



Low temperature deoxidization of biomass and its release characteristics of gas products

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ABSTRACT

With a first attempt of extension for torrefaction temperature aiming to reducing more oxygen, low temperature deoxidization of cellulose, hemicellulose, lignin, corn stalk and cotton stalk were performed at various temperatures (200, 230, 260, 290, 320, 350 and 390 °C) respectively. The deoxidization properties of both solid and gas products were investigated. As could be inferred from the results obtained that the main deoxygenation temperature range of cellulose, xylan, corn stalk and cotton stalk were 290–320 °C, 230–260 °C, 260–390 °C and 260–350 °C respectively. The deoxidation effect of lignin was not obvious during 200–390 °C, only 14.80% oxygen was removed at 390 °C. At lower temperature (200–320 °C), the oxygenated gas products dominated by CO₂ and H₂O were mainly from hemicellulose. In the high temperature range (350–390 °C), cellulose deoxidation was mastery reaction and CO₂ was the main product. The release kinetic analysis of CO₂ and CO during 200–390 °C had been performed for the first time, and as could be inferred from the results obtained that the activation energies for CO₂ and CO release of cellulose, lignin, cotton stalk, corn stalk during low temperature deoxidization were 61.32, 38.41, 43.38, 38.62 kJ/mol and 120.62, 60.55, 83.24, 72.27 kJ/mol, respectively. The release activation energies of hemicellulose in the temperature ranges of 200–290 °C and 320–390 °C were 63.32 and 10.21 kJ/mol for CO₂ and 115.75 and 20.65 kJ/mol for CO respectively.

1. Introduction

Biomass was the only renewable energy that could be converted into liquid fuels and had good compatibility with fossil fuels (Bernides et al., 2003; Zheng et al., 2012). However, the high moisture content, high oxygen content, low energy density, poor grindability and fluidity of biomass limited its further application (Chen and Kuo, 2011). And high oxygen was one of the most significant challenges for widely usage of biomass resources. Torrefaction, a pretreatment processes, was considered effective for improving the quality of biomass feedstock by reducing oxygen and moisture content and had been gained increasing attention in recent years (Chen and Kuo, 2010; Park et al., 2012).

Torrefaction was an easily performed pretreatment process that occurs at 200–300 °C under an inert atmosphere and at low heating rates (Prins, 2005; Thek and Obernberger, 2004). The results from recent research suggested that torrefied biomass had lower atomic O/C and H/C ratios. The higher torrefaction temperature and residence time

decreased O/C and H/C ratios and moved the torrefied product close to that of the char (Chen et al., 2014; Gucho et al., 2015; Mei et al., 2016, 2015). In addition, torrefaction also had an important influence on the subsequent thermochemical conversion, and a two-stage pyrolysis process consisting of torrefaction and fast pyrolysis or catalytic fast pyrolysis was proposed to obtain chemicals and high quality bio-oil (Chen et al., 2016; Neupane et al., 2015; Wang et al., 2016).

However, Liaw et al. (2013) analyzed the effect of pretreatment temperature on the properties of pyrolysis, and concluded that pretreatment at temperatures below 270 °C could not have a major effect on products yield. As the pretreatment temperature over 300 °C, the yield of lignin derived monomers with methoxyl groups seems to be decreased dramatically while the yield of methanol increases. Xiao et al. (2010) pointed out that the temperature range of torrefaction was low and the removal of oxygen was limited. And Yan et al. (2017) concluded that high-yield solid fuel also could be obtained by torrefaction at a temperature of 300 °C. Thus, for an advanced application of

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Table 1
Fiber analyses of raw corn stalk and cotton stalk.

Samples	Chemical composition (d %)			
	Cellulose	Hemicelluloses	Lignin	Others
Corn stalk	21.12	39.81	20.22	18.85
Cotton stalk	35.51	21.98	29.87	12.68

biomass in more possibilities, for instant, chemicals manufactory, biomass deep refinery, etc., it was necessary and viable to explore and describe the deoxidization mechanisms of biomass in a broader temperature range. In addition, there was a lot of research on pyrolysis kinetic (Banyasz et al., 2001; Chan and Krieger, 1981), but very little studies on the kinetics of gaseous products, especially oxygen-containing gases. Han et al. (2015) investigated the oxygen migration in torrefaction products, but the release kinetics of gas products were not involved. In this context, a new pretreatment process of biomass called low temperature deoxidization during 200–390 °C was conducted in a lab-scale fixed bed. Moreover, kinetic parameters of deoxidization and the release property of sulphur were acquired through the analysis of the gas release respectively.

Table 2
Proximate analysis, ultimate analysis of low temperature deoxidization pretreated biomass. (CL, HC, LG, CR, CT and T200 represent the raw cellulose, hemicellulose, lignin, corn stalk, cotton stalk and pretreated sample at 200 °C respectively.).

