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The effects of chemical and physical properties of chars derived from inertinite-rich, high ash coals on gasification reaction kinetics

Gregory Nworah Okolo
B.Eng (Chem. Eng.) (ESUT, Enugu, Nigeria)

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Supervisor: Prof. R. C. Everson

Co-supervisor: Prof. H. W. J. P Neomagus

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“Any fact facing us is not as important as our attitude towards it,

for that determines our success or failure**”**

-Norman Vincent Peale-

DEDICATION

This dissertation is gratefully dedicated to the loving memory of my late sister,
Miss Juliana Okolo, who passed on to glory on the 13th of April, 2008.
May her gentle soul rest in perfect peace!
Eternal rest grant unto her Oh Lord…, And let perpetual light shine upon her…, May she rest in peace…. Amen

DECLARATION

I, **Gregory Nworah Okolo**, do hereby declare that the dissertation with the title: "**The effects of chemical and physical properties of chars derived from inertinite-rich, high ash coals on gasification reaction kinetics**", submitted in partial fulfilment of the requirements for the degree of Master of Engineering (Chemical Engineering) is my work and has not been submitted at any other university either in part or as a whole.

Signed at Potchefstroom on the...............day of ....2010.

....
Gregory N. Okolo

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ABSTRACT

With the increasing global energy demand and the decreasing availability of good quality coals, a better understanding of the important properties that control the behaviour of low-grade coals and the subsequent chars in various utilisation processes, becomes pertinent. An investigation was therefore undertaken, to study the effects of chemical and physical properties imparted on chars during pyrolysis on the subsequent gasification reaction kinetics of typical South African inertinite-rich, high ash Highveld coals. An attempt was made at following these changes in the transition from coals to chars by a detailed characterisation of both the parent coals and the respective chars. These changes were determined using various conventional and advanced techniques, which included among others, carbon crystallite analysis using XRD and char carbon forms analysis using petrography.

Three of the four original coals were characterised as Bituminous Medium rank C (coals B, C and C2), while coal D2 was found to be slightly lower in rank (Bituminous Medium rank D). The coals were rich in inertinites (> 54 *vol. %, mmb* with coal C2 having as high as 79 *vol. %, mmb*) and high in ash content (> 26.7 *wt. %, db*) and cabominerite and minerite contents (26 - 39 *vol. %, mmb*). The inertinite-vitrinite ratios of the coals were found to range from 1.93 to 26.3.

Characterization results show that both volatile matter and inherent moisture content decreased, while ash, fixed carbon and elemental carbon contents increased from coals to chars, indicating that the pyrolysis process was efficient. Elemental hydrogen, oxygen and nitrogen contents decreased, whereas total sulphur contents increased from coals to chars. This reveals that the total sulphur contained in the char samples was associated with the char carbon matrix and the minerals. Hydrogen-carbon and oxygen-carbon ratios decreased considerably from coals to chars showing that the chars are more aromatic and denser products than the original coals. Despite the fact that mineral matter increased from coals to chars, the relative abundance of the different mineral phases and ash components did not exhibit significant variation amongst the samples. The alkali index was, however, found to vary considerably among the subsequent chars. Petrographic analysis of the coals and char carbon forms analysis of the chars reveal that total reactive components (*TRC*) decrease while the

total inert components (*TIC*) increase from coals to chars. The 0% gain in *TIC* observed in char C2 was attributed to its relatively high partially reacted maceral char carbon forms content. Total maceral reflectance shifted to higher values in the chars (4.43 - 5.28 *Rsc%*) relative to the coals (1.15 - 1.63 *Rsc%*) suggesting a higher structural ordering in the chars. Carbon crystallite analyses revealed that the chars were condensed (smaller in size) relative to the parent coals. Lattice parameters: inter-layer spacing, d_{002}, increased, while the average crystallite height, L_c, crystallite diameter, L_a, and number of aromatic layers per crystallite, N_{ave}, decreased from coals to chars. Carbon aromaticity generally increased whereas the fraction of amorphous carbon and the degree of disorder index decreased from parent coals to the respective chars. Both micropore surface area and microporosity were observed to increase while the average micropore diameter decreased from coals to chars. This shows that blind and closed micropores were "opened up" during the charring process.

