SJIF Impact Factor (2026): 8.863| ISI I.F. Value: 1.241| Journal DOI: 10.36713/epra2016 ISSN: 2455-7838(Online)
EPRA International Journal of Research and Development (IJRD)
Volume: 11 | Issue: 4 | April 2026 - Peer Reviewed Journal

KINETIC AND THERMODYNAMIC PARAMETERS ANALYSIS DURING CO-GASIFICATION OF PETROLEUM COKE AND SUGAR CANE BAGASSE CHARS USING TGA TECHNIQUE

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ABSTRACT

This study analyzes the effect of pyrolysis temperatures and blending ratio on petroleum coke and sugar cane bagasse chars. Pyrolysis temperature of 500, 800 and 900 ^{O}C and blending ratio of 1:1, 1:3 and 3:1, were examined using thermogravimetric analyser (TGA) at non-isothermal condition. Kinetic and thermodynamic parameters through first order chemical reaction (O1) and phase boundary controlled reaction (R2 and R3) models were tested. SCB samples showed lower values of activation energy, pre-exponential factor and reaction rate compared to PC. The chars produced at low temperature found more reactive compared to those of high temperature. Average enthalpy change for PC was 9.72 and 3.08% higher with O1 and R3 model, respectively, compared to R2 model. PC: SCB5 at blending ratio of 1:1 showed lower activation energy of 89.96 kJ/mol with R2 model, but, PC: SCB9 at blending ratio of 3:1 and O1 model obtained higher activation energy of 221.90 kJ/mol. The average Gibbs free energy for R2 model was 0.9– 3.98% higher than R3 and O1 model. However, the change of entropy for the samples investigated found insignificant. It's concluded that, the enthalpy and Gibbs free energy change increases as the pyrolysis temperature increased and R2 was the most effect model.

KEYWORDS: *Petroleum coke, sugar cane bagasse char, activation energy, kinetic, thermodynamic parameters.*

1. INTRODUCTION

Due to fossil fuel shortage, interst in renewable energy sources such as biomass is growing [1]. Traditionaly biomass energy was extracted by combustion. However, new methods such as gasification or pyrolysis were used to convert biomass into gases or liquid fuels [2]. Biomass reacts with air and steam at high temperature to form a gas mixture such as hydrogen, carbon monoxide, carbon dioxide, nitrogen and methane. This gases are suitable for power generation, liquid fuels and chemicals production through synthesis gas [3]. Gasification process can be divivded into two stages: char gasification and pyrolysis, hence undersating the reactivity and the kinetic process is curical for gasification reactor design. The reactivity is an important paramter because it determine fuel sutiability, char transformation stage and overall conversion rate [4, 5]. In addition, the kinetics parameters quantify the reactivity of char; hence different biomass sources could be compared. Min et al.[6] compared pyrolysis temperature effect on the physical, reactivity and chemical structure of wheat and corn straw. The temperatures were 500, 600, 700 and 800 °C respectively. The reactivity found decreases as pyrolysis temperature increases. Tushar et al. [7] investigated the effect of residence time and pyrolysis temperature on flax straw char. Strong influence of pyrolysis temperature was observed. The chars produced at low temperature were more reactive compared to those of high temperature. Nemanova et al. [8] studied the co-gasification of petroleum coke and pine pellets biomass using thermogravimetric analyser. Different blends samples at heating rate of 10 °C /min and pressure of 1 bar were tested. The heating was up to 1200 °C. Shorter gasification time was observed with higher biomass content in the blend, hence increased the reactivity. Zhan et al.[9] investigated blending method effect on the co-gasification of petroleum coke and lignite with CO_2 using a thermogravimetric analyzer at a pressure of 0.1 MPa. Blending method found to influence the reactivity significantly. Fermoso et al.[10] studied steam gasification of five solid fuels samples (two bituminous coals with different ash contents, a petcoke and residues of chestnut and olive stones). Thermogravimetric analyzer at different temperatures and steam concentrations was used. Gas –solid models such as volumetric, grain and random pore were investigated. The activation energy was similar for the samples. But the pre-exponential factor was different. Among the models, random pore model found the best describing the behaviour. Xu et al. [11] investigated the interaction and kinetic behavior of CO_2 gasification for three types of coal (lignite, bituminous coal and lean coal) and biomass (Sawdust, Rice straw, Corn stalk) samples using thermogravimetry analyzer. The activation energy was estimated using homogeneous reaction model and shrinking core model. They found that, the activation energy decreases as the sawdust content in the blend increases. Edreis et al.[12] investigated the activation energy of SCB chars using thermogravimetric at a temperature of 500, 800 and 900°C with different heat rates. Vyazovkin and Ozawa–Flynn–Wall models were used to estimate the activation energy. They found that, activation energy increases with increasing pyrolysis temperature. The same author [13] tested the kintic thermal behaviour and physcial structure of sugar

