerated or reactivated in-situ, using steam or other heat treatment processes, thus allowing reuse of the carbon many times over. This principle is used to great advantage in the recovery of volatile organic solvents (the desorbed contaminants being recovered from the steam used to strip the carbon surface). In situations where the adsorbed contaminants are not readily desorbed by steam, thermal reactivation in a kiln is necessary (McKetta, 1976: 141).

In the case of chemically impregnated carbons, reactivation is seldom possible since the chemisorbed (adsorption via chemisorption) contaminants are chemically fixed on the carbon's surface and the impregnant cannot be returned to its original state.

Although the predominant mechanism of an impregnated carbon is one of chemisorption, some physical adsorption, to varying degrees will also take place. In theory the physically adsorbed species could be removed from a saturated impregnated carbon by reactivation (McKetta, 1976: 143). However, the reactivated carbon would remain inactive towards those species requiring a chemisorption

removal mechanism since the impregnant would either still be exhausted or be chemically changed by the reactivation conditions.

Activated carbons are usually reactivated in a multiple hearth furnace and less frequently in a direct fired rotary kiln at temperatures in the 800 – 1 000°C range (McKetta, 1976: 141). Thermal reactivation takes place where the adsorbate is firstly pyrolysed (100 – 800°C) to form volatile material and a residual char which is retained in the carbon pores. Finally, the char is gasified using steam or carbon dioxide above 800°C (McKetta, 1976: 142). During thermal reactivation, physically losses of carbon may be encountered, as well as capacity losses due to blocked pores (leading to decrease in total surface area).

Chemical regeneration of activated carbons is not a widely used practice since strongly adsorbed organics are difficult to extract with a solvent alone (McKetta, 1976: 144).

2.2.7 Principles of adsorption onto activated carbon

Adsorption is the process by which a surface concentrates fluid molecules (adsorbent) by chemical and/or physical forces forming a molecular or atomic film (adsorbate). Absorption is the process whereby fluid molecules are taken up by a liquid or solid and distributed throughout that liquid or solid. The term sorption encompasses both processes, while desorption is the reverse process.

Adsorption is operative in most natural physical, biological, and chemical systems, and is widely used in industrial applications such as activated charcoal, synthetic resins and water purification (Habashi, 1986: 190). Factors that affects adsorption is the nature of the adsorbent, nature of the adsorbate, the pH of the solution (only if the adsorbate is in the liquid phase) and the temperature at which the adsorption process is conducted. Adsorption, ion exchange and chromatography are sorption processes in which certain adsorptives are selectively transferred from the fluid phase to the surface of insoluble, rigid particles suspended in a vessel or packed in a column (McKetta, 1976: 114).

Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent or metallic) of the constituent atoms of the material are filled. But atoms on the (clean) surface experience a bond deficiency, because they are not wholly surrounded by other atoms. Thus it is energetically favourable for them to bond with whatever happens to be available. The exact nature of the bonding depends on the details of the species involved, but the adsorbed material is generally classified as exhibiting physisorption or chemisorption.

2.2.7.1 Physical adsorption

In a physical adsorption (physisorption) process, molecules are held on the carbon's surface by weak forces known as Van Der Waals forces resulting from intermolecular attraction (Habashi, 1986: 189). Adsorption onto activated carbon takes place via the physical adsorption route. In physical adsorption the chemical nature of the adsorbate (the adsorbed molecular species) remains unchanged

(McKetta, 1976: 111). Thus, to affect physical adsorption it is necessary to present the molecule (adsorbate) to be adsorbed to a pore of comparable size. In this way the attractive forces coupled with opposite wall effect will be at a maximum and should be greater than the energy of the molecule. The maximum adsorption capacity of an activated carbon is determined by the degree of liquid packing which can occur in the pores. Adsorption increases with increased pressure and also with increasing molecular weight (Traube's rule), within a series of a chemical family (McKetta, 1976: 111).

2.2.7.2 Chemical adsorption

Chemical adsorption or chemisorption is a process where the molecules chemically react with the carbon's surface (or impregnates onto the carbon's surface) and are held to the surface via strong chemical bonds. The components in a chemical adsorption system are chemically held by the carbon's surface. In chemisorption, electrons are exchanged or shared between the adsorbate and the adsorbent so that chemical reaction actually occurs. The heat of chemisorption is generally of the order 0.44 kJ/mole and chemisorption is often irreversible (McKetta, 1976: 111).

This principle is applied in many industries, particularly in the catalysis field, where the ability to increase the quantity of catalyst on the carbon surface of the carbon can take form. Chemical modification of the activated carbon's surface greatly increases the chance of reaction since the adsorbate has a tremendous choice of reaction sites.

2.2.7.3 Examples of adsorption

- *Heterogeneous catalysis* – It is the process when a solid catalyst interacts with a gaseous feedstock (reactants). The adsorption of reactants to the catalyst surface creates a chemical bond, altering the electron density around the reactant molecule and allowing it to undergo reactions that would normally be available to it.
- *Adsorption refrigeration* – Adsorption refrigeration rely on the adsorption of a refrigerant gas into an adsorbent (activated carbon) at low pressure and subsequent desorption by heating.

2.3 COAL

2.3.1 Definition of coal

Coal is the general descriptive term applied to a group of fossil fuels, black and brown in colour that consists primarily of organic and inorganic materials which have undergone various degrees of decomposition (Meyer ed., 1982: 8). It usually occurs as seams within other consolidated strata (Osborne, 1988: 34).

Coal may be classified as a sedimentary rock consisting essentially of organic components and with only a minor proportion of mineral constituents. It is constant in composition with regular and repeating features which has definable physical and chemical structures (England *et al*, 2002: 1).

The coal layers which are currently mined in South Africa originated about 200 million years ago and are found in the coal formation of the Ecca of the Karoo system. A short discussion on the origin of these coal layers follows (England *et al*, 2002: 1).

2.3.2 The materials from which South African coal was formed

It originated from a variety of plants which include anything from fern plants to large trees, but are totally different from that of today. Fossils from a wide variety of plants are found in the coal and associated shale layers. The dominant plants from which South African (Southern Hemisphere) coal originated are different from those from the Northern Hemisphere. Such differences have been attributed to conditions reigning at the time of coal formation and to the subsequent history of the geological events in each region (England *et al*, 2002: 4).

2.3.2.1 Peat formation

Peat is formed when decaying plant material in moist swamps is biochemically altered (the plant material does not disappear). Suitable conditions form thick beds of peat, which is later buried by sediments such as shale and sandstone. A whole cycle can be repeated and a series of peat beds, separated by shale and sandstone beds

are formed (England *et al*, 2002: 4). Most vegetation contributing to the formation of coal must have grown on site, but trees, floating plants and plant debris may have been carried into the swamps by the feeder river. All coalfields of South Africa were originally fresh water deposits (England *et al*, 2002: 4).

2.3.2.2 Coal formation

For the formation of coal seams, the peat had to subside and become covered with water. Layers of clay, silt and sand then had to be deposited above the peat. A prolonged period of time caused more sediments covering the peat beds, and under the influence of pressure, enhanced temperature and time the second stage of alteration began. In the peat stage of transformation, usually referred to as the biochemical stage, the rate of reaction was rapid and a wide variety of end products could be obtained, depending on the original vegetation present, conditions in the swamp, and the number of variety of fungi, bacteria and other parasitic organisms present. Soon after burial, bacterial activity ceased, and the further transformation proceeded at a very much slower pace. The main net effects of the second, metamorphic, stage are the reduction in the amount of water held by the coal, and a decrease in the oxygen content of the coal (England *et al*, 2002: 4).

2.3.2.3 Formation of coal measure strata

The coal measure strata consist of coal, shale and sandstone, with lesser amounts of grit and occasional conglomerates. The classical succession of deposition consists of peat earth, coal seam, and shale roof which grades into sandstone, followed by similar successions in each coal seam. These deposits are formed due to the gradual subsidence of type strata, so that new bed can be formed by deposition from inflowing water. In South Africa coal seams usually have a shale or sandstone floor, but grits and conglomerates are also found. The same rocks can also form the roof of the seam. For some seams either shale or sandstone forms the typical floor or roof, but in other seams the roof of floor may gradually change laterally from one rock type to another. Some of the thicker seams in the upper portions show a gradual transition from inferior coal to shale. In general, sandstones form the dominant rock type of the coal measures (England *et al*, 2002: 4).

2.3.2.4 Formation of coalfields

South African coalfields were laid down in horizontal beds have virtually remained so. There are minor rolls in the floor causing local dips and raises, and some shallow basins occur, either because of being deposited, or due to later consolidation of underlying beds through pressure of the overburden layers. In certain parts of the coalfields extensive faulting has occurred, nearly always as a result of the intrusion of molten igneous rocks (dolerite) into the coal measures. These intrusions cause displacement of the strata at the fault strike which may be only a few meters or over a hundred meters; the strata on each side of the fault remaining essentially horizontal (England *et al*, 2002: 5). The coalfields are limited by areas where conditions were unfavourable for the formation of coal, by abutment against elevated masses of older rocks forming the borders of the original peat swamps, and by erosion of the coal measures where these occur in elevated positions.

2.3.3 Coal properties

The inert matter in coal

Mineral matter is the inert solid material in coal, and like moisture it reduces the heating value of coal by dilution. On burning coal, the mineral matter remains behind in a slightly altered form as ash (England *et al*, 2002: 12). The objective of coal preparation is to reduce the amount of this inert material to a value acceptable to the consumer (England *et al*, 2002: 12). Both moisture and mineral matter can be subdivided into inherent and adventitious types.

- *Inherent mineral matter* – Inherent mineral matter is mineral matter intimately mixed with the coal. It consists of the minerals present in the original vegetation from which the coal was formed, and finely divided clays and similar materials carried into the swamp by water or by wind. These clays are intimately mixed with the coal substance, and cannot be removed by coal preparation techniques. All South African coals contain varying quantities of such intimately mixed minerals. Such minerals would include finely dispersed clays, quartz, carbonate and pyrite group minerals (England *et al*, 2002: 13).

- *Adventitious mineral matter* – Adventitious (extraneous) mineral matter consists of dirt bands and lenses in the seam, and shales, sandstones and intermediate rocks introduced into the mined product from the roof and floor of the seam. Most of this material is free and easily removed by coal preparation techniques. In some cases the dirt is strongly attached to the coal, but can be freed from the coal by finer crushing (England *et al*, 2002: 13).
- *Other types of mineral matter* – Other forms of extraneous mineral matter are pyrite, and ankerite or calcite (thin white flakes often found in the joints of coal, and sometimes in the bedding plane). These are secondary minerals, deposited in the coal seam after its formation. Both of these mineral types can be so finely disseminated in the coal substance, as to be considered part of the inherent mineral matter from a coal preparation aspect (England *et al*, 2002: 13).

Moisture

Coal materials with a high moisture content result in less material available for combustion and heating because water does not burn. During the combustion of coal, part of the heat available is used up for the conversion of water to steam (England *et al*, 2002: 12).

- *Inherent moisture* – Inherent moisture is contained in the coal lumps or particles. The coal substance is riddled with very small holes or micropores, and these pores contain the inherent moisture. It cannot be removed by draining or centrifuging, or by evaporation at normal temperatures, but can be removed by heating above the boiling point of water (England *et al*, 2002: 12).

2.3.4 Coal locations in South Africa

Nineteen different coal fields are found in South Africa, but this distribution is based on historical and geographical factors and does not necessarily represent geological differences (Snyman and Barclay, 1989: 377). Many assessments of South African coal reserves have been performed since the first attempt in 1928. The 1998 estimate give reserves as 194 000 million tonnes of which 54 300 million tonnes are recoverable (England *et al*, 2002: 5). The quality (predominantly ash content) of the in-situ reserves are generally found to be decreasing.

2.3.5 Industrial uses for coal

Coal is primarily used as a solid fuel to produce electricity and heat through combustion.

Low-ash, low sulphur bituminous coals are subjected to high temperatures (1 000°C) without oxygen where the volatile constituents are driven off to form coke, a solid carbonaceous residue. In this process the fixed carbon and residual ash are fused together. Metallurgic coke is used as a fuel and as a reducing agent in smelting iron ore in a blast furnace. Petroleum coke is the solid residue obtained in oil refining, which resembles coke but contains too many impurities to be useful in metallurgical applications.

Coal gasification breaks down the coal into its components, usually by subjecting it to high temperature and pressure, using steam and measured amounts of oxygen. This leads to the production of a synthetic gas, a mixture mainly consisting of carbon monoxide (CO) and hydrogen (H₂).

Coal can also be converted into liquid fuels like petrol or diesel by several different processes. The Fischer-Tropsch process of indirect synthesis of liquid hydrocarbons is used today by Sasol in South Africa.

2.3.6 Coals used in this study

This information presented below was sourced from a yearly report distributed by the Department of Minerals and Energy (DME, 2006: 47). It refers to the different coals which were used in this study, to determine the viability of using South African coal as a raw material for the production of activated carbon for the extraction of platinum group metals.

Table 2.3.1 to Table 2.3.4 shows the different published properties for each of the coals used in this study. It includes proximate, ultimate, petrographic and ash analyses.

A description of the respective analyses that is available for the characterisation of coal is discussed in **Appendix A**.

Table 2.3.1: Calorific value, proximate analyses and total sulphurs (2001-2002) of coal products (air-dry basis and dry basis) (DME, 2006: 47)

Sample	Air-dry basis							Dry basis					
	Calorific value (MJ/kg)	Gross calorific value (MJ/kg)	Moisture %	Ash %	Volatile matter %	Fixed carbon %	Total Sulphur %	Calorific value (MJ/kg)	Gross calorific value (MJ/kg)	Ash %	Volatile matter %	Fixed carbon %	Total Sulphur %
Witbank Seam 4	27.35	27.30	2.87	14.50	23.80	58.83	0.53	28.16	28.11	14.97	24.53	60.57	0.54
New Vaal	15.84	15.78	7.20	37.90	21.70	33.20	0.62	17.07	17.01	40.80	23.40	35.80	0.67
Grootegeeluk	29.43	29.35	3.10	8.40	37.10	51.40	0.80	30.37	30.29	8.70	38.30	53.00	0.83
Tshikondeni	31.62	31.54	0.80	12.30	22.50	64.40	0.85	31.88	31.79	12.40	22.70	64.90	0.86

Table 2.3.2: Hardgrove grindability indices and ultimate analyses of selected coal products (Air-dry basis and dry-ash-free basis) (DME, 2006: 52)

Sample	HGI	Air-dry basis					Dry ash free basis						
		Carbon %	Hydrogen %	Nitrogen %	Total Sulphur %	Oxygen %	Carbon %	Hydrogen %	Nitrogen %	Total Sulphur %	Oxygen %	Calorific value %	Volatile Matter %
Witbank Seam 4	-	69.43	3.70	1.61	0.53	7.38	84.02	4.48	1.94	0.63	8.92	33.10	28.77
New Vaal	68	42.04	2.27	0.98	0.62	8.99	76.57	4.13	1.79	1.13	16.38	28.85	39.50
Grootegeeluk	53	72.94	4.85	1.37	0.80	8.54	82.41	5.48	1.55	0.90	9.66	33.25	41.90
Tshikondeni	84	78.70	4.20	1.81	0.85	1.34	90.56	4.83	2.08	0.98	1.54	36.39	25.90

Table 2.3.3: Petrographic composition, Mean random vitrinite reflectance and rank designation of selected coal products (DME, 2006: 58)

Sample	Maceral composition (percent by volume) mineral matter free basis				Rank vitrinite random reflectance	Rank designation
	Vitrinite %	Liptinite %	Reactive inertinite %	Inert inertinite %	Mean Random Reflectance	Parameter: Vitrinite Random Reflectance
Witbank Seam 4	24	5	22	49	0.76	Medium - Rank C
New Vaal	19	3	23	55	0.53	Medium - Rank D
Grootegeeluk	91	5	<1	4	0.70	Medium - Rank C
Tshikondeni	90	1	3	6	1.25	Medium - Rank B

Table 2.3.4: Ash analyses, phosphorus and reducing ash fusion temperature of selected coal products (DME, 2006: 55)

Sample	Expressed as Constituents in Ash										In Coal
	SiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	P ₂ O ₅ %	TiO ₂ %	CaO %	MgO %	K ₂ O %	Na ₂ O %	SO ₃ %	P %
Witbank Seam 4	44.63	33.83	3.52	1.94	1.54	7.66	1.94	0.63	0.41	2.92	0.122
New Vaal	57.60	31.70	2.61	0.25	1.55	2.50	0.76	0.44	0.19	1.44	0.044
Grootegeeluk	67.20	19.40	5.34	0.21	1.73	1.36	0.59	0.99	0.24	1.02	0.009
Tshikondeni	57.00	25.00	4.96	0.48	1.40	3.38	1.69	1.40	0.64	3.55	0.026

2.4 ACTIVATED CARBON FROM COAL

2.4.1 Research articles

An article, sourced from Carbon (Sun *et al*, 1997: 341) reported on the research conducted on Illinois Basin bituminous coal where an activated carbon product was prepared using a three step process: ⁽¹⁾oxidation of the coal in air at 150 – 250°C for 2 – 40 hours, ⁽²⁾devolatilisation of these oxidised coals in nitrogen at 500 – 730°C for 1 hour and ⁽³⁾activation (gasification) of the chars in 45% steam, 4% oxygen in nitrogen at 730 – 880°C for 3.5 to 96 hours. The activated carbon products were characterised using CO₂ single-point BET surface area, helium and bulk densities, pore volume and toluene adsorption capacity. Sun *et al* reported that more than 70% of the carbons produced had a surface area exceeding 1 000m².g⁻¹ and that the oxidative pre-treatment of the coal preserved the microstructure of the coal, which leads to the high surface area. A production cost analysis was also performed and they found that the most cost-effective final product is not the activated carbon with the highest surface area.

Lignite and gas coal (high-volatile bituminous coal) has been used in a study by Lorenc-Grabowska *et al* (2004: 194) to develop mesoporosity via coal modification using Ca- and Fe-exchange and reported that an 20 – 25% increase of mesopore volume was observed.

Gañan *et al* (2004: 347) prepared high-porosity carbons from bituminous coal pitches and combined the use of chemical and physical activation. The activated carbons prepared using this process, resulted in surface areas in excess of 400m².g⁻¹ being obtained.

Qiang *et al* (2005: 461) investigated the selective adsorption properties of SO₂ and NO onto coal based activated carbon.

Hsu and Teng (2000: 155) prepared activated carbon from bituminous coal via chemical activation (ZnCl₂, H₃PO₄ and KOH), after which the sample was carbonised in nitrogen at various temperatures. It was found that ZnCl₂ and H₃PO₄ were not

suitable for preparing high-porosity carbons from bituminous coal, while KOH can produce carbons with high porosity.

Mineral matter content can affect the adsorptive capacity of an activated carbon. Linares-Solano *et al* (2000: 635) investigated this subject and reported that the samples with lower ash content yielded higher micropore volumes.

2.4.2 History on the metallurgical utilisation of activated carbon

Activated carbon is an adsorbent commonly used in extractive metallurgy in areas such as gold, silver, platinum, palladium, osmium and uranium (Habashi, 1986: 184). Davis (1880) and Egleston (1890) reported the use of activated carbon in the gold recovery industry by the chlorination process. Johnston (1894) first described the recovering of gold from a cyanide solution. This method was applied in Australia where the gold was adsorbed onto activated carbon and then floated to recover the gold. Gross and Scott did an extensive study in 1927 on the adsorption of gold and silver from a cyanide solution. They found that the maximum adsorption of gold decreased in the presence of silver.

Heyman found in 1932 that activated carbon is an excellent means of precipitating platinum, palladium and osmium from dilute chloride solutions (Habashi, 1986: 185).

Rhenium and molybdenum were selectively separated from solutions of their acids by utilising the difference in their adsorption by carbon (Habashi, 1986:185). Lange and Graf (1964) described a pilot plant in which to recover rhenium from the Mansfeld ore in Germany based on adsorption by activated carbon (Snell *et al*, 1974: 140). The American Metal Climax company developed a pilot plant for the recovery of molybdenum from tailings in 1963 (Habashi, 1986: 186).

Colorado School of Mines Research Foundation developed a process for recovering vanadium from mining wastes (Habashi, 1986: 187).

In 1957 it was reported that the uranyl ion was also adsorbed by activated carbon (Habashi, 1986: 186; Kütahyalı and Eral, 2004: 109).

2.5 ACTIVATED CARBON ANALYSES

2.5.1 BET surface area

Theory

Since surface area plays a significant role in the adsorption process, BET results can be used to compare different activated carbon samples on the grounds of the surface area available for adsorption of phenol from the liquid solution.

Stephan Brunauer, Paul Hugh Emmett and Edward Teller published an article about the BET theory for the first time in 1938 (Brunauer, 1938:309). Currently, the BET theory is a well-known rule for the physical adsorption of gas molecules on a solid surface (in this study, activated carbon). The BET theory is an extension of the Langmuir adsorption theory where only monolayer molecular adsorption is addressed. The following hypothesis is assumed with multilayer adsorption:

- gas molecules physically adsorb on a solid in layers infinitely,
- there is no interaction between the different adsorption layers,
- and the Langmuir theory can be applied to each individual layer

The resulting BET equation is expressed by equation (2.5.1):

$$\frac{1}{v[(P/P_0)-1]} = \frac{1}{v_m c} \left(\frac{P}{P_0} \right) + \frac{1}{v_m} \quad (2.5.1)$$

where P and P_0 are the equilibrium and saturation pressure (kPa) respectively, at the adsorption temperature, v is the adsorbed gas quantity, while v_m is the monolayer adsorbed gas quantity. The BET constant, c , is given by the following equation (2.5.2):

$$c = \exp\left(\frac{E_1 - E_L}{RT}\right) \quad (2.5.2)$$

where E_1 is the heat of adsorption (J/mol) for the first layer, while E_L (J/mol) is that for the second and higher layers, and is equal to the heat of liquefaction. Equation

(2.5.2) is an adsorption isotherm and can be plotted as a straight line with $\left(\frac{1}{v[(P_0/P)-1]}\right)$ on the y-axis and P/P_0 on the x-axis, according to experimental results. This plot is called a BET plot.

The BET method is widely used in surface science for the calculation of surface areas of solids by physical adsorption of gas molecules. The total surface area S_{total} (m^2) and the specific surface area S (m^2/g) are evaluated by equations (2.5.3) and (2.5.4) respectively:

$$S_{total} = \frac{(v_m N s)}{M} \quad (2.5.3)$$

$$S = \frac{S_{total}}{a} \quad (2.5.4)$$

where N is the Avogadro number, s is the adsorption cross section, M is the molecular weight (g/mol) of the adsorbate and a (gram) is the weight of the solid sample.

Equipment

The equipment that was used in the determination of the surface area is a Micromeritics ASAP2020 Surface area analyser. A discussion regarding surface area measurement and equipment detail is described in **Appendix B**.

2.5.2 pH of activated carbon

Theory

The pH of activated carbon can be defined as the pH of a suspension of activated carbon in distilled water. This process where activated carbon is shaken in distilled water is called hydrolytic adsorption. It has been reported that this reaction does not take place in the absence of oxygen (Habashi, 1986: 186). The change in pH of the suspension can either be acidic or basic, depending on the method of activation. The numerical value of the pH will be affected by the experimental environment, for example time and temperature of extraction and the activated carbon/water ratio

(Faust and Aly, 1983: 140). Due to the nature of some adsorption reactions on activated carbon, it is necessary to conduct the reaction at a specific pH to obtain maximum adsorption. To increase or decrease the pH, an acid or alkali can be added (Faust and Aly, 1983: 140).

When an H-type activated carbon is produced, it is hydrophobic in nature and tends to take on a positive charge by adsorbing the H^+ -ions when immersed in water (most surface oxides present are basic in nature). The L-type carbon absorbs the hydroxyl (OH^-) ions when immersed in water (formed surface oxides are acidic in nature).

Equipment

A benchtop pH meter was used to determine/monitor the solution pH during the analysis. Further detail regarding this device is mentioned in **Appendix B**.

2.5.3 Phenol adsorption

Theory

This characterisation method utilised the adsorption property of an activated carbon sample. Activated carbon is suspended in a solution with a known phenol concentration and stirred for a period of time. During this period, the phenol from the parent solution is adsorbed onto the surface of the activated carbon. The phenol solution is then sampled at selected time intervals. These samples are then analysed with an UV-VIS to determine the adsorbance. The phenol concentration and hence the adsorption capacity is calculated from the adsorbance of the specific activated carbon sample (Halhouli *et al*, 1997: 3027).

Equipment

After the phenol adsorption studies were conducted, the concentration of the phenol deficient solution had to be determined. This was performed using a UV-vis spectrophotometer. The operation of the equipment is described in **Appendix B**.

2.5.4 Analyses of other activated carbon properties

Because of the diverse end uses to which a carbon may be applied, it is difficult to conduct specific tests related to any one application. The size and number of pores essentially determine an activated carbon's capacity in adsorbing a specific compound. Since pore size and total pore volume determinations are quite lengthy, they are impractical as a means of quality control during its manufacture. It is therefore necessary to relate the carbon's surface capabilities to a standard reference molecule.

The American Society for Testing and Materials (ASTM), Committee D-28 on activated carbon, has defined several common property tests (some relevant test mentioned in brackets). The following characteristics determine the quality and adsorption capacity of activated carbon:

Adsorption characteristics

Adsorptive capacity is paramount for both gas-phase and liquid-phase activated carbons. It determines the volume of gas or liquid that can be treated per unit weight of carbon (Snell *et al*, 1974: 140). Adsorptive capacity determines both the direct cost for carbon treatment and the sizing of equipment (filters for powdered carbons and columns for the granular type) (Snell *et al*, 1974: 141). The effectiveness of activated carbon is usually specified by the amount of a certain test chemicals it can adsorb per unit weight of activated carbon used.

Carbon Tetrachloride Activity (ASTM D3467-99)

The most widely used method is to measure the carbon's capacity to adsorb carbon tetrachloride (CTC) and express it as w/w% (% CTC activity). CTC activity is accepted as a means of specifying the degree of activation or quality of activated carbon. This test is being substituted in some areas by a similar test which uses n-butane (Butane Number test). To obtain the Butane Number (ASTM D5742-95(2000)) from carbon tetrachloride number, the CTC Activity is divided by 2.55.

Iodine Number (ASTM D4607-94(1999))

A standard measure for activated carbon in liquid phase applications is to determine the Iodine number (mg/gm) of the carbon (characterises activated carbon performance). A higher Iodine number indicates a higher degree of activation.

*Physical characteristics***Inorganic content**

The inorganic content of activated carbon depends on the type of ⁽¹⁾precursor (raw material), ⁽²⁾preparation method and ⁽³⁾activation reagent or component that was used (Lowry, 1981: 10).

pH Value (ASTM D3838-80(1999))

The pH of activated carbon is defined as the pH of the water in which activated carbon is suspended in. The pH value of activated carbon is a measure of whether it is acidic or basic.

Particle Size Distribution (ASTM D2862-97)

The particle size distribution of an activated carbon sample determined using test sieves. The finer the particle size of an activated carbon, the better access to the surface area and the faster the rate of adsorption kinetics.

Moisture (ASTM D2867-99)

This is the amount of water physical bound on the activated carbon under normal condition. The practical limit for the level of moisture content allowed in the activated carbon varies within 3 to 6%.

Ash Content (ASTM D2866-94(1999))

Ash content is the inorganic, inert, amorphous and unusable part present in the activated carbon and is related to the raw material. Ash content reduces the overall activity of activated carbon and reduces the efficiency of reactivation. Ash content is less important except where the carbon is used as a catalyst support since certain constituents of the ash may interfere or destroy the action of precious metal catalysts. Ash content also influences the ignition point of the carbon – this may be a

major consideration where adsorption of certain solvents is concerned (McKetta, 1976: 133).

Surface Area

The internal surface area of a carbon is usually determined by the BET method (Braunauer, Emmett and Teller). This method utilises the low-pressure range of the adsorption isotherm of a molecule of known dimensions (usually nitrogen or in fewer cases carbon dioxide). This region of the isotherm is generally attributed to monolayer adsorption. Thus by assuming the species is adsorbed only one molecule deep on the carbon's surface, the surface area can then be calculated.

Apparent Density (ASTM D2854-96(2000))

Apparent density is the weight of one litre of activated carbon (g/l). Density depends on the raw material being used and the degree of activation. The density does not affect the effectiveness of the activated carbon but will have an effect on adsorption per unit volume. Higher density provides greater volume activity and normally indicates higher quality activated carbon.

Hardness/abrasion number (ASTM D3802-79(1999))

The hardness and resistance to attrition of activated carbons is becoming more and more important. The loosely applied term "hardness" is somewhat difficult to measure on activated carbon. Three forces can mechanically degrade an activated carbon: ⁽¹⁾ impact, ⁽²⁾ crushing and ⁽³⁾ attrition. Of these three, the force of attrition, or abrasion, is the most common cause of degradation in actual end use. At the present time, there are two commonly used methods available to evaluate a carbon's hardness:

- *Ball-pan hardness test* – A screened, weighed sample of carbon is placed in a special hardness pan with a number of stainless steel balls and subjected to combined rotating and tapping action (Kirk and Othmer, 1956: 564).
- *Stirring Bar Abrasion Test* – A carbon sample is placed in a cylindrical vessel where an inverted T-shaped stirrer is turning rapidly at a controlled rate.

Interrelation of properties

There is a relationship between BET surface area and CTC adsorption and this is taken into account when activated carbon specifications are formulated. CTC activity, density and ash content are interrelated and provide a simple means of manufacturing control. As quality of degree of activation increases, CTC activity and ash content increase and density decreases.

Furthermore, CTC activity being equal, coconut carbons show higher density and lower ash content than coal based carbons. Wood based carbon show much lower density than either coal or coconut based carbons but ash contents midway between coal and coconut carbons.

Thus, these properties are not only a means of controlling quality during manufacture but also assist in determining the raw material and quality of an unknown carbon. CTC activity, density, hardness, mesh size and raw material information will enable selection of a suitable carbon from most common application (excepting those utilising chemisorption as a prime mechanism).

2.5.5 Industrial activated carbon analyses

Activated carbon adsorption tests are useful in classifying carbons for general application areas and in manufacturing quality control. Although actual process fluids might appear more useful for testing the adsorptive powers of activated carbon than standard gas or liquid methods, process stream adsorbate and other impurity concentrations often vary with such wide limits that process fluid tests may be less reliable and less convenient than standard gas and liquid tests (Kirk and Othmer, 1956: 565).

Many industrial activated carbon tests have not yet been standardised by ASTM but are in common use. The following are standard industrial tests;

- Shaking a sample with several different sized steel balls for a standard period reveals its hardness or strength; the percent hardness reported is based upon the decrease in average particle size of the carbon calculated from screen

analyses before and after shaking with the steel balls (Kirk and Othmer, 1956: 564).

- Gas adsorptive properties of activated carbon may be statistically determined by exposure to the desired substance at constant temperature and humidity levels in a bell jar. Adsorptive capacity is reported as weight percent pickup of the adsorbate by the carbon adsorbent (Kirk and Othmer, 1956: 565).
- Gas adsorptive properties can be determined dynamically by blowing a test gas through a carbon bed or tube of a standardised depth and diameter. The equilibrium amount of adsorbate per unit weight of carbon is determined when the carbon bed becomes saturated with adsorbate. The most commonly applied gas adsorption tests are carbon tetrachloride and benzene activity tests. The carbon tetrachloride activity of a sample is its weight percent pickup, at equilibrium, of carbon tetrachloride from a dry stream saturated with carbon tetrachloride. Benzene activity is based on the same principle, except that the benzene-saturated air stream is usually diluted to 10 vol% saturation. Another common gas-phase adsorption test is retentivity. Both benzene and carbon tetrachloride retentivities measure the weight percent of adsorbate retained by a carbon sample first saturated with adsorbate and then subjected to air blowing for 6 hours (Kirk and Othmer, 1956: 565).

2.6 CONCLUSION ON THE POTENTIAL SUCCESS OF COAL FOR THE PRODUCTION OF ACTIVATED CARBON

From the literature that was studied during the course of this literature survey, it can be assumed that South African bituminous coals can be regarded as a suitable precursor for the manufacturing of activated carbon with the exception of coal with high vitrinite content which causes the pore structure of the coal to collapse.

Because of the existing inherent micropore and macropore structure of South African coal, with the addition of the different preparation steps as was described, an activated carbon product with large internal surface area and high porosity can be produced.

CHAPTER 3

EXPERIMENTAL

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3.1 INTRODUCTION

To achieve the desired results in manufacturing an activated carbon meeting all criteria, an experimental process needed to be considered. This section considers the experimental procedure which was followed and addresses the equipment which was used.

3.2 EXPERIMENTAL SETUP

From Chapter 2 (Section 2.2.5, p. 13), it was seen that two different routes could be followed to manufacture activated carbon, i.e. physical and chemical activation. During this study, physical activation was considered. An experimental flow sheet was decided on and is presented in Figure 3.2.1

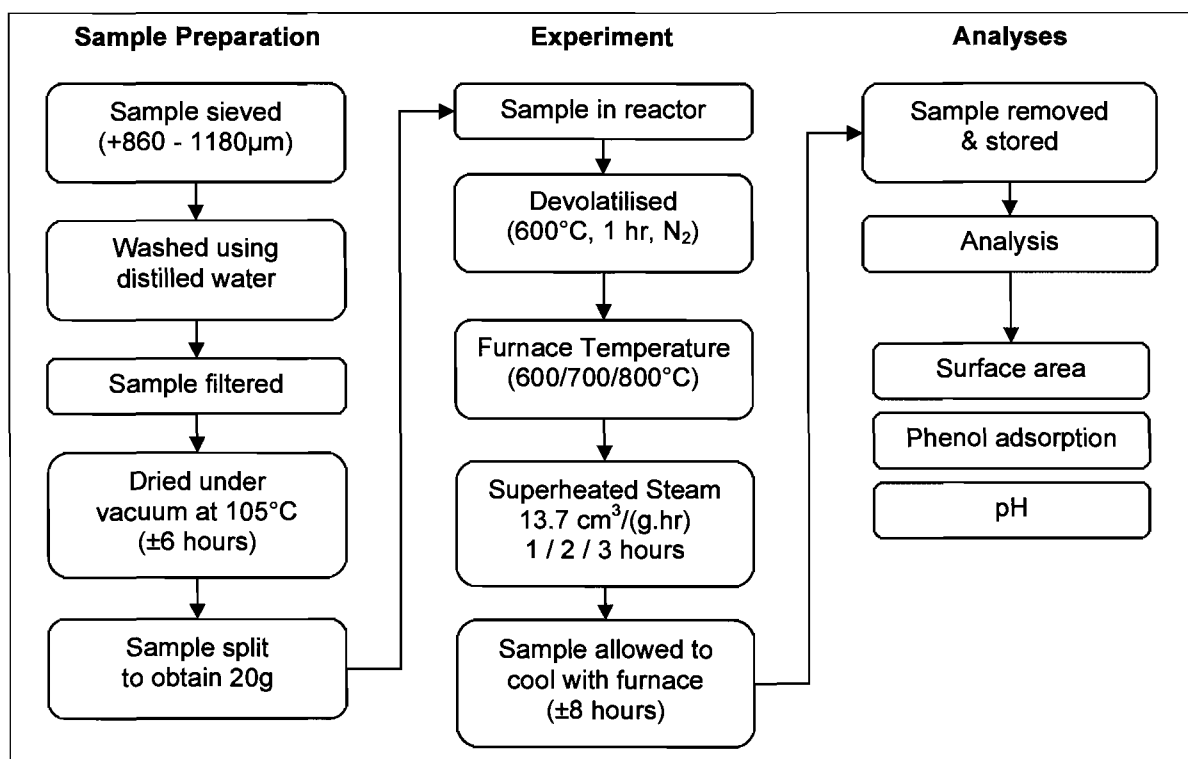


Figure 3.2.1: Summary of experimental flow sheet

As can be seen in the above-mentioned figure, the experimental procedure was divided into three sections namely:

- Sample preparation – The sample was prepared and cleaned before it was stored for the actual experiment
- Actual experiment – Sample was loaded into the reactor, temperatures were fixed and the activated carbon was prepared.
- Analyses – This step included analyzing the product for adsorption area and adsorption characteristics (pH measurements and phenol adsorption).

The coal as raw material under consideration was:

- Witbank Seam 4 (Witbank area, South Africa)
- New Vaal (Vereeniging area, South Africa)
- Grootegeeluk (Ellisras area, South Africa)
- Tshikondeni (Musina, South Africa)

Published ultimate and other related analyses regarding these different raw materials were presented in Section 2.3.5 (p. 27).

When experimental work was conducted on the Grootegeeluk (as well as Tshikondeni) coal, it was found that the particles of this raw material fused together. This can be explained by the high vitrinite content (Table 2.3-3, p. 27) of both these samples and exposure to high temperatures causes the micropores and macropores to collapse. Due to the objectives stated in Chapter 1, producing an activated carbon with high surface area, Grootegeeluk or Tshikondeni coal used as raw material will not be a feasible option. Further experimental procedures and analyses were not performed on these raw materials.

Only a laboratory proximate analysis was performed on the Witbank Seam 4 and New Vaal coals. These results are shown in Chapter 4 (Section 4.2, p. 48).