Despite the original coal samples not showing much variation in their properties (except for their maceral content), it was generally observed that the subsequent chars exhibited substantial differences, both amongst themselves and from the parent coals. The increasing orders of magnitude of micropore surface area, microporosity, fraction of amorphous carbon and structural disorderliness were found to change in the transition, a good indication that the chars' properties varied from that of the respective parent coals.

Isothermal CO_2 gasification experiments were conducted on the chars in a Thermax 500 thermogravimetric analyser in the temperature range of 900 - 950 *°C* with varying concentrations of CO_2 (25 - 100 *mol. %*) in the CO_2-N_2 reaction gas mixture at ambient pressure (0.875 *bar* in Potchefstroom). The effects of temperature and CO_2 concentration were observed to be in conformity with established trends. The initial reactivity of the chars was found to increase in the order: chars C2 < C < B < D2, with char D2 reactivity greater than the reactivity of the other chars by a factor > 4.

Gasification reactivity results were correlated with properties of the parent coals and chars. Except for the rank parameter (the vitrinite reflectance), no significant trend was observed with any other coal petrographic property. Correlations with char properties gave more significant and systematic trends. Major factors affecting the gasification reactivity of the chars as it pertains to this investigation are: parent coal

vitrinite reflectance, and: aromaticity, fraction of amorphous carbon, degree of disorder and alkali indices, micropore surface area, microporosity and average micropore diameter of the chars.

The random pore model (chemical reaction controlling) was found to adequately describe the gasification reaction experimental data (both conversions and conversion rates). The determined activation energy ranged from 163.3 $kJ \cdot mol^{-1}$ for char D2 to 235.7 $kJ \cdot mol^{-1}$ for char B; while the order of reaction with respect to CO_2 concentration ranged between 0.52 to 0.67 for the four chars. The lower activation energy of char D2 was possibly due to its lower rank, lower coal vitrinite reflectance and higher alkali index. The estimated kinetic parameters of the chars in this study correspond very well with published results in open literature. It was possible to express the intrinsic reactivity, r_s, of the chars (rate of carbon conversion per unit total surface area) using kinetic results, in empirical Arrhenius forms.

Keywords: Inertinite-rich coal, Coal and char properties, Char carbon forms, Carbon crystallite analysis, Carbon dioxide gasification, kinetic modelling.

OPSOMMING

Die toeneemlike gebruik van lae graad steenkool word al hoe meer ’n realiteit, as gevolg van hoë kwaliteit steenkole wat verkwis word om te voorsien in die behoeftes van die groeiende energiekwessie dwarsoor die wêreld. Dit is dus van uiterste belang dat ’n goeie begrip rondom die eienskappe en gedrag van lae graad steenkole en hulle gevolglike sintels gevorm word om sodanig gebruik te word in verskeie kommersiële prosesse. ’n Studie was dus onderneem om die effek van chemiese- en fisiese eienskappe van die gevormde sintels van verskeie lae graad steenkole op die gevolglike vergassings reaksie kinetika van tipiese Suid-Afrikaanse inertiniet ryk, hoë as steenkole te ondersoek. ’n Poging was aangewend om die verandering in die karakteristieke eienskappe van steenkool tot die vorming van sintels te monitor, deur gebruik te maak van ’n gedetaileerde karakteriserings ondersoek op beide die rou steenkole en hulle gevormde sintels. Konvensionele- en gevorderde metodes soos koolstof kristallografie met behulp van XRD en sintels koolstof vorm analise met behulp van petrografie is ingespan.

