DOI: 10.1016/j.cej.2015.09.044

Agglomeration detection by pressure fluctuation analysis during *Cynara Cardunculus* L. gasification in a fluidized bed

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Abstract

The bed material and the fuel particles are the main actors in the agglomeration process in a bubbling fluidized bed reactor during a gasification conversion. The physical and chemical reactions between them determine whether the bed operates under a proper fluidization condition or the quality of the fluidization is deteriorated due to agglomerates appearance inside the bed. This work analyzes the pressure signals of a fluidized bed during *Cynara Cardunculus* L. gasification in a reactor with an inner diameter of 52.8 mm. Jetsam and flotsam behaviour of fuel particles are analyzed using sepiolite and silica sand as bed materials, respectively. The wide band energy and the attractor comparison tool are used to detect agglomeration, and, as a consequence, the defluidization of the bed. Similar defluidization times are obtained employing both methods. The wide band energy analysis shows that, for jetsam fuel particles, the endogenous bubbles produced by the fuel devolatilization inside the bed change the energy distribution, while for flotsam fuel particles, the cap-clinker agglomerate formed is detected by high frequencies in the power spectrum.

Keywords: biomass; gasification; fluidized bed; agglomeration detection; pressure signals

Nomenclature

D bed diameter [m].

E_{wb} dimensionless wide band energy [-].

f frequency [Hz].

f_{cI} lower cut-off frequency [Hz].

f_{cII} upper cut-off frequency [Hz].

f_c cutting frequency [Hz].

f_N Nyquist frequency [Hz].

f_s sampling frequency [Hz].

h_b bed height [m].

t time [min].

t_d defluidization time [min].

T temperature [ºC].

U gas velocity [m/s].

U_{mf} minimum fluidization velocity [m/s].

X mean value [-].

Greek symbols

Δf frequency resolution [Hz].

σ standard deviation [-].

ρ_{fuel} density of the biomass particle [kg/m^3].

Abbreviations

CE Cumulative Energy.

ER Equivalence Ratio.

LAL Lower Action Limit.

PSD Power Spectral Distribution.

SPC Statistical Process Control.

UAL Upper Action Limit.

1. Introduction

Fluidized bed is a widely used technology in several applications due to its high heat and mass transfer rates. These applications range from pharmaceutical to thermochemical processes. Yet, operational problems are encountered in industrial applications. In energy conversion systems, such as fluidized bed combustors or gasifiers, the formation of agglomerates at high temperatures is one of the major problems. Agglomerates are typically formed due to the interaction between fuel particles and bed material. Depending on the characteristics of both the fuel and the bed particles, the appearance of adhesive inter-particle forces can lead to the agglomerates formation, which could cause the bed defluidization if the process is not promptly detected.

The chemical composition of fuel and bed particles produces the majority of the agglomeration incidents at high temperature. The interaction between ash and bed material plays an important role in the defluidization process. Alkali elements from biomass tend to volatilize and condensate on the bed particle surface. Different bed materials such as silica sand, magnesite, olivine or dolomite have been tested to elucidate their effect on the agglomeration process. Silica sand reacts with alkalis and retains them, forming alkali silicates which act as a glue between particles while materials such as dolomite promotes a higher alkali volatilization [1,2]. Lin et al. [3] used sand as bed material during wheat straw combustion, founding K_2O-SiO_2 compounds deposited on sand particles which produced the agglomeration of the bed. Fryda et al. [4] found no much difference in the defluidization temperature using sand and olivine, concluding that these bed materials had small chemical interaction with fuel ash. Limestone was used by Fernández et al. [5], founding no sign of agglomeration due to the adsorption of alkali salts on the surface. Liliedhal et al. [6] used magnesite and olivine as bed materials, obtaining a proper operation performance in both cases. Defluidization was avoided, although some clinkers from biomass ash were found inside the bed when using magnesite. The interaction between magnesite and fuel ash was negligible since this bed material has no glass-forming to react with alkali elements. Magnesite showed the best results during the experiments performed by Siedlecki and de Jong [7] while some agglomeration was detected with sand and olivine. Furthermore, some materials are commonly added to the bed in order to reduce/avoid agglomeration. Fernández et al. [8] employed dolomite, lime and kaolin, among other materials, as additives to a silica sand fluidized bed. These materials were effective by the dilution of the biomass ash and only kaolin was capable of reacting with fuel ash, forming compounds with a higher melting point. Siedlecki and de Jong [7] also used kaolin in their experiments, eliminating any sign of agglomeration. The inorganic elements which form the fuel ash have also a significant influence in the bed agglomeration. Different authors have reported agglomeration indicators based on the fuel ash composition [9, 10]. The most affecting elements among these indicators are potassium, sodium, silicon, calcium and magnesium. Bed sintering temperature is also an indicator of the bed agglomeration. Liliedhal et al. [6] obtained an empirical equation for this parameter as a function of the feedstock, bed material, and operating conditions. Another possibility to reduce the agglomeration problems is the use a secondary fuel as Abelha et al. [11], who employed bends of cardoon and eucalyptus to reduce this problem.