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cane bagasses char during non-isothermal steam gasification using thermogravimetric analyzer. The temperaatures were 500, 800 and 900 ^{o}C; whereas, heating rates were 10, 15 and 20 ^{o}C min^{-1}, respectively. Char reactivity found proportinal to heating rate and inversly to pyroloysis temperature. Wang et al. [14] studied co-gasification properties of petroleum coke and millet husk biomass char and their blends using thermogravimetric under steam atmosphere condition. The activation energy was estimated using random pore, volumetric and unreacted-core models. The results indicated that random pore model was better describing char co-gasification compared to volumetric and unreacted-core models. The activation energy of char decreased before it's increased as the proportion of biomass char increases. Edreis et al. [15] studied kinetic and synergistic effects of CO_2 co gasification of low sulphure petroleum coke and biomass wastes. Homogenous and shrinking core models were examied. Synergistic efffect was observed and the reactivity found improved with the addition of petroleum coke. The acivation energy found decrease by the addition of sawdust. In another study, the same author [16] investiaged kinetics, thermodynamic and synergistic effects of petroleum coke and biomass waste with H_2O co-gasification. Five biomass samples of rich husk, rice stalk, coton straw, sawdust and sugar cane baggas were tested. Homogenous and shrinking core models were studied. Sawdust showed the lower reacivity and thermodynamics paramters. Similar results were observed by blending sawdust and coton straw with petroleum coke. In Sudan their about 2.76×10^{6} tones of sugar cane bagasses produced anually [17]. This huge amount of SCB could be used for energy generation [18]. Besides, it has the advantages of good ignition charactertics, cheap and low pollution [19]. Petroleum coke with high calorific value, low ash content and abundant availability found attractive fuel source [20]. However, low reactivity, high sulphur and vanadium oxide (V_2O_5) contents are some drawback hindering it's exploration as a fuel. This drawback causes boiler pipes clogging and environmental problems [21, 22]. Sudanese PC has low sulphur, ash and vanadium oxide contents compared with other PC and coals. Hence, it is found essential fuel source for gasification or combustion [23, 24]. To overcome PC problems, techniques such as large energy, high temperature and longer reaction time or catalyst additives were used [8, 22, 23]. It's found that SCB release large amount of volatile at the beginning of gasification, therefore blending it with PC could provide more energy and overcome the mentioned problems [25, 26]. Hence, understanding the kinetics, reactivity and thermodynamics parameters effect of PC and SCB during thermal conversion is crucial. In this study, PC, SCB char and their blends at different blending ratio, pyrolysis temperature and heating rate were investigated using thermogravimetric analyser. Further, kinetic, reactivity and thermodynamics parameters were evaluated.

2. MATERIALS AND METHODS

2.1. Materials and Samples Preparation

In this study petroleum coke and sugar cane bagasse char were used. The samples were dried (remove the moisture to less than 10%), crushed and sieved. The size of the particles were in range of 180− 450 μm.

2.2. Chars Preparation

SCB chars were devolatilization in a horizontal tube furnace using pure nitrogen. Nitrogen flow rate was 1 L/min for 20 min at a temperature of 500, 800 and 900 ºC, respectively. When the furnace reach the desired temperature, ceramic container content 1g of SCB sample was placed outside the tube and nitrogen was purged. The oxygen was eliminated and the container was pushed into tube heating zone. After the specified time, the char was removed and cooled to a room temperature using nitrogen. The SCB char produced at a temperature of 500, 800 and 900 °C were denoted by SCB5, SCB8 and SCB9, respectively. The char's samples were stored in desiccator to prevent moisture. The mixture of PC and SCB char were prepared at ratio of 1:1, 1:3 and 3:1 by mass.