Drie van die vier steenkole wat gebruik is, is gekarakteriseer as Bitumineuse Gemiddelde Rang C (steenkole B, C en C2) steenkole, terwyl dit gevind is dat steenkool D2 egter ’n effense laer rang gehad het (Bitumineuse Gemiddelde Rang D). Al vier steenkole het hoë inhoude van inertiniet (> 54 *vol. %*, mineraal basis met steenkool C2 wat die hoogste inhoud gehad het met 79 *vol. %* mineraal basis), as (> 26.7 *wt. %*, droë basis), karbomineriet en mineriet (26-39 *vol. %*, mineraal basis) bevat. Die inertiniet-vitriniet verhoudings van die vier steenkole het gewissel tussen ongeveer 1.93 en 26.3.

Vanuit die karakteriserings resultate was dit duidelik dat beide die inherente waterinhoude en vlugtige stofinhoude afneem, terwyl die aswaardes, vaste koolstofinhoude en elementêre koolstof inhoude dienooreenkomstig toeneem vanaf steenkool tot sintels. Hieruit kon dit afgelei word dat die pirolise proses, vir die generering van sintels, effektief was. Hiermee saam het die elementêre waterstof-, suurstof- en stikstof inhoude ook afgeneem, terwyl die swawel inhoud toegeneem het. Die verhoogde swawel vlakke in die sintels toon aan dat dit hoogs waarskynlik meer geassosieer is met die koolstof/sintels koolstofmatriks as met die anorganiese minerale. Die waterstof-koolstof- en suurstof-koolstof verhoudings het ook ’n noemenswaardige afname getoon van steenkool tot sintel, wat aandui dat die

gevormde sintels meer aromaties en digter is as die oorspronklike steenkole. Ten spyte van die feit dat minerale inhoud toeneem van steenkool na sintels kon daar geen noemenswaardige variasie tussen die relatiewe voorkoms van die verskeie mineraalfases en askomponente onderskei word nie. Dit is egter gevind dat die alkali indeks noemenswaardig varieer tussen die verskillende sintels. Petrografiese analise op die steenkole en sintels koolstof vormanalise op die sintels het getoon dat die totale reaktiewe komponente (*TRC*) afneem terwyl die totale inerte komponente (*TIC*) toeneem van steenkool na sintels. Die 0% toename in *TIC* vir sintels C2 kan toegeskryf word aan sy relatiewe hoë inhoud van parsieël gereageerde maserale sintels koolstofvorm. Totale maserale reflektiwiteit was aansienlik hoër vir die sintel (4.43 – 5.28 *% Rsc*) as vir die oorspronklike steenkole (1.15 – 1.63 *% Rsc*), wat op ’n hoër strukturele geordenheid van die sintels dui. Vanuit koolstof kristallografie analise was dit duidelik dat die sintels meer gekondenseer (kleiner in grootte) is as die oorspronklike steenkole. Kristalstruktuur parameters soos die inter-laag spasieëring (d_{002}) het toegeneem, terwyl die gemiddelde kristalhoogte (L_c), kristaldiameter (L_a) en aantal aromatiese lae per kristal afgeneem het van steenkool na sintels. Koolstof aromatisiteit het gevolglik toegeneem, terwyl die fraksie van amorfe koolstof en graad van wanordelikheidsindeks afgeneem het van steenkool na sintels. Vanuit ’n fisiese perspektief het beide die mikroporieuse oppervlakarea en mikroporositeit toegeneem terwyl die gemiddelde mikroporieuse diameter afgeneem het vanaf steenkool tot sintels. Hieruit kan afgelei word dat “blinde” en geslote mikroporieë geopen het gedurende die pirolise proses.

Algeheel was daar geen duidelike verskil tussen karakteristieke eienskappe van die oorpsronklike steenkole nie, behalwe vir die verskil in maserale inhoud. In kontras het daar groot karakteristieke verskille bestaan tussen die verskillende sintels asook van hulle oorpronklike steenkole. Die toenemende orde van grootte van mikroporieuse oppervlakarea, fraksie van amorfe koolstof en strukturele ongeordenheid dui grotendeels daarop dat die sintels grootliks verskillend is van hulle oorspronklike steenkole.