3.3 EXPERIMENTAL EQUIPMENT

The requirements for the manufacturing of activated carbon, necessitated equipment which have to:

- Heat the raw material in a vessel under a oxygen-free (inert) environment to a specific temperature
- Pass steam over the coal sample
- Measure different temperatures and the steam flow rate

From the above-mentioned criteria, the following equipment was selected to fulfil this role:

- Reactor – vessel with top and bottom sieves; used to contain the sample
- Load cell – connected to a load cell amplifier (LCA) with digital read-out
- Water reservoir – 200 cm³ glass vessel with tie-off point for a tube connection
- Piston pump
- Steam generator – 100 cm³ steel vessel with a heating jacket
- Nitrogen gas heating
- Combined steam/N₂ heating
- Vertical tube furnace – maintaining temperatures of 1 000°C
- Thermocouples – measure temperature at various points
- Data logging device – personal computer

Figure 3.3.1 shows the experimental setup including all unit processes:

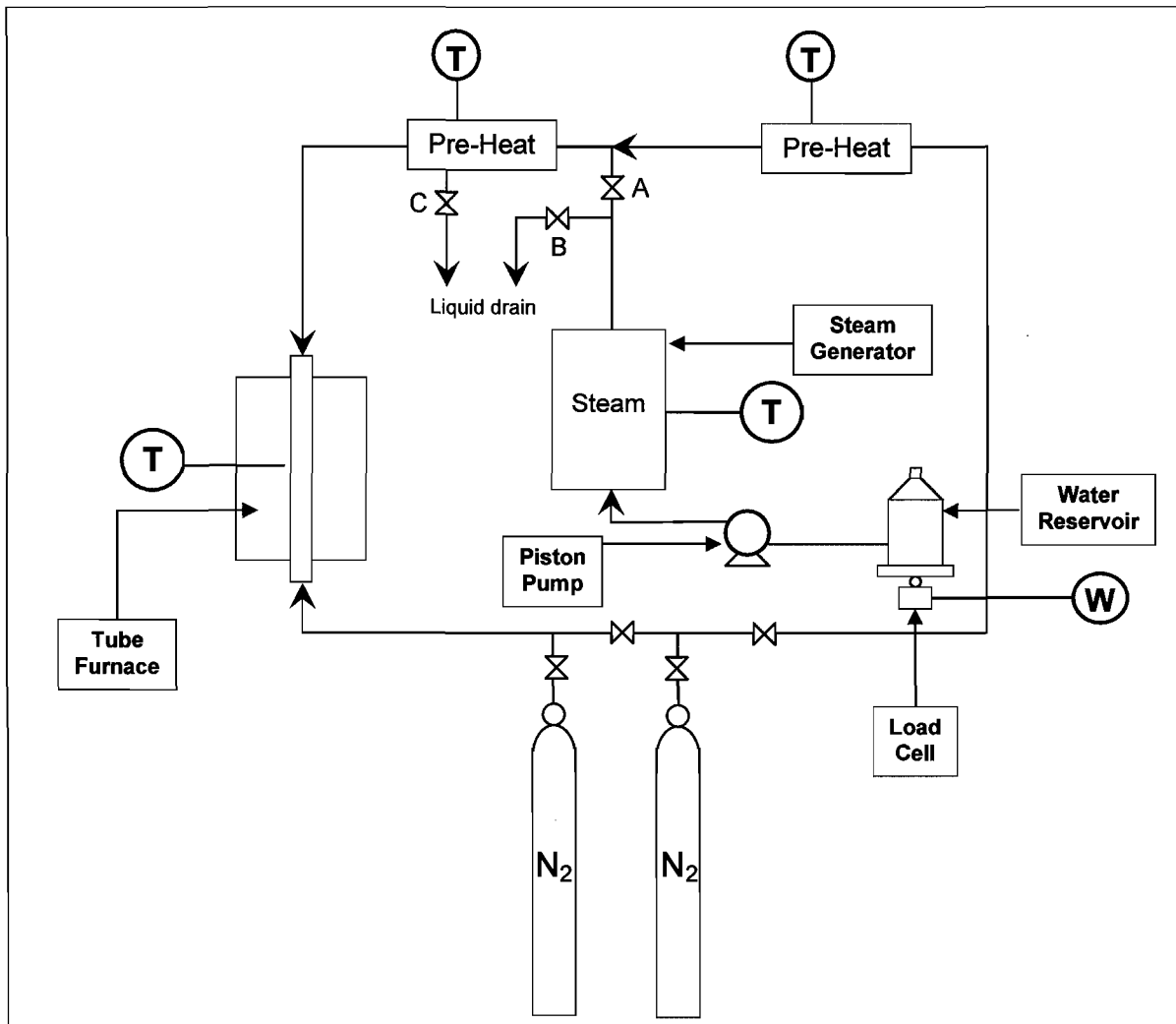


Figure 3.3.1: Experimental setup

The main components in the experimental setup from the above figure (Figure 3.3.1) include a vertical tube furnace capable of reaching temperatures of up to a 1 000°C, and a steam generator with a piston pump delivering the required flow rate of steam.

The water for the steam generator was supplied via a reservoir which was located on a load cell. The mass change (W) was recorded from which the steam flow rate was determined. It was assumed that the mass loss in the reservoir was an indication of the steam flow rate supplied to the furnace.

The steam generator was controlled at a temperature of 160°C. The steam was mixed with nitrogen and passed through a heating stage which was also controlled at 160°C. The piping to the furnace was well insulated that ensured minimal heat loss. The steam/nitrogen mixture was then passed over the sample in the furnace.

A thermocouple in the reactor ensured that the sample temperature was monitored and logged.

The length of the reactor was 200mm, while the length of the heating zone was 300mm. This ensured that both the coil and the reaction zone were at the controlled temperature. The coil ensured that the steam/nitrogen mixture was brought from its initial temperature of $\pm 160^{\circ}\text{C}$ to which temperature the furnace was controlled at.

With each experimental run, 20 grams of raw material was activated. This sample amount occupied more or less half of the volume of the reactor.

3.4 CONTROL PHILOSOPHY

In order to achieve the desired results, a control philosophy had to be adapted to suit the process. Figure 3.4.1 represents this control philosophy that was implemented to control the 'as built' experimental system. The process variables that were measured, were:

- Weight signal from the load cell
- Sample temperature within the reactor

The process variable that were measured and controlled, were:

- Temperature on both the heating stages
- Temperature on the steam generator heating jacket

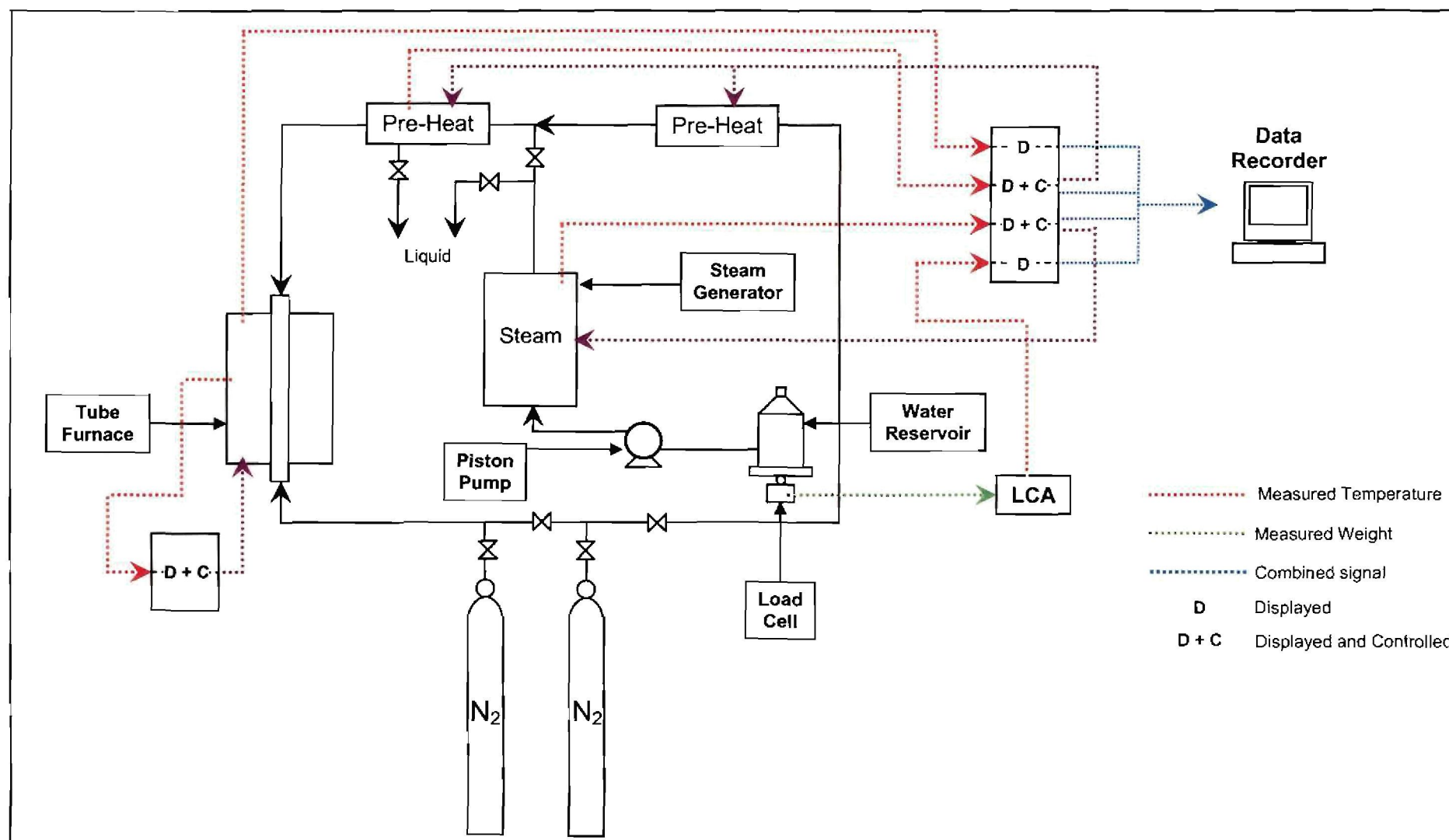


Figure 3.4.1: Experimental setup with controller configuration

3.5 EXPERIMENTAL PROCEDURE

A detailed experimental procedure is presented in **Appendix C**. The experimental procedure can be summarized as follows:

- The coal was crushed and sieved to obtain the correct particle size range. This raw material was then washed with a low concentration (1M) HCl-solution. After the raw material was filtered and oven-dried, it was stored in air-tight containers.
- 20g raw material was weighed off and loaded into the reactor. The reactor was then lowered into the vertical tube furnace. The nitrogen flow was initiated and the furnace was set at 600°C where the sample devolatilised for 1 hour. During this time, the steam generator was started.
- After the devolatilisation step, steam was introduced into the reactor. The duration of this step was determined by the activation time. The furnace was then switched off and the sample was allowed to cool down in a nitrogen-rich atmosphere.
- The sample was removed, stored and labelled. The respective samples was then analysed as described in **Appendix C**.

3.6 EXPERIMENTAL ANALYSES

Phenol adsorption studies

Activated carbon was stirred in a phenol solution and samples were taken at various intervals. These samples were then analyzed with an UV-vis to determine the phenol concentration of the parent solution. The concentration results reflected the amount of phenol that was adsorbed by the activated carbon. Details regarding the phenol adsorption studies are presented in **Appendix C**.

pH Measurement

The determination of the activated carbon pH was performed using a magnetic stirrer and a pH meter which continuously logged the pH at specific intervals (5 minutes) over a specific time period (200 minutes). A detailed procedural description for this analysis method is given in **Appendix C**.

BET Surface analysis

The BET surface analysis was performed at Protechnik (PTY), Highveld Technopark, Pretoria. A detailed description of the equipment and procedure to measure the surface area is discussed in **Appendix B** and **Appendix C** respectively.

CHAPTER 4

RESULTS AND DISCUSSION

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4.1 INTRODUCTION

The following chapter includes information which was obtained from the experimental work performed. From Chapter 3, it can be seen that three sets of results were obtained for each raw material, including BET surface area measurement, pH analyses and lastly phenol adsorption. The different sets of results for the Witbank Seam 4 and New Vaal coal will be discussed below.

4.2 CHARACTERISATION OF RAW MATERIAL

A proximate analysis was performed on the different raw materials used in this study. This analysis method consists out of various ISO and SABS standards which will give information for example on the sample water content (%) as well as volatile matter (%), ash content (%) and fixed carbon content (%). Table 4.2.1 below shows results regarding the proximate analysis of the two different raw materials which were used.

Table 4.2.1: Raw material proximate analysis

Test	Witbank Seam 4	New Vaal
% Moisture (SABS 924)	1.31%	2.37%
% Ash (DB) (ISO 1171)	29.94%	34.40%
% Volatiles (DAFB) (ISO 562)	28.17%	29.96%
% Fixed Carbon (Calculation)	49.67%	44.86%
Calorific Value (ISO 1928)	22.33MJ.kg ⁻¹	19.40MJ.kg ⁻¹

When the proximate analysis results from Table 4.2.1 and Table 2.3.1 are compared, a noticeable difference exists. It should be noted that the analysis results presented in Table 2.3.1 are published monthly averages of the saleable products of the mine. The proximate analysis of the Witbank Seam 4 and New Vaal samples used in this study represent the actual values for the specific samples used in the study. These compare well with the published results (Table 2.3.1, p. 27).

Observations during experiment

During the experiment, the following observations were noted:

- The sample in the reactor was heated at a rate of $25.85^{\circ}\text{C}/\text{min}$ up to 67% of the specified activation temperature.
- Devolatilisation started to occur when the furnace temperature was at $\pm 420^{\circ}\text{C}$. This process continued for ± 25 minutes. 60 minutes was then allowed in total for devolatilisation to complete. At this point the steam generator was started, thus an hour was allowed for the steam generation process to equal the flow rate at which the water was introduced into the system.
- After the experiment was completed, it was found that the initial cooling rate of the sample in the reactor was $1.58^{\circ}\text{C}/\text{min}$; this rate decreases as the furnace temperature decreased thus following an exponential decrease in sample temperature.
- Whenever New Vaal coal was used as raw material, small traces of ash was found between the activated carbon particles when the sample was removed from the reactor unit.

After the experimental run was completed, the activated carbon sample was removed from the reactor and stored in an air-tight container for the following analysis:

- BET surface area
- pH measurements
- Phenol adsorption

4.3 EXPERIMENTAL RESULTS

4.3.1 BET surface area

BET surface measurements were conducted on all of the activated carbon samples prepared from the respective raw materials. Surface area was analysed with a Micromeritics ASAP 2020 surface area analyzer (Equipment discussed in **Appendix B**). The surface area results for the activated carbon prepared from Witbank Seam 4 coal are presented in Table 4.4.1.

Table 4.3.1: Witbank Seam 4 – Surface area (m^2/g)

Witbank Seam 4	1 hr	2 hr	3 hr
600°C	4.70	41.70	175.73
700°C	4.30	107.53	243.30
800°C	9.83	159.40	338.33

These abovementioned results are graphically presented in Figure 4.4.1.

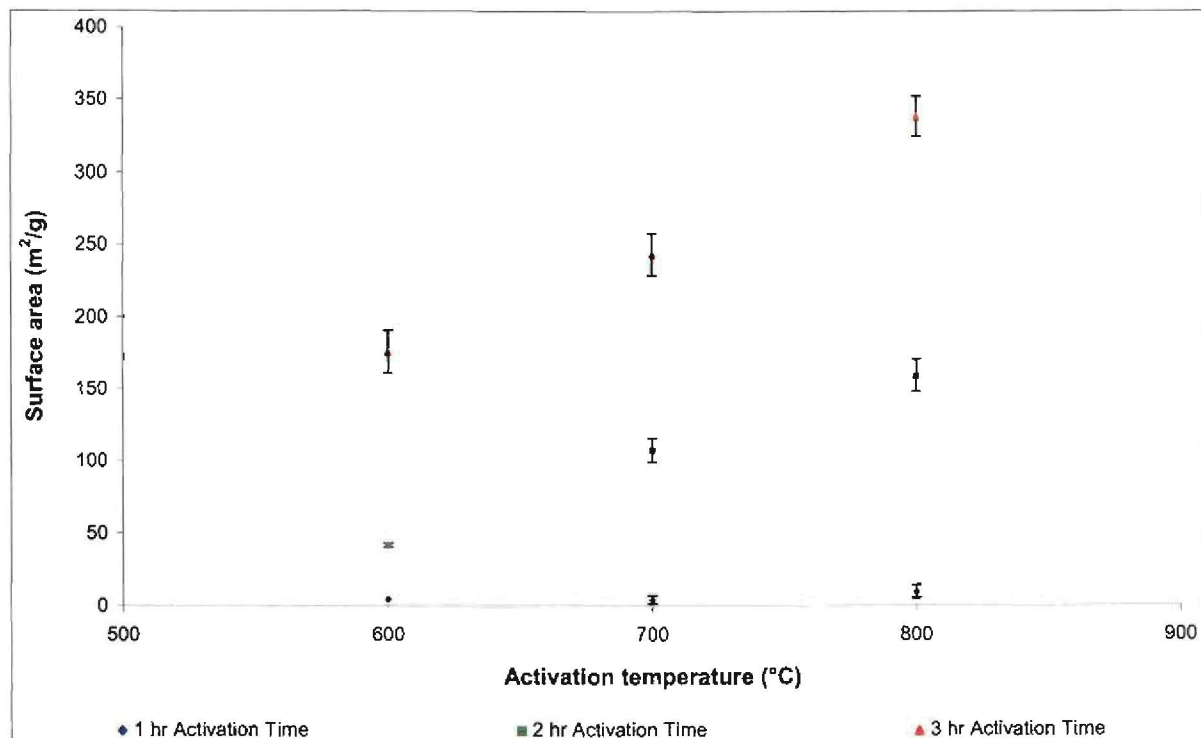


Figure 4.3.1: Witbank Seam 4 – Surface area (m^2/g)

From Figure 4.4.1 it can be seen that surface area increased with increasing activation temperature and activation time. This is also an indication of the quantity of adsorption sites that are created with increasing activation time and temperature. The effect of the large quantity of adsorption sites will be seen when phenol adsorption studies are performed.

The Norit RO 0.8 control sample was also analysed for surface area. The following surface area result, shown in Table 4.4.2, was obtained.

Table 4.3.2: Norit RO 0.8 – Surface area (m^2/g)

Sample	Result (m^2/g)
Norit RO 0.8	757.5

From the above table, it can be seen that the surface area obtained for activated carbon prepared from Witbank Seam 4 coal, does not meet the surface area requirement if compared to that of Norit RO 0.8.

Before experimentation commenced on the New Vaal raw material, it was decided only to assess the final activated carbon product at an activation time of 2 and 3 hours, since the results from the Witbank Seam 4 raw material showed that low surface areas were obtained at an activation time of 1 hour.

A summary of the surface area results obtained for activated carbon prepared from New Vaal coal, are shown in Table 4.4.3.

Table 4.3.3: New Vaal – Surface area (m^2/g)

New Vaal	2 hr	3 hr
600°C	43.97	36.93
700°C	172.77	181.73
800°C	190.10	158.07

The abovementioned results are graphically presented in Figure 4.4.3

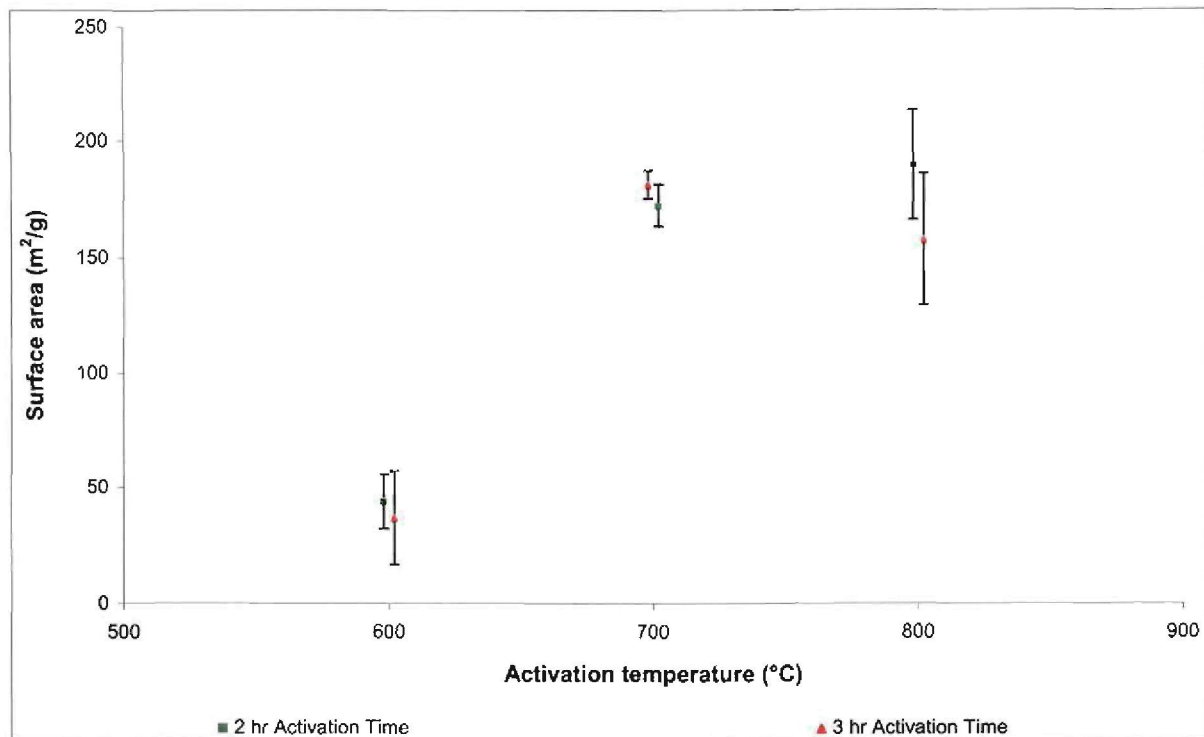


Figure 4.3.2: New Vaal – Surface area (m²/g)

The surface area measurements that were obtained for the activated carbon samples prepared from New Vaal coal, presented the same trend as in the case with the Witbank Seam 4 activated carbon samples. An increase in the overall surface area is observed with an increase in activation time and temperature. However, at high temperature and activation time, the New Vaal activated carbon surface area decreased which was not observed in the case of the Witbank Seam 4 activated carbon.

4.3.2 Activated carbon pH

The pH measurements were performed as described in Chapter 3. A detailed analysis procedure is described in **Appendix C**.

When the activated carbon and de-ionized water was mixed and stirred, it was observed that the maximum pH value was reached within 60 minutes. It was also seen that after reaching the maximum pH value for a specific sample, the pH gradually decreased and never stabilised. The measurement of pH continued until a period of 180 – 200 minutes elapsed. The pH measurements were taken at an interval of 5 minutes. The summary of the results (pH versus contact time) obtained for all the Witbank Seam 4 activated carbon samples, are shown in Figure 4.4.4

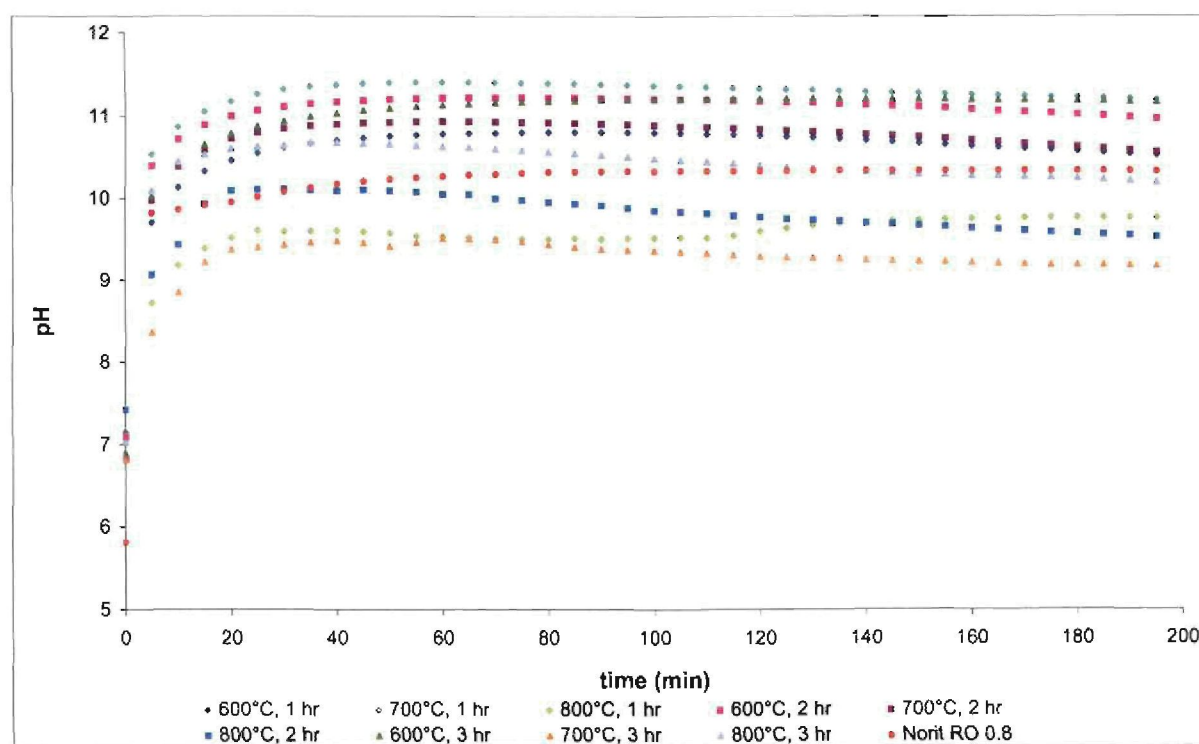


Figure 4.3.3: pH measurements – Witbank Seam 4 activated carbon

Individual graphical representation regarding pH results obtained for all the samples are included in **Appendix G**.

From the measured pH results, the following calculations were applied and graphs were obtained for the different samples.

- ΔpH versus time(s)*

$$\Delta\text{pH} = \text{pH}_{(i+1)} - \text{pH}_{(i)} \quad (4.3-1)$$

- $\Delta\text{pH} / dt$ versus time(s)*

$$\frac{d\text{pH}}{dt} = \frac{\text{pH}_{(i+1)} - \text{pH}_{(i)}}{\Delta t} \quad (4.3-2)$$

- pH versus time(s) to obtain adsorption constant
- pH versus time(s) to obtain desorption constant

* Sample calculations shown in **Appendix E**

The graphs for raw pH data are presented in **Appendix G**.

The adsorption and desorption rates were determined from the initial and final slope of the pH versus contact time graphs. The mathematical process followed to obtain these values is presented in **Appendix E**. Table 4.4.4 and Table 4.4.5 show the initial adsorption and final desorption rates for the activated carbon samples prepared from the Witbank Seam 4 coal.

Table 4.3.4: Witbank Seam 4 – Initial adsorption rates (pH/min)

Witbank Seam 4	1 hr	2 hr	3 hr
600°C	0.5139	0.6612	0.6259
700°C	0.7008	0.6335	0.3595
800°C	0.3473	0.4413	0.6139

Table 4.3.5: Witbank Seam 4 – Final desorption rates (pH/min)

Witbank Seam 4	1 hr	2 hr	3 hr
600°C	-0.0029	-0.0025	-0.0007
700°C	-0.0017	-0.0029	-0.0027
800°C	-0.0004	-0.0039	-0.0034

If these results are compared to the pH results obtained from the Norit RO 0.8 control samples, the aim is to have a high H^+ adsorption rate and thereafter a constant AC/water pH within the given time limit (Table 4.4.6). From the measurement of pH for the respective samples, it is noted that there are no fixed relationship between the pH results and the activation temperature and time.

Table 4.3.6: pH constants for Norit RO 0.8 (pH/min)

Property	Norit RO 0.8
Initial adsorption rate	0.802
Final desorption rate	-0.0003

From literature (Section 2.5.2), it has been mentioned that when H-type carbons is prepared (activated at temperatures above 750°C and exposed to steam or CO_2), these carbons are hydrophobic and take on a positive charge by absorbing hydrogen (H^+) ions when immersed in water. This can be clearly seen with the activated carbons that were prepared from the Witbank Seam 4 and New Vaal raw material. The activated carbon product turned the nature of the water basic during each pH analysis.

The results from Table 4.4.4 and Table 4.4.5 are presented in Figure 4.4.5 and Figure 4.4.6.

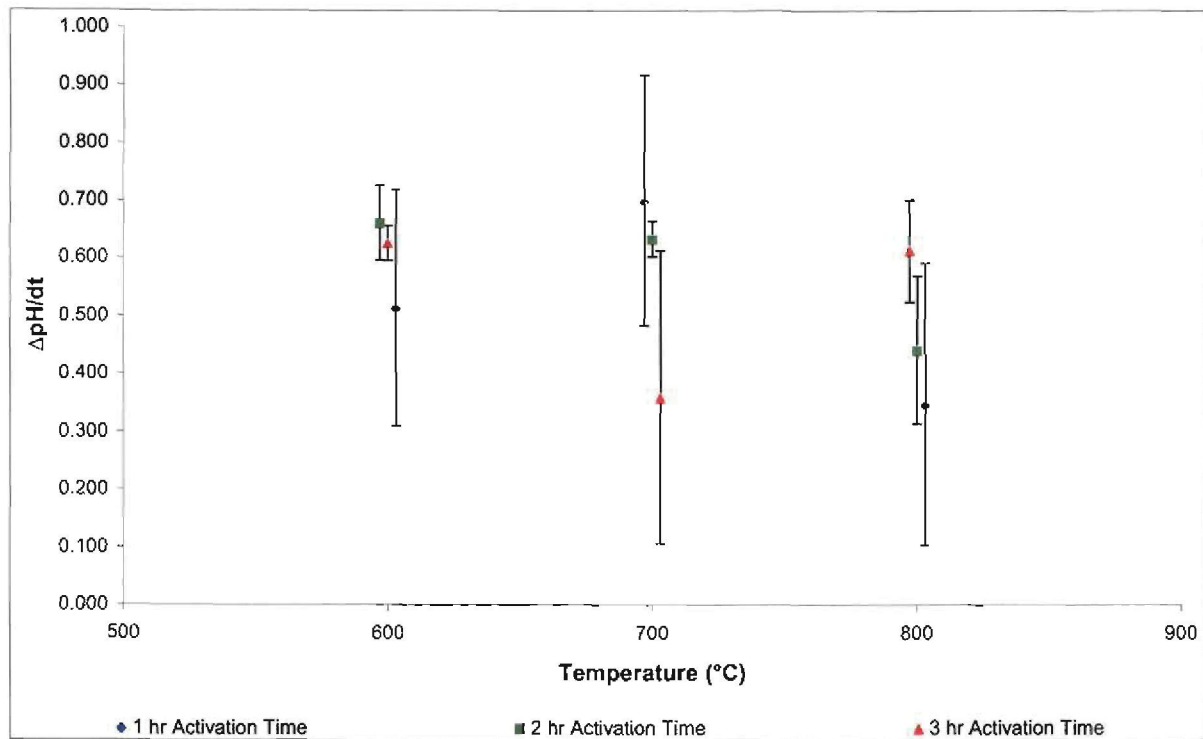


Figure 4.3.4: Witbank Seam 4 – Initial adsorption rates (pH/min)

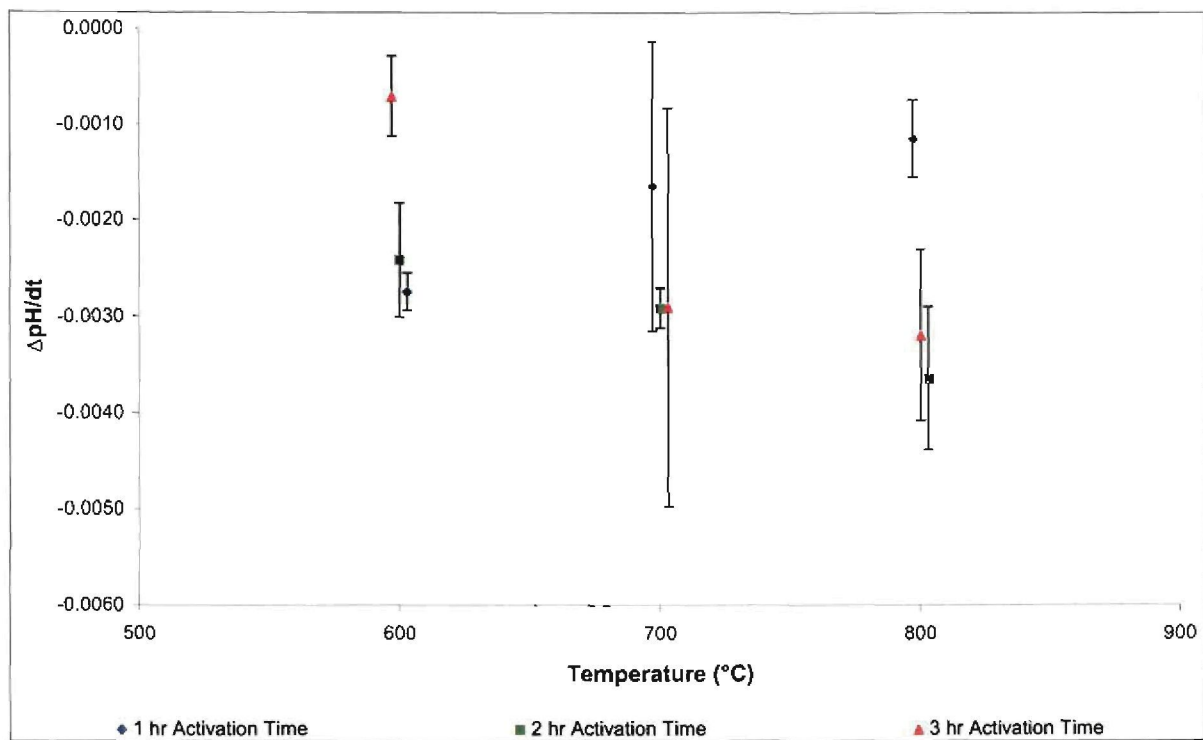


Figure 4.3.5: Witbank Seam 4 – Final desorption rates (pH/min)

When the New Vaal activated carbon samples were analysed, the same phenomenon was observed as in the case with Witbank Seam 4 activated carbon. The maximum pH value was reached within 60 minutes. After reaching the maximum pH value for the New Vaal activated carbon samples, the pH gradually decreased and never stabilized.

The summary of the results (pH versus contact time) obtained for all the New Vaal activated carbon samples, are shown in Figure 4.4.7.

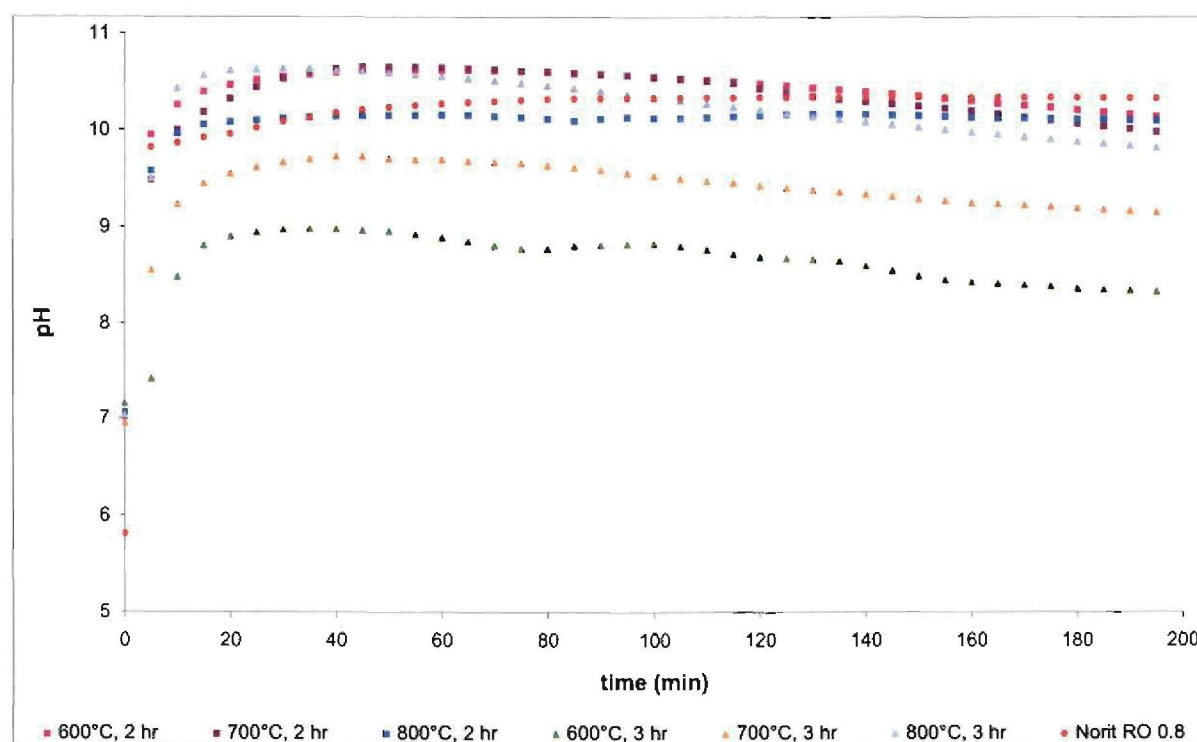


Figure 4.3.6: pH measurements – New Vaal activated carbon

From the measured pH results, the same calculations (Equations 4.4.1 and 4.4.2) were applied and graphs were obtained for the different New Vaal activated carbon samples.

Table 4.4.7 and Table 4.4.8 show the adsorption rates and desorption rates for the New Vaal activated carbons.

Table 4.3.7: New Vaal – Initial adsorption rates (pH/min)

New Vaal	2 hr	3 hr
600°C	0.5883	0.2660
700°C	0.4857	0.4034
800°C	0.6426	0.6207

Table 4.3.8: New Vaal – Final desorption rates (pH/min)

New Vaal	2 hr	3 hr
600°C	-0.0037	-0.0041
700°C	-0.0056	-0.0040
800°C	-0.0028	-0.0048

The abovementioned pH results were compared to the results of the Witbank Seam 4 activated carbon samples. It was observed that the pH results of the New Vaal activated carbon were similar with regards the initial adsorption rates and final desorption rates. The same conclusion (as with the Witbank Seam 4 activated carbons) can be made if compared to the Norit RO 0.8 control sample. These are also H-type samples since it tends to adsorb H^+ ions if immersed in water.

The results from Table 4.4.7 and Table 4.4.8 are graphically presented in Figure 4.4.8 and Figure 4.4.9.