2.3. Proximate and Ultimate Analysis

Proximate analysis of the samples was performed using TGA-2000 (Navas Instruments, Spain) following D5142 standard method. For ultimate analysis Euro-CA 3000 elemental analyser was used. Table 1, shows the proximate and ultimate analyses of PC and SCB chars samples.

Table 1: Proximate and ultimate analysis of PC and SCB char samples.

Sample	Proximate analysis (w%)[a]				Ultimate analysis (w%)[b]				
	M	Ash	VM	FC	C	H	O [c]	N	S
PC	2.15	2.01	9.32	86.52	91.09	3.76	3.03	1.06	1.08
SCB 5	2.33	10.35	23.04	54.28	47.39	4.51	47.54	0.45	0.10
SCB 8	1.19	13.61	9.72	75.48	51.52	1.96	46.05	0.35	0.11
SCB 9	0.73	16.68	6.93	75.66	74.49	1.83	22.29	1.11	0.25

[a] As received basis [b] Dry ash basis [c] Calculated by differences

2.4. Thermo Gravimetric Analysis

Pure and co-gasification of PC and SCB chars were performed using TGA NETZSCHSTA 449/F3 model thermogravimetric analyser under non-isothermal condition. Gasification experiments were conducted at a temperature range of 25 to 1300 °C under heating rate of 20 °C/min. Pure CO_2 was used as a gas agent at a flow rate of 100 mL/min. A 8─ 9 mg of sample was used for each experiment and it

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was dispersed flatly on a crucible. A small amount of sample and a slow heating rate were used to avoid heat transfer limitation and to minimize mass transfer effects. All the experiments were repeated three times to test the reproducibility of the results and the average values was used for the analysis. The mass loss (TG) and derivative curve (DTG) of the samples were presented as a function of temperature. For the active site Boudouard reactions mechanism (Equation 1, 2, 3 and 4) were considered for char gasification in carbon dioxide domain [27, 28].

$$C + CO_2 \rightarrow 2CO \quad (1)$$

$$C_{fas} + CO_2 \xrightarrow{k_{b1}} C(O) + CO \quad (2)$$

$$C(O) + CO \xrightarrow{k_{b2}} C_{fas} + CO_2 \quad (3)$$

$$C(O) \xrightarrow{k_{b3}} CO \quad (4)$$

Where k_i is the rate of the ith reaction.

2.5. Kinetic study

Kinetic parameters of reaction rate (k), activation energy (E) and pre exponential factor (A) of PC, SCB char and their blends during CO_2 co-gasification were investigated using first order chemical reaction and phase boundary controlled reaction based Coats–Redfern techniques. The TGA data were expressed as a function of conversion (X) showed by Equation (5):

$$x = \frac{w_i - w_t}{w_i - w_f} \quad (5)$$

where W_i, W_t and W_f are the initial, given time and final sample mass respectively. A solid state series mechanism was used to fit the data using Equation (6).

$$\frac{dx}{dt} = kf(x) \quad (6)$$

where k is the rate constant (min^{-1}) and f(x) is the reaction mechanism (see Table 2). The activation energy was obtained using Equation (7).

$$\ln\left[\frac{g(X)}{T^2}\right] = \ln\left[\frac{AR}{\beta E}\left(1 - \frac{2RT}{E}\right)\right] - \frac{E}{RT} \quad (7)$$

where, R is the universal gas constant, t is the reaction time (min), T is the absolute temperature (K), β is the heating rate constant and $\beta = \frac{dT}{dt}.g(X)$ is the model reaction mechanism. For the investigated gasification temperature range, the expression $\ln\left[\frac{AR}{\beta E}\left(1 - \frac{2RT}{E}\right)\right]$ in Equation (7) is constant for the activation energy [29, 30]. Therefore, the left side of Equation (7) was plotted as a function of 1/T. The activation energy was found by plotting the slope of the straight line [31]. The pre-exponential factor was obtained by intercept of Equation (7), whereas the reaction rate with Equation (8).

$$k = Aexp\left(\frac{-E}{RT}\right) \quad (8)$$

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Table 2: Expressions of $f(x)$ and $g(x)$ for the kinetic model functions employed for the solid-state reactions [23, 32-35].