Isotermiese CO_2 vergassing is gedoen om die reaktiwiteit van die gevormde sintels te toets. Vir hierdie doeleinde is gebruik gemaak van ’n Thermax 500 termogravimetriese analiseerder. Temperature tussen 900 en 950 *℃* en CO_2 konsentrasies van 25 to 100 *mol. %* (CO_2-N_2 reaksie gasmengsel) by atmosferiese druk is gebruik (0.875 *bar* in Potchefstroom) om die sintelsreaktiwiteit te assesseer.

Die effek van temperatuur en CO_2 konsentrasie op die reaktiwiteit van die sintels het ooreengestem met wat gevind is in literatuur. Hiermee saam het die aanvanklike (inisiële) reaktiwiteit van die sintels afgeneem in die volgende orde: C2 < C < B < D2 met sintel D2 wat se reaktiwiteit ’n faktor 4 groter was as die ander sintels.

Vergassings reaktiwiteitsresultate is verder gekorreleer met die karakteristieke eienskappe van die oorspronklike steenkole en die sintels. Geen ander noemenswaardige korrelasie is verkry tussen die petrografiese eienskappe van die steenkole nie, behalwe vir die vitriniet reflektiwiteit. Korrelasies met die sintels-eienskappe het meer sistematiese verduidelikings gelewer. Vir die betrokke studie was die belangrikste faktore wat ’n rol gespeel het in vergassingsreaktiwiteit: oorspronklike steenkool vitriniet reflektiwiteit; aromatisiteit-, fraksie amorfe koolstof-, graad van wanordelikheidsindeks-, mikroporieuse oppervlakte-, mikroporositeit- en gemiddelde mikroporie diameter van die sintels.

Die eksperimentele vergassingsresultate (beide omsetting en reaksietempo) kon akkuraat beskryf word deur die willekeurige poriemodel (chemiese reaksie beherend). Die bepaalde aktiveringsenergieë het gewissel tussen 163.3 $kJ\cdot mol^{-1}$ vir sintel D2 tot 235.7 $kJ\cdot mol^{-1}$ vir sintels B, terwyl die reaksie-orde met betrekking tot CO_2 konsentrasie gewissel het tussen 0.52 en 0.67 vir die vier sintels. Die lae aktiveringsenergie van sintels D2 kan heel waarskynlik toegeskryf word aan die steenkool se lae vitriniet reflektiwiteit en hoër alkali indeks. Die beraamde kinetiese parameters van die sintels toon goeie ooreenstemming met wat bevind is in literatuur. Dit was verder ook moontlik om die intrinsieke reaktiwiteit, van die sintels (tempo van koolstofomsetting per eenheidsoppervlakarea) uit te druk deur gebruik te maak van kinetiese resultate in empiriese Arrhenius vorms.

Sleutelwoorde: Inertiniet-ryke steenkool, Sintels koolstof vorme, Steenkool en sintels eienskappe, Koolstof kristal analise, Koolstofdioksied vergassing, kinetiese modellering.