Other key parameter in agglomeration is the hydrodynamic behaviour of the fuel particle inside the fluidized bed. Fuel particles may have higher densities than the dense phase, showing a jetsam behaviour. This causes the formation of endogenous bubbles when devolatilization occurs, changing the properties of the fuel particles, and promoting their motion throughout the whole bed [12 - 14]. In contrast, flotsam fuel particles tend to remain at the top of the bed showing a low mixing degree with the bed particles [15, 16]. Both fuel behaviours are addressed in this work through the analysis of the pressure fluctuation measurements. Several techniques have been proposed to characterize the dynamics of fluidized bed processes [17]. Focusing on the detection of agglomeration problems, which are typically shown in gasification processes [3, 18], several techniques of the study of pressure signals in the time and frequency domains, and in the state space have been proposed [17, 19]. One of the most used analysis in the time domain is the standard deviation of the pressure fluctuation signal [17]. It has been commonly employed to identify regime changes due to its dependence on the superficial gas velocity. However, the amplitude of the pressure signal is also influenced by the distribution of bed material and by changes in the average suspension density, and thus, it should be employed carefully [19]. Nevertheless, valuable results have been obtained for the defluidization detection using the standard deviation [20 - 22]. The study of the frequency domain gives information about the characteristic time scales of the bed dynamics. The presence of multiple modes of bed mass oscillations that are sensitive to the fluidization regime makes possible the use of the dominant frequency to monitor the process performance [23 - 25]. In the same way, the signal energy computed in different frequency bands can identify regime transitions and agglomeration processes [19, 26, 27]. In the state space or phase space, the pressure signal is studied using non-linear analyses. The collection of the successive states of the fluidized bed is plotted as an attractor [17]. This attractor can be used to compare between different states, which lead to detect slight changes in fluidized bed dynamics, such as the onset of bed agglomeration [25, 28, 29].

The properties of the fuel and bed particles determine whether agglomerates are formed or not. In this sense, a previous work of Serrano et al. [30] showed different agglomeration and defluidization processes for *Cynara Cardunculus* L. gasification. On the one hand, jetsam behaviour of fuel particles was found in a bed formed by sepiolite particles. In these tests, the whole bed tended to agglomerate in one big cylindrical shape piece. On the other hand, flotsam behaviour of fuel particles in a silica sand bed produced flat plate shape agglomerates located at the top of the bed. To the best author knowledge, such different agglomeration processes have not been studied in detail using pressure fluctuation measurements. Thus, the attractor comparison tool and the wide band energy analysis are employed to further understand the agglomeration mechanism of both types of processes.

In this work, the agglomeration processes of jetsam and flotsam fuel particles during the gasification of *Cynara Cardunculus L*. in a lab-scale fluidized bed are analyzed. Pressure fluctuation signals are studied in the frequency domain, by means of the wide band energy, and in the state space employing the attractor comparison tool. Furthermore, the definition of a proper reference state during the gasification process is also discussed.

2. Experimental Setup

- Biomass and bed material

The biomass used in this work is *Cynara Cardunculus* L. It is an herbaceous perennial plant from Mediterranean regions (with hot and dry summers) and has an annual growth cycle [31]. The low nitrogen oxides pollution derived from its chemical conversion, its low water consumption, the improvement of soil characteristics, the possibility to grow in lands unsuitable for food production, and its high volatile content makes *C. Cardunculus* L. an interesting choice for biomass gasification [32]. *Cynara* characterization was performed by means of proximate, ultimate and high heating value analyses. The results of these analyses are shown in Table 1.

