



# Diagnosis of bed agglomeration during biomass pyrolysis in fluidized-bed at a wide range of temperatures



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## HIGHLIGHTS

- Diagnosis of bed agglomeration was conducted for biomass fast pyrolysis in fluidised bed.
- Bed agglomeration can be modelled via sand loading at a wide range of temperatures.
- At low temperatures (<310 °C), sand loading follows an endothermic reaction pathway.
- At high temperatures (>350 °C), sand loading follows an exothermic reaction pathway.

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## ABSTRACT

This paper reports the diagnosis of bed agglomeration during biomass fast pyrolysis in a fluidized-bed reactor at a wide range of temperatures (200–700 °C). The results show that under all conditions, bed agglomeration can be modelled using sand loading ( $S_L$ ), which is the mass of sand that directly interacts with the incoming biomass feed normalised to the total mass of biomass fed into the reactor. The values of sand loading  $S_L$  during pyrolysis is greatly influenced by the ethanol-soluble extractive contained within the raw biomass. For the pyrolysis of the raw biomass, at temperatures <310 °C,  $S_L$  follows an endothermic reaction pathway ( $E_a = 28$  kJ/mole) during which its values increase with increasing temperature. However, at higher temperatures >350 °C,  $S_L$  follows an exothermic reaction pathway ( $E_a = -45$  kJ/mole) during which its values decrease with increasing temperature.

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## 1. Introduction

Mallee trees are presently planted widely in Western Australia premium agricultural land via alley-farming to help manage and address the dryland salinity issues arisen from the past adverse farming practices [1,2]. These trees lead to an opportunity for supplying a large-scale, secure, renewable and close-to-carbon-neutral biomass feedstock which can be converted to electricity, biofuels and other products via various technologies [2–5]. Fluidised-bed technology is a promising approach for the thermal chemical processing (including pyrolysis, combustion and gasification [6–13]) of low-rank solid fuels such as biomass. Therefore, understanding the thermochemical behaviour of biomass particles in a fluidised-bed reactor is of critical importance to the practical applications of these processes.

For fluidised-bed reactors, bed agglomeration can lead to detrimental defluidisation that is an important consideration for the durable operations of these reactors. Previous studies mostly

focused on bed agglomeration caused during the gasification or combustion of solid fuels such as biomass [8–10]. However, until recently, little work has been done on bed agglomeration during biomass fast pyrolysis. The pioneering work by the same authors [14,15] have clearly demonstrated that bed agglomeration does take place during biomass pyrolysis in a fluidised bed reactor. Such bed agglomeration is a result of interactions between bed materials (sand) and the sticky agents (organic matter) generated from biomass pyrolysis, depending on both biomass properties and pyrolysis reaction conditions. Sand loading ( $S_L$ ), which is defined as the mass of sand sticking to the pyrolysing biomass particles in the bed (to form bed agglomerates) normalised to the total mass of biomass fed into the reactor, has also been introduced [16] for evaluating bed agglomeration. It was shown that  $S_L$  is a powerful parameter for diagnosis into bed agglomeration during biomass fast pyrolysis in fluidised bed under conditions pertinent to bio-oil production at 500 °C [16].

However, application of sand loading  $S_L$  has not been attempted for the diagnosis of bed agglomeration during biomass fast pyrolysis in fluidised bed at a wide range of temperatures. Hence, the objectives of this paper is to apply sand loading  $S_L$  to the diagnosis of

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## Nomenclature

$A_k$	frequency factor ( $\text{min}^{-1}$ )	$PSD$	particle size distribution
$E_a$	activation energy ( $\text{kJ mole}^{-1}$ )	$R$	biomass feed to sand ratio, $g_{\text{total,biomass(db)}}/g_{\text{total,sand}}$
$F$	feed rate of biomass ( $\text{g min}^{-1}$ )	$S_L$	sand loading, i.e. mass of sand (g) interacting with the biomass per unit mass of biomass feed (g, db), $g_{\text{sand}}/g_{\text{biomass(db)}}$
$K$	ratio constant for biomass sand loading ( $S_L$ ), $g_{\text{sand}}/g_{\text{biomass(db)}}$	$S_{LO}$	sand loading at holding time $t_0$ , $g_{\text{sand}}/g_{\text{biomass(db)}}$
$K_S$	reaction constant related to holding time ( $t_h$ ), $g_{\text{sand}}/g_{\text{biomass(db)}}/\text{min}$	$T$	temperature ( $^{\circ}\text{C}$ or $\text{K}$ )
$K_R$	reaction constant with respect to biomass to sand ( $R$ ), $g_{\text{sand}}/g_{\text{biomass(db)}}$	$t_f$	time for biomass feeding, min
$M_{B0}$	actual total mass of biomass fed to reactor during experiment (g)	$t_h$	holding time, min
		$t_0$	initial holding time, 0 min, taken at the instant when the biomass feeding completes

bed agglomeration during biomass pyrolysis in fluidised-bed at a wide range of temperatures (200–700  $^{\circ}\text{C}$ ).