Model	Symbol	$f(x)$	$g(x)$
Chemical reaction or homogenous model (HM)			
First-order	O1	$(1-x)$	$-\ln(1-x)$
Phase Boundary Controlled Reactions			
Two Dimensions (Contracting Cylinder)	R_2	$2(1-x)^{\frac{1}{2}}$	$1-(1-x)^{\frac{1}{2}}$
Three Dimensions (Contracting Sphere)	R_3	$3(1-x)^{\frac{2}{3}}$	$1-(1-x)^{\frac{1}{3}}$

2.6. Thermodynamic parameters estimation

Thermodynamic parameters such as Enthalpy change (ΔH^o, kJ/mol), Gibbs free energy (ΔG^o, kJ/mol) and Entropies change (ΔS^o, kJ/mol.K) for PC, SCB char and their blends during co-gasification were calculated using equations (9), (10) and (11), respectively [36, 37].

$$\Delta H^o = E - RT \tag{9}$$

$$\Delta G^o = E + R.T_m.\ln\left(\frac{K_B.T_m}{h.A}\right) \tag{10}$$

$$\Delta S^o = \frac{\Delta H^o - \Delta G^o}{T_m} \tag{11}$$

where K_B is Boltzmann constant ($1.38X10^{-23}$J/K), h is Plank constant ($6.626X10^{-34}$Js) and T_m is the peak temperature (K).

3. RESULTS AND DISCUSSIONS

3.1 Kinetic Analysis

The kinetics parameters of pre-exponential factor, activation energy and reaction rate are related to material structure and the reactivity. Reaction with high activation energy require high temperature or long reaction time [23]. The kinetics parameters investigated are presented in Table 3. Phase boundary controlled reaction (R2 and R3) showed the lowest values of activation energy, pre exponential factor and reaction rate, however opposite trend was observed with first order chemical reaction model. Compared to PC, SCB9, SCB8, SCB5 showed the lower values of activation energy, pre-exponential factor and reaction rate. The higher activation energy values achieved by O1 model was 242.84 kJ/mol for PC, whereas the lowest was 120.18 kJ/mol obtained by SCB5 with R2 model. The blends activation energy values were in the range of 160.81−221.90, 89.96−144.01, 111.37−153.13 kJ/mol for blending ratio of 3:1, 1:1 and 1:3 respectively. The blend of PC: SCB5 at blending ratio of 1:1 showed lower activation energy (89.96 kJ/mol) with R2 model, while the higher activation energy (221.90 kJ/mol) was obtained by PC: SCB9 at blending ratio of 3:1 and O1 model. This is indicated that O1 outperform R2 by 59.46%. This is because the chars prepared at low temperature were more reactive compared to those of high temperature[13]. Furthermore, it's found that as pyrolysis temperature increases the activation energy increased, hence significantly affect the kinetic parameters. The correlation coefficient for the models of O1, R2 and R3 was in range of 0.9531– 0.9998 as presented in Figures 1, 2 and 3. This is indicated that the reaction models highly fitted the experimental data. The models of R2 and R3 obtained similar correlation coefficient of 0.9973 for PC, whereas for SCB5, SCB8 and SCB9 these values were 0.9992, 0.9998 and 0.9997, respectively, with O1 model. At blend ratio of 1:1 and 3:1, O1 model showed correlation coefficient of 0.9983 and 0.9877, respectively. For the blend of PC: SCB5 the correlation was 0.9983 at blending ratio 3:1 with O1 model. Whereas for PC: SCB8 at blending ratio of 3:1, 1:1 and 1:3, the correlation coefficient obtained were 0.9979, 0.9848 and 0.9706 with O1 model, however for PC: SCB9 these values were 0.9989, 0.9848 and 0.9706 respectively. Among the investigated models, O1 model showed the best fitted results at blending ratio of 3:1 and 1:3. However, model R2 was found the most effective solid-state mechanisms for PC and the blend of PC: SCB9 at blending ratios of 1:1. Higher reaction rate of 4.92E+08 (1/min) was obtained by PC sample using O1 model, however, the lower value of 3.46E+04 (1/min) was obtained by R2 model with SCB5. In addition, the blend of PC: SCB9 at blending ratio of 3:1 obtained higher reaction rate of 1.17E+08 (1/min), but the PC: SCB5 at blending ratio of 1:1 showed lower reaction rate of 2.28E+02 (1/min). Therefore, it's concluded that, the char prepared at low temperature was more reactive compared those of high temperature, hence PC: SCB5 at blending ratio of 1:1 was the most reactive with R2 model.