Samples	Proximate analysis(d %)				Ultimate analysis(daf %)				HHV d/(MJ/Kg)
	V	A	Fc	C	H	N	S	O ^a	
Raw CL	95.50	0.00	4.50	42.70	6.20	0.02	0.05	51.03	15.80
T 200	94.02	0.13	5.85	43.98	6.26	0.01	0.05	49.69	16.15
T 230	93.47	0.23	6.31	45.67	6.35	0.01	0.05	47.93	16.61
T 260	92.02	0.16	7.83	48.24	6.55	0.01	0.05	45.16	17.35
T 290	88.18	0.17	11.65	50.42	6.23	0.01	0.05	43.29	17.78
T 320	73.38	2.10	24.53	67.93	4.64	0.01	0.04	27.38	21.59
T 350	60.15	2.20	37.66	74.62	4.52	0.01	0.04	20.81	23.05
T 390	59.02	2.30	38.68	75.73	3.80	0.01	0.04	20.42	23.24
Raw HC	79.48	4.90	15.63	41.61	5.73	0.02	0.03	52.61	15.33
T 200	76.50	5.50	18.00	48.01	6.18	0.02	0.03	45.77	17.15
T 230	69.98	7.72	22.30	53.72	6.76	0.01	0.03	39.48	18.84
T 260	58.48	8.97	32.55	75.71	5.56	0.02	0.03	18.67	23.96
T 290	39.52	14.82	45.66	76.26	5.58	0.10	0.02	18.05	24.10
T 320	38.09	14.92	46.99	76.79	5.49	0.03	0.02	17.67	24.20
T 350	36.31	15.18	48.51	77.03	5.44	0.03	0.02	17.49	24.24
T 390	35.03	15.83	49.14	77.42	5.41	0.02	0.02	17.13	24.33
Raw LG	58.90	20.23	20.87	47.31	4.91	0.08	0.71	46.99	16.44
T 200	57.92	20.46	21.63	48.01	4.70	0.05	0.70	46.55	16.54
T 230	54.11	20.31	25.58	48.37	4.68	0.02	0.70	46.23	16.62
T 260	51.64	21.34	27.01	49.14	4.68	0.02	0.70	45.47	16.81
T 290	49.96	21.49	28.55	50.04	4.46	0.05	0.69	44.75	16.96
T 320	44.48	23.70	31.82	51.41	4.43	0.01	0.69	43.46	17.29
T 350	43.91	23.75	32.34	53.46	4.29	0.06	0.69	41.51	17.76
T 390	39.04	25.79	35.17	54.95	4.39	0.02	0.67	39.97	18.18
Raw CR	73.88	8.69	17.44	49.83	6.99	1.27	0.29	41.63	17.94
T 200	71.53	9.47	19.00	52.95	6.72	0.67	0.28	39.37	18.63
T 230	69.81	8.04	22.15	55.49	6.01	0.70	0.28	37.52	18.98
T 260	61.24	9.55	29.21	61.91	5.60	0.87	0.28	31.35	20.45
T 290	46.39	11.15	42.46	66.96	5.36	1.09	0.27	26.32	21.64
T 320	45.75	9.39	44.86	71.22	5.21	0.85	0.27	22.45	22.67
T 350	42.28	12.00	45.72	74.97	5.02	1.19	0.27	18.55	23.55
T 390	39.32	13.16	47.51	76.55	4.33	0.95	0.26	17.91	23.66
Raw CT	78.62	4.81	16.57	49.22	7.16	1.40	0.34	41.88	17.86
T 200	78.01	4.95	17.04	53.20	6.00	1.12	0.34	39.35	18.39
T 230	75.49	5.52	18.99	57.59	5.61	1.24	0.33	35.24	19.35
T 260	70.98	6.53	22.49	64.48	5.51	1.39	0.33	28.28	21.07
T 290	54.99	10.13	34.88	72.38	5.34	1.79	0.32	20.16	23.02
T 320	51.45	8.23	40.32	75.17	5.06	1.82	0.32	17.63	23.61
T 350	44.61	9.90	45.50	77.72	4.65	1.70	0.32	15.62	24.09
T 390	39.26	11.26	49.48	79.04	4.49	1.90	0.31	14.26	24.36

^a The oxygen content was calculated by difference.

2. Material and methods

2.1. Materials

The corn stalk and cotton stalk used in this study was obtained in Zhoukou city, Henan Province, China. The samples were dried at 105 °C for 24 h and ground to less than 60 mesh (< 250 μm) using a commercial wood grinder (HJF800, Huijie Machine Corp., China). The fiber analyses method was adopted as Chen et al. (2010), and the results were shown in Table 1.

Component model compounds of biomass, cellulose, xylan and lignin were provided by Sigma-Aldrich Co., Ltd. It was in the form of white and microcrystalline powders of 20 μm particle size. All samples were dried at 105 °C for 24 h before the low temperature deoxidization experiment. The proximate and ultimate analyses and the high heat value were shown in Table 2.

2.2. Low temperature deoxidization experiment

A lab-scale tube furnace system was used for the deoxidization experiment as shown in Fig. 1. It consisted of a vertical alundum tube (38 mm ID and 350 mm long), an electrical furnace with a temperature control device. The experimental method and parameter selection was

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