## 2. Methods

### 2.1. Sample preparation

Leaf biomass material was collected from the Narrogin region of Western Australia. The detailed sample preparation can be found elsewhere [14,15]. In brief, the leaf samples were collected, sorted, air dried, cut and then sieved to prepare the size fraction of 355–500  $\mu\text{m}$  (the “raw” biomass sample) for subsequent experiments. The size fraction was used as the Biot number is less than 0.1, under which conditions that the effect of the intra-particle heat transfer can be ignored [17–20]. The raw biomass was also washed using ethanol to remove extractives in the biomass to prepare an “ethanol-washed” biomass sample, following the procedure described elsewhere [15]. The resulting leachate was also subjected to evaporation at 35  $^{\circ}\text{C}$  in a fume cupboard to prepare the “extract” sample after ethanol was evaporated. Table 1 is a summary of the fuel properties of each of the components used in the experiment.

### 2.2. Experiments using a fluidised bed reactor for drying and fast pyrolysis

Two types of experiments were carried out using a laboratory-scale fluidised bed reactor. Type I experiments were the fast pyrolysis (heating rate > 200  $\text{K/s}$ ) experiments of the raw biomass and ethanol-washed biomass samples at 200–700  $^{\circ}\text{C}$  and 200–600  $^{\circ}\text{C}$ , respectively. The reactor was loaded with 19.5 g sand (125–250  $\mu\text{m}$ ) as bed material. The reactor was firstly fluidised using argon and preheated at a desired temperature, followed by the feeding of 1.95 g raw or ethanol-washed biomass sample (355–500  $\mu\text{m}$ ) into the reactor at 0.3  $\text{g/min}$ . After the feeding is completed, the reactor was held at the temperature for a further 15 min holding time ( $t_h$ ). Upon the completion of an experiment, the reactor was immediately lifted out of the furnace and allowed to cool down to below 50  $^{\circ}\text{C}$  while the fluidised gas continuously flows through the reactor. Type II experiments were on the pyrolysis of the extract sample, also performed in the same fluidised bed reactor at 170–600  $^{\circ}\text{C}$  under fast heating conditions (heating rate > 200  $\text{K/s}$ ). The extract sample was dissolved in ethanol (1:24 based on mass) and then fed into the reactor using a syringe feeder. The feed rate was adjusted to be equivalent to the same amount extract fed into the reactor when the raw biomass was used, with all other operation parameters being the same as those used in Type I experiments. In each experiment (Type I or II), the weight loss (on a dry basis) of the feed sample during pyrolysis was determined by the difference in weight of the reactor before and after

**Table 1**  
Fuel properties of the raw biomass, ethanol-extracted biomass and extract samples.

Sample	Proximate analysis (wt%) <sup>a</sup>				
	Moisture	Ash	VM <sup>c</sup>	FC <sup>d</sup>	
Raw biomass	4.8	3.7	71.2	20.3	
Ethanol-washed biomass	2.1	4.4	72.7	20.8	
Extract	10.2	1.6	67.5	20.7	
Sample	Ultimate analysis (wt% daf) <sup>b</sup>				
	C	H	N	S	O <sup>e</sup>
Raw biomass	52.19	6.55	1.35	0.72	39.19
Ethanol-washed biomass	47.82	6.12	1.71	0.79	43.57
Extract	57.32	7.91	0.31	0.88	33.58
Sample	Ash analysis (wt%)				
	Na	K	Mg	Ca	
Raw biomass	0.645	0.545	0.136	0.889	
Ethanol-washed biomass	0.728	0.672	0.156	1.274	
Extract	0.414	0.241	0.082	0.008	

<sup>a</sup> As-received.

<sup>b</sup> Dry-ash-free.

<sup>c</sup> Volatile matter.

<sup>d</sup> Fixed carbon.

<sup>e</sup> Calculated by difference.

the experiment. The resulting fluidised bed now termed the “bed sample” was also collected for further analysis.

### 2.3. Sample characterisation

The resulting bed samples were screened using 8 different size screens (106  $\mu\text{m}$ , 125  $\mu\text{m}$ , 355  $\mu\text{m}$ , 500  $\mu\text{m}$ , 0.71 mm, 1.0 mm, 1.7 mm and 3.15 mm). Following a combustion method detailed elsewhere [16], the amount of sand in bed agglomerates (i.e. those that interact with the pyrolysing feed material to form agglomerates) was determined. Similar to those used previously [16], sand loading  $S_L$  is defined as the mass of sand as part of bed agglomerates normalised to the total mass of feed introduced into the reactor. Separate samples of the feed samples were also subjected to thermogravimetric (TG) analysis following an adapted ASTM E 871–82 method [21] for proximate analysis. Elemental analysis of the feed samples were carried out using a CHNS/O analyser (model: PerkinElmer 2400 series II).

## 3. Results and discussion

### 3.1. Sand loading during biomass fast pyrolysis at a wide range of temperatures

Fig. 1 presents sand loading as a function of pyrolysis temperature (200–700  $^{\circ}\text{C}$ ) for the fast pyrolysis of raw biomass,

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