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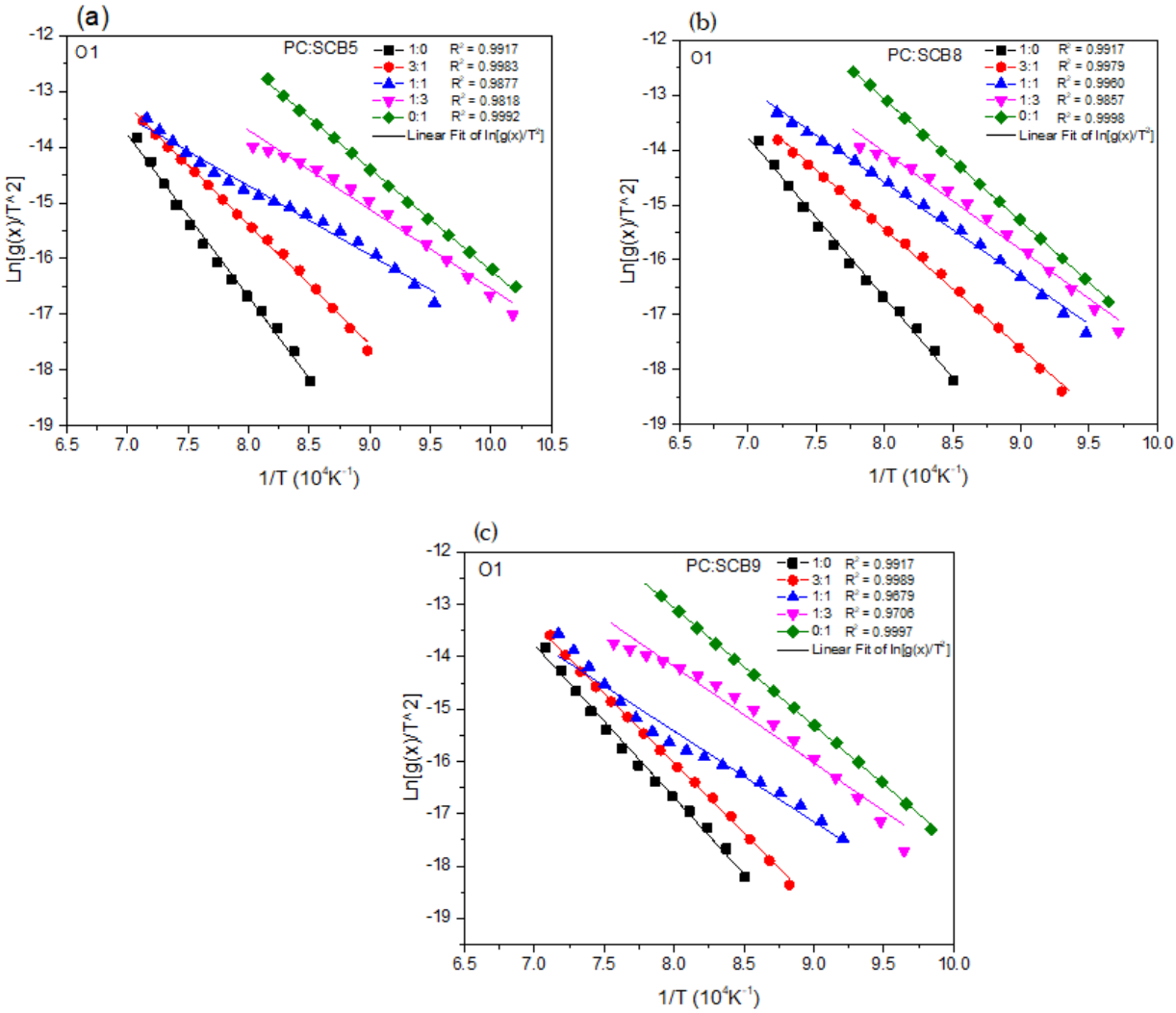


Figure 1: The plot of *ln* [g(x)/T^2] against 1/T obtained by the model O1.