Table 1. *Cynara Cardunculus* L. properties.

Biomass particles consisted on pellets of 6 mm in diameter and 15 mm in length with a mean mass of 0.50 g, which were manually fed into the reactor by its upper part. These biomass pellets have an apparent density of 1220.48 kg/m^3.

Silica sand and sepiolite (clay, $Mg_8Si_{12}O_{30}(OH)_4(OH_2)_48H_2$)particles were used as bed materials for the experiments. Silica sand and sepiolite had densities of 2645 and 1551 kg/m^3 respectively, with a particle diameter between 425 and 600 μm in both cases. With these properties, the two bed materials belong to type B particles according to Geldart's classification [33]. The minimum fluidization velocity at 850 ºC was 0.089 m/s for silica sand and 0.057 m/s for sepiolite.

- Experimental facility

A stainless steel lab-scale fluidized bed was used for the experiments using air as gasifying agent. The reactor, with an inner diameter of 52.8 mm, was divided in two sections: a lower part where the gasifying agent was preheated and an upper part where the bed was located (Figure 1). A perforate plate of 2 mm thickness with 38 holes of 0.5 mm in diameter was used as distributor, separating the lower and upper part of the reactor. Two electrical furnaces provided the energy to reach the desired temperature inside the bed and a temperature control system was employed to simulate adiabatic operation conditions. Different K-type thermocouples were placed along the reactor to measure temperature. Pressure was also measured at a height of 30 mm above the gas distributor using a piezoelectric (Kistler type 7261) pressure sensor. The sampling frequency was f_s = 400 Hz.

Figure 1. Schematic of the experimental facility.

- Experimental procedure

Prior to each experiment, the bed material (silica sand or sepiolite) was loaded into the reactor to obtain a bed aspect ratio of h_b/D = 1.5. The air supply system was turned on and the electrical furnaces were set to 850 ºC. Once the bed temperature, measured by thermocouples T2 and T3, reached the selected temperature (850 ºC), minimum fluidization was determined and the air flow was set to the specific value for each experiment. Biomass feeding rate was calculated according to an equivalence ratio (ER) of 0.3. This value is defined as the ratio between the air flow rate introduced into the gasifier and the stoichiometric air flow needed for the complete combustion of biomass. Hence, the feeding rates for silica sand varied from 10.54 to 25.90 g/min, while for sepiolite ranged from 6.46 to 16.44 g/min. Pressure and temperature signals were acquired during 300 s before starting the biomass feeding in order to have reference conditions for each experiment. Then, biomass was fed continuously to the reactor and the experiment finished when pressure fluctuations became zero and the bed seemed to be defluidized. At this point, biomass feeding was stopped and signals were acquired for another 300 s. After this time, the electrical furnaces and the air supply were shut down and the reactor was cooled down. More detailed information about the experimental setup is described in [30].

- Methods of analysis

The wide band energy (E_{wb}) was obtained computing the energy contained within the power spectral density (PSD). This variable is defined as the ratio between the energy in a frequency region and the energy of the whole frequency domain and can be used to detect changes in the fluidization behavior [19]. In a previous work, Gomez-Hernandez et al. [26] studied both the visual and the Student's *t*-distribution approaches available for such a frequency division. The visual frequency division approach showed that the frequency regions obtained were able to detect neither the change in the bed aspect ratio nor the start of the rotating distributor, preventing its use to compute the wide band energy. Therefore, in this work the Student's *t*-distribution approximation of the cumulative energy distribution (CE) of the PSD was employed to divide the frequency spectra. This methodology divided the frequency domain considering the difference between the CE and the Student's *t*-cumulative density function. As a result, the CE frequency distribution can be divided in three regions: two regions of poor matching that correspond to the tails of the CE distribution, and a region of proper matching corresponding to the highest energy content of the distribution. According to this approach, each region is related to different fluidization phenomena, which depends on the CE distribution as well as on the cut-off frequencies. In general, each region represents:

- Region 1 ($\Delta f < f < f_{cI}$): contains the low frequencies, which are associated to the long term dynamics and the larger structures of the bed.
- Region 2 ($f_{cI} < f < f_{cII}$): contains the dominant frequency of the bed, suggesting its relation to the bulk dynamics of the bed.

- Region 3 ($f_{cII} < f < f_N$): includes the high frequency region of the spectrum, and thus, it is related to fast fluidization phenomena such as the bubble eruption on the bed surface and the presence of channels.