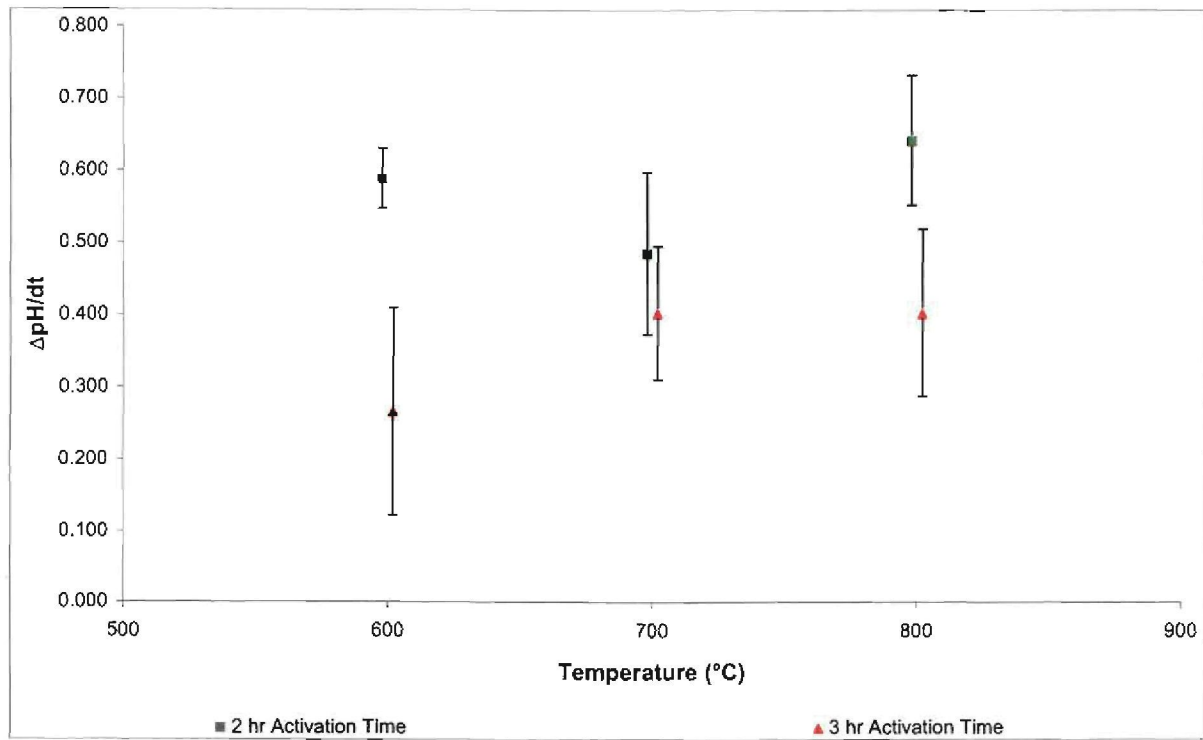


Figure 4.3.7: New Vaal – Initial adsorption rates (pH/min)

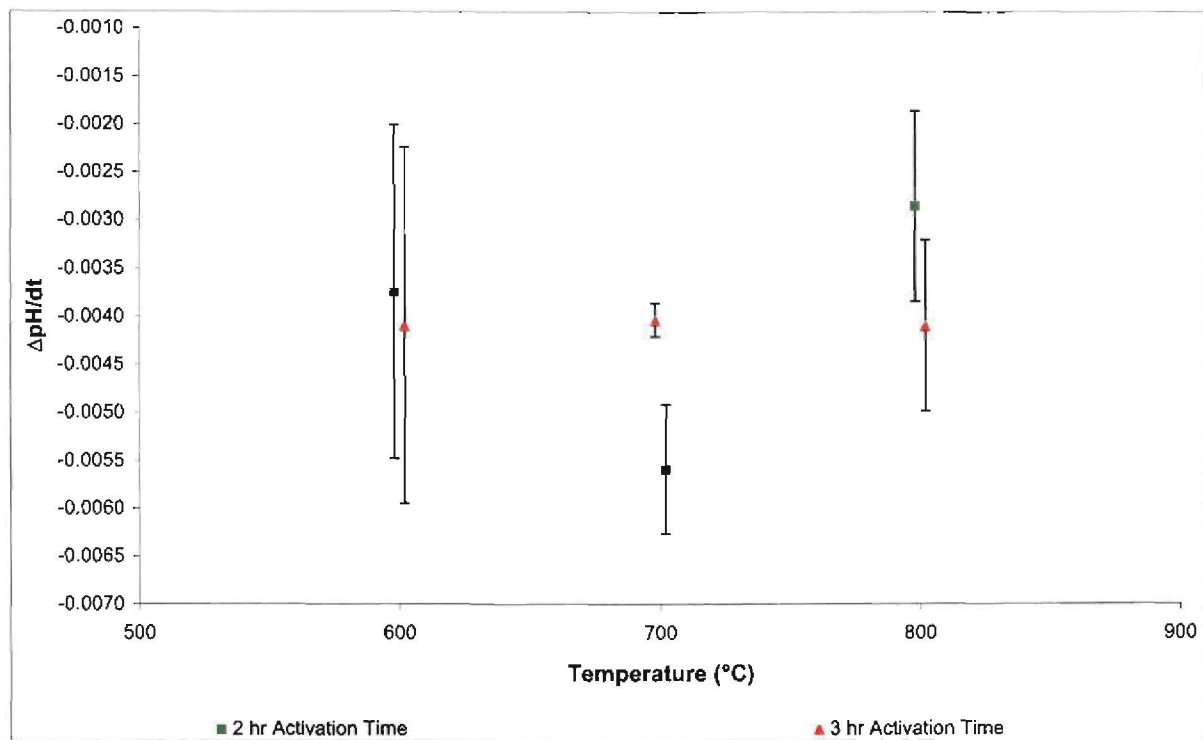


Figure 4.3.8: New Vaal – Final desorption rates (pH/min)

4.3.3 Phenol adsorption

The phenol adsorption analyses were performed on all the activated carbon samples prepared. A detailed analysis procedure is presented in **Appendix C**.

Table 4.4.9 shows the initial phenol adsorption results which were obtained for the different activated carbon samples prepared from Witbank Seam 4 raw material.

Table 4.3.9: Witbank Seam 4 – Initial phenol adsorption rates (ppm/min)

Witbank Seam 4	1 hr	2 hr	3 hr
600°C	4.7216	4.4124	5.5979
700°C	5.8557	5.8127	6.7663
800°C	8.9141	17.2474	21.9467

The above-mentioned results are graphically presented in Figure 4.4.10.

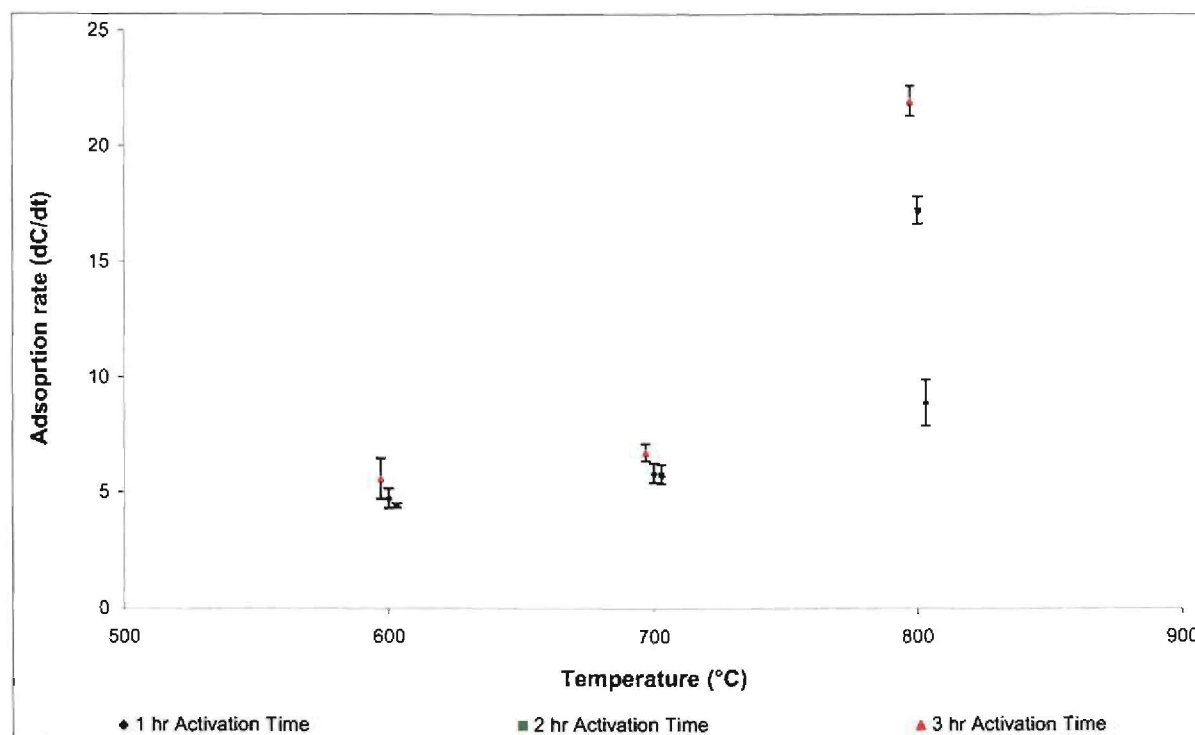


Figure 4.3.9: Witbank Seam 4 – Initial adsorption rates (ppm/min)

From Figure 4.4.10 it can be seen that the initial phenol adsorption rate increased with activation temperature and activation time. As mentioned previously there is a relationship between the BET surface area and phenol adsorption rate (BET surface area, p 51). A higher BET surface area results in a higher phenol adsorption rate.

When the phenol adsorption rates for Witbank Seam 4 activated carbon were compared to Norit RO 0.8 (Table 4.4.10), only the Witbank Seam 4 activated carbon sample, prepared at 800 °C and 3 hours showed a resemblance. Desorption for the respective samples were not evident since phenol loading on the total carbon surface occurred up to a certain point where all adsorption site are occupied.

Table 4.3.10: Phenol adsorption constants for Norit RO 0.8 (ppm/min)

Property	Norit RO 0.8
Adsorption rate	27.763

Phenol adsorption studies were also performed on the activated carbon prepared from New Vaal coal. Table 4.4.11 shows the phenol adsorption results which were obtained for the different activated carbon samples prepared from New Vaal coal.

Table 4.3.11: New Vaal – Initial phenol adsorption rates (ppm/min)

New Vaal	2 hr	3 hr
600°C	6.4828	5.4089
700°C	7.2818	9.4381
800°C	20.6495	20.4691

The same conclusions regarding the rate at which the NV activated carbon adsorbed phenol can be made as in the case of Witbank Seam 4 activated carbon. When the activation temperature and time is increased, the phenol adsorption rate increases up to the point where maximum activated carbon loading occurs.

The results from Table 4.4.11 are graphically presented in Figure 4.4.11.

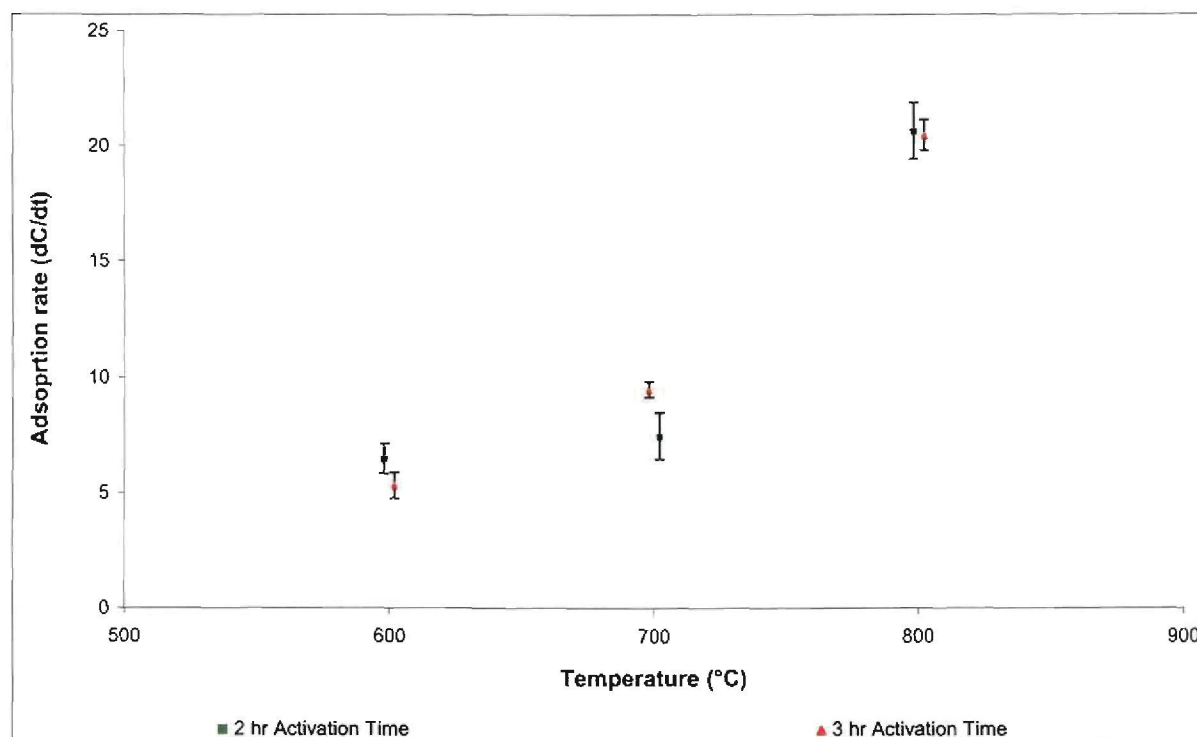


Figure 4.3.10: New Vaal – Initial adsorption rates (ppm/min)

After discussion of all relevant analyses performed and results obtained on the activated carbon manufactured, final remarks and conclusions will be presented and discussed in Chapter 5.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

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5.1 INTRODUCTION

This section concludes the project on the preparation of activated carbon from South African coal for PGM extraction. It addresses the main results from experimentation and analyses as well as recommendations regarding further studies.

5.2 CONCLUSIONS

5.2.1 BET surface area

The BET surface area results which were obtained for the activated carbon samples prepared from Witbank Seam 4 coal were presented in Table 4.4.1 (p. 50). Those samples prepared at the lower activation temperature (600°C) and activation time (1 hour) resulted in very low surface areas ($\pm 5\text{m}^2/\text{g}$). With regards to the samples prepared at high temperatures (800°C) and longer activations times (3 hours), higher surface areas were obtained ($\pm 340\text{m}^2/\text{g}$). Therefore it can be concluded that a higher activation time and temperature will result in activated carbon product with a higher surface area.

The BET surface area results which were obtained for the activated carbon samples prepared from New Vaal coal were presented in Table 4.4.3 (p. 51). For the New Vaal raw material samples, activated carbon was not prepared at an activation time of 1 hour (only 2 and 3 hours). The reason for this was that these activation condition delivered poor surface area results for the Witbank Seam 4 activated carbon, and posed not feasible for New Vaal coal. The highest surface area which could be obtained for the New Vaal raw material was the sample activated at 800°C and 2 hours which resulted in a BET surface area of $\pm 190\text{m}^2/\text{g}$. The conclusion regarding the activation time and temperature is thus not similar as in the case of the Witbank Seam 4 activated carbon where high activation temperatures and activation time delivers a sample with a high BET surface area.

When the above results are taken into consideration and compared with surface area results obtained for the Norit RO 0.8 control sample, it can be seen that only the activated carbon prepared from the Witbank Seam 4 raw material corresponds well. The highest surface area results for the individually prepared activated carbons and the Norit RO 0.8 control sample is shown in the table (Table 5.2.1) below.

Table 5.2.1: Surface area results

Raw material	Activation conditions	Surface area (m²/g)
Witbank Seam 4	800°C, 3 hr	338
New Vaal	800°C, 2 hr	190
Norit RO 0.8	Control sample	757

5.2.2 Activated carbon pH

The pH measurements of the Witbank Seam 4 activated carbon were performed as was described in Chapter 3. From the discussion in Chapter 4, it can be concluded that an H-type activated carbon was manufactured using Witbank Seam 4 raw material and it corresponds well with what literature predicts. The Witbank Seam 4 activated carbon sample results were compared with the Norit RO 0.8 control sample. It was seen that the initial adsorption rates compared well with the results obtained from the control sample but the solution pH decreased after the maximum solution pH was reached.

A similar phenomenon, as seen with the Witbank Seam 4 activated carbon pH measurements, presents in the case of the activated carbon from the New Vaal raw material. As mentioned in literature, an H-type activated carbon causes the solution pH to increase. This is because the H⁺ ions are adsorbed from the water and this process turns the nature of the water basic. The New Vaal activated carbon sample results were also compared with the Norit RO 0.8 control sample. It was also seen that the initial adsorption rates compared well with the results obtained from the control sample but the solution pH decreased after the maximum solution pH was reached.

5.2.3 Phenol adsorption

From the results which were obtained for the Witbank Seam 4 activated carbon phenol adsorptions studies, it can be clearly seen that there is a strong relationship between the extent to which the sample were activated (activation time and temperature) and phenol adsorption characteristics. With increasing activation time and temperature, an increase in the amount of phenol adsorbed is observed. Similar adsorption results were obtained when the Witbank Seam 4 activated carbon

adsorption results were compared to those adsorption results obtained from the Norit RO 0.8 activated carbon. The initial adsorption results for the activated carbon prepared at the condition (800°C, 3 hours) showed the closest resemblance to the adsorption characteristics of the Norit RO 0.8 activated carbon.

The relationship between BET surface area and phenol adsorption can also be seen with the activated carbons prepared from the New Vaal coal. With increasing activation time and temperature, an increase in the amount of phenol adsorbed is observed. When these results were compared to the adsorption results from the Norit RO 0.8 activated carbon, similar trends were observed. The phenol adsorption results for the activated carbon prepared at 800°C and 2 hours, had the highest adsorption rate if compared to the other New Vaal activated carbon samples. This also corresponds with the fact that this specific sample had on average a higher BET surface area with regards to other New Vaal activated carbon samples.

5.2.4 Conclusion on success of investigation

During this investigation, activated carbon was prepared from two different types of South African coal. An activation method was chosen and activated carbon samples were prepared at different temperature and activation time parameters. The activated carbons were analysed according BET surface area, pH and phenol adsorption. The results were compared to that of the Norit RO 0.8 control sample. Although the surface area measurements did not compare well with that of the control sample, pH and phenol adsorption studies of the prepared activated carbons proved to exhibit the same adsorption characteristics.

5.3 RECOMMENDATIONS

5.3.1 Regeneration of spent activated carbon

As discussed in Chapter 2, two methods of reactivating activated carbon were discussed: thermal (Section 2.2.6, p. 14) and chemical (Section 2.2.6, p. 15) reactivation. In principle, thermal reactivation is similar to the physical activation of the raw material. The activated carbon is heated in an inert environment (pyrolysed) and then activated with steam at elevated temperatures. Further studies would be to manufacture the activated carbon from South African coal, apply adsorption and explore ways in which the activated carbon could be successfully reactivated for repeated use in an adsorption process.

5.3.2 Ash content reduction

South African coal inherently contains a large amount of ash (Table 4.2.1, p. 48). From the proximate analysis it could be seen that this figure is as high as 35% (New Vaal coal). Methods have to be explored to determine if a reduction in ash of South African coals can deliver an activated carbon with enhanced adsorption properties if compared to activated carbon adsorption characteristics obtained from coal with no ash reduction.

5.3.3 Surface functional group determination

An FTIR analysis of the activated carbon sample can determine the functional groups on the surface of the carbon. This will explain the adsorption behavior of the activated carbon when pH measurements and phenol adsorption studies were performed. Surface functional groups can also be determined via the Boehm titration method. The Boehm titration method can also be used to determine the number of oxygenated surface groups. An activated carbon sample is placed in one of the following solutions: hydrochloric acid, sodium hydroxide, sodium carbonate, and sodium bicarbonate. The sample is stirred for an extended period of time and then filtered. Each filtrate is then titrated with HCl and NaOH depending on the original titrant. The number of acidic sites of various types can then be calculated using the assumption that NaOH neutralizes carboxylic, phenolic, and lactonic; Na_2CO_3

neutralizes carboxylic and lactonic groups; and NaHCO_3 neutralizes only carboxylic groups. The number of basic sites can be calculated from the amount of HCl that reacted with the carbon.

5.4 ACTIVATED CARBON STUDY IN PERSPECTIVE

From the objectives that were mentioned in Chapter 1, it can be concluded that the study was successful.

- A literature survey was conducted and different aspects of activated carbon were addressed.
- A range of precursors was selected (Witbank Seam 4, New Vaal, Grootegeeluk and Tshikondeni). The precursors were prepared as instructed in Chapter 3. The preparation included sieving (obtain correct particle size range), washing and drying. After the activation method was applied, it was seen that coal which contains a high vitrinite content (Grootegeeluk and Tshikondeni), was not a suitable precursor because of their coking properties. Further experimentation was only continued on the Witbank Seam 4 and New Vaal coal.
- A suitable activation method was selected. This method entailed devolatilising the sample at elevated temperatures to form a char, which was then activated at a specific temperature and time. This method does not make use of any treatment of the raw material prior to activation except obtaining the correct particle size and sample HCl washing.
- The manufactured activated carbon sample was characterized using BET surface area measurement (N_2 at 77K), determining the pH of the activated carbon and phenol adsorption (determine adsorption characteristics).
- Norit RO 0.8 was chosen as the control sample (commercially available) since it is already used in other PGM adsorption studies (Mbaya and Kasaini, 2006). The properties of the activated carbon prepared in this study and Norit RO 0.8 compared well with regards to phenol adsorption. A large surface area, as with Norit RO 0.8 ($760\text{m}^2/\text{g}$) could not be obtained. pH results for the different prepared samples also compared fair with Norit RO 0.8.

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APPENDIX A

RELEVANT LITERATURE

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A.1 INTRODUCTION

Appendix A presents all the literature that was not included in Chapter 2: Literature survey. It focuses on some background on the different coals that were used in this study and different characterisation methods for coal and activated carbon.

A.2 COALS USED IN THIS STUDY

A.2.1 Witbank Seam 4

Witbank Seam 4 coal was sourced from a colliery situated in the Witbank coalfield of the Main Karoo basin. The sedimentary sequence unconformable overlies rocks of the bushveld igneous complex. Reserve blocks are separated from each other by transgressive Karoo dolerite sills, causing displacement and burning of coal seams. The Witbank seams which are economically important for this colliery are the No 2, No 4 and No 5 seams. The Witbank Seam 2 seam exhibits well-defined zoning in the area up to six zones of differing qualities, the vertical quality variation forms the basis of the selective mining, practised when mining this seam. The seam thickness ranges from 5m to 7.5m, with an average thickness of 6.5m. The Witbank Seam 4 seam ranges from 2.4m to 3.3m in thickness. The Witbank Seam 5 seam has been extensively eroded and restricted in distribution with an average thickness of 1.4m.

A.2.2 New Vaal

New Vaal Colliery is located in the Maccauvlei area immediately south of Vereeniging on the bank of the Vaal River in the Free State Province. New Vaal Colliery was established during the 1980s to mine the remaining coal in the Maccauvlei area to supply coal to Eskom's (South Africa's electric power utility) Lethabo power station.

A.2.3 Grooteegeluk

Grooteegeluk mine is situated 25 km from Lephalale (Ellisras) in South Africa's Limpopo province. This open-pit mine employs 1 800 people and produces 18,6Mtpa of thermal and semi-soft coking coal using a conventional truck and shovel operation. This mine has an estimated mineable coal reserve of 811Mt and a total measured coal resource of 426 million tonnes. Semi-soft coking coal, thermal coal and metallurgical coal can be produced from these reserves.

Grooteegeluk has the world's largest beneficiation complex where 7 600 tonnes per hour of run-of-mine (ROM) coal is upgraded in six different plants. Some 15,3Mt of annual production is power station coal, transported directly to Eskom's Matimba power station on a 7 km conveyor belt in terms of the existing supply contract. An

additional 1,5Mtpa of metallurgical coal is sold domestically to the metals and other industries on short-term contracts. Grootegeluk produces 2,7Mtpa of semi-soft coking coal, the bulk of which is railed directly to Mittal SA under a long-term supply agreement. Approximately 1,1Mtpa of semi-soft coking coal and thermal coal is exported through Richards Bay Coal Terminal or sold domestically (Exxaro, 2007).

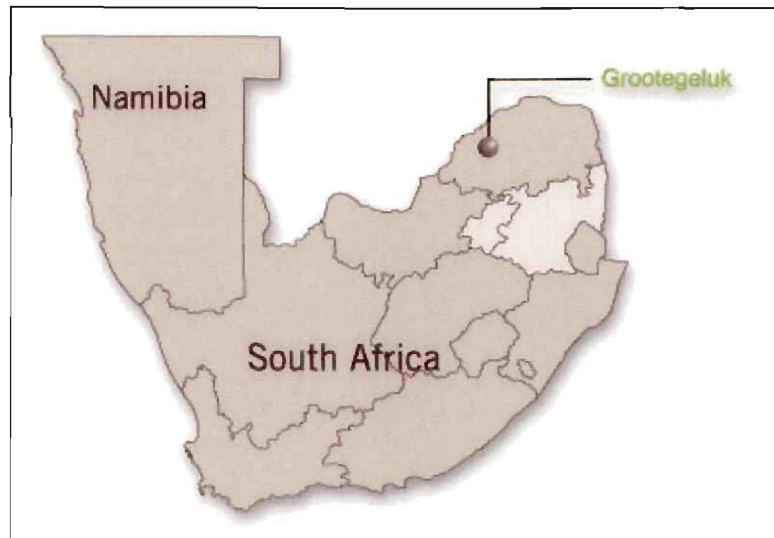


Figure A.2.1: Grootegeluk mine

A.2.4 Tshikondeni

Tshikondeni is located 140 km east of Musina in Limpopo province. This underground mine employs 770 people and currently produces 414ktpa of premium hard coking coal. Conventional board and pillar extraction methods are used and coal is beneficiated using cyclones, spirals and froth flotation. The beneficiated product is trucked to Musina and railed to Mittal South Africans steelworks at Vanderbijlpark under a long-term agreement at the cost of production plus a management fee of 3%. The mine has coal reserves of 6Mt and a resource of 36Mt (Exxaro, 2007).

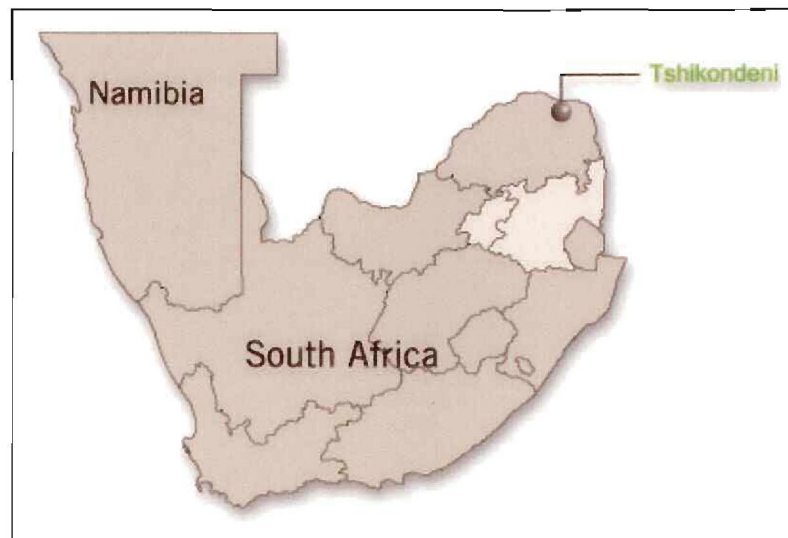


Figure A.2.2: Tshikondeni mine

A.3 COAL ANALYSES

The table below indicates the different methods that are used to characterise coal samples.

Table A.3.1: List of standard methods and other references

Analysis/ Requirement	Standard Method/ Reference	Title
Sampling and sample preparation	SABS 0135 Part I and Part II	Code of Practice for the sampling of coal and preparation of a sample for analysis
Calorific value	ISO 1928	Solid mineral fuels – Determination of gross calorific value by the bomb calorimetric method, and calculation of the calorific value
Gross calorific value	By calculation	Calorific value (MJ/kg) – (0.095 X % total sulphur)
Moisture in analysis sample	SABS 924	Moisture content of coal samples intended for general analysis (vacuum-oven method)
Ash	ISO 1171	Solid mineral fuels – Determination of ash
Volatile matter	ISO 562	Hard coal and coke – Determination of volatile matter content
Fixed carbon	By calculation	FC = 100% - % (moisture + ash + volatiles)
Total sulphur	ASTM D4239	Sulphur in the analysis sample of coal and coke using high-temperature combustion tube furnace combustion methods (Method C)
Carbon, hydrogen, nitrogen	ASTM D5373	Instrumental determination of carbon, hydrogen and nitrogen in laboratory samples of coal and coke
Oxygen	By difference	O = 100% - % (moisture + ash + C + H + N + total sulphur)
Ash fusion temperatures	ISO 540	Solid mineral fuels – Determination of fusibility of ash – High temperature method
Ash Oxides	ASTM D3682	Major and minor elements in coal and coke ash by atomic absorption
Calculation to different bases	ISO 1170	Coal and coke – Calculations of analysis to different bases
Preparation for petrographic analysis	ISO 7404-2	Methods for the petrographic analysis of bituminous coal and anthracite – Part 2: Preparation of coal samples
Maceral analysis	ISO 7404-3	Methods for the petrographic analysis of bituminous coal and anthracite – Part 3: Methods for determining maceral group composition

Vitrinite Random Reflectance	ISO 7404-5	Methods for the petrographic analysis of bituminous coal and anthracite – Part 5: Methods for determining microscopically the reflectance of vitrinite
Long-wave Fluorescence Relative Intensity of vitrinite	In-house method	Method described in: Kruszewska, KJ and du Cann, VM – Detection of the incipient oxidation of coal by petrographic techniques. <i>Fuel</i> , 75:6. 1996
Classification of coals	ENERGY/WP.1/R.50	International Classification of Seam Coals (only rank parameter used)

A.4 BET SURFACE AREA MEASUREMENT USING N₂

Adsorption of N₂ at -196°C using the BET equation has been used extensively for measuring surface areas of porous and nonporous solids. However N₂ adsorption on coals measures surface areas which are appreciably lower than those expected (Meyers, 1982:60). It has been suggested (Anderson et al., 1965; Walker and Geller, 1956) that at -196°C micropores in coals are not completely accessible to N₂ molecule due to an activated diffusion process and/or shrinkage of pores. Anderson et al. (1956) suggested that N₂ adsorption at -196°C provides a measure of the area contained in pores having diameters greater than about 5 Å (Meyers, 1982:61). Due to limitations of using N₂ adsorption at -196°C for measuring surface areas of coals, it was realized that in order to overcome diffusional effects, their surface areas would be measured by adsorption of gases at the highest possible temperature. Attempts were thus made to measure surface areas of coals from adsorption of neon at room temperature (Bond and Spencer, 1958), hydrocarbon gases at or near room temperature (Van der Sommen et al., 1966; Gregg and Pope, 1959; Juentgen and Langhoff, 1964), and Kr and Xe at -78°C and/or 0°C (Ahuja et al., 1961; Kini, 1963, 1964). Adsorption of CO₂ at -78°C has been used extensively for measuring surface area of coals (Anderson et al, 1965; Marsh and Wynne-Jones, 1964; Kipling et al., 1964; Thomas and Damberger, 1976; Debelak and Schrodtt, 1979) (Meyers, 1982:61). These surface areas greatly exceed those measured from the uptake of N₂ at -196°C for two reasons.

First, CO₂ molecule is a smaller molecule (minimum dimension - 3.3°Å) than a N₂ molecule (minimum dimension = 3.64°Å). Second, because of higher kinetic energy of gas molecules at -78°C relative to -196°C, diffusional rates are much higher for the smaller CO₂ molecule at the higher adsorption temperature (Meyers, 1982:61). In this context, Thomas and Damberger (1976) calculated that for a capillary pore of 0.001 cm length and 5 Å diameter, the time of passage for N₂ at -196°C is more than 105 times that for CO₂ at -78°C. From diffusional studies of N₂ and CO₂ from coals varying in rank from anthracite to highly volatile bituminous, Nandi and Walker (1964) found that N₂ has a higher activation energy than CO₂ (Meyers, 1982:61). They estimated that for an arbitrary "equilibration" time of 30 min for each sorption point,

about 85% of the pore volume of the anthracite would be filled with CO₂ at -78°C, whereas only 32% of the pore volume of bituminous coal with a carbon content (daf) of 83% would be filled with CO₂. However, at 25°C, essentially the entire pore volume of each coal studied would be filled with CO₂ (Meyers, 1982:61). In the light of his observation, Walker and Kini (1956) used adsorption of CO₂ at 25°C to measure BET surface areas of coals (Meyers, 1982:61). For the sake of comparison, they also determined surface areas from adsorption of N₂ (-196°C), Kr (-78°C), CO₂ (-78°C) and Xe (0°C). To use the BET equation for calculating surface area, it is essential to measure sorption isotherms up to a relative vapour pressure (rvp) of about 0.2 (Brunauer et al., 1938) (Meyers, 1982:61). Because the saturation vapour pressures of CO₂ at 25°C and of Kr and Xe at the selected adsorption temperatures are appreciably higher than the atmospheric pressure. Walker and Kini measured their isotherms in a high pressure adsorption system (Meyers, 1982:61). They concluded that adsorption of Xe at 0°C and of CO₂ at -78°C should usually measure essentially the total surface area of coals, whereas adsorption of CO₂ at 25°C should always measure essentially the total surface areas of coals (Meyers, 1982:62).

A.5 ANALYSES OF ACTIVATED CARBON PROPERTIES

Because of the diverse end uses to which a carbon may be applied, it is difficult to conduct specific tests related to any one application. The size and number of pores essentially determine an activated carbon's capacity in adsorbing a specific compound. Since pore size and total pore volume determinations are quite lengthy, they are impractical as a means of quality control during its manufacture. It is therefore necessary to relate the carbon's surface capabilities to a standard reference molecule.

The American Society for Testing and Materials (ASTM), Committee D-28 on activated carbon, has defined several common property tests (some relevant test mentioned in brackets). The following characteristics determine the quality and adsorption capacity of activated carbon:

A.5.1 Adsorption characteristics

Adsorptive capacity is paramount for both gas-phase and liquid-phase activated carbons. It determines the volume of gas or liquid that can be treated per unit weight of carbon (Snell *et al*, 1974:140). Adsorptive capacity determines both the direct cost for carbon treatment and the sizing of equipment (filters for powdered carbons and columns for the granular type) (Snell *et al*, 1974:141). The effectiveness of activated carbon is usually specified by the amount of a certain test chemicals it can adsorb per unit weight of activated carbon used.

Carbon Tetrachloride Activity (ASTM D3467-99)

The most widely used method is to measure the carbon's capacity to adsorb carbon tetrachloride (CTC) and express this as w/w%. This is determined by flowing CTC laden air through a sample of carbon of known weight, under standard conditions, until constant weight is achieved. The apparatus essentially consists of a mean to control the supply of air pressure, produce a specified concentration of CTC and control the air/CTC mixture flow rate through the sample. The weight of CTC adsorbed is referred to as the carbon's % CTC activity. However, this test does not necessarily provide an absolute or relative measure of the effectiveness of the carbon for other adsorbates or under different conditions. CTC activity is accepted as a means of specifying the degree of activation or quality of activated carbon. In some areas, this test is being substituted by a similar test which uses n-butane, known as the Butane Number test. To obtain the Butane Number (ASTM D5742-95(2000)) from carbon tetrachloride number, the CTC Activity is divided by 2.55.

Iodine Number (ASTM D4607-94(1999))

For activated carbon used in filtering water and liquids (standard measure for activated carbon in a liquid phase application), the test chemical used is usually iodine mixed with water and is specified as Iodine number (mg/gm) of carbon. It is the most fundamental parameter used to characterise activated carbon performance. It is a measure of activity level (higher number indicates higher degree of activation). It is a measure of the micropore content of the activated carbon (0 to 20Å, or up to 2 nm) by adsorption of iodine from solution, which is the equivalent to an activated carbon surface area of between 900m²/g and 1100m²/g.

A.5.2 Physical characteristics

Inorganic content

The inorganic content of activated carbon differs from carbon to carbon. This depends on the type of precursor (raw material), preparation method and activation reagent or component that was used (Lowry, 1981: 0). For some adsorption processes, activated carbons with the same inorganic content are required and in other adsorption processes, this inorganic content does not play any role. To ensure that the activated carbon does not contain any inorganic constituents, the following methods can be used, either: ⁽¹⁾Pre-deashing of precursor or ⁽²⁾Water/acid washing of precursor. Inorganic content of coal may include iron, sulphur, phosphates, calcium, chlorides, sodium, copper and silica. The removal of these inorganic constituents by washing with acid, alkali or water is never 100% effective.

pH Value (ASTM D3838-80(1999))

Coconut shell based activated carbon normally is specified for a pH of 9 – 11. The pH value of activated carbon is a measure of whether it is acidic or basic. The pH of activated carbon is defined as the pH of the water in which activated carbon is suspended in.

Particle Size Distribution (ASTM D2862-97)

Activated carbons are available in a wide range of granular and powdered grades. The particle size distribution of a grade is defined using test sieves. Different particle size of activated carbon offer different flow resistance to the air or water it is filtering. Depending upon the situation, system designers will specify different particle sizes of the activated carbon to be used. Typically, for filtering air or gases, the larger granule sizes are used; for filtering water or fluids, the smaller granule sizes are used. The finer the particle size of an activated carbon, the better access to the surface area and the faster the rate of adsorption kinetics. In vapour phase systems this needs to be considered against pressure drop, which will affect energy cost. Careful consideration of particle size distribution can provide significant operating benefits.

Moisture (ASTM D2867-99)

This is the amount of water physical bound on the activated carbon under normal condition. The practical limit for the level of moisture content allowed in the activated carbon varies within 3 to 6%.

Ash Content (ASTM D2866-94(1999))

Ash content is less important except where the carbon is used as a catalyst support since certain constituents of the ash may interfere or destroy the action of precious metal catalysts. Ash content also influences the ignition point of the carbon – this may be a major consideration where adsorption of certain solvents is concerned.

This is the inorganic, inert, amorphous and unusable part present in the activated carbon. This ash comes initially from the basic material. Lower the ash content, the better the activated carbon. The practical limit for the level of ash content allowed in the activated carbon varies within 2 to 5%. The ash content of the charcoal increases with increasing activation temperature since during this process more volatile matter is removed. Ash components are K_2O , Na_2O , CaO , MgO , Fe_2O_3 , Al_2O_3 , P_2O_5 , SO_3 and Cl^- . Ash can be removed by washing with concentrated HCl or HF , and then by large quantities of water (McKetta, 1976:133). Ash content reduces the overall activity of activated carbon. It also reduces the efficiency of reactivation. The metals (Fe_2O_3) can leach out of activated carbon resulting in decolouration. Acid/water soluble ash content is more significant than total ash content.

Surface Area

The internal surface area of a carbon is usually determined by the BET method (Braunauer, Emmett and Teller). This method utilises the low-pressure range of the adsorption isotherm of a molecule of known dimensions (usually nitrogen or in fewer cases carbon dioxide). This region of the isotherm is generally attributed to monolayer adsorption. Thus by assuming the species is adsorbed only one molecule deep on the carbon's surface, the surface area can be calculated.

Apparent Density (ASTM D2854-96(2000))

The density of activated carbon is of great importance to many users in estimating the weight required to fill a vessel. Apparent density is the weight of one litre of activated carbon (g/l). Density depends on the raw material being used and the degree of activation. The density does not affect the effectiveness of the activated carbon measured in adsorption per unit weight, but will have an effect on adsorption per unit volume. Higher density provides greater volume activity and normally indicates better quality activated carbon.

Hardness/abrasion number (ASTM D3802-79(1999))

The hardness and resistance to attrition of activated carbons is becoming more and more important. The loosely applied term “hardness” is somewhat difficult to measure on activated carbon.

Three forces can mechanically degrade an activated carbon: ⁽¹⁾impact, ⁽²⁾crushing and ⁽³⁾attrition. Of these three, the force of attrition, or abrasion, is the most common cause of degradation in actual end use. At the present time, there are two commonly used methods available to evaluate a carbon’s hardness:

- *Ball-pan hardness test* – A screened, weighed sample of carbon is placed in a special hardness pan with a number of stainless steel balls and subjected to combined rotating and tapping action for ½ hour. The particle size degradation is measured by determining the weight of the carbon retained on a sieve (with an opening closest to one half the opening of the sieve defining the minimum nominal particle size of the original sample). The ball-pan hardness method has been used widely in the past and has a broad history in the activated carbon industry for measuring the property loosely described as “hardness”. In this context, the test is useful in establishing a measurable characteristic, conceding that it does not actually measure in-service resistance to degradation, it can be used to establish comparability of differing batches of the same material. This test actually applies all of the three forces mentioned earlier, in a variable manner determined by the size, shape and density of the particles.
- *Stirring Bar Abrasion Test* – In this procedure, a sample of carbon is placed in a cylindrical vessel where an inverted T-shaped stirrer is turning rapidly at a

controlled rate. The percentage reduction in average particle size, resulting from the T-bar action, is recorded after 1 hour. This method measures attrition of the carbon, as long as the particle size is smaller than a 12 mesh. There is evidence showing that the results of this method are influenced by particle geometry.