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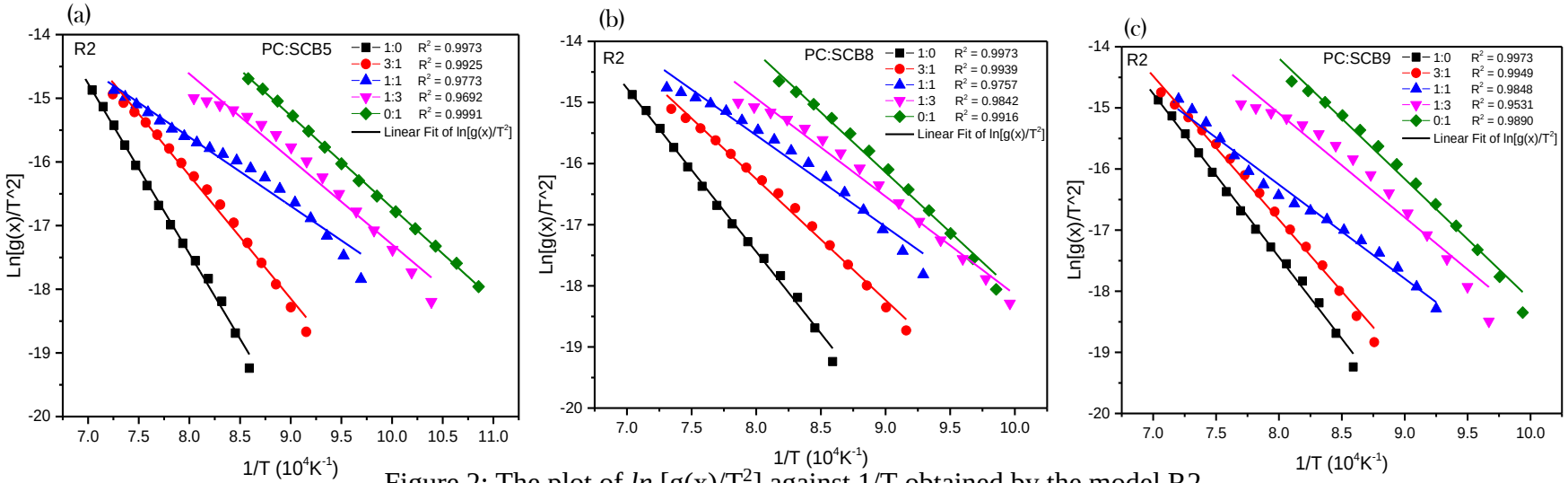


Figure 2: The plot of *ln* [g(x)/T^2] against 1/T obtained by the model R2.

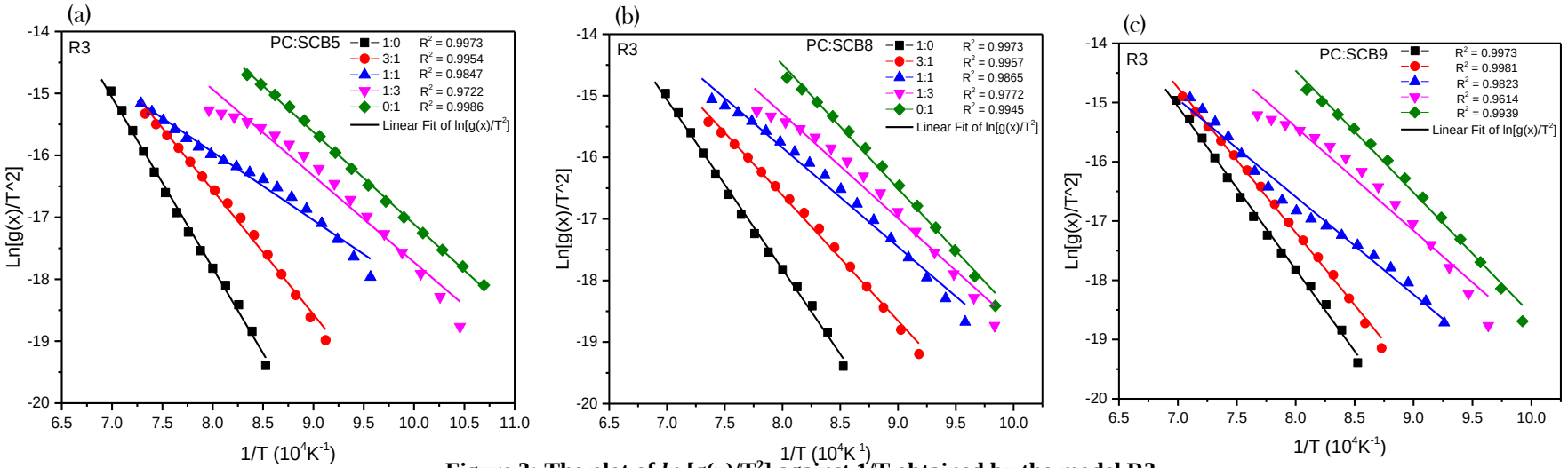


Figure 3: The plot of *ln* [g(x)/T^2] against 1/T obtained by the model R3.