Figure 2 shows the influence of the fluidization velocity on the CE for silica sand and sepiolite bed particles. For both materials, the energy of the spectrum is mainly distributed near the dominant frequency of each test, while the tails of the distributions represents around 20-25% of the total energy. The results of applying the Student's-*t* approach are shown in Table 2.

Figure 2. Cumulative energy distribution of the PSD for different bed materials: (a) silica sand (b) sepiolite.

Table 2.Computational settings for the frequency division method.

The wide band energy is employed together with the Statistical Process Control (SPC) scheme in order to define a reference state. This control scheme determines a control zone estimating the control limits of the process. In this way, the identification of the bed defluidization is possible. The main parameters used to estimate the control limits are summarized in Table 3 and further details can be found in [34]:

Table 3. Settings for the SPC monitoring.

The attractor comparison tool is used to decide if two time series are produced by the same mechanism. Diks et al. [35] proposed a statistical parameter *S* for testing the null hypothesis, which establishes that two muldimensional probability distributions are identical. On the basis of this approach, van Ommen et al. [28] defined a monitoring method that gives an early warning of the onset of agglomeration in a fluidized bed. The attractor of a reference time series of pressure fluctuations is compared with that of successive time series measured during the bed operation. In this way, for *S* parameters larger than 3, the attractor of time series under evaluation is statistically different from the reference attractor, indicating that the fluid-dynamic conditions have changed in the fluidized bed. Therefore, it is possible to detect agglomeration at the very early stages for a given reference state [22, 36].

3. Results

Cynara Cardunculus L. gasification tests in sepiolite and silica sand beds, operated at $U/U_{mf} = 6$, are analyzed in detail employing the on-line monitoring tools described above. Similar results were obtained for the rest of fluidization velocities. Finally, the defluidization time for all the tests is compared to previous results reported by [30].

- Visual observations

Biomass particles showed two different hydrodynamic behaviors according to the bed density (the bulk density for the silica sand and sepiolite bed were 1481.2 and 558.4 kg/m^3 respectively). When sepiolite was used as bed, material biomass particles ($\rho_{fuel} =$ 1220.5 kg/m^3) circulated throughout the whole bed height and its motion was not restricted to the bed surface, they were also found immersed in the dense bed. In these conditions, devolatilization and gasification reactions occurred inside the bed where endogenous bubbles were formed. These endogenous bubbles added to the exogenous bubbles due to fluidization forced the biomass particles to move throughout the whole bed. Thereby, volatiles were produced all along the bed, interacting with the bed particles. This interaction can enhance different catalytic reactions which benefit the final gas composition and tar reduction. On the other hand, during the experiments in a silica sand bed, a completely different behavior was observed. Due to buoyancy effects, biomass particles just floated on the surface of the silica sand bed. A stagnant flame was observed in the freeboard for the whole experiment. This flame appeared due to the combustion of a fraction of the biomass which reacted with the oxygen present in the fluidization gas. Some part of the volatiles was also burned in the freeboard of the bed.

As a consequence of these different behaviors of the biomass particles, the agglomeration process was also different for sepiolite and silica sand beds. In the former case, with sepiolite, ash was generated all along the bed. This more homogeneous distribution of ash inside the bed produced a slower agglomeration process in which the whole bed was involved. As a result, the size and shape of the agglomerates differed from those found when operating in silica sand beds. In a bed composed of sepiolite particles, a big cylindrical clinker was formed, whose dimensions were close to those of the whole bed. However, in a silica sand bed, the maldistribution of fuel particles leaded to generate ash in a narrow part of the bed, close to the bed surface. As the interaction between the bed material and biomass/ash was poor, the agglomeration process was faster, generating a cap-like clinker on the surface of the bed. The rest of the bed, behind this agglomerate, seemed to remain unaltered.

- Combustible behavior: jetsam

During the biomass gasification in a sepiolite bed, bed temperatures and pressure fluctuation signals were monitored. Since the objective of this work is the description of the agglomeration process using the attractor comparison tool and the wide band energy analysis, the definition of a reference state, capable of defining the steady gasification properties, is mandatory. To that end, temperature profiles were employed to set this reference state.