Whichever of these tests is performed on carbon, it is generally accepted that granular coconut based carbons show the least rate of physical degradation.

This is possible due to two factors. First, granular coconut carbon is produced from pieces of raw coconut shell whereas; most other carbons are produced from reconstituted powders. In consequence, carbons other than coconut based types can only breakdown to a powder or dust.

Coconut carbon essentially chips and breaks into smaller pieces and thus degradation to powder, is a relatively lengthy process. Second, as outlined earlier, the coconut carbon structure is different to other types, producing a material of relatively high density and physical strength.

The harder the activated carbon, the less it will crumble into fine particles during handling and use. This value gives an idea of the abrasion-resistance of the activated carbon and is measured by means of a ball pan. Coconut shell based activated carbon has the unique property of the highest hardness (over 99%) which increases the durability and reduces loss in application, handling and activated carbon regeneration. It is a measure of the activated carbon's resistance to attrition. It is an important indicator of activated carbon to maintain its physical integrity and withstand frictional forces imposed by backwashing, etc. There are large differences in the hardness of activated carbons, depending on the raw material and activity level.

A.5.3 Interrelation of properties

There is a relationship between BET surface area and CTC adsorption and this is taken into account when activated carbon specifications are formulated. CTC activity, density and ash content are interrelated and provide a simple means of manufacturing control.

As quality of degree of activation increases, CTC activity and ash content increase and density decreases.

Furthermore, CTC activity being equal, coconut carbons show higher density and lower ash content than coal based carbons. Wood based carbon show much lower density than either coal or coconut based carbons but ash contents midway between coal and coconut carbons.

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For additional information, see Section 16.

4. FIRST AID MEASURES

- Skin: • Wash material off the skin with soap and water. Seek medical attention if irritation occurs.
 Eyes: • Flush with copious amounts of water. Seek medical attention if irritation occurs.
 Ingestion: • Give one or two glasses of water to drink. Seek medical attention if gastrointestinal symptoms develop.
 Inhalation: • Remove to fresh air. Seek medical attention if cough or respiratory symptoms develop.

5. FIRE FIGHTING MEASURES

- Flashpoint • Not Applicable.
 Non-flammable • 16CFR1500.44.
 Not Self Heating • UN Manual of Tests and Criteria, Test N.3.
 Flammability Limits in Air • LFL and UFL Not Applicable.

GENERAL HAZARD:

Activated carbon is difficult to ignite and tends to burn slowly (smolder) without producing smoke or flame. Toxic gases will form upon combustion.

FIRE FIGHTING INSTRUCTIONS:

If possible to do safely, move smoldering activated carbon to a non-hazardous area, preferably out of doors. Extinguish fire using water fog, fine water spray, carbon dioxide or foam. Avoid stirring up dust clouds.

FIRE FIGHTING EQUIPMENT:

Fire fighting personnel should wear full protective equipment, including self-contained breathing apparatus (SCBA) for all inside fires and large outdoor fires.

HAZARDOUS COMBUSTION PRODUCTS:

Combustion products may include smoke and oxides of carbon (for example, carbon monoxide). Materials allowed to smolder for long periods in enclosed spaces, may produce amounts of carbon monoxide which reach the lower explosive limit (carbon monoxide LEL = 12.5% in air). Under certain conditions, any airborne dust may be an explosion hazard. Used activated carbon may produce additional combustion products.

6. ACCIDENTAL RELEASE MEASURES

IF A SPILL OR LEAK OCCURS:

Clean up spills in a manner that does not disperse dust into the air. Handle in accordance with good industrial hygiene and safety practices. These practices include avoiding unnecessary exposure, and removal of material from eyes, skin, and clothing.

DISPOSAL METHOD:

Spent granular activated carbon may be recyclable. Dispose of virgin (unused) carbon (waste or spillage) in a facility permitted for non-hazardous wastes. Spent (used) carbon should be disposed of in accordance with applicable laws.

CONTAINER DISPOSAL:

Do not reuse empty bags. Dispose of in facility permitted for non-hazardous wastes.

7. HANDLING AND STORAGE

- Storage Temperature: • Ambient
 Storage Pressure: • Atmospheric
 Handling: • Follow good handling and housekeeping practices to minimize spills, generation of airborne dusts, and accumulation of dusts on exposed surfaces.
 • Use with adequate exhaust ventilation to draw dust away from workers' breathing zones.
 • Prevent or minimize exposures to dusts by using appropriate personal protection equipment.
 • Wash exposed skin areas thoroughly with soap and water after handling.
 Storage: • Store product in a closed dry container. Maintain good housekeeping. Store away from strong oxidizers such as ozone, liquid oxygen, chlorine, permanganate, etc.

Figure A.6.1: Norit RO 0.8 MSDS (p 2) (Norit-Americas, 2007)

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8. EXPOSURE CONTROLS / PERSONAL PROTECTION

- Engineering Controls:** • Use local exhaust ventilation to control emissions near the source.
- Eye Protection:** • Safety glasses with side shields are recommended for any type of handling. Where eye contact or dusty conditions may be likely, dust tight goggles are recommended. Have eye flushing equipment available.
- Skin Protection:** • Avoid skin contact with this product. Wear appropriate dust resistant clothing. Wash contaminated clothing and clean protective equipment before reuse. Wash skin thoroughly after handling.
- Respiratory Protection:** • Keep dust exposure to a minimum with engineering and administrative controls. Use appropriate NOISH/MSHA approved particulate respirators if necessary. Observe respirator use limitations specified by NOISH/MSHA or the manufacturer.

Airborne Exposure Guidelines:

Recommended Exposure Limits 8-hr TWA	Activated Carbon
Total Dust	7.5 mg/m ³ *
Respirable Fraction	2.5 mg/m ³ *

*OSHA and ACGIH have not established specific exposure limits for this material. These guidelines are based on a conservatively high concentration of silica quartz (2%). Actual airborne silica concentrations may be much lower. If so, the PEL or TLV would be higher. No ceiling or short-term exposure limits have been set by OSHA or ACGIH.

9. PHYSICAL AND CHEMICAL PROPERTIES

Boiling Point, C:	• NA	Freezing Point, C:	• NA
Bulk Density - Granular Grades	• 21-25 lbs/ft ³	% Volatiles	• NA
Bulk Density - Powder Grades	• 15-35 lbs/ft ³	Solubility in Water	• Insoluble
Vapor Pressure	• NA	Appearance and Odor	• Black granules or powder with no odor
Vapor Density	• NA		
Evaporation Rate	• NA		

NA - Not applicable

10. STABILITY AND REACTIVITY DATA

- Stability:** • This product is stable under the specified conditions of storage, shipment and use.
- Incompatibility:** • Contact with strong oxidizers such as ozone, liquid oxygen, chlorine, permanganate, etc. may result in rapid combustion. Avoid contact with strong acids.
- Hazardous Decomposition Products:** • Oxides of Carbon
- Hazardous Polymerization:** • Does not occur.

11. TOXICOLOGICAL INFORMATION

This material is non-toxic in its original state. Used activated carbon may exhibit characteristics of the adsorbed material.

12. ECOLOGICAL INFORMATION

This material, in its original state, is not harmful to the environment. Used activated carbon may exhibit characteristics of the adsorbed material.

13. DISPOSAL CONSIDERATIONS

Activated carbon, in its original state, is not a hazardous material or hazardous waste. Follow applicable governmental regulations for waste disposal.

Used activated carbon may become classified as a hazardous waste depending upon the application. Follow applicable regulations for disposal.

Recycling (reactivation) may be a viable alternative to disposal. Contact NORIT Americas Inc. for information.

Figure A.6.1: Norit RO 0.8 MSDS (p 3) (Norit-Americas, 2007)

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14. TRANSPORT INFORMATION

DOT (Department of Transportation)

- | | |
|-------------------------|---|
| Proper Shipping Name: | • Activated carbon (Not DOT Regulated). |
| Hazard Class: | • Not applicable. |
| UN/NA Number: | • Not applicable. |
| Packing Group: | • Not applicable. |
| Freight Classification: | • STCC Code - #2899643 NMFC #040560 |

15. REGULATORY INFORMATION

FEDERAL REGULATIONS:

- | | |
|---|--|
| OSHA Hazard Communication Standard, 29CFR1910.1200: | • See "Particulates not otherwise regulated," in Table Z-1, of 29CFR1910.1000, "Limits For Air Contaminates". |
| CERCLA/SUPERFUND, 40CFR117, 302: | • Notification of spills of this material is not required. |
| SARA/SUPERFUND: | • Section 302 - Extremely Hazardous Substances (40CFR355): This product is not listed as an extremely hazardous substance. |
| | • Section 313 - List Of Toxic Chemicals: This product is not listed. |
| Toxic Substances Control Act, 40CFR710: | • Activated carbon is on the inventory list. |
| Resource Conservation and Recovery Act: | • Activated carbon, in its original state, does not meet the criteria of hazardous waste. |

STATE REGULATIONS:

- | | |
|--|--------------|
| California Occupational Safety and Health: | • Not listed |
| Massachusetts Substance List: | • Not listed |
| New Jersey Right-to-Know: | • Not listed |
| Pennsylvania Right-to-Know: | • Not listed |

16. OTHER INFORMATION

Activated carbon can be safely stored in any normal storage area, but away from sources of direct heat.

WARNING: Activated carbon (especially when wet) can deplete oxygen from the air, and dangerously low levels of oxygen may result. When workers enter a vessel containing activated carbon, procedures for potentially low oxygen areas should be followed.

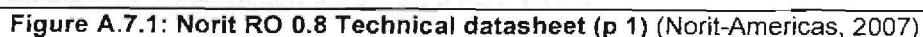
Activated carbons are not listed as potential carcinogens by any agency. Activated carbon may contain crystalline silica, which has been listed as a potential carcinogen of the lungs by the International Agency for Research on Cancer (IARC) and the National Toxicology Program (NTP). Much of the silica is inextricably bound within the particles of activated carbon, and does not present a substantial health hazard. Because NORIT Americas adheres to a very conservative position regarding all health and safety matters, we recommend and follow a practice of requiring respiratory protection whenever there is any evidence of airborne dust.

REVISION SUMMARY:

REV 02: Added new products NORIT A SPECIAL E 153, NORIT GCN 612G, NORIT RB 4W, NORIT RO 0.8H, NORIT ROW 0.8 SUPRA, NORIT RO 3515, NORIT RO 3520, NORIT RX 4 EXTRA and NORIT VETERINAIR to Section 1.
 REV 01: Added new products, NORIT DLC PLUS, NORIT DLC SUPER and NORIT DLC SUPER to Section 1.
 REV 00: New MSDS

The information herein is given in good faith but no warranty, expressed or implied, is made.

Figure A.6.1: Norit RO 0.8 MSDS (p 4) (Norit-Americas, 2007)



DATASHEET

No. 3340
Oct 2006**NORIT® RO 0.8***(continued)***Material Handling**

Wet activated carbon depletes oxygen from air and, therefore, dangerously low levels of oxygen may be encountered. Whenever workers enter a vessel containing activated carbon, the vessel's oxygen content should be determined and work procedures for potentially low oxygen areas should be followed. Appropriate protective equipment should be worn. Avoid inhalation of excessive carbon dust. No problems are known to be associated in handling this material. This product may contain silica. Please see the product Material Safety Data Sheet for details. Long-term inhalation of high dust concentrations can lead to respiratory impairment. Use forced ventilation or a dust mask when necessary for protection against airborne dust exposure (see Code of Federal Regulations - Title 29, Subpart Z, par. 1910.1000, Table Z-3).

Note: Any specification given was valid at time of issuance of the publication. However, we maintain a policy of continuous development and reserve the right to amend any specification without notice.

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Figure A.7.1: Norit RO 0.8 Technical datasheet (p 2) (Norit-Americas, 2007)

APPENDIX B

EXPERIMENTAL/ANALYSIS EQUIPMENT

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B.1 Orion 3-star Benchtop pH meter

A pH meter is an electronic instrument used to measure the pH (acidity or basicity) of a liquid. A typical pH meter consists of a probe (a glass electrode) connected to an electronic meter that measures and displays the pH reading. The Orion 3-star Benchtop pH meter also measures temperature and performs a pH-temperature correction.

B.2 Heidolph 1000MR magnetic stirrer

A magnetic stirrer consists of a:

- Stand or plate containing a rotating magnet or stationary electromagnets creating a rotating magnetic field. Often, the plate can also be heated.
- Small bar magnet (or stir bar), which is normally coated in a plastic such as PTFE (polytetrafluoroethylene)

During operation of a typical magnetic stirrer, the bar magnet (or flea) is placed in a vessel containing a liquid to be stirred. The vessel is placed on top of the magnetic plate, where the rapidly rotating magnetic field causes the bar magnet to rotate.

B.3 Varian Cary 50 Conc Ultraviolet visible spectrophotometer (UV-vis)

Ultra-violet visible spectroscopy (UV-vis) corresponds to electronic excitations between the energy levels that correspond to the molecular orbitals of a system. For this reason, the ultraviolet visible spectrophotometer can be applied to two distinctive wavelength range categories, namely the ultra-violet wavelength range (non-visible) and the colour spectrum range (visible).

The ultra-violet wavelength range varies from 200 to 400nm, while the visible colour spectrum wavelength varies from 400 to 800nm. Phenol in solution with its characteristic wavelength of 269nm, falls within the ultra-violet range, and not the visible colour spectrum range (Gunzler and Williams, 2001).

The main components of an UV-vis system include:

- Deuterium light source
- Quartz cell
- Lens
- Sensor
- Computer to display the measured results.

The liquid sample to be analyzed is poured into the quartz cell, also known as a cuvette. A cuvette is a small, transparent, square tube, made of plastic, glass or quartz, and is designed to hold liquid samples for spectroscopic measurements. The ultra-violet spectrophotometer has six available slots where the cuvettes can be inserted. The first slot is always occupied by a cuvette containing the reference liquid. The measurements of the remaining slots containing the samples will be compared to this reference liquid sample (Gunzler & Williams, 2001).

The parallel light source is reflected through the liquid sample containing the cuvette and the light rays moving through the cuvette are received by the light sensor. The measured data are processed and displayed in the computer screen. The UV-vis system measures the adsorbance of the sample at a certain wavelength (i.e. 269nm as in the case of phenol concentration).

This particular method is used in this study to obtain the percentage phenol adsorbed by the activated carbon sample. This is done by measuring the phenol concentration of the solution that was exposed to the activated carbon sample, and subtract this value from the initial known phenol concentration of the solution.

B.4 Micromeritics ASAP 2020 surface area analyzer

The BET surface area is determined by means of a Micromeritics ASAP 2020 Surface area analyzer. A sample contained in an evacuated sample tube is cooled to cryogenic temperature, after which it is exposed to an analysis gas at a series of precisely controlled pressures. With each incremental pressure increase, the number of gas molecules adsorbed on the surface increases. The pressure at which adsorption equilibrium occurs is measured and the universal gas law is applied to determine the quantity of gas adsorbed (Micromeritics, 2006). As adsorption proceeds, the thickness of the adsorbed film increases. Firstly any micropores in the surface are filled quickly, then the free surface becomes completely covered, and finally the large pores are filled. The process may continue to the point of bulk condensation of the analysis gas. A desorption process may begin in which pressure systematically is reduced resulting in liberation of the adsorbed molecules. As with the adsorption process, the changing quantity of gas on the solid surface is quantified. These two sets of data describe the adsorption and desorption isotherms. Analysis of the isotherms yields information about the surface characteristics of the material (Micromeritics, 2006).

B.5 Mettler-Toledo Halogen moisture analyzer

This abovementioned apparatus is used to quantify sample moisture content thermo gravimetrically. A circular halogen lamp heats up the sample while the mass loss is measure via a load cell. The heating process continues until the sample weight does not change over time. The percentage moisture can then be calculated from the difference in initial and final mass.

APPENDIX C

EXPERIMENTAL INFORMATION

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C.1 INTRODUCTION

Appendix C presents the detailed experimental procedures that were followed to prepare the raw material prior to activation, activate the raw material and use different methods to characterise the activated carbon product.

C.2 RAW MATERIAL PREPARATION

Obtaining particle size

The raw material was crushed to size by means of stage breaking. This is all dependent on the original nominal size of the raw material. It will pass firstly through a jaw crusher, obtaining a particle size of 5mm and smaller. After the initial breaking step, the produced material has to be passed through a cone crusher. This will crush the particles down to a size of 2mm and smaller. A short sieving process can follow this crushing step to remove any material that is too small/large in particle size. The size that was used for the material in the activated carbon process was governed by 860 microns – 1150 microns. The sample which was in the correct size interval was stored in seal-tight storage to prevent/minimize the amount of oxidation that will take place.

Each time a sample was taken for experimentation from the storage, care was taken to ensure that the sample was representative.

Raw material cleaning - HCl washing

After the initial preparation step, the raw material has to be cleaned. This is done by stirring the raw material with a low concentration HCl just to remove any inorganic material that was not removed by sieving. The low concentration (1M) HCl was prepared in the following matter.

Stirring

Place a bar magnet (or flea) in the 250ml beaker and place the beaker on the magnetic stirrer. Fill the beaker with ± 100 ml 1M HCl solution. Switch the magnetic stirrer on and adjust the speed control to between 40 and 50%. The maximum stirring speed of the Heidolph MR1000 magnetic stirrer is 2200rpm. 40 to 50% of this speed equates to a stirring speed of between 880 to 1100rpm. Add sample to the beaker with the use of a spatula. Be careful not to add too much sample, since this action will results in overloading the raw material/HCl solution and the stirrer will come to a halt. The mixture should be stirred for at least 1 hour. This period can be extended to 2 hours. After the time has expired, the sample needs to be filtered and washed subsequently with distilled water.

Sample drying

The sample is then placed in a 105 – 110°C preheated vacuum oven. Set the vacuum at approximately 40kPa. The sample will take ± 8 hours to dry. After this period, the sample is allowed to cool in a desiccator for ± 1 hour. The sample is now ready for use.

C.3 ACTUAL EXPERIMENT

Reactor loading

A sample of 20g raw material is put into the reactor. Place this material in the reactor and ensure that the base of the reactor is tightly fixed to the shell part of the reactor by means of the 5mm hex bolts. Also ensure that the sieve on the bottom of the reactor is not cracked or teared because this will result in sample loss. The reactor unit is then placed inside the vertical tube furnace. All piping is then connected to the tube furnace.

Place the reactor within the vertical APM tube in the furnace. The temperature couple can now be connected to the temperature indicator. The nitrogen/steam line can also be connected and fastened with the use of 2 x 10mm spanners or 2 x 30mm shifting spanners. The nitrogen bottles can now be opened. The flow to the tube furnace from above and below should be in the order of 80ml/min. Always check that there are at least 10000kPa of nitrogen left before running any experiment. This will ensure that there will not be any lack of flow of nitrogen which can result in the raw material burning to ash.

Devolatilisation of raw material

The temperature of the vertical tube furnace is then set to the desired temperature (600, 700 or 800°C) and the controller switched on. Within the first 10 - 15 minutes, a white plume will form at the top of the APM tube (Temperature = 420°C). This is the stage when devolatilisation starts to occur. This will continue for $\pm 20 - 25$ min (plume varying in colour: white, yellow and light brown). The sample is allowed to devolatilise for a total time of 60 minutes to ensure that devolatilisation is complete.

Steam generation

Whilst the furnace heats up to the desired temperature in the previous step, the steam generator needs to be started. This will be discussed next.

Fill the water reservoir as closely to the rim of the vessel as possible. Be sure not to waste any water on the load cell. The load cell indicator should read 68% and the weight indicator should read in the order of 2 000g (2.000kg). Start the pump by means of the pump on/off switch. On the controller software installed on the computer (Shinko Controller Software), change the desired jacket temperature to 160°C.

Also change the steam temperature to 160°C. The temperature of the heating coil around the nitrogen/steam line (preheat) and the heating jacket on the boiler will now increase to the desired controlled variable.

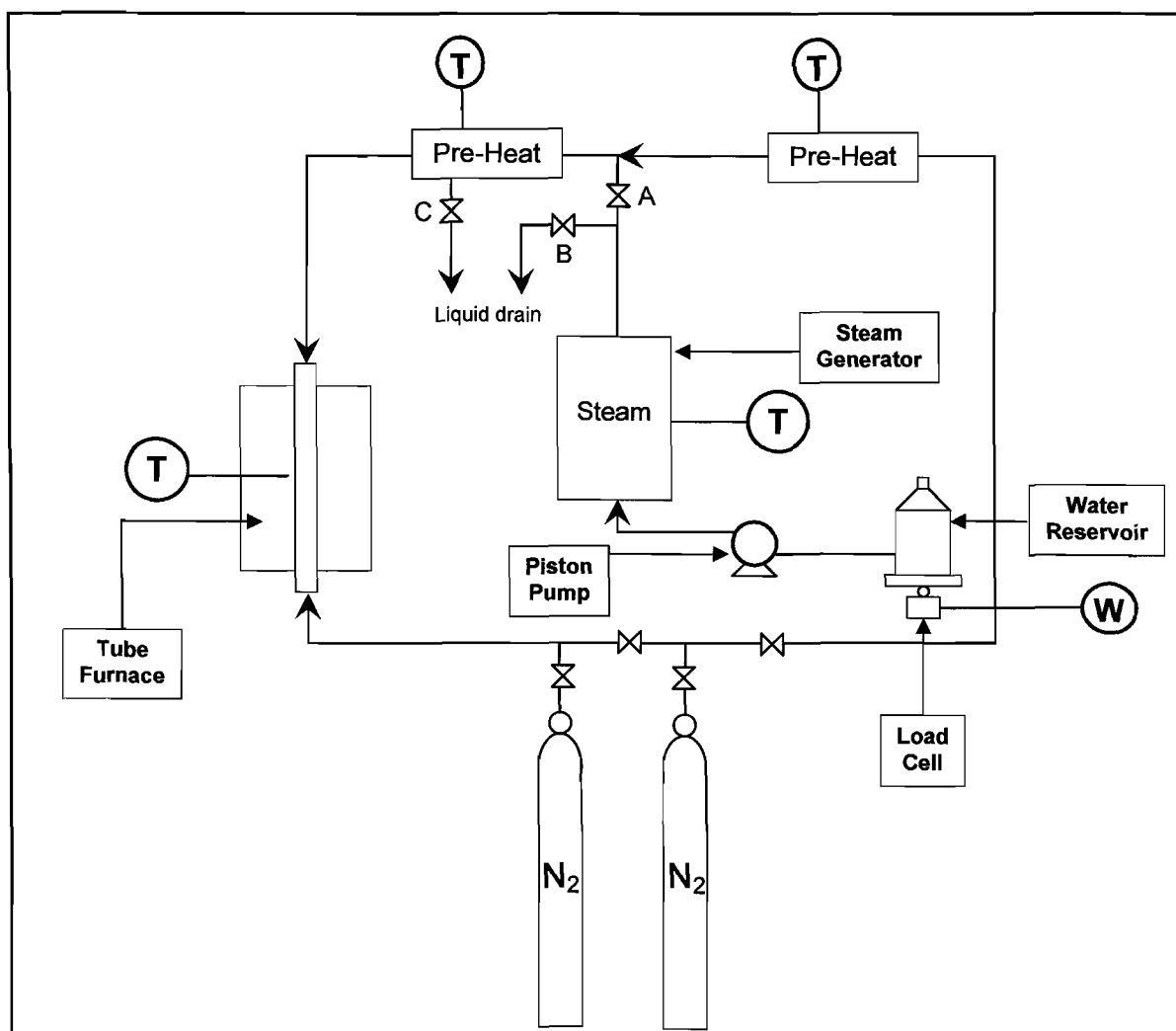


Figure C.3.1: Experimental equipment

Steam activation of sample

After the previous 60 minutes, valve A is then closed and B opened (Refer to Figure C.3.1). Time recording starts at this step (1, 2, 3 hours). It usually takes ± 4 minutes until steam is visible at the top of the reaction tube.

After the desired activation time has been reached, Valve A is opened and B closed. Time recording stops at this step. The furnace is allowed to cool down to room temperature. This process usually takes 6 - 8 hours, depending on the activation temperature.

After the furnace has reached room temperature, the sample is removed from the sample unit. The final sample weight is recorded and it is then stored for further

analysis. The various analyses which the sample will undergo is described in Section 3.6

Duration of experiment

The flow of nitrogen to the vertical tube furnace needs to be checked every half an hour. When the steam is introduced into the system, the pressure in the feed line increases and the flow rate has to be adjusted to ensure adequate flow of nitrogen to the reaction tube. The duration of the experiment is determined by the amount of time that the sample needs to be activated. This time relates to the period that the sample needs to be in a superheated steam atmosphere. After the desired time has expired, close the steam valve and open the purge valve. The steam generator and piston pump can now be switched off. The on/off knob on the furnace temperature controller can also be turned in an anti-clockwise direction. The furnace should now be allowed to cool. This process can take up to 8 hours or as soon as the furnace temperature reads 45°C. After this period the nitrogen flow can be closed and the reactor removed.

Removal of sample

The sample can now be removed from the reactor. It is important to ensure that no sample loss takes place during this step. This will influence the final weight and % mass loss of the original raw material. The activated carbon product needs to be stored in an air-tight container.

C.4 ACTIVATED CARBON ANALYSES

Phenol adsorption studies

Physical adsorption

It was the task to first find the time limit at which the activated carbon particles reached a saturation stage. For this purpose, an AC/phenol solution ratio was selected and tested (Maritz, 2006).

A 100ml, 500ppm phenol solution was selected and stirred with 1 g activated carbon (Witbank Seam 4 activated carbon 600°C, 1 hr). The concentration of the phenol solution (after 1 hour of stirring) was measured and it was found that only 2.5% of the phenol was adsorbed (12ppm of the phenol solution). The phenol concentration was reduced to 100ppm. After 1 hour of stirring, it was found that only 12ppm of the phenol solution was adsorbed (adsorption of 12%). It was decided to use a 100ppm phenol solution for all of the phenol adsorption studies.

For the purpose of the adsorption studies, phenol concentrations for specific time intervals were recorded (C_0 , 2, 5, 10, 30, 60 minutes was selected).

From the above-mentioned step, it was seen that only the activated carbon samples prepared at higher temperatures, adsorbed a maximum amount of phenol at 30 minutes. 60 minutes was then selected as the cut-off time and all samples were treated accordingly.

Ultraviolet spectrometer analysis

When the phenol solution samples are withdrawn, the concentration of the samples is measured by means of an ultraviolet-spectrophotometer (UV-vis). A description of the UV-vis used for this application, is described in **Appendix B**.

The liquid samples are each poured into a small container called a cuvette. A cuvette is a small, transparent, square tube, made of plastic, glass or quartz, and is designed to hold liquid samples for spectroscopic measurements. For the purpose of these measurements, disposable plastic cuvettes were used. The ultra-violet spectrometer has six available slots where the cuvettes can be inserted. The first slot is always

occupied by a cuvette containing the reference liquid, in this case, distilled water. The measurements of the remaining slots containing samples will be compared to this reference liquid sample. The process in analyzing the phenol samples is summarized as follows:

- The cuvettes, used for the reference liquid, as well as the measured liquid solution, is rinsed thoroughly with distilled water and dried.
- Distilled water is poured into the first cuvette, while the phenol solution to be measured is poured into another.
- These two cuvettes are inserted into the machine slots marked “1” and “2” respectively.
- The wave scan option is selected (scan for a wavelength interval of 180 to 320nm) and adsorbance measurements are taken.
- The measured data is saved and displays the measured absorbance next to its corresponding wavelength, and the absorbance for a wavelength of 269 nm is obtained from the table.

pH Measurement

An activated carbon sample of known mass is stirred with water while the pH of the solution/slurry is measured continuously. The benchtop pH meter should be calibrated first before any measurements can be taken. The following steps should be followed to obtain the necessary pH results:

- Measure off 2g of the prepared activated carbon sample.
- Add 40ml distilled water to a small beaker (100ml), (AC/H₂O ratio of 0.05).
- Place the 100ml beaker with bar magnet on the magnetic stirrer.
- The magnetic stirrer can be switched on; a speed of 1100rpm has proven to be sufficient.
- Measure the pH of the distilled water by using the pH electrode. This creates this zero point on which all measurements will be based.
- As soon as the pH meter has shown a stable value for the distilled water, the activated carbon sample can be added to the solution.

- The pH of the solution is measured once every 5 minutes and stored in the temporary memory of the benchtop meter. Continuously measure the pH of the slurry/solution for 180 – 200 minutes.
- Remove the pH electrode from the slurry after 180 – 200 minutes has elapsed
- Dispose off the activated carbon/distilled water slurry.
- The pH values are then stored in a spreadsheet file from which all calculations are performed.

APPENDIX D

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D.1 WITBANK SEAM 4 SAMPLES

D.1.1 pH measurements

The acronym used (WS4821) in the tables below refers to the first sample of the activated carbons from Witbank Seam 4 coal, prepared at 800°C and 2 hours.

Table D.1.1: Witbank Seam 4 – pH measurements (600°C, 700°C, 800°C, 1 hr)

Time (min)	WS4611 611	WS4612 612	WS4613 613	WS4711 711	WS4712 712	WS4713 713	WS4811 811	WS4812 812	WS4813 813
0	7.502	6.980	6.955	6.827	6.967	7.337	6.536	7.553	7.232
5	8.538	10.263	10.344	11.237	11.192	9.214	6.679	8.721	10.797
10	9.158	10.562	10.710	11.512	11.467	9.654	6.984	9.395	11.193
15	9.468	10.700	10.850	11.644	11.598	9.949	7.461	9.398	11.337
20	9.674	10.796	10.940	11.722	11.686	10.162	7.856	9.294	11.426
25	9.813	10.868	11.003	11.772	11.763	10.289	8.229	9.132	11.490
30	9.914	10.930	11.041	11.809	11.813	10.383	8.262	8.978	11.574
35	10.004	10.968	11.062	11.836	11.841	10.425	8.262	8.999	11.568
40	10.067	10.991	11.079	11.856	11.862	10.444	8.197	9.061	11.598
45	10.128	11.005	11.093	11.869	11.878	10.464	8.102	9.090	11.621
50	10.184	11.013	11.095	11.880	11.890	10.468	7.996	9.116	11.640
55	10.218	11.013	11.102	11.887	11.893	10.471	7.865	9.120	11.669
60	10.250	11.010	11.107	11.892	11.898	10.463	7.807	9.161	11.663
65	10.281	11.005	11.107	11.897	11.898	10.457	7.754	9.164	11.671
70	10.304	10.999	11.105	11.901	11.900	10.444	7.720	9.150	11.678
75	10.319	10.991	11.114	11.902	11.898	10.426	7.727	9.132	11.681
80	10.335	10.982	11.115	11.906	11.897	10.409	7.726	9.107	11.696
85	10.344	10.973	11.115	11.906	11.893	10.393	7.775	9.092	11.700
90	10.351	10.965	11.119	11.908	11.888	10.372	7.749	9.084	11.703
95	10.356	10.951	11.119	11.909	11.885	10.353	7.771	9.084	11.705
100	10.358	10.939	11.119	11.909	11.881	10.334	7.780	9.093	11.709
105	10.358	10.925	11.119	11.911	11.874	10.319	7.794	9.090	11.711
110	10.351	10.911	11.114	11.913	11.871	10.298	7.798	9.067	11.712
115	10.344	10.896	11.112	11.911	11.865	10.275	7.824	9.139	11.712
120	10.335	10.882	11.108	11.911	11.860	10.251	7.895	9.221	11.714
125	10.321	10.865	11.103	11.911	11.857	10.228	7.948	9.288	11.714
130	10.305	10.847	11.095	11.911	11.851	10.206	7.989	9.341	11.712
135	10.290	10.830	11.082	11.911	11.846	10.183	8.020	9.384	11.712
140	10.272	10.813	11.067	11.909	11.843	10.154	8.027	9.421	11.705
145	10.257	10.792	11.053	11.911	11.838	10.131	8.056	9.453	11.702
150	10.236	10.775	11.036	11.909	11.834	10.114	8.050	9.483	11.700
155	10.217	10.754	11.018	11.908	11.831	10.089	8.054	9.512	11.696
160	10.201	10.735	11.004	11.906	11.825	10.072	8.061	9.533	11.692
165	10.187	10.721	10.987	11.906	11.822	10.051	8.062	9.550	11.691
170	10.175	10.702	10.975	11.906	11.817	10.032	8.068	9.566	11.687
175	10.159	10.683	10.961	11.902	11.813	10.013	8.073	9.582	11.685
180	10.144	10.668	10.946	11.901	11.810	9.990	8.080	9.575	11.681
185	10.126	10.643	10.930	11.901	11.808	9.975	8.083	9.570	11.680
190	10.114	10.626	10.916	11.899	11.803	9.954	8.095	9.561	11.674
195	10.102	10.604	10.899	11.897	11.796	9.926	8.090	9.547	11.672

Table D.1.2: Witbank Seam 4 – pH measurements (600°C, 700°C, 800°C, 2 hr)

Time (min)	WS4621 621	WS4622 622	WS4623 623	WS4721 721	WS4722 722	WS4723 723	WS4821 821	WS4822 822	WS4823 823
0	6.943	7.204	7.142	6.769	6.807	6.873	7.826	7.766	6.671
5	10.308	10.280	10.619	10.128	9.739	10.085	10.039	7.361	9.825
10	10.672	10.642	10.872	10.521	10.240	10.428	10.747	7.389	10.200
15	10.898	10.820	10.981	10.710	10.483	10.612	10.844	8.642	10.332
20	11.039	10.942	11.049	10.828	10.668	10.711	10.880	9.052	10.376
25	11.136	11.013	11.090	10.908	10.767	10.762	10.894	9.049	10.404
30	11.191	11.060	11.119	10.957	10.820	10.798	10.897	9.022	10.446
35	11.223	11.105	11.144	10.993	10.850	10.829	10.897	8.971	10.451
40	11.245	11.140	11.160	11.025	10.867	10.841	10.888	8.971	10.437
45	11.259	11.165	11.174	11.046	10.876	10.852	10.880	8.999	10.446
50	11.269	11.184	11.186	11.065	10.881	10.855	10.871	9.014	10.413
55	11.271	11.196	11.196	11.080	10.884	10.857	10.853	9.010	10.390
60	11.271	11.207	11.202	11.094	10.881	10.855	10.841	8.963	10.360
65	11.271	11.219	11.207	11.101	10.870	10.855	10.827	8.978	10.346
70	11.268	11.222	11.210	11.107	10.862	10.850	10.809	8.889	10.316
75	11.262	11.226	11.210	11.107	10.851	10.845	10.791	8.880	10.281
80	11.255	11.227	11.210	11.107	10.837	10.838	10.768	8.853	10.258
85	11.245	11.231	11.210	11.107	10.829	10.829	10.752	8.841	10.219
90	11.236	11.233	11.207	11.103	10.820	10.816	10.729	8.816	10.207
95	11.224	11.234	11.203	11.101	10.806	10.805	10.712	8.792	10.166
100	11.212	11.243	11.200	11.098	10.799	10.788	10.691	8.748	10.138
105	11.196	11.245	11.196	11.091	10.790	10.773	10.669	8.739	10.113
110	11.184	11.248	11.193	11.082	10.781	10.762	10.648	8.736	10.087
115	11.169	11.248	11.186	11.072	10.771	10.750	10.631	8.707	10.059
120	11.151	11.247	11.179	11.063	10.755	10.735	10.608	8.706	10.032
125	11.134	11.243	11.170	11.047	10.741	10.717	10.581	8.692	10.006
130	11.117	11.240	11.163	11.033	10.724	10.705	10.562	8.695	9.981
135	11.094	11.236	11.154	11.018	10.703	10.692	10.541	8.686	9.962
140	11.073	11.231	11.144	11.000	10.687	10.680	10.519	8.672	9.944
145	11.051	11.227	11.133	10.988	10.668	10.662	10.498	8.674	9.923
150	11.028	11.219	11.125	10.976	10.649	10.643	10.475	8.663	9.911
155	11.007	11.210	11.112	10.960	10.628	10.626	10.458	8.655	9.884
160	10.985	11.193	11.098	10.946	10.605	10.606	10.433	8.640	9.870
165	10.960	11.179	11.083	10.931	10.581	10.585	10.414	8.640	9.833
170	10.947	11.163	11.069	10.913	10.556	10.562	10.392	8.633	9.818
175	10.929	11.147	11.055	10.898	10.533	10.540	10.373	8.625	9.793
180	10.910	11.130	11.042	10.880	10.509	10.518	10.354	8.625	9.770
185	10.889	11.118	11.027	10.859	10.486	10.494	10.336	8.619	9.749
190	10.869	11.104	11.009	10.840	10.464	10.471	10.318	8.623	9.731
195	10.842	11.090	10.995	10.821	10.436	10.447	10.299	8.611	9.707

Table D.1.3: Witbank Seam 4 – pH measurements (600°C, 700°C, 800°C, 3 hr)

Time (min)	WS4631 631	WS4632 632	WS4633 633	WS4731 731	WS4732 732	WS4733 733	WS4831 831	WS4832 832	WS4833 833
0	6.831	6.876	6.993	6.724	7.000	6.730	7.000	7.041	7.054
5	9.906	10.495	9.687	10.419	7.881	6.813	9.402	10.314	10.587
10	10.389	10.761	10.185	10.743	8.453	7.399	9.751	10.736	10.880
15	10.642	10.938	10.413	10.884	8.591	8.216	9.880	10.821	10.940
20	10.782	11.046	10.558	10.986	8.559	8.602	10.022	10.877	10.933
25	10.875	11.122	10.662	11.049	8.403	8.791	10.107	10.902	10.898
30	10.929	11.176	10.742	11.091	8.310	8.924	10.162	10.907	10.880
35	10.983	11.219	10.806	11.126	8.263	9.021	10.274	10.902	10.889
40	11.015	11.257	10.855	11.144	8.206	9.118	10.289	10.893	10.884
45	11.045	11.288	10.902	11.154	8.199	9.049	10.275	10.881	10.862
50	11.059	11.310	10.937	11.167	8.206	8.896	10.307	10.865	10.831
55	11.076	11.331	10.963	11.168	8.377	8.870	10.298	10.849	10.804
60	11.085	11.348	10.993	11.174	8.460	8.927	10.295	10.833	10.778
65	11.090	11.365	11.014	11.177	8.502	8.870	10.319	10.811	10.748
70	11.104	11.377	11.034	11.179	8.531	8.810	10.302	10.793	10.730
75	11.101	11.388	11.055	11.179	8.552	8.720	10.296	10.760	10.712
80	11.102	11.398	11.070	11.177	8.560	8.600	10.288	10.729	10.689
85	11.102	11.405	11.083	11.175	8.562	8.498	10.298	10.694	10.673
90	11.102	11.410	11.095	11.172	8.547	8.448	10.293	10.655	10.650
95	11.099	11.415	11.104	11.170	8.547	8.415	10.281	10.617	10.635
100	11.099	11.419	11.114	11.168	8.540	8.392	10.274	10.592	10.599
105	11.095	11.422	11.123	11.165	8.529	8.379	10.249	10.573	10.590
110	11.090	11.425	11.133	11.163	8.512	8.346	10.237	10.562	10.569
115	11.085	11.429	11.142	11.158	8.495	8.313	10.219	10.534	10.553
120	11.076	11.429	11.149	11.156	8.488	8.273	10.195	10.503	10.534
125	11.069	11.431	11.158	11.153	8.481	8.254	10.179	10.484	10.513
130	11.064	11.431	11.165	11.146	8.460	8.242	10.170	10.459	10.495
135	11.055	11.431	11.174	11.142	8.458	8.233	10.162	10.429	10.476
140	11.048	11.431	11.176	11.117	8.444	8.230	10.148	10.410	10.454
145	11.039	11.431	11.183	11.105	8.425	8.221	10.137	10.375	10.446
150	11.031	11.431	11.188	11.098	8.413	8.225	10.139	10.351	10.431
155	11.025	11.429	11.191	11.088	8.403	8.226	10.130	10.333	10.431
160	11.015	11.431	11.191	11.079	8.394	8.223	10.121	10.307	10.428
165	11.004	11.425	11.197	11.068	8.379	8.216	10.121	10.300	10.428
170	10.996	11.424	11.198	11.058	8.368	8.209	10.114	10.281	10.424
175	10.985	11.422	11.202	11.047	8.372	8.187	10.118	10.272	10.419
180	10.975	11.419	11.204	11.035	8.374	8.192	10.100	10.253	10.412
185	10.964	11.415	11.202	11.024	8.377	8.187	10.104	10.212	10.396
190	10.950	11.413	11.200	11.012	8.379	8.186	10.092	10.196	10.380
195	10.938	11.408	11.200	10.998	8.379	8.187	10.078	10.167	10.361

D.1.2 Phenol adsorption measurements

Table D.1.4: Witbank Seam 4 – UV-vis spectrophotometer results

		Measured adsorbance						
Time (min)		0	2	5	10	30	50	60
WS4611	611	0.000	1.772	1.754	1.733	1.729	1.729	1.729
WS4612	612	0.000	1.806	1.768	1.765	1.760	1.760	1.760
WS4613	613	0.000	1.765	1.743	1.727	1.724	1.724	1.724
WS4711	712	0.000	1.715	1.692	1.682	1.662	1.656	1.653
WS4712	712	0.000	1.734	1.740	1.721	1.704	1.690	1.684
WS4713	713	0.000	1.762	1.710	1.708	1.676	1.666	1.661
WS4811	811	0.000	1.649	1.500	1.371	1.045	0.917	0.913
WS4812	812	0.000	1.646	1.536	1.391	1.108	1.005	0.990
WS4813	813	0.000	1.560	1.377	1.216	0.898	0.857	0.850
WS4621	621	0.000	1.799	1.790	1.787	1.786	1.786	1.786
WS4622	622	0.000	1.787	1.780	1.761	1.756	1.756	1.756
WS4623	623	0.000	1.793	1.759	1.758	1.755	1.755	1.755
WS4721	721	0.000	1.764	1.722	1.685	1.614	1.591	1.586
WS4722	722	0.000	1.728	1.701	1.671	1.610	1.576	1.569
WS4723	723	0.000	1.724	1.695	1.681	1.627	1.584	1.577
WS4821	821	0.000	1.330	0.855	0.509	0.128	0.089	0.088
WS4822	822	0.000	1.281	0.840	0.518	0.141	0.081	0.071
WS4823	823	0.000	1.274	0.915	0.566	0.165	0.116	0.105
WS4631	631	0.000	1.799	1.770	1.731	1.729	1.729	1.729
WS4632	632	0.000	1.731	1.721	1.703	1.697	1.697	1.697
WS4633	633	0.000	1.711	1.688	1.663	1.658	1.658	1.658
WS4731	731	0.000	1.723	1.689	1.651	1.490	1.340	1.321
WS4732	732	0.000	1.688	1.658	1.604	1.424	1.300	1.283
WS4733	733	0.000	1.694	1.656	1.615	1.455	1.264	1.245
WS4831	831	0.000	1.101	0.602	0.300	0.051	0.020	0.018
WS4832	832	0.000	1.087	0.574	0.257	0.044	0.018	0.015
WS4833	833	0.000	1.150	0.662	0.290	0.068	0.029	0.026

D.1.3 BET surface area measurements

Table D.1.5: Witbank Seam 4 – BET surface area results

		Surface Area (m ² /g)
WS4611	611	8.8
WS4612	612	2.2
WS4613	613	3.1
WS4711	711	6.9
WS4712	712	5.9
WS4713	713	0.12
WS4811	811	7.4
WS4812	812	16.7
WS4813	813	5.4
WS4621	621	41
WS4622	622	40.3
WS4623	623	43.8
WS4721	721	112.3
WS4722	722	94.8
WS4723	723	115.5
WS4821	821	176.2
WS4822	822	152.4
WS4823	823	149.6
WS4631	631	198.2
WS4632	632	160.5
WS4633	633	168.5
WS4731	731	255.6
WS4732	732	221.5
WS4733	733	252.8
WS4831	831	320.2
WS4832	832	335.9
WS4833	833	358.9

D.2 NEW VAAL SAMPLES

D.2.1 pH measurements

The acronym used (NV821) in the tables below refers to the first sample of the activated carbons from New Vaal coal, prepared at 800°C and 2 hours.