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Samples	Models	*E*	*A*	*K*	*E*	*A*	*K*	*E*	*A*	*K*
1:0	O1	242.84	5.03E+08	4.92E+08						
	R2	222.35	3.08E+07	3.02E+07						
	R3	228.84	4.06E+07	3.97E+07						
			PC:SCB5			PC:SCB8			PC:SCB9	
3:1	O1	178.29	2.83E+06	2.79E+06	180.52	3.36E+06	3.30E+06	221.90	1.20E+08	1.17E+08
	R2	160.81	2.12E+05	2.08E+05	164.67	3.01E+05	2.96E+05	195.27	3.75E+06	3.68E+06
	R3	166.70	2.71E+05	2.67E+05	169.35	3.35E+05	3.30E+05	203.31	5.83E+06	5.72E+06
1:1	O1	103.27	2.66E+03	2.63E+03	143.28	1.80E+05	1.77E+05	144.01	8.47E+04	8.36E+04
	R2	89.96	2.30E+02	2.28E+02	124.51	1.02E+04	1.01E+04	127.89	7.05E+03	6.96E+03
	R3	91.40	2.39E+02	2.37E+02	133.77	1.94E+04	1.91E+04	137.00	1.28E+04	1.27E+04
1:3	O1	118.20	3.31E+04	3.27E+04	148.29	5.18E+05	5.10E+05	153.13	7.32E+05	7.21E+05
	R2	111.37	6.49E+03	6.41E+03	133.38	4.59E+04	4.52E+04	141.61	9.14E+04	9.01E+04
	R3	115.93	7.55E+03	7.45E+03	141.13	7.07E+04	6.97E+04	145.38	9.72E+04	9.58E+04
0:1	O1	152.24	3.39E+06	3.33E+06	185.36	5.84E+07	5.73E+07	187.08	7.00E+07	6.86E+07
	R2	120.18	3.51E+04	3.46E+04	163.12	2.02E+06	1.98E+06	164.06	2.20E+06	2.16E+06
	R3	123.75	3.75E+04	3.69E+04	167.51	2.31E+06	2.26E+06	171.06	3.41E+06	3.34E+06

Table 3: Kinetic parameters values.

E: (kJ/ mol), A (1/min), k (1/ min)

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3.2 Thermodynamic parameters analysis

Thermodynamic parameters of the investigated samples of PC, SCB and their blends are showed in Table 4. The changes of enthalpies demonstrated the energy difference between the activated complex of samples structure [37]. While the Gibbs free energies showed the growth of overall energy which could be obtain from samples gasification [36]. Besides the entropies measures the random energy and the matter in samples gasification [36]. From the table, the enthalpy change values of PC samples were 210.80, 217.29 and 231.29 kJ/mol for R2, R3 and O1 models, respectively. Compared to R2, O1 and R3 shows 9.72% and 3.08% higher enthalpy change respectively. Gibbs free energy values were 381.17, 384.74 and 369.40 kJ/mol for R2, R3 and O1 models, respectively. However, insignificant entropy change was observed for the tested models. For the SCB5, SCB8 and SCB9, the enthalpy change values were in range of 110.86−154.26, 114.43−161.2 and 142.92−177.28 kJ/mol, respectively. Whereas for Gibbs free energy, these values were in the range of 309.52−323.13, 312.47−325.83 and 298.98−312.23 kJ/mol, respectively. For PC: SCB5, PC: SCB8 and PC: SCB9, the enthalpy change values were in range of 79.38−167.45, 113.52−169.62 and 116.77−210.50 kJ/mol, respectively. Whereas for Gibbs free energy the range were 309.33−366.43, 322.00−367.90 and 323.60−378.90 k|J/mol for PC: SCB5, PC: SCB8 and PC: SCB9, respectively. For the blends of PC: SCB5, PC: SCB8 and PC: SCB9 insignificant entropy change was observed. Among the models investigated, the model of R2 shows the lowest enthalpy change values, but the lower Gibbs free energy value was obtained by O1 model for all blends. It's concluded that, the enthalpy change and Gibbs free energy change increases as the pyrolysis temperature increased. However, the change of entropies were insignificant for the investigated samples.