Figure 3 shows the temporal evolution of the temperatures in the bed during a biomass gasification test with a dimensionless gas velocity U/U_{mf} = 6 and using sepiolite particles as bed material. An increase of the bed temperature can be observed at the beginning of the biomass feeding (t = 5 min), reaching a constant temperature after around 2 min. Plenum temperature needed around 10 min to reach a constant value of 820 ºC. Later, the defluidization process was detected by a sudden increase of T2 and T3. In this case, the agglomeration process defluidized the bed after around 42 min. This figure also shows the three states used as a reference for the analysis of the pressure signals. The first reference state is defined prior to the beginning of the biomass feeding. Reference 2 starts 5 min after the biomass feeding, when the bed temperatures showed a constant value of 925 ºC, while Reference 3 is chosen when the plenum temperature reached the steady temperature of 820 ºC.

Figure 3. Temporal evolution of temperature during a gasification process in a sepiolite bed (U/U_{mf} = 6).

Figure 4 presents the *S*-test results employing the reference states described above. As can be seen, the *S*-values are lower than 3 at the beginning of the test for Reference 1, since the attractor comparison tool is still analyzing the reference attractor. However, once the biomass was fed, the *S*-values showed a sharp increase above the threshold. This result identifies the change of the fluid-dynamic behavior of the bed showing the difference between Reference 1, with no biomass, and the gasification beginning, characterized by the formation of endogenous bubbles. The *S*-values increase continuously up to 20 min, suggesting a transition in the fluidization behavior, in a similar time to that needed by the plenum temperature to reach a constant value. Furthermore, the defluidization is also shown as a sharp increase of the *S*-value. Even though changes in the fluidization dynamics can be detected using Reference 1, its use is not recommended since *S*-values larger than 3 will be obtained as soon as biomass is fed into the reactor.

The *S*-test using Reference 2 shows values greater than 3 at the beginning of the test, when no biomass was fed in the bed. As the gasification process progressed, the *S*-values continuously decreased until the reference state was reached. However, the threshold (*S*-value > 3) was exceeded again at t = 17 min when no defluidization was visually observed. Therefore, Reference 2 does not represent the steady state of the gasification process. Greater *S*-values were obtained at the beginning of the test when using Reference 3 as a reference state. In this case, the *S*-values decreased as the gasification process continued, reaching values lower than 3 from t = 17 min until the bed defluidization at t = 42 min. Thus, for monitoring purposes, the reference control state employed should be Reference 3, where the steady state was reached for all the temperatures in the bed, in order to be able to detect the bed defluidization properly using the *S*-test method.

Figure 4. *S*-test for the sepiolite bed using different reference states (U/U_{mf} = 6).

Prior to the computation of the wide band energy, the cumulative energy distribution of the power spectrum was estimated. As for the *S*-test, different time periods were considered to analyze the influence of the reference state on the CE. Figure 5 shows the CE estimated using the reference states described in Figure 3. Furthermore, two extra periods, before and after the bed defluidization, were considered. Figure 5 shows that the biomass feeding produced a clear effect on CE distribution. As can be seen comparing the CE distributions of Reference 1 and 2, the dominant frequency of the bed moved to lower frequencies. This effect is similar to the increase of the gas fluidization velocity, shown in Figure 2. Such a result can be explained by the fuel particle behavior in the sepiolite bed. The jetsam behavior of the fuel particle ensured that the devolatilization occurred within the dense bed, and thus endogenous bubbles were produced, as explained in [13, 14]. These bubbles affect the fluid-dynamic behavior of the entire bed increasing the effective flow rate, and thus, changing the CE distribution. It is worth to mention that this is an averaged result since the reference period considers 5 min of data and the biomass was fed continuously, ensuring the continuous formation of endogenous bubbles.

As gasification progressed from Reference 2 to Reference 3, CE distribution slightly moved to lower frequencies, as shown in Figure 5. At the maldistributed period, which is near the bed defluidization, the CE shows variations at the lower frequencies, suggesting the modification of the fluidized regime. Finally, the CE obtained when the bed is defluidized informs of a total absence of fluidization in this period.

Figure 5. Cumulative energy distribution of the PSD for different periods during sepiolite test (U/U_{mf} = 6).