Table D.2.1: New Vaal – pH measurements (600°C, 700°C, 800°C, 2hr)

Time (min)	NV621 621	NV622 622	NV623 623	NV721 721	NV722 722	NV723 723	NV821 821	NV822 822	NV823 823
0	6.909	7.015	7.126	7.059	7.052	7.077	7.056	7.034	7.094
5	9.758	9.732	10.384	8.787	9.338	10.349	9.588	10.803	10.432
10	10.078	10.087	10.630	9.419	9.942	10.660	9.969	11.078	10.732
15	10.202	10.259	10.738	9.701	10.108	10.766	10.062	11.207	10.833
20	10.262	10.351	10.800	9.893	10.238	10.851	10.086	11.270	10.873
25	10.317	10.411	10.839	10.054	10.363	10.920	10.105	11.304	10.887
30	10.356	10.443	10.862	10.180	10.457	10.958	10.123	11.353	10.890
35	10.386	10.474	10.876	10.275	10.537	10.981	10.135	11.376	10.888
40	10.414	10.501	10.881	10.334	10.587	10.996	10.147	11.386	10.880
45	10.450	10.520	10.879	10.357	10.617	11.000	10.150	11.404	10.869
50	10.483	10.527	10.883	10.355	10.629	10.996	10.159	11.405	10.862
55	10.489	10.527	10.870	10.344	10.632	10.988	10.159	11.404	10.850
60	10.490	10.526	10.865	10.351	10.632	10.976	10.161	11.402	10.838
65	10.506	10.522	10.856	10.357	10.623	10.958	10.161	11.397	10.824
70	10.510	10.517	10.842	10.362	10.601	10.939	10.145	11.388	10.803
75	10.508	10.513	10.830	10.369	10.573	10.917	10.137	11.386	10.782
80	10.512	10.504	10.817	10.387	10.542	10.887	10.121	11.379	10.758
85	10.508	10.490	10.798	10.405	10.506	10.866	10.100	11.369	10.737
90	10.503	10.480	10.780	10.440	10.469	10.830	10.119	11.358	10.713
95	10.494	10.464	10.759	10.481	10.434	10.800	10.135	11.346	10.687
100	10.492	10.450	10.738	10.497	10.395	10.759	10.130	11.329	10.664
105	10.485	10.432	10.711	10.507	10.356	10.733	10.131	11.313	10.640
110	10.481	10.418	10.683	10.545	10.322	10.681	10.135	11.299	10.612
115	10.478	10.400	10.653	10.552	10.287	10.637	10.147	11.285	10.586
120	10.469	10.379	10.621	10.476	10.254	10.580	10.156	11.268	10.558
125	10.464	10.356	10.588	10.429	10.223	10.539	10.168	11.250	10.532
130	10.455	10.335	10.550	10.394	10.192	10.486	10.175	11.235	10.509
135	10.448	10.312	10.513	10.382	10.162	10.436	10.173	11.214	10.483
140	10.460	10.289	10.473	10.374	10.134	10.398	10.170	11.200	10.457
145	10.444	10.269	10.432	10.357	10.107	10.377	10.164	11.184	10.436
150	10.439	10.243	10.393	10.343	10.079	10.349	10.157	11.165	10.408
155	10.436	10.220	10.352	10.325	10.055	10.320	10.149	11.148	10.382
160	10.432	10.195	10.308	10.302	10.030	10.287	10.138	11.134	10.361
165	10.425	10.170	10.269	10.281	10.004	10.242	10.130	11.115	10.340
170	10.418	10.144	10.230	10.256	9.982	10.190	10.130	11.095	10.318
175	10.416	10.121	10.192	10.233	9.954	10.138	10.126	11.076	10.297
180	10.411	10.094	10.151	10.210	9.930	10.089	10.121	11.059	10.274
185	10.405	10.068	10.112	10.190	9.907	10.046	10.119	11.038	10.255
190	10.400	10.040	10.071	10.171	9.885	10.003	10.110	11.017	10.234
195	10.397	10.015	10.034	10.148	9.860	9.964	10.103	10.998	10.217
200	10.390	9.986	9.999	10.130	9.838	9.931	10.097	10.977	10.197
205	10.382	9.960	9.963	10.109	9.815	9.900	10.088	10.955	10.180
210	10.377	9.932	9.930	10.089	9.791	9.871	10.079	10.934	10.161
215	10.370	9.907	9.895	10.068	9.768	9.843	10.072	10.914	10.140
220	10.361	9.879	9.861	10.045	9.748	9.817	10.065	10.890	10.121
225	10.352	9.852	9.831	10.025	9.727	9.794	10.055	10.868	10.103
230	10.344	9.824	9.797	10.004	9.704	9.772	10.043	10.845	10.088
235	10.335	9.799	9.765	9.983	9.682	9.746	10.032	10.817	10.069

Table D.2.2: New Vaal – pH measurements (600°C, 700°C, 800°C, 3 hr)

time	NV631 631	NV632 632	NV633 633	NV731 731	NV732 732	NV733 733	NV831 831	NV832 832	NV833 833
0	6.873	7.096	7.516	7.050	7.012	6.809	7.013	7.090	7.007
5	7.615	6.303	8.356	9.123	7.081	9.460	10.878	7.441	10.215
10	8.114	8.711	8.623	9.431	8.408	9.889	11.188	9.679	10.435
15	8.248	9.387	8.788	9.560	8.781	10.030	11.312	9.884	10.509
20	8.302	9.533	8.873	9.638	8.947	10.086	11.358	9.937	10.564
25	8.316	9.604	8.915	9.698	9.024	10.149	11.358	9.949	10.588
30	8.325	9.627	8.968	9.755	9.083	10.185	11.351	9.956	10.609
35	8.330	9.634	8.988	9.815	9.083	10.225	11.327	9.972	10.614
40	8.316	9.627	9.004	9.848	9.109	10.250	11.312	9.937	10.604
45	8.314	9.575	9.007	9.841	9.094	10.262	11.296	9.934	10.611
50	8.316	9.546	9.014	9.832	9.030	10.279	11.279	9.922	10.602
55	8.309	9.434	9.036	9.818	8.981	10.300	11.245	9.903	10.585
60	8.313	9.323	9.036	9.824	8.969	10.319	11.220	9.886	10.568
65	8.311	9.214	9.031	9.812	8.916	10.338	11.195	9.839	10.571
70	8.311	9.090	9.019	9.798	8.889	10.345	11.167	9.799	10.571
75	8.347	8.967	9.014	9.793	8.837	10.366	11.139	9.753	10.566
80	8.350	8.960	9.019	9.772	8.786	10.378	11.115	9.708	10.556
85	8.397	9.000	9.017	9.748	8.728	10.391	11.084	9.675	10.542
90	8.416	9.011	9.005	9.719	8.680	10.387	11.053	9.644	10.525
95	8.406	9.072	8.993	9.686	8.634	10.372	11.019	9.598	10.504
100	8.390	9.113	8.978	9.660	8.586	10.359	10.986	9.562	10.482
105	8.388	9.068	8.961	9.633	8.547	10.344	10.952	9.529	10.454
110	8.407	8.958	8.944	9.608	8.520	10.323	10.912	9.493	10.426
115	8.395	8.847	8.925	9.586	8.509	10.312	10.879	9.455	10.401
120	8.384	8.782	8.904	9.553	8.468	10.286	10.840	9.419	10.378
125	8.356	8.793	8.885	9.526	8.446	10.265	10.800	9.384	10.356
130	8.322	8.802	8.870	9.500	8.434	10.236	10.774	9.340	10.330
135	8.280	8.816	8.856	9.474	8.424	10.210	10.747	9.300	10.308
140	8.234	8.733	8.841	9.448	8.408	10.182	10.721	9.271	10.287
145	8.191	8.628	8.829	9.424	8.400	10.152	10.692	9.233	10.266
150	8.128	8.549	8.813	9.398	8.383	10.123	10.659	9.202	10.249
155	8.087	8.484	8.796	9.376	8.362	10.098	10.637	9.169	10.230
160	8.069	8.445	8.781	9.354	8.352	10.069	10.602	9.143	10.213
165	8.062	8.418	8.769	9.333	8.373	10.044	10.576	9.117	10.192
170	8.048	8.407	8.755	9.311	8.378	10.020	10.547	9.083	10.177
175	8.035	8.391	8.743	9.287	8.381	9.996	10.519	9.060	10.159
180	8.014	8.354	8.731	9.259	8.381	9.969	10.490	9.035	10.144
185	8.017	8.331	8.717	9.233	8.385	9.947	10.469	9.016	10.125
190	8.028	8.309	8.707	9.216	8.383	9.923	10.442	8.995	10.108
195	8.012	8.291	8.695	9.195	8.386	9.902	10.421	8.973	10.087
200	7.999	8.300	8.685	9.178	8.383	9.881	10.397	8.950	10.070
205	7.998	8.282	8.676	9.161	8.383	9.863	10.376	8.933	10.054
210	7.998	8.273	8.668	9.138	8.378	9.846	10.356	8.914	10.034
215	7.998	8.272	8.658	9.120	8.374	9.825	10.333	8.900	10.008
220	7.990	8.263	8.647	9.101	8.374	9.811	10.310	8.883	9.989
225	7.987	8.261	8.637	9.082	8.376	9.794	10.297	8.873	9.968
230	7.978	8.266	8.627	9.063	8.373	9.776	10.289	8.857	9.949
235	7.980	8.261	8.615	9.044	8.373	9.761	10.263	8.842	9.929

D.2.2 Phenol adsorption measurements

Table D.2.3: New Vaal – UV-vis spectrophotometer results

Time (min)		Measured adsorbance						
		0	2	5	10	30	50	60
NV621	621	0.000	1.680	1.677	1.665	1.648	1.648	1.648
NV622	622	0.000	1.742	1.714	1.672	1.660	1.660	1.660
NV623	623	0.000	1.716	1.704	1.671	1.659	1.659	1.659
NV721	721	0.000	1.740	1.607	1.536	1.501	1.481	1.476
NV722	722	0.000	1.663	1.576	1.463	1.492	1.442	1.428
NV723	723	0.000	1.642	1.558	1.419	1.479	1.456	1.443
NV821	821	0.000	1.205	0.759	0.581	0.178	0.082	0.077
NV822	822	0.000	1.091	0.796	0.534	0.251	0.113	0.090
NV823	823	0.000	1.193	0.743	0.568	0.161	0.071	0.064
NV631	631	0.000	1.782	1.726	1.725	1.711	1.688	1.681
NV632	632	0.000	1.755	1.727	1.713	1.677	1.634	1.629
NV633	633	0.000	1.726	1.685	1.658	1.640	1.590	1.583
NV731	731	0.000	1.619	1.459	1.295	0.883	0.732	0.709
NV732	732	0.000	1.573	1.450	1.336	0.983	0.788	0.771
NV733	733	0.000	1.602	1.388	1.231	0.779	0.641	0.628
NV831	831	0.000	1.156	0.718	0.415	0.173	0.095	0.090
NV832	832	0.000	1.212	0.780	0.439	0.147	0.099	0.089
NV833	833	0.000	1.142	0.708	0.409	0.144	0.088	0.081

D.2.3 BET surface area measurements

Table D.2.4: New Vaal – BET surface area results

		Surface Area (m ² /g)
NV621	621	44.6
NV622	622	60.9
NV623	623	26.4
NV721	721	169.7
NV722	722	162.4
NV723	723	186.2
NV821	821	154.9
NV822	822	192.3
NV823	823	223.1
NV631	631	49.7
NV632	632	54.2
NV633	633	6.9
NV731	731	184.5
NV732	732	187.7
NV733	733	173.0
NV831	831	180.5
NV832	832	115.5
NV833	833	178.2

D.3 NORIT RO 0.8 SAMPLE

D.3.1 pH measurements

Table D.3.1: Norit RO 0.8 – pH measurements

Time (min)	Norit RO 0.8_1	Norit RO 0.8_2
0	5.269	6.370
5	9.780	9.878
10	9.843	9.904
15	9.919	9.937
20	9.928	9.997
25	10.001	10.055
30	10.069	10.115
35	10.124	10.152
40	10.168	10.198
45	10.200	10.228
50	10.227	10.254
55	10.248	10.273
60	10.266	10.290
65	10.282	10.304
70	10.295	10.314
75	10.307	10.325
80	10.321	10.332
85	10.328	10.333
90	10.335	10.332
95	10.345	10.328
100	10.348	10.328
105	10.355	10.328
110	10.360	10.325
115	10.360	10.326
120	10.363	10.326
125	10.369	10.326
130	10.372	10.325
135	10.375	10.323
140	10.379	10.323
145	10.375	10.323
150	10.377	10.323
155	10.377	10.318
160	10.379	10.316
165	10.379	10.314
170	10.375	10.321
175	10.375	10.318
180	10.374	10.316
185	10.372	10.313
190	10.369	10.314
195	10.367	10.306

D.3.2 Phenol adsorption measurements

Table D.3.2: Norit RO 0.8 – UV-vis spectrophotometer results

		Measured adsorbance						
time (min)		0	2	5	10	30	50	60
Norit RO 0.8		0.000	0.887	0.277	0.059	0.015	0.015	0.015

D.3.3 BET surface area measurements

Table D.3.3: Norit RO 0.8 – BET surface area results

		Surface Area (m ² /g)
Norit RO 0.8		757.5

APPENDIX E

SAMPLE CALCULATIONS

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E.1 pH MEASUREMENTS – RATE CALCULATIONS

The change in pH was calculated to determine whether a constant solution pH has been reached. The example presented is for the first activated carbon sample prepared from Witbank Seam 4 at a temperature of 600°C and 1 hour:

$$\begin{aligned}\Delta pH &= pH_{(i+1)} - pH_{(i)} \\ &= 8.538 - 7.502 \\ &= \underline{1.036}\end{aligned}$$

The rate change of pH was calculated for the same abovementioned sample to determine adsorption tempo:

$$\begin{aligned}\frac{dpH}{dt} &= \frac{pH_{(i+1)} - pH_{(i)}}{t_{(i+1)} - t_{(i)}} \\ &= \frac{8.538 - 7.502}{5} \\ &= \underline{0.2072}\end{aligned}$$

The following calculation was to determine the rate at which desorption occurred in some of the activated carbon samples:

$$\begin{aligned}\frac{dpH}{dt} &= \frac{pH_{final} - pH_{max}}{\Delta t(pH_{final}, pH_{max})} \\ &= \frac{10.102 - 10.358}{90} \\ &= -0.0028\end{aligned}$$

The ideal situation would have been if the latter value was zero or a positive zero, corresponding to results obtained for Norit RO 0.8.

E.2 PHENOL ADSORPTION – ADSORPTION/DESORPTION RATE CALCULATIONS

Before the phenol concentration could be determined, it was necessary to obtain a calibration curve. This was performed by measuring the adsorbance of different concentration of phenol with the use of the UV-vis. The adsorbance data was plotted against the different concentrations and a straight line was fitted. The equation obtained from this fitted line:

$$\begin{aligned}\text{Adsorbance} &= 0.0194x + 0.0242 \\ \text{Concentration} &= \frac{\text{Adsorbance} - 0.0242}{0.0194}\end{aligned}$$

For the calculation of the % adsorbed, and with an initial phenol solution concentration of 100ppm:

$$\% \text{ Adsorbed} = \frac{100 - \text{Concentration}}{100} \times 100$$

To determine the initial phenol adsorption rate:

$$\frac{dC}{dt} = \frac{C_{(i+1)} - C_{(i)}}{\Delta t}$$

The calculation presented is for the activated carbon sample from Witbank Seam 4, prepared at 600°C and 1 hour:

$$\begin{aligned}\text{Concentration} &= \frac{1.772 - 0.0242}{0.0194} \\ &= \underline{90.093\text{ppm}}\end{aligned}$$

$$\begin{aligned}\% \text{ Adsorbed} &= \frac{100 - 90.093}{100} \times 100 \\ &= \underline{9.907\%}\end{aligned}$$

$$\begin{aligned}\frac{dC}{dt} &= \frac{C_{(i+t)} - C_{(i)}}{\Delta t} \\ &= \frac{9.907 - 0.000}{2} \\ &= \underline{4.954}\end{aligned}$$

The initial adsorption rate for the Witbank Seam 4 activated carbon (600°C, 1 hr) was calculated to be 4.954ppm/min.

APPENDIX F

TABULATED CALCULATED DATA

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F.1 WITBANK SEAM 4 SAMPLES

F.1.1 pH results

Refer to **Appendix E: Sample calculations** for the detail calculations regarding the pH measurements (Pages 119 – 120).

The acronym used (WS4821) in the tables below refers to the first sample of the activated carbons from Witbank Seam 4 coal, prepared at 800°C and 2 hours.

The calculated results for the pH measurement results are shown in Table F.1.1 – F.1.9.

Table F.1.1: Calculated pH derivatives (600°C, 1 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4611	WS4612	WS4613	WS4611	WS4612	WS4613	WS4611	WS4612	WS4613
0	7.502	6.980	6.955	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	8.538	10.263	10.344	1.036	3.283	3.389	0.2072	0.6566	0.6778
10	9.158	10.562	10.710	0.620	0.299	0.366	0.1240	0.0598	0.0732
15	9.468	10.700	10.850	0.310	0.138	0.140	0.0620	0.0276	0.0280
20	9.674	10.796	10.940	0.206	0.096	0.090	0.0412	0.0192	0.0180
25	9.813	10.868	11.003	0.139	0.072	0.063	0.0278	0.0144	0.0126
30	9.914	10.930	11.041	0.101	0.062	0.038	0.0202	0.0124	0.0076
35	10.004	10.968	11.062	0.090	0.038	0.021	0.0180	0.0076	0.0042
40	10.067	10.991	11.079	0.063	0.023	0.017	0.0126	0.0046	0.0034
45	10.128	11.005	11.093	0.061	0.014	0.014	0.0122	0.0028	0.0028
50	10.184	11.013	11.095	0.056	0.008	0.002	0.0112	0.0016	0.0004
55	10.218	11.013	11.102	0.034	0.000	0.007	0.0068	0.0000	0.0014
60	10.250	11.010	11.107	0.032	-0.003	0.005	0.0064	-0.0006	0.0010
65	10.281	11.005	11.107	0.031	-0.005	0.000	0.0062	-0.0010	0.0000
70	10.304	10.999	11.105	0.023	-0.006	-0.002	0.0046	-0.0012	-0.0004
75	10.319	10.991	11.114	0.015	-0.008	0.009	0.0030	-0.0016	0.0018
80	10.335	10.982	11.115	0.016	-0.009	0.001	0.0032	-0.0018	0.0002
85	10.344	10.973	11.115	0.009	-0.009	0.000	0.0018	-0.0018	0.0000
90	10.351	10.965	11.119	0.007	-0.008	0.004	0.0014	-0.0016	0.0008
95	10.356	10.951	11.119	0.005	-0.014	0.000	0.0010	-0.0028	0.0000
100	10.358	10.939	11.119	0.002	-0.012	0.000	0.0004	-0.0024	0.0000
105	10.358	10.925	11.119	0.000	-0.014	0.000	0.0000	-0.0028	0.0000
110	10.351	10.911	11.114	-0.007	-0.014	-0.005	-0.0014	-0.0028	-0.0010
115	10.344	10.896	11.112	-0.007	-0.015	-0.002	-0.0014	-0.0030	-0.0004
120	10.335	10.882	11.108	-0.009	-0.014	-0.004	-0.0018	-0.0028	-0.0008
125	10.321	10.865	11.103	-0.014	-0.017	-0.005	-0.0028	-0.0034	-0.0010
130	10.305	10.847	11.095	-0.016	-0.018	-0.008	-0.0032	-0.0036	-0.0016
135	10.290	10.830	11.082	-0.015	-0.017	-0.013	-0.0030	-0.0034	-0.0026
140	10.272	10.813	11.067	-0.018	-0.017	-0.015	-0.0036	-0.0034	-0.0030
145	10.257	10.792	11.053	-0.015	-0.021	-0.014	-0.0030	-0.0042	-0.0028
150	10.236	10.775	11.036	-0.021	-0.017	-0.017	-0.0042	-0.0034	-0.0034
155	10.217	10.754	11.018	-0.019	-0.021	-0.018	-0.0038	-0.0042	-0.0036
160	10.201	10.735	11.004	-0.016	-0.019	-0.014	-0.0032	-0.0038	-0.0028
165	10.187	10.721	10.987	-0.014	-0.014	-0.017	-0.0028	-0.0028	-0.0034
170	10.175	10.702	10.975	-0.012	-0.019	-0.012	-0.0024	-0.0038	-0.0024
175	10.159	10.683	10.961	-0.016	-0.019	-0.014	-0.0032	-0.0038	-0.0028
180	10.144	10.668	10.946	-0.015	-0.015	-0.015	-0.0030	-0.0030	-0.0030
185	10.126	10.643	10.930	-0.018	-0.025	-0.016	-0.0036	-0.0050	-0.0032
190	10.114	10.626	10.916	-0.012	-0.017	-0.014	-0.0024	-0.0034	-0.0028
195	10.102	10.604	10.899	-0.012	-0.022	-0.017	-0.0024	-0.0044	-0.0034

Table F.1.2: Calculated pH derivatives (700°C, 1 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4711	WS4712	WS4713	WS4711	WS4712	WS4713	WS4711	WS4712	WS4713
0	6.827	6.967	7.337	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	11.237	11.192	9.214	4.410	4.225	1.877	0.8820	0.8450	0.3754
10	11.512	11.467	9.654	0.275	0.275	0.440	0.0550	0.0550	0.0880
15	11.644	11.598	9.949	0.132	0.131	0.295	0.0264	0.0262	0.0590
20	11.722	11.686	10.162	0.078	0.088	0.213	0.0156	0.0176	0.0426
25	11.772	11.763	10.289	0.050	0.077	0.127	0.0100	0.0154	0.0254
30	11.809	11.813	10.383	0.037	0.050	0.094	0.0074	0.0100	0.0188
35	11.836	11.841	10.425	0.027	0.028	0.042	0.0054	0.0056	0.0084
40	11.856	11.862	10.444	0.020	0.021	0.019	0.0040	0.0042	0.0038
45	11.869	11.878	10.464	0.013	0.016	0.020	0.0026	0.0032	0.0040
50	11.880	11.890	10.468	0.011	0.012	0.004	0.0022	0.0024	0.0008
55	11.887	11.893	10.471	0.007	0.003	0.003	0.0014	0.0006	0.0006
60	11.892	11.898	10.463	0.005	0.005	-0.008	0.0010	0.0010	-0.0016
65	11.897	11.898	10.457	0.005	0.000	-0.006	0.0010	0.0000	-0.0012
70	11.901	11.900	10.444	0.004	0.002	-0.013	0.0008	0.0004	-0.0026
75	11.902	11.898	10.426	0.001	-0.002	-0.018	0.0002	-0.0004	-0.0036
80	11.906	11.897	10.409	0.004	-0.001	-0.017	0.0008	-0.0002	-0.0034
85	11.906	11.893	10.393	0.000	-0.004	-0.016	0.0000	-0.0008	-0.0032
90	11.908	11.888	10.372	0.002	-0.005	-0.021	0.0004	-0.0010	-0.0042
95	11.909	11.885	10.353	0.001	-0.003	-0.019	0.0002	-0.0006	-0.0038
100	11.909	11.881	10.334	0.000	-0.004	-0.019	0.0000	-0.0008	-0.0038
105	11.911	11.874	10.319	0.002	-0.007	-0.015	0.0004	-0.0014	-0.0030
110	11.913	11.871	10.298	0.002	-0.003	-0.021	0.0004	-0.0006	-0.0042
115	11.911	11.865	10.275	-0.002	-0.006	-0.023	-0.0004	-0.0012	-0.0046
120	11.911	11.860	10.251	0.000	-0.005	-0.024	0.0000	-0.0010	-0.0048
125	11.911	11.857	10.228	0.000	-0.003	-0.023	0.0000	-0.0006	-0.0046
130	11.911	11.851	10.206	0.000	-0.006	-0.022	0.0000	-0.0012	-0.0044
135	11.911	11.846	10.183	0.000	-0.005	-0.023	0.0000	-0.0010	-0.0046
140	11.909	11.843	10.154	-0.002	-0.003	-0.029	-0.0004	-0.0006	-0.0058
145	11.911	11.838	10.131	0.002	-0.005	-0.023	0.0004	-0.0010	-0.0046
150	11.909	11.834	10.114	-0.002	-0.004	-0.017	-0.0004	-0.0008	-0.0034
155	11.908	11.831	10.089	-0.001	-0.003	-0.025	-0.0002	-0.0006	-0.0050
160	11.906	11.825	10.072	-0.002	-0.006	-0.017	-0.0004	-0.0012	-0.0034
165	11.906	11.822	10.051	0.000	-0.003	-0.021	0.0000	-0.0006	-0.0042
170	11.906	11.817	10.032	0.000	-0.005	-0.019	0.0000	-0.0010	-0.0038
175	11.902	11.813	10.013	-0.004	-0.004	-0.019	-0.0008	-0.0008	-0.0038
180	11.901	11.810	9.990	-0.001	-0.003	-0.023	-0.0002	-0.0006	-0.0046
185	11.901	11.808	9.975	0.000	-0.002	-0.015	0.0000	-0.0004	-0.0030
190	11.899	11.803	9.954	-0.002	-0.005	-0.021	-0.0004	-0.0010	-0.0042
195	11.897	11.796	9.926	-0.002	-0.007	-0.028	-0.0004	-0.0014	-0.0056

Table F.1.3: Calculated pH derivatives (800°C, 1 hr)

time	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
	WS4811	WS4812	WS4813	WS4811	WS4812	WS4813	WS4811	WS4812	WS4813
0	6.536	7.553	7.232	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	6.679	8.721	10.797	0.143	1.168	3.565	0.0286	0.2336	0.7130
10	6.984	9.395	11.193	0.305	0.674	0.396	0.0610	0.1348	0.0792
15	7.461	9.398	11.337	0.477	0.003	0.144	0.0954	0.0006	0.0288
20	7.856	9.294	11.426	0.395	-0.104	0.089	0.0790	-0.0208	0.0178
25	8.229	9.132	11.490	0.373	-0.162	0.064	0.0746	-0.0324	0.0128
30	8.262	8.978	11.574	0.033	-0.154	0.084	0.0066	-0.0308	0.0168
35	8.262	8.999	11.568	0.000	0.021	-0.006	0.0000	0.0042	-0.0012
40	8.197	9.061	11.598	-0.065	0.062	0.030	-0.0130	0.0124	0.0060
45	8.102	9.090	11.621	-0.095	0.029	0.023	-0.0190	0.0058	0.0046
50	7.996	9.116	11.640	-0.106	0.026	0.019	-0.0212	0.0052	0.0038
55	7.865	9.120	11.669	-0.131	0.004	0.029	-0.0262	0.0008	0.0058
60	7.807	9.161	11.663	-0.058	0.041	-0.006	-0.0116	0.0082	-0.0012
65	7.754	9.164	11.671	-0.053	0.003	0.008	-0.0106	0.0006	0.0016
70	7.720	9.150	11.678	-0.034	-0.014	0.007	-0.0068	-0.0028	0.0014
75	7.727	9.132	11.681	0.007	-0.018	0.003	0.0014	-0.0036	0.0006
80	7.726	9.107	11.696	-0.001	-0.025	0.015	-0.0002	-0.0050	0.0030
85	7.775	9.092	11.700	0.049	-0.015	0.004	0.0098	-0.0030	0.0008
90	7.749	9.084	11.703	-0.026	-0.008	0.003	-0.0052	-0.0016	0.0006
95	7.771	9.084	11.705	0.022	0.000	0.002	0.0044	0.0000	0.0004
100	7.780	9.093	11.709	0.009	0.009	0.004	0.0018	0.0018	0.0008
105	7.794	9.090	11.711	0.014	-0.003	0.002	0.0028	-0.0006	0.0004
110	7.798	9.067	11.712	0.004	-0.023	0.001	0.0008	-0.0046	0.0002
115	7.824	9.139	11.712	0.026	0.072	0.000	0.0052	0.0144	0.0000
120	7.895	9.221	11.714	0.071	0.082	0.002	0.0142	0.0164	0.0004
125	7.948	9.288	11.714	0.053	0.067	0.000	0.0106	0.0134	0.0000
130	7.989	9.341	11.712	0.041	0.053	-0.002	0.0082	0.0106	-0.0004
135	8.020	9.384	11.712	0.031	0.043	0.000	0.0062	0.0086	0.0000
140	8.027	9.421	11.705	0.007	0.037	-0.007	0.0014	0.0074	-0.0014
145	8.056	9.453	11.702	0.029	0.032	-0.003	0.0058	0.0064	-0.0006
150	8.050	9.483	11.700	-0.006	0.030	-0.002	-0.0012	0.0060	-0.0004
155	8.054	9.512	11.696	0.004	0.029	-0.004	0.0008	0.0058	-0.0008
160	8.061	9.533	11.692	0.007	0.021	-0.004	0.0014	0.0042	-0.0008
165	8.062	9.550	11.691	0.001	0.017	-0.001	0.0002	0.0034	-0.0002
170	8.068	9.566	11.687	0.006	0.016	-0.004	0.0012	0.0032	-0.0008
175	8.073	9.582	11.685	0.005	0.016	-0.002	0.0010	0.0032	-0.0004
180	8.080	9.575	11.681	0.007	-0.007	-0.004	0.0014	-0.0014	-0.0008
185	8.083	9.570	11.680	0.003	-0.005	-0.001	0.0006	-0.0010	-0.0002
190	8.095	9.561	11.674	0.012	-0.009	-0.006	0.0024	-0.0018	-0.0012
195	8.090	9.547	11.672	-0.005	-0.014	-0.002	-0.0010	-0.0028	-0.0004

Table F.1.4: Calculated pH derivatives (600°C, 2 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4621	WS4622	WS4623	WS4621	WS4622	WS4623	WS4621	WS4622	WS4623
0	6.943	7.204	7.142	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	10.308	10.280	10.619	3.365	3.076	3.477	0.6730	0.6152	0.6954
10	10.672	10.642	10.872	0.364	0.362	0.253	0.0728	0.0724	0.0506
15	10.898	10.820	10.981	0.226	0.178	0.109	0.0452	0.0356	0.0218
20	11.039	10.942	11.049	0.141	0.122	0.068	0.0282	0.0244	0.0136
25	11.136	11.013	11.090	0.097	0.071	0.041	0.0194	0.0142	0.0082
30	11.191	11.060	11.119	0.055	0.047	0.029	0.0110	0.0094	0.0058
35	11.223	11.105	11.144	0.032	0.045	0.025	0.0064	0.0090	0.0050
40	11.245	11.140	11.160	0.022	0.035	0.016	0.0044	0.0070	0.0032
45	11.259	11.165	11.174	0.014	0.025	0.014	0.0028	0.0050	0.0028
50	11.269	11.184	11.186	0.010	0.019	0.012	0.0020	0.0038	0.0024
55	11.271	11.196	11.196	0.002	0.012	0.010	0.0004	0.0024	0.0020
60	11.271	11.207	11.202	0.000	0.011	0.006	0.0000	0.0022	0.0012
65	11.271	11.219	11.207	0.000	0.012	0.005	0.0000	0.0024	0.0010
70	11.268	11.222	11.210	-0.003	0.003	0.003	-0.0006	0.0006	0.0006
75	11.262	11.226	11.210	-0.006	0.004	0.000	-0.0012	0.0008	0.0000
80	11.255	11.227	11.210	-0.007	0.001	0.000	-0.0014	0.0002	0.0000
85	11.245	11.231	11.210	-0.010	0.004	0.000	-0.0020	0.0008	0.0000
90	11.236	11.233	11.207	-0.009	0.002	-0.003	-0.0018	0.0004	-0.0006
95	11.224	11.234	11.203	-0.012	0.001	-0.004	-0.0024	0.0002	-0.0008
100	11.212	11.243	11.200	-0.012	0.009	-0.003	-0.0024	0.0018	-0.0006
105	11.196	11.245	11.196	-0.016	0.002	-0.004	-0.0032	0.0004	-0.0008
110	11.184	11.248	11.193	-0.012	0.003	-0.003	-0.0024	0.0006	-0.0006
115	11.169	11.248	11.186	-0.015	0.000	-0.007	-0.0030	0.0000	-0.0014
120	11.151	11.247	11.179	-0.018	-0.001	-0.007	-0.0036	-0.0002	-0.0014
125	11.134	11.243	11.170	-0.017	-0.004	-0.009	-0.0034	-0.0008	-0.0018
130	11.117	11.240	11.163	-0.017	-0.003	-0.007	-0.0034	-0.0006	-0.0014
135	11.094	11.236	11.154	-0.023	-0.004	-0.009	-0.0046	-0.0008	-0.0018
140	11.073	11.231	11.144	-0.021	-0.005	-0.010	-0.0042	-0.0010	-0.0020
145	11.051	11.227	11.133	-0.022	-0.004	-0.011	-0.0044	-0.0008	-0.0022
150	11.028	11.219	11.125	-0.023	-0.008	-0.008	-0.0046	-0.0016	-0.0016
155	11.007	11.210	11.112	-0.021	-0.009	-0.013	-0.0042	-0.0018	-0.0026
160	10.985	11.193	11.098	-0.022	-0.017	-0.014	-0.0044	-0.0034	-0.0028
165	10.960	11.179	11.083	-0.025	-0.014	-0.015	-0.0050	-0.0028	-0.0030
170	10.947	11.163	11.069	-0.013	-0.016	-0.014	-0.0026	-0.0032	-0.0028
175	10.929	11.147	11.055	-0.018	-0.016	-0.014	-0.0036	-0.0032	-0.0028
180	10.910	11.130	11.042	-0.019	-0.017	-0.013	-0.0038	-0.0034	-0.0026
185	10.889	11.118	11.027	-0.021	-0.012	-0.015	-0.0042	-0.0024	-0.0030
190	10.869	11.104	11.009	-0.020	-0.014	-0.018	-0.0040	-0.0028	-0.0036
195	10.842	11.090	10.995	-0.027	-0.014	-0.014	-0.0054	-0.0028	-0.0028