TABLE OF CONTENTS

Chapter 6: CHAR GASIFICATION WITH CARBON DIOXIDE: KINETIC MODELLING AND PARAMETERS EVALUATION..166

Chapter 7: CONCLUSION AND RECOMMENDATIONS..........................191

LIST OF FIGURES

LIST OF TABLES

NOMENCLATURE

Symbol	Description	Unit
A	Breadth of X-ray beam	mm
$A(\theta)$	Absorption factor	-
A, k_{so}	Pre-exponential factor	$min^{-1}\cdot bar^{-m}$
A_{002}	Area under the (*002*) peak	$Å^2$
A_f	Final ash content of coal or char after demineralisation	wt. %
A_i	Original ash content of coal or char before demineralisation	wt. %
AI	Alkali index	-
A_x	Breadth of X-ray	cm
$A\gamma$	Area under the γ gamma side band of (*002*) peak	$Å^2$
C_g	Concentration of gaseous reactant	$mole\cdot m^{-3}$
CV	Calorific value	$MJ\cdot kg^{-1}$
d_{002}	Inter-layer spacing for a group of N_{ave} parallel layers	Å
d_p	Average diameter of coal or char particles	µm, mm
D_p	Pore diameter / average pore diameter	Å
E	Activation Energy	$kJ\cdot mol^{-1}$
E_d	Effectiveness of demineralisation	%
$f(X)$	Structural factor	m^{-1}
f_a	Carbon aromaticity	-
GCV	Gross calorific value	$MJ\cdot kg^{-1}$
H_a	Hydrogen aromaticity	-
I	X-ray reduced intensity / X-ray intensity	Atomic units / counts
I_{002}	Reduced intensity due to (*002*) reflection	atomic units
I_{am}	X-ray reduced intensity due to amorphous carbon	atomic units
I_{cr}	X-ray reduced intensity due to crystalline carbon	atomic units
I_{max}	Maximum reduced intensity of (*002*) peak	atomic units
K	Constant depending on X-ray refection plane	-
K	Absolute temperature scale	K
k, k_1, k_2, k_3	Reaction rate constant	min^{-1}
k'_{so}	Lumped pre-exponential factor	$min^{-1}\cdot bar^{-m}$

Symbol	Description	Unit
k_{so}	Pre-exponential factor	$m\cdot min^{-1}\cdot bar^{-m}$
k_v	Intrinsic rate constant of the volume reaction model.	min^{-1}
$K\alpha_1$	X-ray radiation from Cobalt due to $K_{\alpha 1}$	counts
$K\alpha_2$	X-ray radiation from Cobalt due to $K_{\alpha 2}$	counts
L_a	Crystallite diameter	Å
L_c	Crystallite height	Å
L_o	Total pore length per unit volume	$m\cdot m^{-3}$
M	Molarity of acid	M
m	Order of reaction with respect to CO2 concentration	-
m_{ash}	mass of ash	mg
MI	Maceral index	-
m_o	Initial mass of char	mg
m_t	Mass of char at time, t	mg
N_{ave}	Average number of aromatic layers per carbon crystallite	-
P_n	Fraction of aromatic carbon contained within the d_{002} peak	-
R	Ideal gas constant	$J\cdot K\cdot mol^{-1}$
R	Initial reactivity of the chars	min^{-1}
r_1, r_2, r_3	Reaction rates	min^{-1}
RMI	Reactive maceral index	-
RMI*	Modified reactive maceral index	-
Rr	Mean random vitrinite reflectance	%
r_s	reaction rate	$m\cdot min^{-1}$
Rsc	Mean random maceral reflectance	%
s	$2\sin\theta/\lambda$	$Å^{-1}$
S_{max}	Value of s ($2\sin\theta/\lambda$) at which I_{max} occurs	$Å^{-1}$
S_o	Initial surface area	$m^2\cdot m^{-3}$
T	Temperature	°C or K
t	Time	min
$t_{0.5}$	Time for fractional carbon conversion of 50%	min
$t_{0.9}$	Time for fractional carbon conversion of 90%	min
t_f	Time factor	min^{-1}

Symbol	Description	Unit
V	Volume per unit mass	$m^3 \cdot g^{-1}$
X	Fractional conversion of carbon	-
X_A	Fraction of amorphous carbon	-
y_{CO}	Molar fraction / partial pressure of CO	- / bar
y_{CO_2}	Molar fraction / partial pressure of CO_2	- / bar

GREEK SYMBOLS

Symbol	Description	Unit
λ	Wavelength of incident X-ray	Å
β	Full width at half maximum of the corresponding peak or band	degrees (°)
ε_o	Initial porosity of char samples	%
θ	Peak position / XRD angle of scan	degrees (°)
θ_{002}	Peak position of (*002*) peak	degrees (°)
θ_{10}	Peak position of (*10*) peak	degrees (°)
θ_{11}	Peak position of (*11*) Peak	degrees (°)
μ/ρ	Absorption coefficient for Cobalt-$K\alpha$ radiation	-
ρ	Skeletal density / density of coal or char samples	$kg{\cdot}m^{-3}$
ρ', ρ_c	Bulk density of coal or char sample	$kg{\cdot}m^{-3}$
σ	Standard deviation	various unit
τ	Dimensionless time	-
Δs_{E_a}	Slope of $\ln(t_f)$ against T^{-1} at constant CO_2 concentration	K^{-1}
Δs_t	Slope of the plot of real time t, against $\sqrt{1-\psi\ln(1-X)}-1$,	min
$\tau_{0.9}$	Dimensionless time at 90% conversion	-
ψ	Dimensionless structural parameter for char pores	-