Table 4: Thermodynamic parameters values.

Samples	Models									
		ΔH^o	ΔG^o	ΔS^o	ΔH^o	ΔG^o	ΔS^o	ΔH^o	ΔG^o	ΔS^o
1:0	O1	231.29	369.40	-0.0994						
	R2	210.80	381.17	-0.1227						
	R3	217.29	384.47	-0.1204						
		PC:SCB5			PC:SCB8			PC:SCB9		
3:1	O1	167.45	352.59	-0.1420	169.62	353.94	-0.1406	210.50	363.01	-0.1112
	R2	149.97	363.20	-0.1635	153.77	364.39	-0.1607	183.87	375.89	-0.1401
	R3	155.86	366.43	-0.1615	158.45	367.90	-0.1598	191.91	378.90	-0.1364
1:1	O1	92.69	346.93	-0.1997	132.29	350.42	-0.1650	132.89	362.00	-0.1714
	R2	79.38	359.53	-0.2201	113.52	363.20	-0.1889	116.77	373.52	-0.1920
	R3	80.82	360.57	-0.2198	122.78	365.39	-0.1835	125.88	376.00	-0.1871
1:3	O1	108.82	309.33	-0.1778	138.46	322.00	-0.1553	143.29	323.60	-0.1524
	R2	101.99	317.78	-0.1913	123.55	330.91	-0.1754	131.77	332.54	-0.1697
	R3	106.55	320.92	-0.1900	131.30	334.41	-0.1718	135.54	335.70	-0.1692
0:1	O1	142.92	298.98	-0.1392	175.58	311.94	-0.1160	177.28	312.23	-0.1145
	R2	110.86	309.52	-0.1772	153.34	322.59	-0.1439	154.26	323.13	-0.1432
	R3	114.43	312.47	-0.1767	157.73	325.67	-0.1428	161.26	325.83	-0.1396

ΔH^o: (kJ/ mol), ΔG^o (kJ/mol), ΔS^o (kJ/mol.K)

4. CONCLUSIONS

The effects of pyrolysis temperature and blending ratio on the kinetics and thermodynamics parameters during co-gasification of petroleum coke and sugar cane bagasse chars were studied using TGA technique. Among the investigated models, O1 found the best fit model for SCB and their blend of PC: SCB5, PC: SCB8 and PC: SCB9 at blending ratio of 1:3 and 3:1. The lowest activation energy achieved by SCB5, SCB8 and SCB9 were 120.18, 163.13 and 164.06 kJ/mol, respectively, for the model of R2 at blending ratio of 1:1. While, the higher activation energy value was 242.84 kJ/mol obtained by PC and O1 model. The blend of PC: SCB5 at blending ratio of 1:1 showed lower activation energy of 89.96 kJ/mol with R2 model and blending ratio of 1:1, while the higher activation energy (221.90 kJ/mol) was obtained by PC: SCB9 at blending ratio of 3:1.The lowest enthalpy change value achieved by the SCB5, SCB8 and SCB9 were 79.38, 113.52 and 116.77 kJ/mol, respectively, for R2 model at blending ratio of 1:1. Whereas for Gibbs free energy, the samples of SCB5, SCB8 and SCB9 obtained the values of 309.33, 322.00 and 323.60 kJ/mol respectively with O1 model at blending ratio of 1:3 which were lower compared to R2 and R3. It's concluded that,

SJIF Impact Factor (2026): 8.863| ISI I.F. Value: 1.241| Journal DOI: 10.36713/epra2016 ISSN: 2455-7838(Online)
EPRA International Journal of Research and Development (IJRD)
Volume: 11 | Issue: 4 | April 2026 - Peer Reviewed Journal

the enthalpies change and Gibbs free energies change increases as the pyrolysis temperature increased. But the changes for the entropies were found insignificant.

Acknowledgement
The authors would like to acknowledge Prof. Hong Yao for the financial support and the staff at key laboratory of coal combustion, Huazhong University of Science and Technology.

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