Table F.1.5: Calculated pH derivatives (700°C, 2 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4721	WS4722	WS4723	WS4721	WS4722	WS4723	WS4721	WS4722	WS4723
0	6.769	6.807	6.873	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	10.128	9.739	10.085	3.359	2.932	3.212	0.6718	0.5864	0.6424
10	10.521	10.240	10.428	0.393	0.501	0.343	0.0786	0.1002	0.0686
15	10.710	10.483	10.612	0.189	0.243	0.184	0.0378	0.0486	0.0368
20	10.828	10.668	10.711	0.118	0.185	0.099	0.0236	0.0370	0.0198
25	10.908	10.767	10.762	0.080	0.099	0.051	0.0160	0.0198	0.0102
30	10.957	10.820	10.798	0.049	0.053	0.036	0.0098	0.0106	0.0072
35	10.993	10.850	10.829	0.036	0.030	0.031	0.0072	0.0060	0.0062
40	11.025	10.867	10.841	0.032	0.017	0.012	0.0064	0.0034	0.0024
45	11.046	10.876	10.852	0.021	0.009	0.011	0.0042	0.0018	0.0022
50	11.065	10.881	10.855	0.019	0.005	0.003	0.0038	0.0010	0.0006
55	11.080	10.884	10.857	0.015	0.003	0.002	0.0030	0.0006	0.0004
60	11.094	10.881	10.855	0.014	-0.003	-0.002	0.0028	-0.0006	-0.0004
65	11.101	10.870	10.855	0.007	-0.011	0.000	0.0014	-0.0022	0.0000
70	11.107	10.862	10.850	0.006	-0.008	-0.005	0.0012	-0.0016	-0.0010
75	11.107	10.851	10.845	0.000	-0.011	-0.005	0.0000	-0.0022	-0.0010
80	11.107	10.837	10.838	0.000	-0.014	-0.007	0.0000	-0.0028	-0.0014
85	11.107	10.829	10.829	0.000	-0.008	-0.009	0.0000	-0.0016	-0.0018
90	11.103	10.820	10.816	-0.004	-0.009	-0.013	-0.0008	-0.0018	-0.0026
95	11.101	10.806	10.805	-0.002	-0.014	-0.011	-0.0004	-0.0028	-0.0021
100	11.098	10.799	10.788	-0.003	-0.007	-0.017	-0.0006	-0.0014	-0.0035
105	11.091	10.790	10.773	-0.007	-0.009	-0.015	-0.0014	-0.0018	-0.0030
110	11.082	10.781	10.762	-0.009	-0.009	-0.011	-0.0018	-0.0018	-0.0022
115	11.072	10.771	10.750	-0.010	-0.010	-0.012	-0.0020	-0.0020	-0.0024
120	11.063	10.755	10.735	-0.009	-0.016	-0.015	-0.0018	-0.0032	-0.0030
125	11.047	10.741	10.717	-0.016	-0.014	-0.018	-0.0032	-0.0028	-0.0036
130	11.033	10.724	10.705	-0.014	-0.017	-0.012	-0.0028	-0.0034	-0.0024
135	11.018	10.703	10.692	-0.015	-0.021	-0.013	-0.0030	-0.0042	-0.0026
140	11.000	10.687	10.680	-0.018	-0.016	-0.012	-0.0036	-0.0032	-0.0024
145	10.988	10.668	10.662	-0.012	-0.019	-0.018	-0.0024	-0.0038	-0.0036
150	10.976	10.649	10.643	-0.012	-0.019	-0.019	-0.0024	-0.0038	-0.0038
155	10.960	10.628	10.626	-0.016	-0.021	-0.017	-0.0032	-0.0042	-0.0034
160	10.946	10.605	10.606	-0.014	-0.023	-0.020	-0.0028	-0.0046	-0.0040
165	10.931	10.581	10.585	-0.015	-0.024	-0.021	-0.0030	-0.0048	-0.0042
170	10.913	10.556	10.562	-0.018	-0.025	-0.023	-0.0036	-0.0050	-0.0046
175	10.898	10.533	10.540	-0.015	-0.023	-0.022	-0.0030	-0.0046	-0.0044
180	10.880	10.509	10.518	-0.018	-0.024	-0.022	-0.0036	-0.0048	-0.0044
185	10.859	10.486	10.494	-0.021	-0.023	-0.024	-0.0042	-0.0046	-0.0048
190	10.840	10.464	10.471	-0.019	-0.022	-0.023	-0.0038	-0.0044	-0.0046
195	10.821	10.436	10.447	-0.019	-0.028	-0.024	-0.0038	-0.0056	-0.0048

Table F.1.6: Calculated pH derivatives (800°C, 2 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4821	WS4822	WS4823	WS4821	WS4822	WS4823	WS4821	WS4822	WS4823
0	7.826	7.766	6.671	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	10.039	7.361	9.825	2.213	-0.405	3.154	0.4426	-0.0810	0.6308
10	10.747	7.389	10.200	0.708	0.028	0.375	0.1416	0.0056	0.0750
15	10.844	8.642	10.332	0.097	1.253	0.132	0.0194	0.2506	0.0264
20	10.880	9.052	10.376	0.036	0.410	0.044	0.0072	0.0820	0.0088
25	10.894	9.049	10.404	0.014	-0.003	0.028	0.0028	-0.0006	0.0056
30	10.897	9.022	10.446	0.003	-0.027	0.042	0.0006	-0.0054	0.0084
35	10.897	8.971	10.451	0.000	-0.051	0.005	0.0000	-0.0102	0.0010
40	10.888	8.971	10.437	-0.009	0.000	-0.014	-0.0018	0.0000	-0.0028
45	10.880	8.999	10.446	-0.008	0.028	0.009	-0.0016	0.0056	0.0018
50	10.871	9.014	10.413	-0.009	0.015	-0.033	-0.0018	0.0030	-0.0066
55	10.853	9.010	10.390	-0.018	-0.004	-0.023	-0.0036	-0.0008	-0.0046
60	10.841	8.963	10.360	-0.012	-0.047	-0.030	-0.0024	-0.0094	-0.0060
65	10.827	8.978	10.346	-0.014	0.015	-0.014	-0.0028	0.0030	-0.0028
70	10.809	8.889	10.316	-0.018	-0.089	-0.030	-0.0036	-0.0178	-0.0060
75	10.791	8.880	10.281	-0.018	-0.009	-0.035	-0.0036	-0.0018	-0.0070
80	10.768	8.853	10.258	-0.023	-0.027	-0.023	-0.0046	-0.0054	-0.0046
85	10.752	8.841	10.219	-0.016	-0.012	-0.039	-0.0032	-0.0024	-0.0078
90	10.729	8.816	10.207	-0.023	-0.025	-0.012	-0.0046	-0.0050	-0.0024
95	10.712	8.792	10.166	-0.017	-0.024	-0.041	-0.0034	-0.0048	-0.0082
100	10.691	8.748	10.138	-0.021	-0.044	-0.028	-0.0042	-0.0088	-0.0056
105	10.669	8.739	10.113	-0.022	-0.009	-0.025	-0.0044	-0.0018	-0.0050
110	10.648	8.736	10.087	-0.021	-0.003	-0.026	-0.0042	-0.0006	-0.0052
115	10.631	8.707	10.059	-0.017	-0.029	-0.028	-0.0034	-0.0058	-0.0056
120	10.608	8.706	10.032	-0.023	-0.001	-0.027	-0.0046	-0.0002	-0.0054
125	10.581	8.692	10.006	-0.027	-0.014	-0.026	-0.0054	-0.0028	-0.0052
130	10.562	8.695	9.981	-0.019	0.003	-0.025	-0.0038	0.0006	-0.0050
135	10.541	8.686	9.962	-0.021	-0.009	-0.019	-0.0042	-0.0018	-0.0038
140	10.519	8.672	9.944	-0.022	-0.014	-0.018	-0.0044	-0.0028	-0.0036
145	10.498	8.674	9.923	-0.021	0.002	-0.021	-0.0042	0.0004	-0.0042
150	10.475	8.663	9.911	-0.023	-0.011	-0.012	-0.0046	-0.0022	-0.0024
155	10.458	8.655	9.884	-0.017	-0.008	-0.027	-0.0034	-0.0016	-0.0054
160	10.433	8.640	9.870	-0.025	-0.015	-0.014	-0.0050	-0.0030	-0.0028
165	10.414	8.640	9.833	-0.019	0.000	-0.037	-0.0038	0.0000	-0.0074
170	10.392	8.633	9.818	-0.022	-0.007	-0.015	-0.0044	-0.0014	-0.0030
175	10.373	8.625	9.793	-0.019	-0.008	-0.025	-0.0038	-0.0016	-0.0050
180	10.354	8.625	9.770	-0.019	0.000	-0.023	-0.0038	0.0000	-0.0046
185	10.336	8.619	9.749	-0.018	-0.006	-0.021	-0.0036	-0.0012	-0.0042
190	10.318	8.623	9.731	-0.018	0.004	-0.018	-0.0036	0.0008	-0.0036
195	10.299	8.611	9.707	-0.019	-0.012	-0.024	-0.0038	-0.0024	-0.0048

Table F.1.7: Calculated pH derivatives (600°C, 3 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4631	WS4632	WS4633	WS4631	WS4632	WS4633	WS4631	WS4632	WS4633
0	6.831	6.876	6.993	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	9.906	10.495	9.687	3.075	3.619	2.694	0.6150	0.7238	0.5388
10	10.389	10.761	10.185	0.483	0.266	0.498	0.0966	0.0532	0.0996
15	10.642	10.938	10.413	0.253	0.177	0.228	0.0506	0.0354	0.0456
20	10.782	11.046	10.558	0.140	0.108	0.145	0.0280	0.0216	0.0290
25	10.875	11.122	10.662	0.093	0.076	0.104	0.0186	0.0152	0.0208
30	10.929	11.176	10.742	0.054	0.054	0.080	0.0108	0.0108	0.0160
35	10.983	11.219	10.806	0.054	0.043	0.064	0.0108	0.0086	0.0128
40	11.015	11.257	10.855	0.032	0.038	0.049	0.0064	0.0076	0.0098
45	11.045	11.288	10.902	0.030	0.031	0.047	0.0060	0.0062	0.0094
50	11.059	11.310	10.937	0.014	0.022	0.035	0.0028	0.0044	0.0070
55	11.076	11.331	10.963	0.017	0.021	0.026	0.0034	0.0042	0.0052
60	11.085	11.348	10.993	0.009	0.017	0.030	0.0018	0.0034	0.0060
65	11.090	11.365	11.014	0.005	0.017	0.021	0.0010	0.0034	0.0042
70	11.104	11.377	11.034	0.014	0.012	0.020	0.0028	0.0024	0.0040
75	11.101	11.388	11.055	-0.003	0.011	0.021	-0.0006	0.0022	0.0042
80	11.102	11.398	11.070	0.001	0.010	0.015	0.0002	0.0020	0.0030
85	11.102	11.405	11.083	0.000	0.007	0.013	0.0000	0.0014	0.0026
90	11.102	11.410	11.095	0.000	0.005	0.012	0.0000	0.0010	0.0024
95	11.099	11.415	11.104	-0.003	0.005	0.009	-0.0006	0.0010	0.0018
100	11.099	11.419	11.114	0.000	0.004	0.010	0.0000	0.0008	0.0020
105	11.095	11.422	11.123	-0.004	0.003	0.009	-0.0008	0.0006	0.0018
110	11.090	11.425	11.133	-0.005	0.003	0.010	-0.0010	0.0006	0.0020
115	11.085	11.429	11.142	-0.005	0.004	0.009	-0.0010	0.0008	0.0018
120	11.076	11.429	11.149	-0.009	0.000	0.007	-0.0018	0.0000	0.0014
125	11.069	11.431	11.158	-0.007	0.002	0.009	-0.0014	0.0004	0.0018
130	11.064	11.431	11.165	-0.005	0.000	0.007	-0.0010	0.0000	0.0014
135	11.055	11.431	11.174	-0.009	0.000	0.009	-0.0018	0.0000	0.0018
140	11.048	11.431	11.176	-0.007	0.000	0.002	-0.0014	0.0000	0.0004
145	11.039	11.431	11.183	-0.009	0.000	0.007	-0.0018	0.0000	0.0014
150	11.031	11.431	11.188	-0.008	0.000	0.005	-0.0016	0.0000	0.0010
155	11.025	11.429	11.191	-0.006	-0.002	0.003	-0.0012	-0.0004	0.0006
160	11.015	11.431	11.191	-0.010	0.002	0.000	-0.0020	0.0004	0.0000
165	11.004	11.425	11.197	-0.011	-0.006	0.006	-0.0022	-0.0012	0.0012
170	10.996	11.424	11.198	-0.008	-0.001	0.001	-0.0016	-0.0002	0.0002
175	10.985	11.422	11.202	-0.011	-0.002	0.004	-0.0022	-0.0004	0.0008
180	10.975	11.419	11.204	-0.010	-0.003	0.002	-0.0020	-0.0006	0.0004
185	10.964	11.415	11.202	-0.011	-0.004	-0.002	-0.0022	-0.0008	-0.0004
190	10.950	11.413	11.200	-0.014	-0.002	-0.002	-0.0028	-0.0004	-0.0004
195	10.938	11.408	11.200	-0.012	-0.005	0.000	-0.0024	-0.0010	0.0000

Table F.1.8: Calculated pH derivatives (700°C, 3 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	WS4731	WS4732	WS4733	WS4731	WS4732	WS4733	WS4731	WS4732	WS4733
0	6.724	7.000	6.730	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	10.419	7.881	6.813	3.695	0.881	0.083	0.7390	0.1762	0.0166
10	10.743	8.453	7.399	0.324	0.572	0.586	0.0648	0.1144	0.1172
15	10.884	8.591	8.216	0.141	0.138	0.817	0.0282	0.0276	0.1634
20	10.986	8.559	8.602	0.102	-0.032	0.386	0.0204	-0.0064	0.0772
25	11.049	8.403	8.791	0.063	-0.156	0.189	0.0126	-0.0312	0.0378
30	11.091	8.310	8.924	0.042	-0.093	0.133	0.0084	-0.0186	0.0266
35	11.126	8.263	9.021	0.035	-0.047	0.097	0.0070	-0.0094	0.0194
40	11.144	8.206	9.118	0.018	-0.057	0.097	0.0036	-0.0114	0.0194
45	11.154	8.199	9.049	0.010	-0.007	-0.069	0.0020	-0.0014	-0.0138
50	11.167	8.206	8.896	0.013	0.007	-0.153	0.0026	0.0014	-0.0306
55	11.168	8.377	8.870	0.001	0.171	-0.026	0.0002	0.0342	-0.0052
60	11.174	8.460	8.927	0.006	0.083	0.057	0.0012	0.0166	0.0114
65	11.177	8.502	8.870	0.003	0.042	-0.057	0.0006	0.0084	-0.0114
70	11.179	8.531	8.810	0.002	0.029	-0.060	0.0004	0.0058	-0.0120
75	11.179	8.552	8.720	0.000	0.021	-0.090	0.0000	0.0042	-0.0180
80	11.177	8.560	8.600	-0.002	0.008	-0.120	-0.0004	0.0016	-0.0240
85	11.175	8.562	8.498	-0.002	0.002	-0.102	-0.0004	0.0004	-0.0204
90	11.172	8.547	8.448	-0.003	-0.015	-0.050	-0.0006	-0.0030	-0.0100
95	11.170	8.547	8.415	-0.002	0.000	-0.033	-0.0004	0.0000	-0.0066
100	11.168	8.540	8.392	-0.002	-0.007	-0.023	-0.0004	-0.0014	-0.0046
105	11.165	8.529	8.379	-0.003	-0.011	-0.013	-0.0006	-0.0022	-0.0026
110	11.163	8.512	8.346	-0.002	-0.017	-0.033	-0.0004	-0.0034	-0.0066
115	11.158	8.495	8.313	-0.005	-0.017	-0.033	-0.0010	-0.0034	-0.0066
120	11.156	8.488	8.273	-0.002	-0.007	-0.040	-0.0004	-0.0014	-0.0080
125	11.153	8.481	8.254	-0.003	-0.007	-0.019	-0.0006	-0.0014	-0.0038
130	11.146	8.460	8.242	-0.007	-0.021	-0.012	-0.0014	-0.0042	-0.0024
135	11.142	8.458	8.233	-0.004	-0.002	-0.009	-0.0008	-0.0004	-0.0018
140	11.117	8.444	8.230	-0.025	-0.014	-0.003	-0.0050	-0.0028	-0.0006
145	11.105	8.425	8.221	-0.012	-0.019	-0.009	-0.0024	-0.0038	-0.0018
150	11.098	8.413	8.225	-0.007	-0.012	0.004	-0.0014	-0.0024	0.0008
155	11.088	8.403	8.226	-0.010	-0.010	0.001	-0.0020	-0.0020	0.0002
160	11.079	8.394	8.223	-0.009	-0.009	-0.003	-0.0018	-0.0018	-0.0006
165	11.068	8.379	8.216	-0.011	-0.015	-0.007	-0.0022	-0.0030	-0.0014
170	11.058	8.368	8.209	-0.010	-0.011	-0.007	-0.0020	-0.0022	-0.0014
175	11.047	8.372	8.187	-0.011	0.004	-0.022	-0.0022	0.0008	-0.0044
180	11.035	8.374	8.192	-0.012	0.002	0.005	-0.0024	0.0004	0.0010
185	11.024	8.377	8.187	-0.011	0.003	-0.005	-0.0022	0.0006	-0.0010
190	11.012	8.379	8.186	-0.012	0.002	-0.001	-0.0024	0.0004	-0.0002
195	10.998	8.379	8.187	-0.014	0.000	0.001	-0.0028	0.0000	0.0002

Table F.1.9: Calculated pH derivatives (800°C, 3 hr)

time	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
	WS4831	WS4832	WS4833	WS4831	WS4832	WS4833	WS4831	WS4832	WS4833
0	7.000	7.041	7.054	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	9.402	10.314	10.587	2.402	3.273	3.533	0.4804	0.6546	0.7066
10	9.751	10.736	10.880	0.349	0.422	0.293	0.0698	0.0844	0.0586
15	9.880	10.821	10.940	0.129	0.085	0.060	0.0258	0.0170	0.0120
20	10.022	10.877	10.933	0.142	0.056	-0.007	0.0284	0.0112	-0.0014
25	10.107	10.902	10.898	0.085	0.025	-0.035	0.0170	0.0050	-0.0070
30	10.162	10.907	10.880	0.055	0.005	-0.018	0.0110	0.0010	-0.0036
35	10.274	10.902	10.889	0.112	-0.005	0.009	0.0224	-0.0010	0.0018
40	10.289	10.893	10.884	0.015	-0.009	-0.005	0.0030	-0.0018	-0.0010
45	10.275	10.881	10.862	-0.014	-0.012	-0.022	-0.0028	-0.0024	-0.0044
50	10.307	10.865	10.831	0.032	-0.016	-0.031	0.0064	-0.0032	-0.0062
55	10.298	10.849	10.804	-0.009	-0.016	-0.027	-0.0018	-0.0032	-0.0054
60	10.295	10.833	10.778	-0.003	-0.016	-0.026	-0.0006	-0.0032	-0.0052
65	10.319	10.811	10.748	0.024	-0.022	-0.030	0.0048	-0.0044	-0.0060
70	10.302	10.793	10.730	-0.017	-0.018	-0.018	-0.0034	-0.0036	-0.0036
75	10.296	10.760	10.712	-0.006	-0.033	-0.018	-0.0012	-0.0066	-0.0036
80	10.288	10.729	10.689	-0.008	-0.031	-0.023	-0.0016	-0.0062	-0.0046
85	10.298	10.694	10.673	0.010	-0.035	-0.016	0.0020	-0.0070	-0.0032
90	10.293	10.655	10.650	-0.005	-0.039	-0.023	-0.0010	-0.0078	-0.0046
95	10.281	10.617	10.635	-0.012	-0.038	-0.015	-0.0024	-0.0076	-0.0030
100	10.274	10.592	10.599	-0.007	-0.025	-0.036	-0.0014	-0.0050	-0.0072
105	10.249	10.573	10.590	-0.025	-0.019	-0.009	-0.0050	-0.0038	-0.0018
110	10.237	10.562	10.569	-0.012	-0.011	-0.021	-0.0024	-0.0022	-0.0042
115	10.219	10.534	10.553	-0.018	-0.028	-0.016	-0.0036	-0.0056	-0.0032
120	10.195	10.503	10.534	-0.024	-0.031	-0.019	-0.0048	-0.0062	-0.0038
125	10.179	10.484	10.513	-0.016	-0.019	-0.021	-0.0032	-0.0038	-0.0042
130	10.170	10.459	10.495	-0.009	-0.025	-0.018	-0.0018	-0.0050	-0.0036
135	10.162	10.429	10.476	-0.008	-0.030	-0.019	-0.0016	-0.0060	-0.0038
140	10.148	10.410	10.454	-0.014	-0.019	-0.022	-0.0028	-0.0038	-0.0044
145	10.137	10.375	10.446	-0.011	-0.035	-0.008	-0.0022	-0.0070	-0.0016
150	10.139	10.351	10.431	0.002	-0.024	-0.015	0.0004	-0.0048	-0.0030
155	10.130	10.333	10.431	-0.009	-0.018	0.000	-0.0018	-0.0036	0.0000
160	10.121	10.307	10.428	-0.009	-0.026	-0.003	-0.0018	-0.0052	-0.0006
165	10.121	10.300	10.428	0.000	-0.007	0.000	0.0000	-0.0014	0.0000
170	10.114	10.281	10.424	-0.007	-0.019	-0.004	-0.0014	-0.0038	-0.0008
175	10.118	10.272	10.419	0.004	-0.009	-0.005	0.0008	-0.0018	-0.0010
180	10.100	10.253	10.412	-0.018	-0.019	-0.007	-0.0036	-0.0038	-0.0014
185	10.104	10.212	10.396	0.004	-0.041	-0.016	0.0008	-0.0082	-0.0031
190	10.092	10.196	10.380	-0.012	-0.016	-0.016	-0.0024	-0.0032	-0.0033
195	10.078	10.167	10.361	-0.014	-0.029	-0.019	-0.0028	-0.0058	-0.0038

F.1.2 Phenol adsorption results

Refer to **Appendix E: Sample calculations** for the detail calculations regarding the phenol adsorption results (Pages 120 – 121).

The calculated results for the phenol adsorption tests are shown in Table F.1.10 – F.1.12.

Table F.1.10: Phenol adsorption results (600°C, 700°C, 800°C, 1 hr)

		Calculated data							Initial adsorption (dC/dt)		
time (m/n)		0	2	5	10	30	50	60	C _i	C _{rad}	dC/dt
WS4611	611	0.000	1.772	1.754	1.733	1.729	1.729	1.729			
Concentration		0.000	90.093	89.165	88.082	87.876	87.876	87.876			
% Adsorbed		0.000	9.907	10.835	11.918	12.124	12.124	12.124	0.000	9.907	4.954
WS4612	612	0.000	1.806	1.768	1.765	1.760	1.760	1.760			
Concentration		0.000	91.845	89.887	89.732	89.474	89.474	89.474			
% Adsorbed		0.000	8.155	10.113	10.268	10.526	10.526	10.526	0.000	8.155	4.077
WS4613	613	0.000	1.765	1.743	1.727	1.724	1.724	1.724			
Concentration		0.000	89.732	88.598	87.773	87.619	87.619	87.619			
% Adsorbed		0.000	10.268	11.402	12.227	12.381	12.381	12.381	0.000	10.268	5.134
Average	610	0.000	9.443	10.784	11.471	11.677	11.677	11.677	0.000	9.443	4.722
WS4711	711	0.000	1.715	1.692	1.682	1.662	1.656	1.653			
Concentration		0.000	87.155	85.969	85.454	84.423	84.113	83.959			
% Adsorbed		0.000	12.845	14.031	14.546	15.577	15.887	16.041	0.000	12.845	6.423
WS4712	712	0.000	1.734	1.740	1.721	1.704	1.690	1.684			
Concentration		0.000	88.134	88.443	87.464	86.588	85.866	85.557			
% Adsorbed		0.000	11.866	11.557	12.536	13.412	14.134	14.443	0.000	11.866	5.933
WS4713	713	0.000	1.762	1.710	1.708	1.676	1.666	1.661			
Concentration		0.000	89.577	86.897	86.794	85.144	84.629	84.371			
% Adsorbed		0.000	10.423	13.103	13.206	14.856	15.371	15.629	0.000	10.423	5.211
Average	710	0.000	11.711	12.897	13.430	14.615	15.131	15.371	0.000	11.711	5.856
WS4811	811	0.000	1.649	1.500	1.371	1.045	0.917	0.913			
Concentration		0.000	83.753	76.072	69.423	52.619	46.021	45.814			
% Adsorbed		0.000	16.247	23.928	30.577	47.381	53.979	54.186	0.000	16.247	8.124
WS4812	812	0.000	1.646	1.536	1.391	1.108	1.005	0.990			
Concentration		0.000	83.598	77.928	70.454	55.866	50.557	49.784			
% Adsorbed		0.000	16.402	22.072	29.546	44.134	49.443	50.216	0.000	16.402	8.201
WS4813	813	0.000	1.560	1.377	1.216	0.898	0.857	0.850			
Concentration		0.000	79.165	69.732	61.433	45.041	42.928	42.567			
% Adsorbed		0.000	20.835	30.268	38.567	54.959	57.072	57.433	0.000	20.835	10.418
Average	810	0.000	17.828	25.423	32.897	48.825	53.498	53.945	0.000	17.828	8.914

Table F.1.11: Phenol adsorption results (600°C, 700°C, 800°C, 2 hr)

		Calculated data							Initial adsorption (dC/dt)		
time (min)		0	2	5	10	30	50	60	C _i	C _{i+dt}	dC/dt
WS4621	621	0.000	1.799	1.790	1.787	1.786	1.786	1.786			
Concentration		0.000	91.485	91.021	90.866	90.814	90.814	90.814			
% Adsorbed		0.000	8.515	8.979	9.134	9.186	9.186	9.186	0.000	8.515	4.258
WS4622	622	0.000	1.787	1.780	1.761	1.756	1.756	1.756			
Concentration		0.000	90.866	90.505	89.526	89.268	89.268	89.268			
% Adsorbed		0.000	9.134	9.495	10.474	10.732	10.732	10.732	0.000	9.134	4.567
WS4623	623	0.000	1.793	1.759	1.758	1.755	1.755	1.755			
Concentration		0.000	91.175	89.423	89.371	89.216	89.216	89.216			
% Adsorbed		0.000	8.825	10.577	10.629	10.784	10.784	10.784	0.000	8.825	4.412
Average	620	0.000	8.825	9.684	10.079	10.234	10.234	10.234	0.000	8.825	4.412
WS4721	721	0.000	1.764	1.722	1.685	1.614	1.591	1.586			
Concentration		0.000	89.680	87.515	85.608	81.948	80.763	80.505			
% Adsorbed		0.000	10.320	12.485	14.392	18.052	19.237	19.495	0.000	10.320	5.160
WS4722	722	0.000	1.728	1.701	1.671	1.610	1.576	1.569			
Concentration		0.000	87.825	86.433	84.887	81.742	79.990	79.629			
% Adsorbed		0.000	12.175	13.567	15.113	18.258	20.010	20.371	0.000	12.175	6.088
WS4723	723	0.000	1.724	1.695	1.681	1.627	1.584	1.577			
Concentration		0.000	87.619	86.124	85.402	82.619	80.402	80.041			
% Adsorbed		0.000	12.381	13.876	14.598	17.381	19.598	19.959	0.000	12.381	6.191
Average	720	0.000	11.625	13.309	14.701	17.897	19.615	19.942	0.000	11.625	5.813
WS4821	821	0.000	1.330	0.855	0.509	0.128	0.089	0.088			
Concentration		0.000	67.309	42.825	24.990	5.351	3.340	3.289			
% Adsorbed		0.000	32.691	57.175	75.010	94.649	96.660	96.711	0.000	32.691	16.345
WS4822	822	0.000	1.281	0.840	0.518	0.141	0.081	0.071			
Concentration		0.000	64.784	42.052	25.454	6.021	2.928	2.412			
% Adsorbed		0.000	35.216	57.948	74.546	93.979	97.072	97.588	0.000	35.216	17.608
WS4823	823	0.000	1.274	0.915	0.566	0.165	0.116	0.105			
Concentration		0.000	64.423	45.918	27.928	7.258	4.732	4.165			
% Adsorbed		0.000	35.577	54.082	72.072	92.742	95.268	95.835	0.000	35.577	17.789
Average	820	0.000	34.495	56.402	73.876	93.790	96.333	96.711	0.000	34.495	17.247

Table F.1.12: Phenol adsorption results (600°C, 700°C, 800°C, 3 hr)

		Calculated data							Initial adsorption (dC/dt)		
time (min)		0	2	5	10	30	50	60	C _i	C _∞	dC/dt
WS4631	631	0.000	1.799	1.770	1.731	1.729	1.729	1.729			
Concentration		0.000	91.485	89.990	87.979	87.876	87.876	87.876			
% Adsorbed		0.000	8.515	10.010	12.021	12.124	12.124	12.124	0.000	8.515	4.258
WS4632	632	0.000	1.731	1.721	1.703	1.697	1.697	1.697			
Concentration		0.000	87.979	87.464	86.536	86.227	86.227	86.227			
% Adsorbed		0.000	12.021	12.536	13.464	13.773	13.773	13.773	0.000	12.021	6.010
WS4633	633	0.000	1.711	1.688	1.663	1.658	1.658	1.658			
Concentration		0.000	86.948	85.763	84.474	84.216	84.216	84.216			
% Adsorbed		0.000	13.052	14.237	15.526	15.784	15.784	15.784	0.000	13.052	6.526
Average	630	0.000	11.196	12.261	13.670	13.893	13.893	13.893	0.000	11.196	5.598
WS4731	731	0.000	1.723	1.689	1.651	1.490	1.340	1.321			
Concentration		0.000	87.567	85.814	83.856	75.557	67.825	66.845			
% Adsorbed		0.000	12.433	14.186	16.144	24.443	32.175	33.155	0.000	12.433	6.216
WS4732	732	0.000	1.688	1.658	1.604	1.424	1.300	1.283			
Concentration		0.000	85.763	84.216	81.433	72.155	65.763	64.887			
% Adsorbed		0.000	14.237	15.784	18.567	27.845	34.237	35.113	0.000	14.237	7.119
WS4733	733	0.000	1.694	1.656	1.615	1.455	1.264	1.245			
Concentration		0.000	86.072	84.113	82.000	73.753	63.907	62.928			
% Adsorbed		0.000	13.928	15.887	18.000	26.247	36.093	37.072	0.000	13.928	6.964
Average	730	0.000	13.533	15.285	17.570	26.179	34.168	35.113	0.000	13.533	6.766
WS4831	831	0.000	1.101	0.602	0.300	0.051	0.020	0.018			
Concentration		0.000	55.505	29.784	14.216	1.381	0.000	0.000			
% Adsorbed		0.000	44.495	70.216	85.784	98.619	100.000	100.000	0.000	44.495	22.247
WS4832	832	0.000	1.087	0.574	0.257	0.044	0.018	0.015			
Concentration		0.000	54.784	28.340	12.000	1.021	0.000	0.000			
% Adsorbed		0.000	45.216	71.660	88.000	98.979	100.000	100.000	0.000	45.216	22.608
WS4833	833	0.000	1.150	0.662	0.290	0.068	0.029	0.026			
Concentration		0.000	58.031	32.876	13.701	2.258	0.247	0.093			
% Adsorbed		0.000	41.969	67.124	86.299	97.742	99.753	99.907	0.000	41.969	20.985
Average	830	0.000	43.893	69.667	86.694	98.447	99.918	99.969	0.000	43.893	21.947

F.2 NEW VAAL SAMPLES

F.2.1 pH results

Refer to **Appendix E: Sample calculations** for the detail calculations regarding the pH measurements (Pages 119 – 120).

The acronym used (NV821) in the data tables below refers to the first sample of the activated carbons from New Vaal coal, prepared at 800°C and 2 hours.

The calculated results for the pH measurement results are shown in Table F.2.1 – F.2.6.

Table F.2.1: Calculated pH derivatives (600°C, 2 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	NV621	NV622	NV623	NV621	NV622	NV623	NV621	NV622	NV623
0	6.909	7.015	7.126	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	9.758	9.732	10.384	2.849	2.717	3.258	0.5698	0.5434	0.6516
10	10.078	10.087	10.630	0.320	0.355	0.246	0.0640	0.0710	0.0492
15	10.202	10.259	10.738	0.124	0.172	0.108	0.0248	0.0344	0.0216
20	10.262	10.351	10.800	0.060	0.092	0.062	0.0120	0.0184	0.0124
25	10.317	10.411	10.839	0.055	0.060	0.039	0.0110	0.0120	0.0078
30	10.356	10.443	10.862	0.039	0.032	0.023	0.0078	0.0064	0.0046
35	10.386	10.474	10.876	0.030	0.031	0.014	0.0060	0.0062	0.0028
40	10.414	10.501	10.881	0.028	0.027	0.005	0.0056	0.0054	0.0010
45	10.450	10.520	10.879	0.036	0.019	-0.002	0.0072	0.0038	-0.0004
50	10.483	10.527	10.883	0.033	0.007	0.004	0.0066	0.0014	0.0008
55	10.489	10.527	10.870	0.006	0.000	-0.013	0.0012	0.0000	-0.0026
60	10.490	10.526	10.865	0.001	-0.001	-0.005	0.0002	-0.0002	-0.0010
65	10.506	10.522	10.856	0.016	-0.004	-0.009	0.0032	-0.0008	-0.0018
70	10.510	10.517	10.842	0.004	-0.005	-0.014	0.0008	-0.0010	-0.0028
75	10.508	10.513	10.830	-0.002	-0.004	-0.012	-0.0004	-0.0008	-0.0024
80	10.512	10.504	10.817	0.004	-0.009	-0.013	0.0008	-0.0018	-0.0026
85	10.508	10.490	10.798	-0.004	-0.014	-0.019	-0.0008	-0.0028	-0.0038
90	10.503	10.480	10.780	-0.005	-0.010	-0.018	-0.0010	-0.0020	-0.0036
95	10.494	10.464	10.759	-0.009	-0.016	-0.021	-0.0018	-0.0032	-0.0042
100	10.492	10.450	10.738	-0.002	-0.014	-0.021	-0.0004	-0.0028	-0.0042
105	10.485	10.432	10.711	-0.007	-0.018	-0.027	-0.0014	-0.0036	-0.0054
110	10.481	10.418	10.683	-0.004	-0.014	-0.028	-0.0008	-0.0028	-0.0056
115	10.478	10.400	10.653	-0.003	-0.018	-0.030	-0.0006	-0.0036	-0.0060
120	10.469	10.379	10.621	-0.009	-0.021	-0.032	-0.0018	-0.0042	-0.0064
125	10.464	10.356	10.588	-0.005	-0.023	-0.033	-0.0010	-0.0046	-0.0066
130	10.455	10.335	10.550	-0.009	-0.021	-0.038	-0.0018	-0.0042	-0.0076
135	10.448	10.312	10.513	-0.007	-0.023	-0.037	-0.0014	-0.0046	-0.0074
140	10.460	10.289	10.473	0.012	-0.023	-0.040	0.0024	-0.0046	-0.0080
145	10.444	10.269	10.432	-0.016	-0.020	-0.041	-0.0032	-0.0040	-0.0082
150	10.439	10.243	10.393	-0.005	-0.026	-0.039	-0.0010	-0.0052	-0.0078
155	10.436	10.220	10.352	-0.003	-0.023	-0.041	-0.0006	-0.0046	-0.0082
160	10.432	10.195	10.308	-0.004	-0.025	-0.044	-0.0008	-0.0050	-0.0088
165	10.425	10.170	10.269	-0.007	-0.025	-0.039	-0.0014	-0.0050	-0.0078
170	10.418	10.144	10.230	-0.007	-0.026	-0.039	-0.0014	-0.0052	-0.0078
175	10.416	10.121	10.192	-0.002	-0.023	-0.038	-0.0004	-0.0046	-0.0076
180	10.411	10.094	10.151	-0.005	-0.027	-0.041	-0.0010	-0.0054	-0.0082
185	10.405	10.068	10.112	-0.006	-0.026	-0.039	-0.0012	-0.0052	-0.0078
190	10.400	10.040	10.071	-0.005	-0.028	-0.041	-0.0010	-0.0056	-0.0082
195	10.397	10.015	10.034	-0.003	-0.025	-0.037	-0.0006	-0.0050	-0.0074
200	10.39	9.986	9.999	-0.007	-0.029	-0.035	-0.0014	-0.0058	-0.007
205	10.382	9.96	9.963	-0.008	-0.026	-0.036	-0.0016	-0.0052	-0.0072
210	10.377	9.932	9.93	-0.005	-0.028	-0.033	-0.001	-0.0056	-0.0066
215	10.37	9.907	9.895	-0.007	-0.025	-0.035	-0.0014	-0.005	-0.007
220	10.361	9.879	9.861	-0.009	-0.028	-0.034	-0.0018	-0.0056	-0.0068
225	10.352	9.852	9.831	-0.009	-0.027	-0.03	-0.0018	-0.0054	-0.006
230	10.344	9.824	9.797	-0.008	-0.028	-0.034	-0.0016	-0.0056	-0.0068
235	10.335	9.799	9.765	-0.009	-0.025	-0.032	-0.0018	-0.005	-0.0064