ABBREVIATIONS

Acronym	Meaning
% Ave. Dev.	Percent average deviation
A.S.M.E.	American Society of Mechanical Engineers
ACT	Advanced Coal Technology, Pretoria
adb	Air dry basis
afb	Ash free basis
Afrox	African Oxygen
AI	Alkali index
ASA	Active surface area
ASAP	Accelerated surface area and porosimetry
ASTM	American Society for Testing Materials
Ave.	Average value
Ave. Dev.	Average deviation
BET	Brunauer-Emmett-Teller Method
BFBC	Bubbling fluidised bed combustion
Bit. Med.	Bituminous Medium Rank
BSU	Basic structural unit
Cat.	Category
CCT	Clean Coal Technology
CDM	Clean Development Mechanism
CFBC	Circulating fluidised bed combustion
daf	Dry ash free basis
db	Dry basis
Demin	Demineralised coal or char sample
DME	Department of Minerals and Energy
dmmb	Dry mineral matter basis
DOI	Degree of disorder index
D-R	Dubinin-Radushkevich method
DTF	Drop tube furnace
EFR	Entrained flow reactor

Acronym	**Meaning**
ESKOM	South African Electricity Supply Commission
ESS	Error sum of squares
FBC	Fluidised bed combustion
FBDB	Fixed bed dry Bottom gasifier
FBG	Fluidised bed gasification
FC	Fixed carbon
Fig.	Figure
FWHM	Full width at half maximum
gfb	Graphite (carbon) free basis
H/C	Hydrogen-carbon atomic ratio
HCL	Hydrochloric acid
HF	Hydrofluoric acid
H-K	Horvath-Kawazoe method
HP	Helium pycnometry
HPTGA	High pressure thermogravimetric analyser
HRTEM	High resolution transmission electron microscopy
HTR	Horizontal tube reactor
ID	Identity
IGCC	Integrated Gasification Combined Cycle
IR	Infra-red
ISO	International Standard Organisation
lfb	*LOI* free basis
LMO	Local molecular orientation
LOI	Loss on ignition
LTB	Lithium tetraborate
MIP	Mercury intrusion porosimetry
mmb	Visible mineral matter basis
mmfb	Visible mineral matter free basis
MOD	Molecular orientation domain
NMR	Nuclear magnetic resonance
NO_X	Oxides of nitrogen
NWU	North-West University

Acronym	**Meaning**
O/C	Oxygen-carbon atomic ratio
PBBR	Packed bed balance reactor
PCC	Pulverised coal combustion
PCI	Pulverised coal injection
PDTF	Pressurised drop tube furnace
PF	Pulverised fuel
PFB	Pressurised fluidised bed
Pp	Page number / pages
PSD	Position sensitive detectors
rpm	Revolution per minute
RPM	Random pore model
SA	South Africa
SABS	South African Bureau of Standards
SCM	Shrinking core model
SO_X	Oxides of sulphur
sfb	Sulphur free basis
TGA	Thermogravimetric Analyser
TIC	Total inert components
TPD	Temperature programmed desorption
TRC	Total reactive components
TSA	Total surface area
UCG	Underground Coal Gasification
UNFCCC	The United Nations Framework Convention on Climate Change
VM	Volatile matter content
vol. %	Volume percent
VRM	Volumetric reaction model
VTR	Vertical tube reactor
WCI	World Coal Institute
wt. %	Weight percent
XRD	X-ray diffraction
XRF	X-ray fluorescence

The nomenclatures for the petrographic analyses are provided in the relevant sections of Chapter 3 and Appendix A.

Conference Presentation Resulting from this Investigation

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