Table F.2.2: Calculated pH derivatives (700°C, 2 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	NV721	NV722	NV723	NV724	NV722	NV723	NV721	NV722	NV723
0	7.059	7.052	7.077	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	8.787	9.338	10.349	1.728	2.286	3.272	0.3456	0.4572	0.6544
10	9.419	9.942	10.660	0.632	0.604	0.311	0.1264	0.1208	0.0622
15	9.701	10.108	10.766	0.282	0.166	0.106	0.0564	0.0332	0.0212
20	9.893	10.238	10.851	0.192	0.130	0.085	0.0384	0.0260	0.0170
25	10.054	10.363	10.920	0.161	0.125	0.069	0.0322	0.0250	0.0138
30	10.180	10.457	10.958	0.126	0.094	0.038	0.0252	0.0188	0.0076
35	10.275	10.537	10.981	0.095	0.080	0.023	0.0190	0.0160	0.0046
40	10.334	10.587	10.996	0.059	0.050	0.015	0.0118	0.0100	0.0030
45	10.357	10.617	11.000	0.023	0.030	0.004	0.0046	0.0060	0.0008
50	10.355	10.629	10.996	-0.002	0.012	-0.004	-0.0004	0.0024	-0.0008
55	10.344	10.632	10.988	-0.011	0.003	-0.008	-0.0022	0.0006	-0.0016
60	10.351	10.632	10.976	0.007	0.000	-0.012	0.0014	0.0000	-0.0024
65	10.357	10.623	10.958	0.006	-0.009	-0.018	0.0012	-0.0018	-0.0036
70	10.362	10.601	10.939	0.005	-0.022	-0.019	0.0010	-0.0044	-0.0038
75	10.369	10.573	10.917	0.007	-0.028	-0.022	0.0014	-0.0056	-0.0044
80	10.387	10.542	10.887	0.018	-0.031	-0.030	0.0036	-0.0062	-0.0060
85	10.405	10.506	10.866	0.018	-0.036	-0.021	0.0036	-0.0072	-0.0042
90	10.440	10.469	10.830	0.035	-0.037	-0.036	0.0070	-0.0074	-0.0072
95	10.481	10.434	10.800	0.041	-0.035	-0.030	0.0082	-0.0070	-0.0060
100	10.497	10.395	10.759	0.016	-0.039	-0.041	0.0032	-0.0078	-0.0082
105	10.507	10.356	10.733	0.010	-0.039	-0.026	0.0020	-0.0078	-0.0052
110	10.545	10.322	10.681	0.038	-0.034	-0.052	0.0076	-0.0068	-0.0104
115	10.552	10.287	10.637	0.007	-0.035	-0.044	0.0014	-0.0070	-0.0088
120	10.476	10.254	10.580	-0.076	-0.033	-0.057	-0.0152	-0.0066	-0.0114
125	10.429	10.223	10.539	-0.047	-0.031	-0.041	-0.0094	-0.0062	-0.0082
130	10.394	10.192	10.486	-0.035	-0.031	-0.053	-0.0070	-0.0062	-0.0106
135	10.382	10.162	10.436	-0.012	-0.030	-0.050	-0.0024	-0.0060	-0.0100
140	10.374	10.134	10.398	-0.008	-0.028	-0.038	-0.0016	-0.0056	-0.0076
145	10.357	10.107	10.377	-0.017	-0.027	-0.021	-0.0034	-0.0054	-0.0042
150	10.343	10.079	10.349	-0.014	-0.028	-0.028	-0.0028	-0.0056	-0.0056
155	10.325	10.055	10.320	-0.018	-0.024	-0.029	-0.0036	-0.0048	-0.0058
160	10.302	10.030	10.287	-0.023	-0.025	-0.033	-0.0046	-0.0050	-0.0066
165	10.281	10.004	10.242	-0.021	-0.026	-0.045	-0.0042	-0.0052	-0.0090
170	10.256	9.982	10.190	-0.025	-0.022	-0.052	-0.0050	-0.0044	-0.0104
175	10.233	9.954	10.138	-0.023	-0.028	-0.052	-0.0046	-0.0056	-0.0104
180	10.210	9.930	10.089	-0.023	-0.024	-0.049	-0.0046	-0.0048	-0.0098
185	10.190	9.907	10.046	-0.020	-0.023	-0.043	-0.0040	-0.0046	-0.0086
190	10.171	9.885	10.003	-0.019	-0.022	-0.043	-0.0038	-0.0044	-0.0086
195	10.148	9.860	9.964	-0.023	-0.025	-0.039	-0.0046	-0.0050	-0.0078
200	10.130	9.838	9.931	-0.018	-0.022	-0.033	-0.0036	-0.0044	-0.0066
205	10.109	9.815	9.900	-0.021	-0.023	-0.031	-0.0042	-0.0046	-0.0062
210	10.089	9.791	9.871	-0.02	-0.024	-0.029	-0.004	-0.0048	-0.0058
215	10.068	9.768	9.843	-0.021	-0.023	-0.028	-0.0042	-0.0046	-0.0056
220	10.045	9.748	9.817	-0.023	-0.02	-0.026	-0.0046	-0.004	-0.0052
225	10.025	9.727	9.794	-0.02	-0.021	-0.023	-0.004	-0.0042	-0.0046
230	10.004	9.704	9.772	-0.021	-0.023	-0.022	-0.0042	-0.0046	-0.0044
235	9.983	9.682	9.746	-0.021	-0.022	-0.026	-0.0042	-0.0044	-0.0052

Table F.2.3: Calculated pH derivatives (800°C, 2 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time	NV821	NV822	NV823	NV821	NV822	NV823	NV821	NV822	NV823
0	7.056	7.034	7.094	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	9.588	10.803	10.432	2.532	3.769	3.338	0.5064	0.7538	0.6676
10	9.969	11.078	10.732	0.381	0.275	0.300	0.0762	0.0550	0.0600
15	10.062	11.207	10.833	0.093	0.129	0.101	0.0186	0.0258	0.0202
20	10.086	11.270	10.873	0.024	0.063	0.040	0.0048	0.0126	0.0080
25	10.105	11.304	10.887	0.019	0.034	0.014	0.0038	0.0068	0.0028
30	10.123	11.353	10.890	0.018	0.049	0.003	0.0036	0.0098	0.0006
35	10.135	11.376	10.888	0.012	0.023	-0.002	0.0024	0.0046	-0.0004
40	10.147	11.386	10.880	0.012	0.010	-0.008	0.0024	0.0020	-0.0016
45	10.150	11.404	10.869	0.003	0.018	-0.011	0.0006	0.0036	-0.0022
50	10.159	11.405	10.862	0.009	0.001	-0.007	0.0018	0.0002	-0.0014
55	10.159	11.404	10.850	0.000	-0.001	-0.012	0.0000	-0.0002	-0.0024
60	10.161	11.402	10.838	0.002	-0.002	-0.012	0.0004	-0.0004	-0.0024
65	10.161	11.397	10.824	0.000	-0.005	-0.014	0.0000	-0.0010	-0.0028
70	10.145	11.388	10.803	-0.016	-0.009	-0.021	-0.0032	-0.0018	-0.0042
75	10.137	11.386	10.782	-0.008	-0.002	-0.021	-0.0016	-0.0004	-0.0042
80	10.121	11.379	10.758	-0.016	-0.007	-0.024	-0.0032	-0.0014	-0.0048
85	10.100	11.369	10.737	-0.021	-0.010	-0.021	-0.0042	-0.0020	-0.0042
90	10.119	11.358	10.713	0.019	-0.011	-0.024	0.0038	-0.0022	-0.0048
95	10.135	11.346	10.687	0.016	-0.012	-0.026	0.0032	-0.0024	-0.0052
100	10.130	11.329	10.664	-0.005	-0.017	-0.023	-0.0010	-0.0034	-0.0046
105	10.131	11.313	10.640	0.001	-0.016	-0.024	0.0002	-0.0032	-0.0048
110	10.135	11.299	10.612	0.004	-0.014	-0.028	0.0008	-0.0028	-0.0056
115	10.147	11.285	10.586	0.012	-0.014	-0.026	0.0024	-0.0028	-0.0052
120	10.156	11.268	10.558	0.009	-0.017	-0.028	0.0018	-0.0034	-0.0056
125	10.168	11.250	10.532	0.012	-0.018	-0.026	0.0024	-0.0036	-0.0052
130	10.175	11.235	10.509	0.007	-0.015	-0.023	0.0014	-0.0030	-0.0046
135	10.173	11.214	10.483	-0.002	-0.021	-0.026	-0.0004	-0.0042	-0.0052
140	10.170	11.200	10.457	-0.003	-0.014	-0.026	-0.0006	-0.0028	-0.0052
145	10.164	11.184	10.436	-0.006	-0.016	-0.021	-0.0012	-0.0032	-0.0042
150	10.157	11.165	10.408	-0.007	-0.019	-0.028	-0.0014	-0.0038	-0.0056
155	10.149	11.148	10.382	-0.008	-0.017	-0.026	-0.0016	-0.0034	-0.0052
160	10.138	11.134	10.361	-0.011	-0.014	-0.021	-0.0022	-0.0028	-0.0042
165	10.130	11.115	10.340	-0.008	-0.019	-0.021	-0.0016	-0.0038	-0.0042
170	10.130	11.095	10.318	0.000	-0.020	-0.022	0.0000	-0.0040	-0.0044
175	10.126	11.076	10.297	-0.004	-0.019	-0.021	-0.0008	-0.0038	-0.0042
180	10.121	11.059	10.274	-0.005	-0.017	-0.023	-0.0010	-0.0034	-0.0046
185	10.119	11.038	10.255	-0.002	-0.021	-0.019	-0.0004	-0.0042	-0.0038
190	10.110	11.017	10.234	-0.009	-0.021	-0.021	-0.0018	-0.0042	-0.0042
195	10.103	10.998	10.217	-0.007	-0.019	-0.017	-0.0014	-0.0038	-0.0034
200	10.097	10.977	10.197	-0.006	-0.021	-0.020	-0.0012	-0.0042	-0.0040
205	10.088	10.955	10.180	-0.009	-0.022	-0.017	-0.0018	-0.0044	-0.0034
210	10.079	10.934	10.161	-0.009	-0.021	-0.019	-0.0018	-0.0042	-0.0038
215	10.072	10.914	10.140	-0.007	-0.020	-0.021	-0.0014	-0.0040	-0.0042
220	10.065	10.890	10.121	-0.007	-0.024	-0.019	-0.0014	-0.0048	-0.0038
225	10.055	10.868	10.103	-0.010	-0.022	-0.018	-0.0020	-0.0044	-0.0036
230	10.043	10.845	10.088	-0.012	-0.023	-0.015	-0.0024	-0.0046	-0.0030
235	10.032	10.817	10.069	-0.011	-0.028	-0.019	-0.0022	-0.0056	-0.0038

Table F.2.4: Calculated pH derivatives (600°C, 3 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
<i>time</i>	NV631	NV632	NV633	NV631	NV632	NV633	NV631	NV632	NV633
0	6.873	7.096	7.516	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	7.615	6.303	8.356	0.742	-0.793	0.840	0.1484	-0.1586	0.1680
10	8.114	8.711	8.623	0.499	2.408	0.267	0.0998	0.4816	0.0534
15	8.248	9.387	8.788	0.134	0.676	0.165	0.0268	0.1352	0.0330
20	8.302	9.533	8.873	0.054	0.146	0.085	0.0108	0.0292	0.0170
25	8.316	9.604	8.915	0.014	0.071	0.042	0.0028	0.0142	0.0084
30	8.325	9.627	8.968	0.009	0.023	0.053	0.0018	0.0046	0.0106
35	8.330	9.634	8.988	0.005	0.007	0.020	0.0010	0.0014	0.0040
40	8.316	9.627	9.004	-0.014	-0.007	0.016	-0.0028	-0.0014	0.0032
45	8.314	9.575	9.007	-0.002	-0.052	0.003	-0.0004	-0.0104	0.0006
50	8.316	9.546	9.014	0.002	-0.029	0.007	0.0004	-0.0058	0.0014
55	8.309	9.434	9.036	-0.007	-0.112	0.022	-0.0014	-0.0224	0.0044
60	8.313	9.323	9.036	0.004	-0.111	0.000	0.0008	-0.0222	0.0000
65	8.311	9.214	9.031	-0.002	-0.109	-0.005	-0.0004	-0.0218	-0.0010
70	8.311	9.090	9.019	0.000	-0.124	-0.012	0.0000	-0.0248	-0.0024
75	8.347	8.967	9.014	0.036	-0.123	-0.005	0.0072	-0.0246	-0.0010
80	8.350	8.960	9.019	0.003	-0.007	0.005	0.0006	-0.0014	0.0010
85	8.397	9.000	9.017	0.047	0.040	-0.002	0.0094	0.0080	-0.0004
90	8.416	9.011	9.005	0.019	0.011	-0.012	0.0038	0.0022	-0.0024
95	8.406	9.072	8.993	-0.010	0.061	-0.012	-0.0020	0.0122	-0.0024
100	8.390	9.113	8.978	-0.016	0.041	-0.015	-0.0032	0.0082	-0.0030
105	8.388	9.068	8.961	-0.002	-0.045	-0.017	-0.0004	-0.0090	-0.0034
110	8.407	8.958	8.944	0.019	-0.110	-0.017	0.0038	-0.0220	-0.0034
115	8.395	8.847	8.925	-0.012	-0.111	-0.019	-0.0024	-0.0222	-0.0038
120	8.384	8.782	8.904	-0.011	-0.065	-0.021	-0.0022	-0.0130	-0.0042
125	8.356	8.793	8.885	-0.028	0.011	-0.019	-0.0056	0.0022	-0.0038
130	8.322	8.802	8.870	-0.034	0.009	-0.015	-0.0068	0.0018	-0.0030
135	8.280	8.816	8.856	-0.042	0.014	-0.014	-0.0084	0.0028	-0.0028
140	8.234	8.733	8.841	-0.046	-0.083	-0.015	-0.0092	-0.0166	-0.0030
145	8.191	8.628	8.829	-0.043	-0.105	-0.012	-0.0086	-0.0210	-0.0024
150	8.128	8.549	8.813	-0.063	-0.079	-0.016	-0.0126	-0.0158	-0.0032
155	8.087	8.484	8.796	-0.041	-0.065	-0.017	-0.0082	-0.0130	-0.0034
160	8.069	8.445	8.781	-0.018	-0.039	-0.015	-0.0036	-0.0078	-0.0030
165	8.062	8.418	8.769	-0.007	-0.027	-0.012	-0.0014	-0.0054	-0.0024
170	8.048	8.407	8.755	-0.014	-0.011	-0.014	-0.0028	-0.0022	-0.0028
175	8.035	8.391	8.743	-0.013	-0.016	-0.012	-0.0026	-0.0032	-0.0024
180	8.014	8.354	8.731	-0.021	-0.037	-0.012	-0.0042	-0.0074	-0.0024
185	8.017	8.331	8.717	0.003	-0.023	-0.014	0.0006	-0.0046	-0.0028
190	8.028	8.309	8.707	0.011	-0.022	-0.010	0.0022	-0.0044	-0.0020
195	8.012	8.291	8.695	-0.016	-0.018	-0.012	-0.0032	-0.0036	-0.0024
200	7.999	8.300	8.685	-0.013	0.009	-0.010	-0.0026	0.0018	-0.0020
205	7.998	8.282	8.676	-0.001	-0.018	-0.009	-0.0002	-0.0036	-0.0018
210	7.998	8.273	8.668	0.000	-0.009	-0.008	0.0000	-0.0018	-0.0016
215	7.998	8.272	8.658	0.000	-0.001	-0.010	0.0000	-0.0002	-0.0020
220	7.990	8.263	8.647	-0.008	-0.009	-0.011	-0.0016	-0.0018	-0.0022
225	7.987	8.261	8.637	-0.003	-0.002	-0.010	-0.0006	-0.0004	-0.0020
230	7.978	8.266	8.627	-0.009	0.005	-0.010	-0.0018	0.0010	-0.0020
235	7.980	8.261	8.615	0.002	-0.005	-0.012	0.0004	-0.0010	-0.0024

Table F.2.5: Calculated pH derivatives (700°C, 3 hr)

	Raw data			$\Delta pH = pH_{(i+1)} - pH_{(i)}$			$\frac{dpH}{dt} = \frac{pH_{(i+1)} - pH_{(i)}}{\Delta t}$		
time (min)	NV731	NV732	NV733	NV731	NV732	NV733	NV731	NV732	NV733
0	7.050	7.012	6.809	0.000	0.000	0.000	0.0000	0.0000	0.0000
5	9.123	7.081	9.460	2.073	0.069	2.651	0.4146	0.0138	0.5302
10	9.431	8.408	9.889	0.308	1.327	0.429	0.0616	0.2654	0.0858
15	9.560	8.781	10.030	0.129	0.373	0.141	0.0258	0.0746	0.0282
20	9.638	8.947	10.086	0.078	0.166	0.056	0.0156	0.0332	0.0112
25	9.698	9.024	10.149	0.060	0.077	0.063	0.0120	0.0154	0.0126
30	9.755	9.083	10.185	0.057	0.059	0.036	0.0114	0.0118	0.0072
35	9.815	9.083	10.225	0.060	0.000	0.040	0.0120	0.0000	0.0080
40	9.848	9.109	10.250	0.033	0.026	0.025	0.0066	0.0052	0.0050
45	9.841	9.094	10.262	-0.007	-0.015	0.012	-0.0014	-0.0030	0.0024
50	9.832	9.030	10.279	-0.009	-0.064	0.017	-0.0018	-0.0128	0.0034
55	9.818	8.981	10.300	-0.014	-0.049	0.021	-0.0028	-0.0098	0.0042
60	9.824	8.969	10.319	0.006	-0.012	0.019	0.0012	-0.0024	0.0038
65	9.812	8.916	10.338	-0.012	-0.053	0.019	-0.0024	-0.0106	0.0038
70	9.798	8.889	10.345	-0.014	-0.027	0.007	-0.0028	-0.0054	0.0014
75	9.793	8.837	10.366	-0.005	-0.052	0.021	-0.0010	-0.0104	0.0042
80	9.772	8.786	10.378	-0.021	-0.051	0.012	-0.0042	-0.0102	0.0024
85	9.748	8.728	10.391	-0.024	-0.058	0.013	-0.0048	-0.0116	0.0026
90	9.719	8.680	10.387	-0.029	-0.048	-0.004	-0.0058	-0.0096	-0.0008
95	9.686	8.634	10.372	-0.033	-0.046	-0.015	-0.0066	-0.0092	-0.0030
100	9.660	8.586	10.359	-0.026	-0.048	-0.013	-0.0052	-0.0096	-0.0026
105	9.633	8.547	10.344	-0.027	-0.039	-0.015	-0.0054	-0.0078	-0.0030
110	9.608	8.520	10.323	-0.025	-0.027	-0.021	-0.0050	-0.0054	-0.0042
115	9.586	8.509	10.312	-0.022	-0.011	-0.011	-0.0044	-0.0022	-0.0022
120	9.553	8.468	10.286	-0.033	-0.041	-0.026	-0.0066	-0.0082	-0.0052
125	9.526	8.446	10.265	-0.027	-0.022	-0.021	-0.0054	-0.0044	-0.0042
130	9.500	8.434	10.236	-0.026	-0.012	-0.029	-0.0052	-0.0024	-0.0058
135	9.474	8.424	10.210	-0.026	-0.010	-0.026	-0.0052	-0.0020	-0.0052
140	9.448	8.408	10.182	-0.026	-0.016	-0.028	-0.0052	-0.0032	-0.0056
145	9.424	8.400	10.152	-0.024	-0.008	-0.030	-0.0048	-0.0016	-0.0060
150	9.398	8.383	10.123	-0.026	-0.017	-0.029	-0.0052	-0.0034	-0.0058
155	9.376	8.362	10.098	-0.022	-0.021	-0.025	-0.0044	-0.0042	-0.0050
160	9.354	8.352	10.069	-0.022	-0.010	-0.029	-0.0044	-0.0020	-0.0058
165	9.333	8.373	10.044	-0.021	0.021	-0.025	-0.0042	0.0042	-0.0050
170	9.311	8.378	10.020	-0.022	0.005	-0.024	-0.0044	0.0010	-0.0048
175	9.287	8.381	9.996	-0.024	0.003	-0.024	-0.0048	0.0006	-0.0048
180	9.259	8.381	9.969	-0.028	0.000	-0.027	-0.0056	0.0000	-0.0054
185	9.233	8.385	9.947	-0.026	0.004	-0.022	-0.0052	0.0008	-0.0044
190	9.216	8.383	9.923	-0.017	-0.002	-0.024	-0.0034	-0.0004	-0.0048
195	9.195	8.386	9.902	-0.021	0.003	-0.021	-0.0042	0.0006	-0.0042
200	9.178	8.383	9.881	-0.017	-0.003	-0.021	-0.0034	-0.0006	-0.0042
205	9.161	8.383	9.863	-0.017	0.000	-0.018	-0.0034	0.0000	-0.0036
210	9.138	8.378	9.846	-0.023	-0.005	-0.017	-0.0046	-0.0010	-0.0034
215	9.120	8.374	9.825	-0.018	-0.004	-0.021	-0.0036	-0.0008	-0.0042
220	9.101	8.374	9.811	-0.019	0.000	-0.014	-0.0038	0.0000	-0.0028
225	9.082	8.376	9.794	-0.019	0.002	-0.017	-0.0038	0.0004	-0.0034
230	9.063	8.373	9.776	-0.019	-0.003	-0.018	-0.0038	-0.0006	-0.0036
235	9.044	8.373	9.761	-0.019	0.000	-0.015	-0.0038	0.0000	-0.0030

Table F.2.6: Calculated pH derivatives (800°C, 3 hr)

	Raw data			$\Delta pH = pH_{(t+\Delta t)} - pH_{(t)}$			$\frac{dpH}{dt} = \frac{pH_{(t+\Delta t)} - pH_{(t)}}{\Delta t}$		
time (min)	NV831	NV832	NV833	NV831	NV832	NV833	NV831	NV832	NV833
0	7.013	7.090	7.007	0.000	0.000	0.000	0.000	0.000	0.000
5	10.878	7.441	10.215	3.865	0.351	3.208	0.7730	0.0702	0.6416
10	11.188	9.679	10.435	0.310	2.238	0.220	0.0620	0.4476	0.0440
15	11.312	9.884	10.509	0.124	0.205	0.074	0.0248	0.0410	0.0148
20	11.358	9.937	10.564	0.046	0.053	0.055	0.0092	0.0106	0.0110
25	11.358	9.949	10.588	0.000	0.012	0.024	0.0000	0.0024	0.0048
30	11.351	9.956	10.609	-0.007	0.007	0.021	-0.0014	0.0014	0.0042
35	11.327	9.972	10.614	-0.024	0.016	0.005	-0.0048	0.0032	0.0010
40	11.312	9.937	10.604	-0.015	-0.035	-0.010	-0.0030	-0.0070	-0.0020
45	11.296	9.934	10.611	-0.016	-0.003	0.007	-0.0032	-0.0006	0.0014
50	11.279	9.922	10.602	-0.017	-0.012	-0.009	-0.0034	-0.0024	-0.0018
55	11.245	9.903	10.585	-0.034	-0.019	-0.017	-0.0068	-0.0038	-0.0034
60	11.220	9.886	10.568	-0.025	-0.017	-0.017	-0.0050	-0.0034	-0.0034
65	11.195	9.839	10.571	-0.025	-0.047	0.003	-0.0050	-0.0094	0.0006
70	11.167	9.799	10.571	-0.028	-0.040	0.000	-0.0056	-0.0080	0.0000
75	11.139	9.753	10.566	-0.028	-0.046	-0.005	-0.0056	-0.0092	-0.0010
80	11.115	9.708	10.556	-0.024	-0.045	-0.010	-0.0048	-0.0090	-0.0020
85	11.084	9.675	10.542	-0.031	-0.033	-0.014	-0.0062	-0.0066	-0.0028
90	11.053	9.644	10.525	-0.031	-0.031	-0.017	-0.0062	-0.0062	-0.0034
95	11.019	9.598	10.504	-0.034	-0.046	-0.021	-0.0068	-0.0092	-0.0042
100	10.986	9.562	10.482	-0.033	-0.036	-0.022	-0.0066	-0.0072	-0.0044
105	10.952	9.529	10.454	-0.034	-0.033	-0.028	-0.0068	-0.0066	-0.0056
110	10.912	9.493	10.426	-0.040	-0.036	-0.028	-0.0080	-0.0072	-0.0056
115	10.879	9.455	10.401	-0.033	-0.038	-0.025	-0.0066	-0.0076	-0.0050
120	10.840	9.419	10.378	-0.039	-0.036	-0.023	-0.0078	-0.0072	-0.0046
125	10.800	9.384	10.356	-0.040	-0.035	-0.022	-0.0080	-0.0070	-0.0044
130	10.774	9.340	10.330	-0.026	-0.044	-0.026	-0.0052	-0.0088	-0.0052
135	10.747	9.300	10.308	-0.027	-0.040	-0.022	-0.0054	-0.0080	-0.0044
140	10.721	9.271	10.287	-0.026	-0.029	-0.021	-0.0052	-0.0058	-0.0042
145	10.692	9.233	10.266	-0.029	-0.038	-0.021	-0.0058	-0.0076	-0.0042
150	10.659	9.202	10.249	-0.033	-0.031	-0.017	-0.0066	-0.0062	-0.0034
155	10.637	9.169	10.230	-0.022	-0.033	-0.019	-0.0044	-0.0066	-0.0038
160	10.602	9.143	10.213	-0.035	-0.026	-0.017	-0.0070	-0.0052	-0.0034
165	10.576	9.117	10.192	-0.026	-0.026	-0.021	-0.0052	-0.0052	-0.0042
170	10.547	9.083	10.177	-0.029	-0.034	-0.015	-0.0058	-0.0068	-0.0030
175	10.519	9.060	10.159	-0.028	-0.023	-0.018	-0.0056	-0.0046	-0.0036
180	10.490	9.035	10.144	-0.029	-0.025	-0.015	-0.0058	-0.0050	-0.0030
185	10.469	9.016	10.125	-0.021	-0.019	-0.019	-0.0042	-0.0038	-0.0038
190	10.442	8.995	10.108	-0.027	-0.021	-0.017	-0.0054	-0.0042	-0.0034
195	10.421	8.973	10.087	-0.021	-0.022	-0.021	-0.0042	-0.0044	-0.0042
200	10.397	8.950	10.070	-0.024	-0.023	-0.017	-0.0048	-0.0046	-0.0034
205	10.376	8.933	10.054	-0.021	-0.017	-0.016	-0.0042	-0.0034	-0.0032
210	10.356	8.914	10.034	-0.020	-0.019	-0.020	-0.0040	-0.0038	-0.0040
215	10.333	8.900	10.008	-0.023	-0.014	-0.026	-0.0046	-0.0028	-0.0052
220	10.310	8.883	9.989	-0.023	-0.017	-0.019	-0.0046	-0.0034	-0.0038
225	10.297	8.873	9.968	-0.013	-0.010	-0.021	-0.0026	-0.0020	-0.0042
230	10.289	8.857	9.949	-0.008	-0.018	-0.019	-0.0016	-0.0032	-0.0038
235	10.263	8.842	9.929	-0.026	-0.015	-0.020	-0.0052	-0.0030	-0.0040

F.2.2 Phenol adsorption results

Refer to **Appendix E: Sample calculations** for the detail calculations regarding the phenol adsorption results (Pages 120 – 121).

The calculated results for the phenol adsorption tests are shown in Table F.2.7 – F.2.8.

Table F.2.7: Phenol adsorption results (600°C, 700°C, 800°C, 2 hr)

		Calculated data							Initial adsorption (dC/dt)		
time (min)		0	2	5	10	30	50	60	C _i	C _{i+dt}	dC/dt
NV621	621	0.000	1.680	1.677	1.665	1.648	1.648	1.648			
Concentration		0.000	85.351	85.196	84.577	83.701	83.701	83.701			
% Adsorbed		0.000	14.649	14.804	15.423	16.299	16.299	16.299	0.000	14.649	7.325
NV622	622	0.000	1.742	1.714	1.672	1.660	1.660	1.660			
Concentration		0.000	88.546	87.103	84.938	84.320	84.320	84.320			
% Adsorbed		0.000	11.454	12.897	15.062	15.680	15.680	15.680	0.000	11.454	5.727
NV623	623	0.000	1.716	1.704	1.671	1.659	1.659	1.659			
Concentration		0.000	87.206	86.588	84.887	84.268	84.268	84.268			
% Adsorbed		0.000	12.794	13.412	15.113	15.732	15.732	15.732	0.000	12.794	6.397
Average	620	0.000	12.966	13.704	15.199	15.904	15.904	15.904	0.000	12.966	6.483
NV721	721	0.000	1.740	1.607	1.536	1.501	1.481	1.476			
Concentration		0.000	88.443	81.588	77.928	76.124	75.093	74.835			
% Adsorbed		0.000	11.557	18.412	22.072	23.876	24.907	25.165	0.000	11.557	5.778
NV722	722	0.000	1.663	1.576	1.463	1.492	1.442	1.428			
Concentration		0.000	84.474	79.990	74.165	75.660	73.082	72.361			
% Adsorbed		0.000	15.526	20.010	25.835	24.340	26.918	27.639	0.000	15.526	7.763
NV723	723	0.000	1.642	1.558	1.419	1.479	1.456	1.443			
Concentration		0.000	83.392	79.062	71.897	74.990	73.804	73.134			
% Adsorbed		0.000	16.608	20.938	28.103	25.010	26.196	26.866	0.000	16.608	8.304
Average	720	0.000	14.564	19.787	25.337	24.409	26.007	26.557	0.000	14.564	7.282
NV821	821	0.000	1.205	0.759	0.581	0.178	0.082	0.077			
Concentration		0.000	60.866	37.876	28.701	7.928	2.979	2.722			
% Adsorbed		0.000	39.134	62.124	71.299	92.072	97.021	97.278	0.000	39.134	19.567
NV822	822	0.000	1.091	0.796	0.534	0.251	0.113	0.090			
Concentration		0.000	54.990	39.784	26.278	11.691	4.577	3.392			
% Adsorbed		0.000	45.010	60.216	73.722	88.309	95.423	96.608	0.000	45.010	22.505
NV823	823	0.000	1.193	0.743	0.568	0.161	0.071	0.064			
Concentration		0.000	60.247	37.052	28.031	7.052	2.412	2.052			
% Adsorbed		0.000	39.753	62.948	71.969	92.948	97.588	97.948	0.000	39.753	19.876
Average	820	0.000	41.299	61.763	72.330	91.110	96.677	97.278	0.000	41.299	20.649

Table F.2.8: Phenol adsorption results (600°C, 700°C, 800°C, 3 hr)

		Calculated data							Initial adsorption (dC/dt)		
time (min)		0	2	5	10	30	50	60	C _i	C _{i+dt}	dC/dt
NV631	631	0.000	1.782	1.726	1.725	1.711	1.688	1.681			
Concentration		0.000	90.608	87.722	87.670	86.948	85.763	85.402			
% Adsorbed		0.000	9.392	12.278	12.330	13.052	14.237	14.598	0.000	9.392	4.696
NV632	632	0.000	1.755	1.727	1.713	1.677	1.634	1.629			
Concentration		0.000	89.216	87.773	87.052	85.196	82.979	82.722			
% Adsorbed		0.000	10.784	12.227	12.948	14.804	17.021	17.278	0.000	10.784	5.392
NV633	633	0.000	1.726	1.685	1.658	1.640	1.590	1.583			
Concentration		0.000	87.722	85.608	84.216	83.289	80.711	80.351			
% Adsorbed		0.000	12.278	14.392	15.784	16.711	19.289	19.649	0.000	12.278	6.139
Average	630	0.000	10.818	12.966	13.687	14.856	16.849	17.175	0.000	10.818	5.409
NV731	731	0.000	1.619	1.459	1.295	0.883	0.732	0.709			
Concentration		0.000	82.206	73.959	65.505	44.268	36.485	35.299			
% Adsorbed		0.000	17.794	26.041	34.495	55.732	63.515	64.701	0.000	17.794	8.897
NV732	732	0.000	1.573	1.450	1.336	0.983	0.788	0.771			
Concentration		0.000	79.835	73.495	67.619	49.423	39.371	38.495			
% Adsorbed		0.000	20.165	26.505	32.381	50.577	60.629	61.505	0.000	20.165	10.082
NV733	733	0.000	1.602	1.388	1.231	0.779	0.641	0.628			
Concentration		0.000	81.330	70.299	62.206	38.907	31.794	31.124			
% Adsorbed		0.000	18.670	29.701	37.794	61.093	68.206	68.876	0.000	18.670	9.335
Average	730	0.000	18.876	27.416	34.890	55.801	64.117	65.027	0.000	18.876	9.438
NV831	831	0.000	1.156	0.718	0.415	0.173	0.095	0.090			
Concentration		0.000	58.340	35.763	20.144	7.670	3.649	3.392			
% Adsorbed		0.000	41.660	64.237	79.856	92.330	96.351	96.608	0.000	41.660	20.830
NV832	832	0.000	1.212	0.780	0.439	0.147	0.099	0.089			
Concentration		0.000	61.227	38.959	21.381	6.330	3.856	3.340			
% Adsorbed		0.000	38.773	61.041	78.619	93.670	96.144	96.660	0.000	38.773	19.387
NV833	833	0.000	1.142	0.708	0.409	0.144	0.088	0.081			
Concentration		0.000	57.619	35.247	19.835	6.175	3.289	2.928			
% Adsorbed		0.000	42.381	64.753	80.165	93.825	96.711	97.072	0.000	42.381	21.191
Average	830	0.000	40.938	63.344	79.546	93.275	96.402	96.780	0.000	40.938	20.469

F.3 NORIT RO 0.8 SAMPLE

F.3.1 pH results

Refer to **Appendix E: Sample calculations** for the detail calculations regarding the pH measurements (Pages 119 – 120).

The calculated results for the pH measurements for Norit RO 0.8 are shown in Table F.3.1.

Table F.3.1: Calculated pH derivatives (Norit RO 0.8)

time (min)	Raw data		$\Delta pH = pH_{(t+1)} - pH_{(t)}$		$\frac{dpH}{dt} = \frac{pH_{(t+1)} - pH_{(t)}}{\Delta t}$	
	Norit RO 0.8_1	Norit RO 0.8_2	Norit RO 0.8_1	Norit RO 0.8_2	Norit RO 0.8_1	Norit RO 0.8_2
0	5.269	6.370	0.000	0.000	0.0000	0.0000
5	9.780	9.878	4.511	3.508	0.9022	0.7016
10	9.843	9.904	0.063	0.026	0.0126	0.0052
15	9.919	9.937	0.076	0.033	0.0152	0.0066
20	9.928	9.997	0.009	0.060	0.0018	0.0120
25	10.001	10.055	0.073	0.058	0.0146	0.0116
30	10.069	10.115	0.068	0.060	0.0136	0.0120
35	10.124	10.152	0.055	0.037	0.0110	0.0074
40	10.168	10.198	0.044	0.046	0.0088	0.0092
45	10.200	10.228	0.032	0.030	0.0064	0.0060
50	10.227	10.254	0.027	0.026	0.0054	0.0052
55	10.248	10.273	0.021	0.019	0.0042	0.0038
60	10.266	10.290	0.018	0.017	0.0036	0.0034
65	10.282	10.304	0.016	0.014	0.0032	0.0028
70	10.295	10.314	0.013	0.010	0.0026	0.0020
75	10.307	10.325	0.012	0.011	0.0024	0.0022
80	10.321	10.332	0.014	0.007	0.0028	0.0014
85	10.328	10.333	0.007	0.001	0.0014	0.0002
90	10.335	10.332	0.007	-0.001	0.0014	-0.0002
95	10.345	10.328	0.010	-0.004	0.0020	-0.0008
100	10.348	10.328	0.003	0.000	0.0006	0.0000
105	10.355	10.328	0.007	0.000	0.0014	0.0000
110	10.360	10.325	0.005	-0.003	0.0010	-0.0006
115	10.360	10.326	0.000	0.001	0.0000	0.0002
120	10.363	10.326	0.003	0.000	0.0006	0.0000
125	10.369	10.326	0.006	0.000	0.0012	0.0000
130	10.372	10.325	0.003	-0.001	0.0006	-0.0002
135	10.375	10.323	0.003	-0.002	0.0006	-0.0004
140	10.379	10.323	0.004	0.000	0.0008	0.0000
145	10.375	10.323	-0.004	0.000	-0.0008	0.0000
150	10.377	10.323	0.002	0.000	0.0004	0.0000
155	10.377	10.318	0.000	-0.005	0.0000	-0.0010
160	10.379	10.316	0.002	-0.002	0.0004	-0.0004
165	10.379	10.314	0.000	-0.002	0.0000	-0.0004
170	10.375	10.321	-0.004	0.007	-0.0008	0.0014
175	10.375	10.318	0.000	-0.003	0.0000	-0.0006
180	10.374	10.316	-0.001	-0.002	-0.0002	-0.0004
185	10.372	10.313	-0.002	-0.003	-0.0004	-0.0006
190	10.369	10.314	-0.003	0.001	-0.0006	0.0002
195	10.367	10.306	-0.002	-0.008	-0.0004	-0.0016

F.3.2 Phenol adsorption

Refer to **Appendix E: Sample calculations** for the detail calculations regarding the phenol adsorption results (Pages 120 – 121).

The calculated results for the phenol adsorption tests on the Norit RO 0.8 sample are shown in Table F.3.2.

Table F.3.2: Phenol adsorption results (Norit RO 0.8)

		Calculated data							Initial adsorption (dC/dt)		
time (min)		0	2	5	10	30	50	60	C _i	C _{eq}	dC/dt
Norit RO 0.8		0.000	0.887	0.277	0.059	0.015	0.015	0.015			
Concentration		0.000	44.474	13.031	1.794	0.000	0.000	0.000			
% Adsorbed		0.000	55.526	86.969	98.206	100.000	100.000	100.000	0.000	55.526	27.763
Average		0.000	55.526	86.969	98.206	100.000	100.000	100.000	0.000	55.526	27.763

APPENDIX G

GRAPHS

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G.1 WITBANK SEAM 4 SAMPLES

G.1.1 pH Measurements

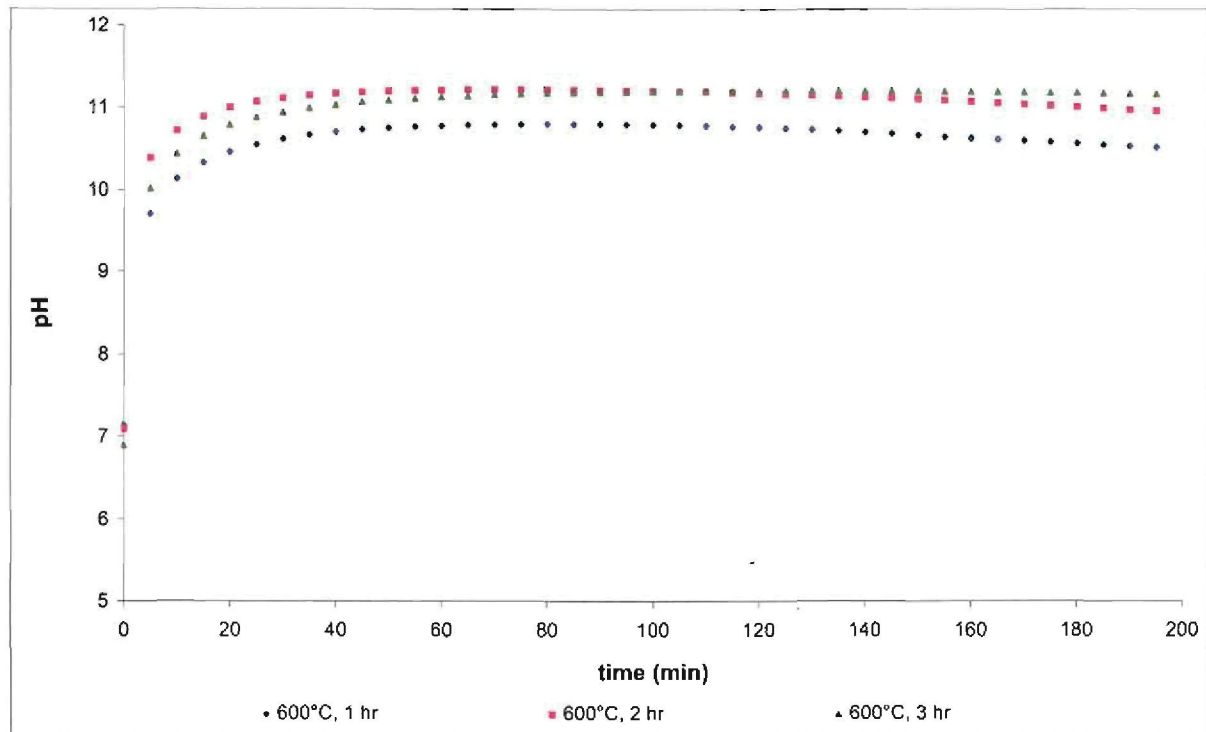


Figure G.1.1: Witbank Seam 4 – pH measurements (600°C)

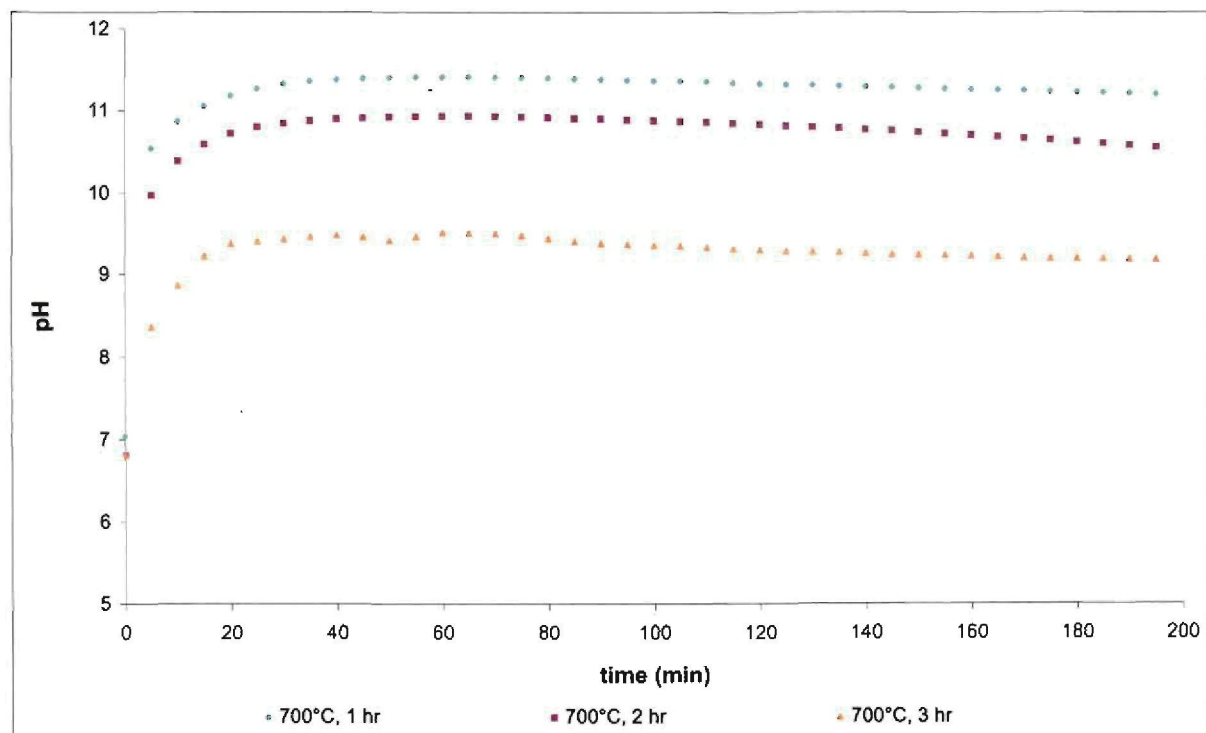


Figure G.1.2: Witbank Seam 4 – pH measurements (700°C)

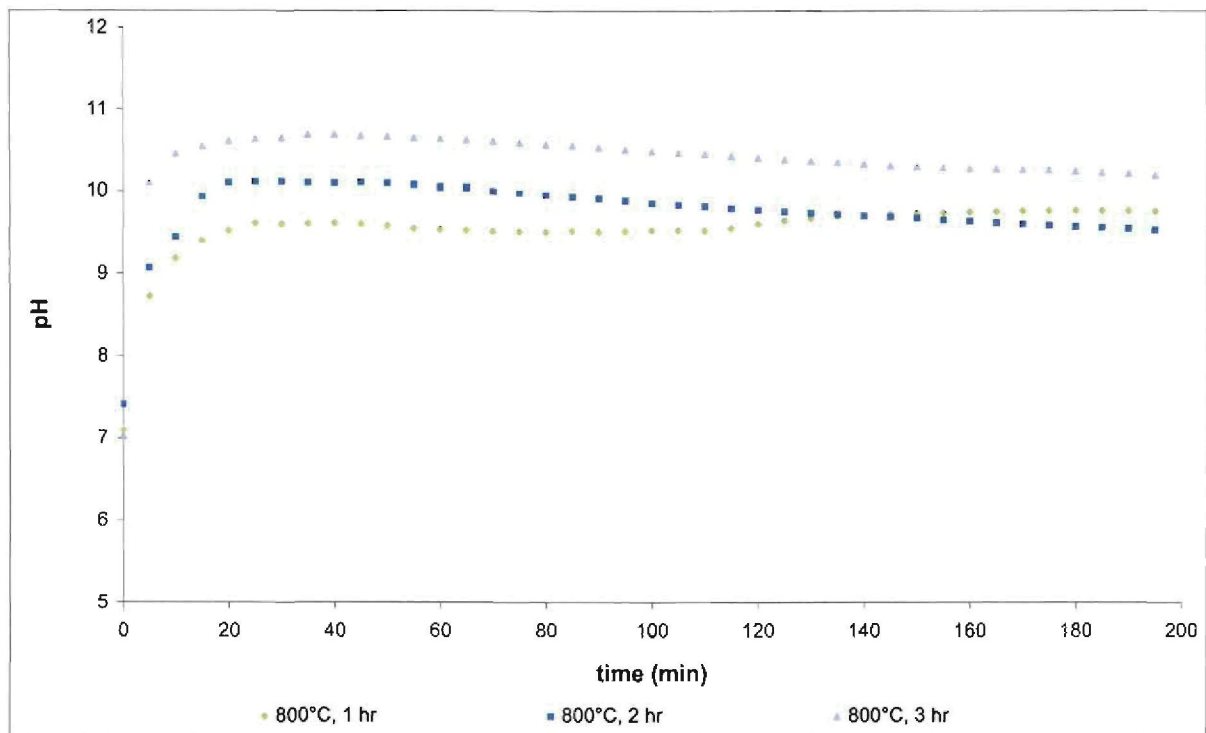


Figure G.1.3: Witbank Seam 4 – pH measurements (800°C)

G.1.2 Phenol adsorption

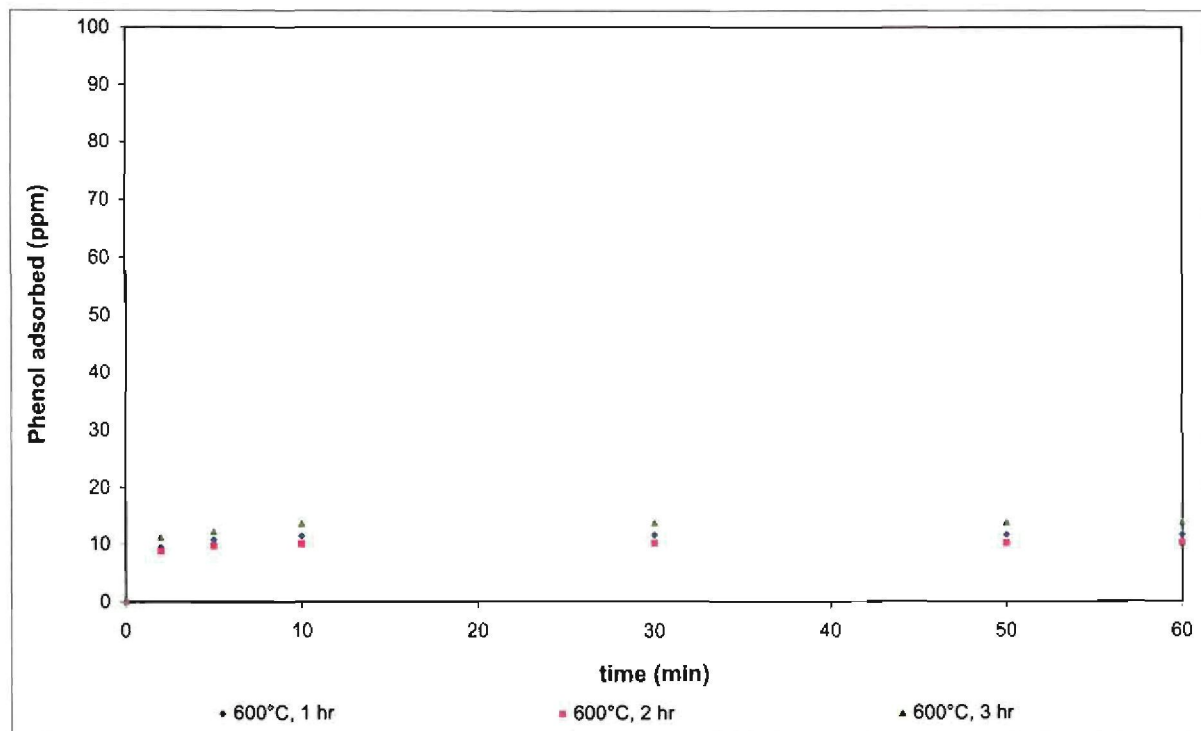
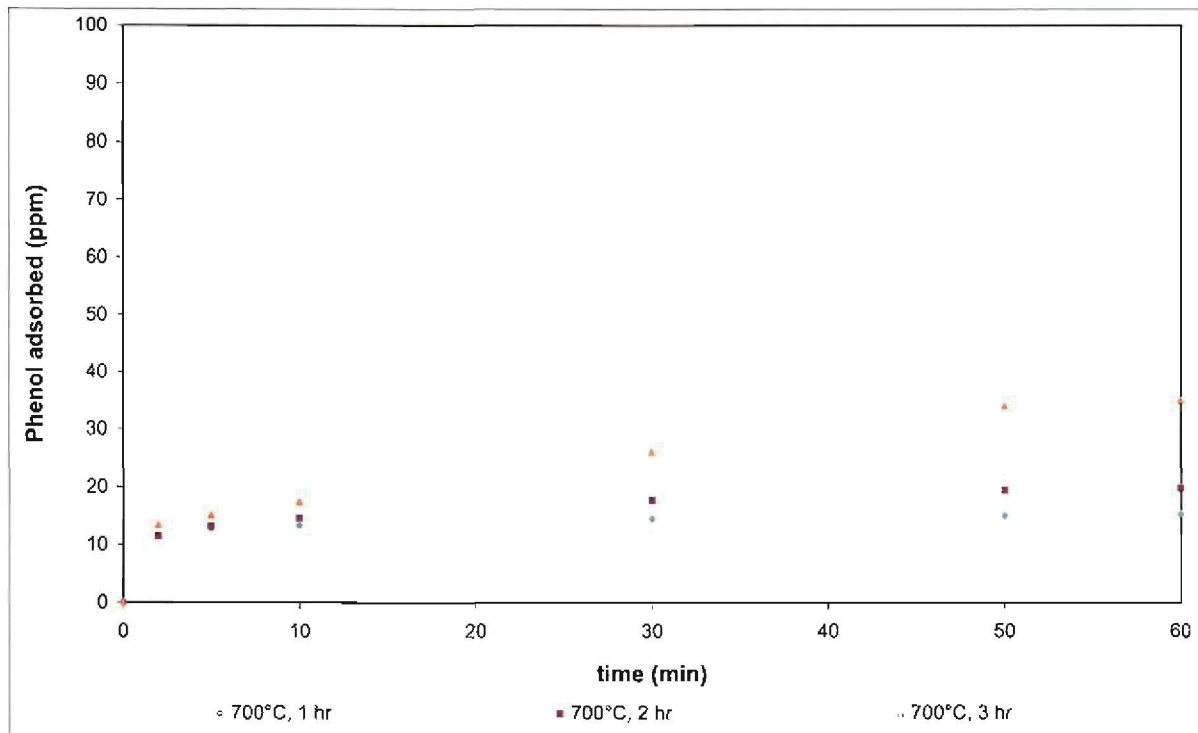
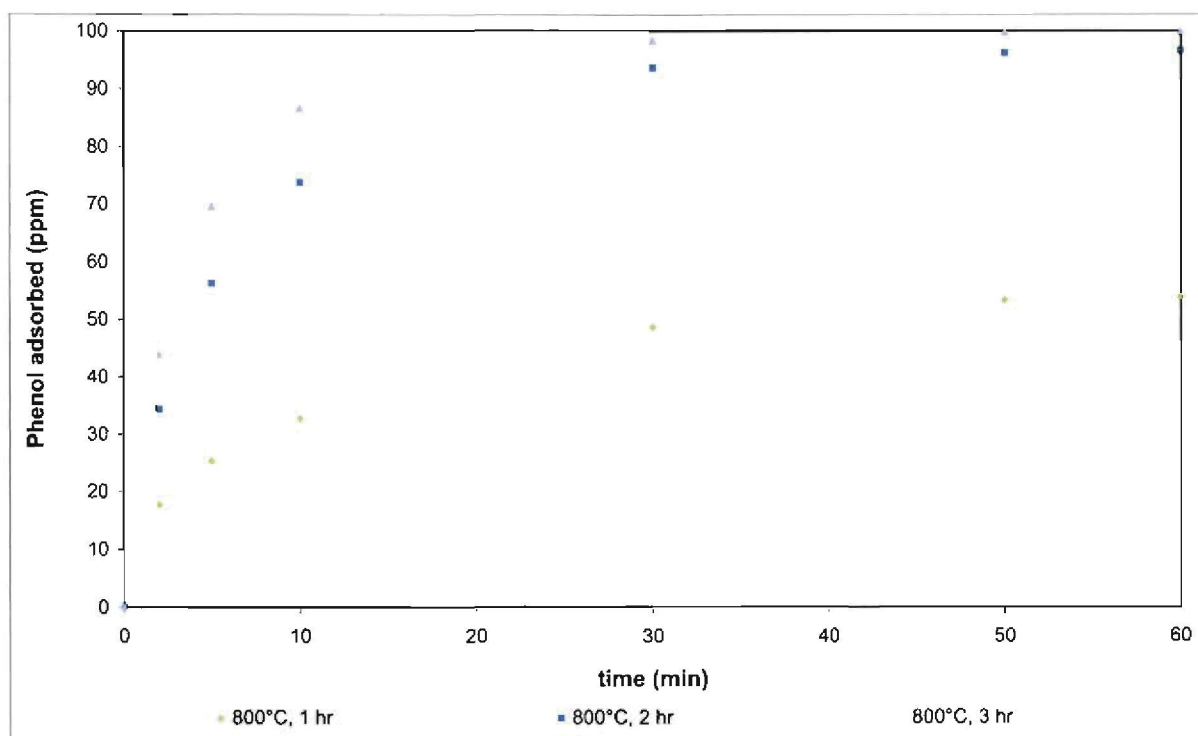


Figure G.1.4: Witbank Seam 4 – Phenol adsorption (600°C)

**Figure G.1.5: Witbank Seam 4 – Phenol adsorption (700°C)****Figure G.1.6: Witbank Seam 4 – Phenol adsorption (800°C)**

G.2 NEW VAAL SAMPLES

G.2.1 pH Measurements

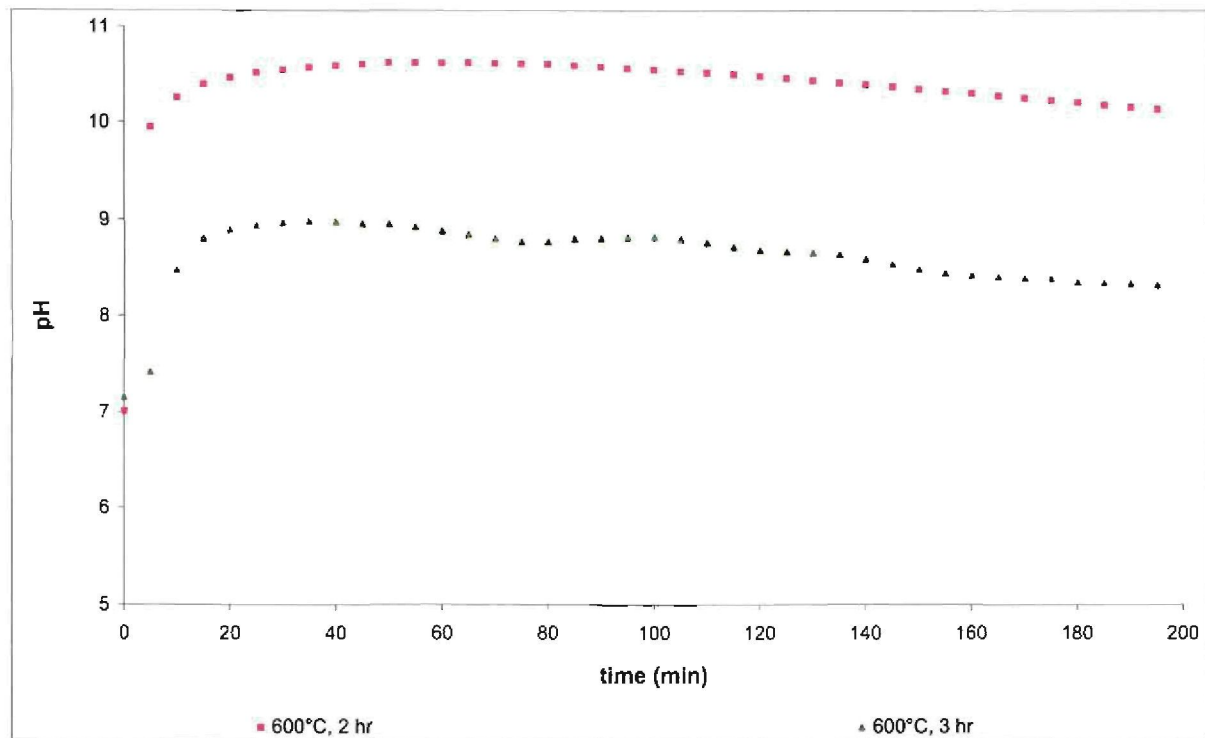


Figure G.2.1: New Vaal – pH measurements (600°C)

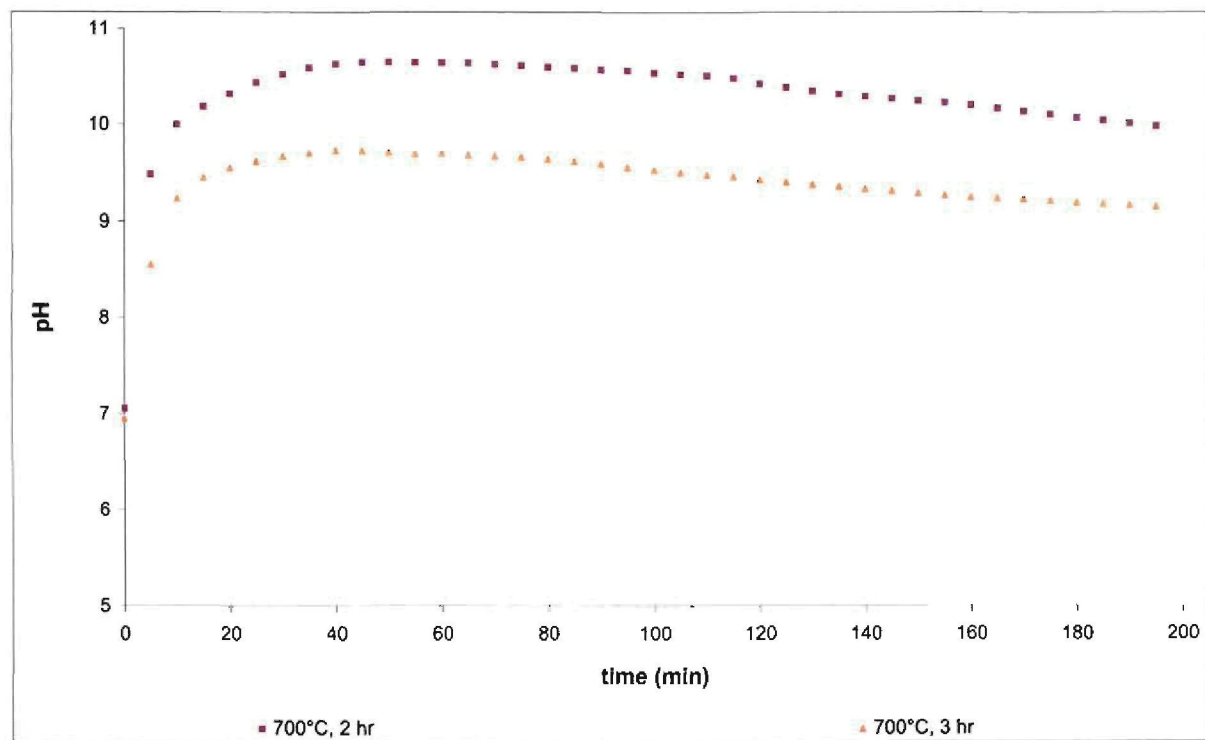


Figure G.2.2: New Vaal – pH measurements (700°C)

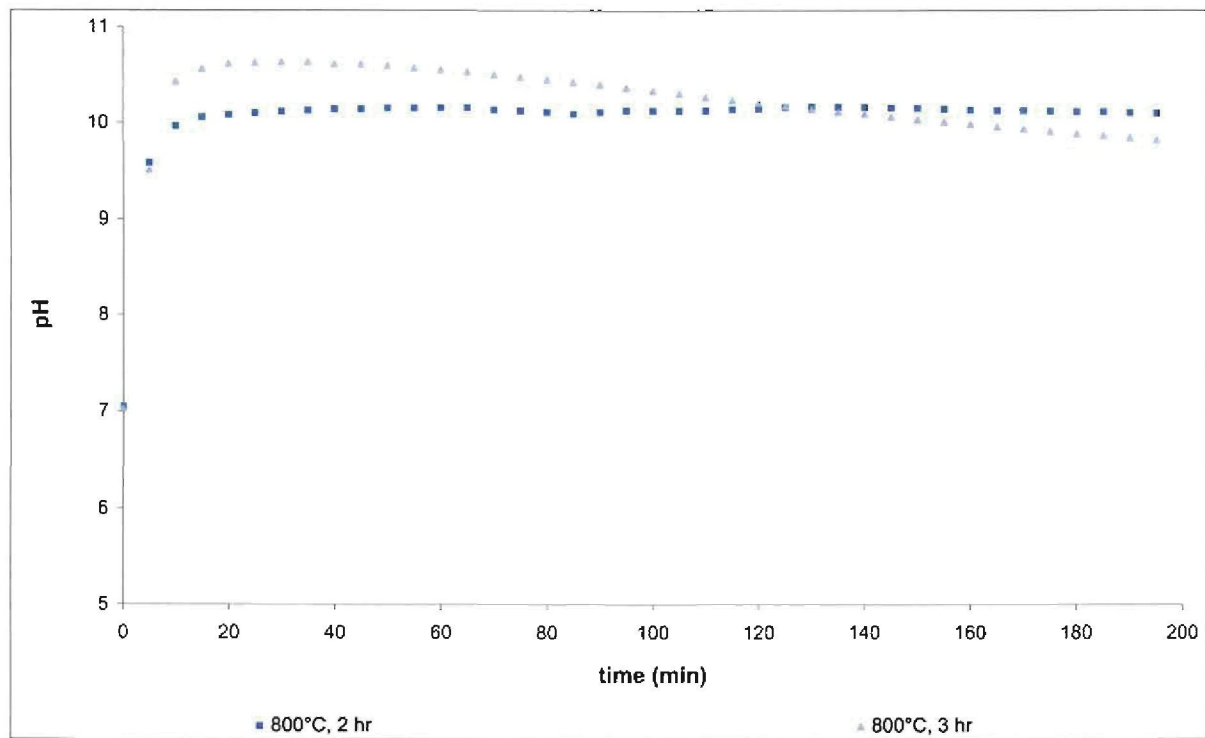


Figure G.2.3: New Vaal – pH measurements (800°C)

G.2.2 Phenol adsorption

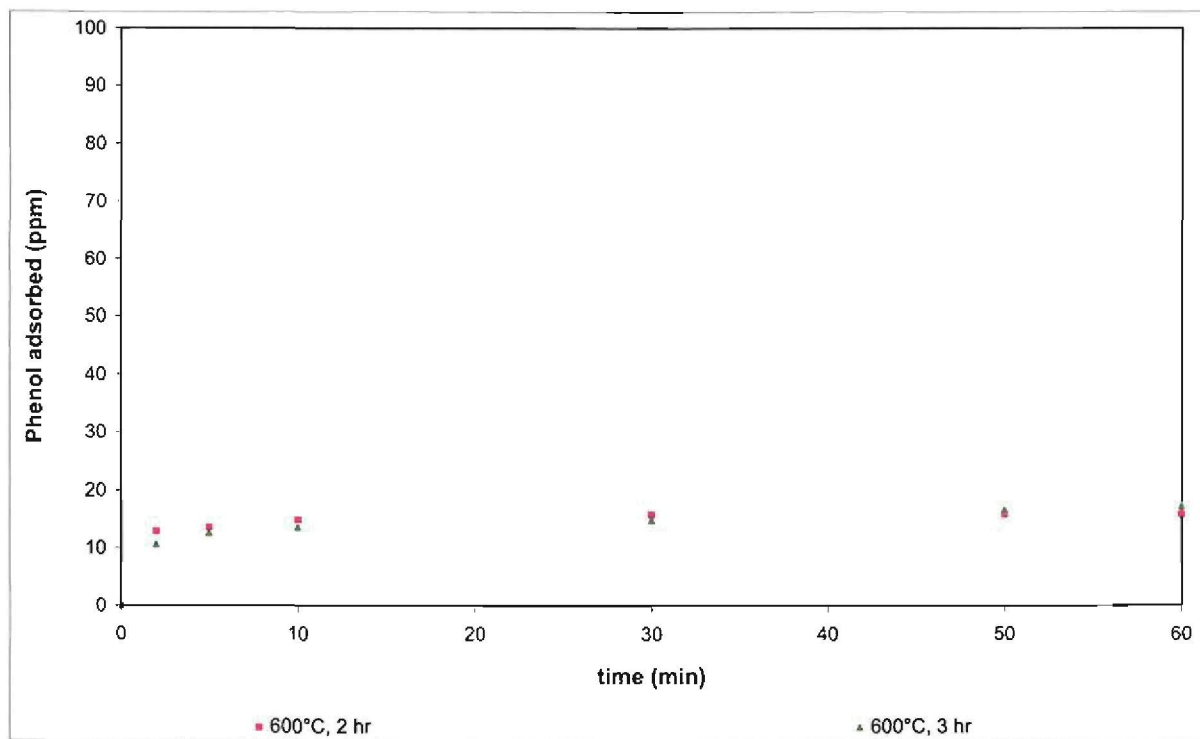


Figure G.2.4: New Vaal – Phenol adsorption (600°C)

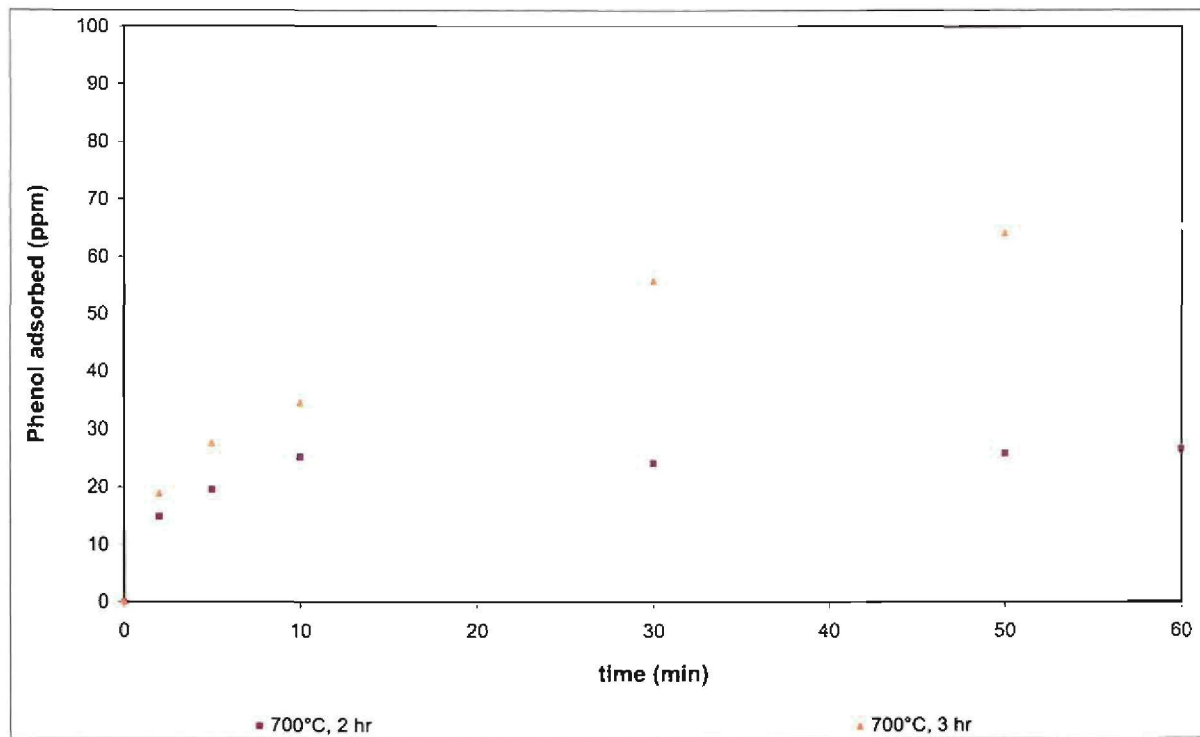


Figure G.2.5: New Vaal – Phenol adsorption (700°C)

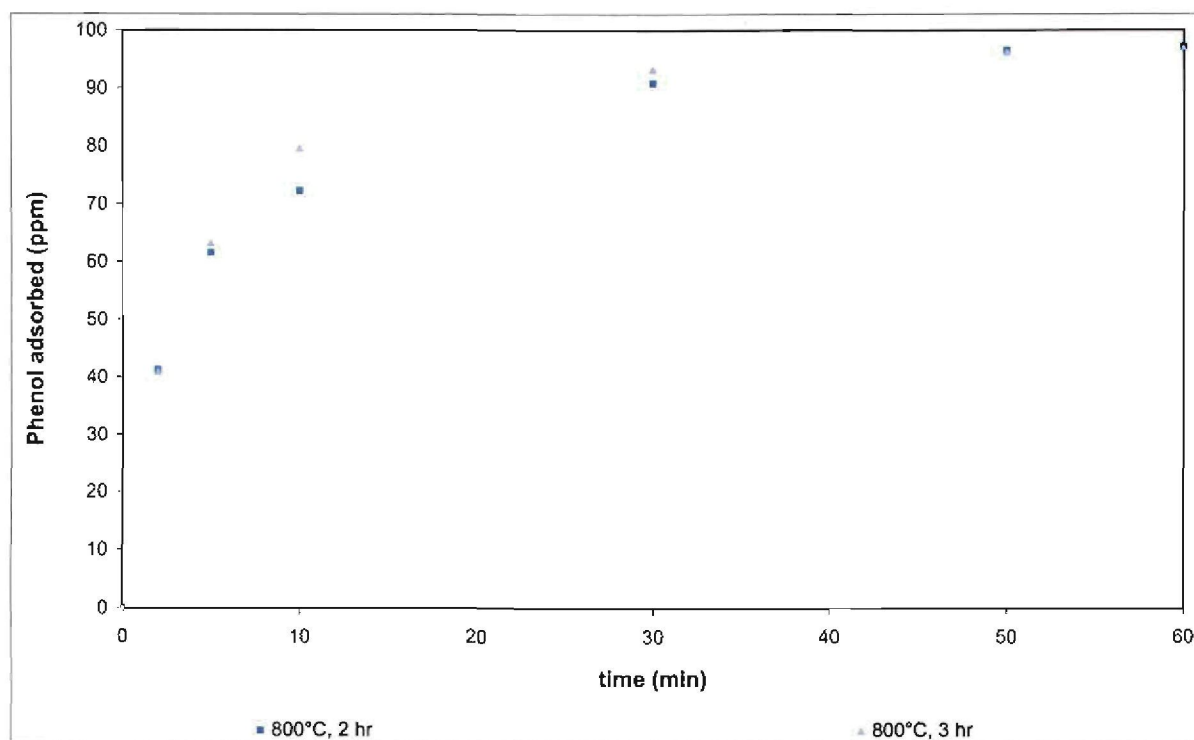


Figure G.2.6: New Vaal – Phenol adsorption (800°C)

G.3 NORIT RO 0.8

G.3.1 pH Measurements

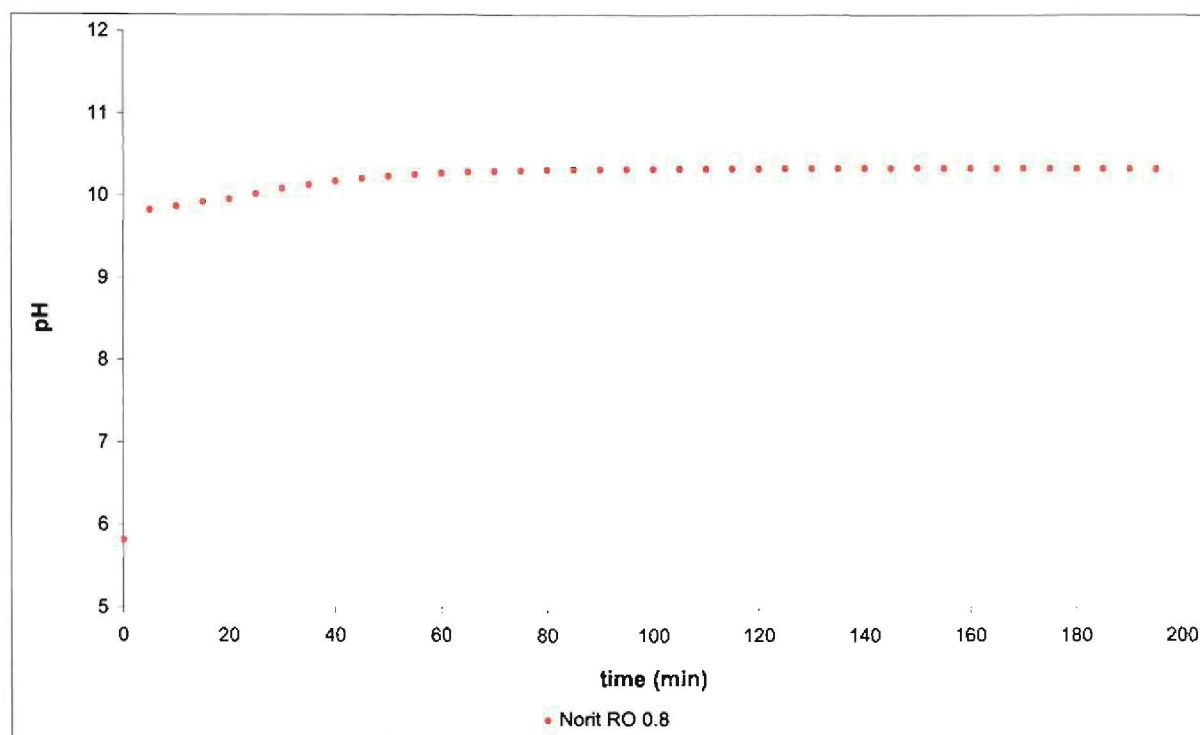


Figure G.3.1: Norit RO 0.8 – pH measurements

G.3.2 Phenol adsorption

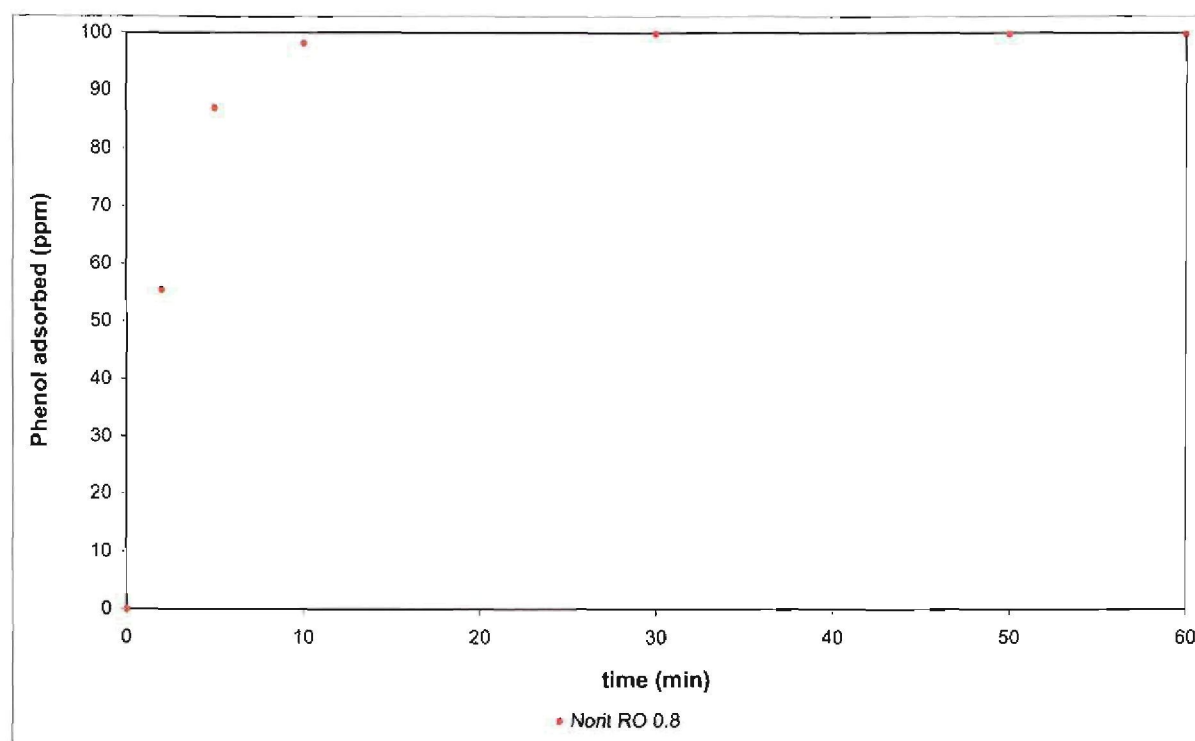


Figure G.3.2: Norit RO 0.8 – Phenol adsorption