Instead of using this averaged information to characterize the process performance, the frequency domain can be divided, computing the energy of each frequency band. In this sense, the wide band energy analysis reflects the energy contained in the three regions in which the frequency domain is divided. This variable shows the evolution of the CE distribution, although, previous to its computation, a definition of the reference state is needed. Considering the temporal evolution of the bed temperatures and the results of the *S*-test, Reference 3 was employed to estimate the cut-off frequencies and the control limits. The energy contained in the frequency regions and the limits that define the control state are presented in Figure 6. According to the SPC monitoring scheme, these limits were estimated as $\bar{X} \pm 3\sigma$, where the mean, $\bar{X}$, and the standard deviation, σ, were calculated for each energy region during the reference state. As suggested by [19, 26], the energy contained in each region can be identified in terms of the time scale dynamics of different fluidization phenomena. In this way, E_{wb3} represents the energy of the high frequencies, which are related to the appearance of channels, E_{wb2} contains the dominant frequencies of the bed, suggesting its relation to the bulk dynamics, and E_{wb1} is identified with the larger structures of the bed.

As can be seen in Figure 6, all the regions present energy values out of the control state prior to the biomass feeding. As the biomass was fed, the CE changed and the wide band energy values moved towards the control zone. During the beginning of the gasification process, the energy was transferred from the higher, E_{wb3}, to the lower E_{wb2} and E_{wb1} frequencies. This result can be explained by the modification of the frequency spectra, and thus, by the change of the CE distribution, as shown in Figure 5. The energy is mainly contained within the high frequency region at the beginning of the test. As the gasification process progressed, the CE moved to lower frequencies, showing similar values to the reference state. Concerning the energy values plotted in Figure 6, the energy contained in the high frequencies, E_{wb3}, was transferred to medium and low frequency regions, E_{wb2} and E_{wb1} respectively. The three frequency regions reached the control zone at t = 15 min. No significant changes were shown during the experiment up to the bed defluidization. At this moment, t = 42 min, the defluidization of the bed can be detected by the three energies as an out of the control zone. The energy decrease of E_{wb2}, which is related to the bulk dynamics, suggests the complete agglomeration of the bed. This energy was transferred to E_{wb3} and E_{wb1} pointing to a sharp change in the fluidization dynamics. Quite similar results were obtained using the attractor analysis based on Reference 3 (Figure 4) and the energy values of the frequency regions (Figure 6).

Figure 6. Wide band energy analysis during a biomass gasification process in a sepiolite bed (*U/U_{mf}* = 6).

- Combustible behavior: flotsam

A flotsam behavior was observed when using the same fuel particles in a silica sand bed due to the lower density of the biomass pellets in comparison with the bed bulk density. The low axial mixing of the fuel, which remained at the top of the bed, and the formation of alkali silicates during the gasification enhances the defluidization process. In these tests, the agglomerate formed is plate-shaped, showing a different defluidization process to that described previously for the sepiolite bed. The same methodology applied to the sepiolite bed was also applied to the silica sand bed.

The temporal evolution of the temperature measurements in the bed during gasification of biomass in a silica sand bed operated at U/U_{mf} = 6 is presented in Figure 7. The short defluidization time, t = 5 min, makes difficult to define a steady process during these gasification tests. For this reason, the reference state employed for the attractor comparison tool and the wide band energy analysis was placed in this case at the beginning of the test, prior to the biomass feeding.

Figure 7. Temporal evolution of temperature during a gasification process in a silica sand bed (*U/U_{mf}* = 6).

Figure 8 presents the results of the attractor comparison method. The *S*-values showed a sharp increase at t = 8 min. This result is explained by the rapid defluidization when operating in a silica sand bed. However, once the bed was defluidized and the biomass feeding was stopped, the *S*-values decreased up to a constant value. The formation of a cap-clinker near the bed surface explains this decrease of the *S*-values. When this agglomerate was formed, the rest of the bed seemed to remain unaltered, showing a barely fluidized state. This result differs from that obtained for the sepiolite bed (Figure 4) when the *S*-values did not decrease to a constant value after the bed defluidization due to the formation of a big cylindrical clinker in the whole bed.

Figure 8. *S*-test for the silica sand bed using different reference states (*U/U_{mf}* = 6).

The results, obtained using the wide band energy analysis to the biomass gasification process in the silica sand bed, are displayed in Figure 9. The same reference state employed for the *S*-test was used for the computation of the cut-off frequencies and the control limits. Similarly to the *S*-test results, an out of control state was detected after t = 8 min for E_{wb3} and E_{wb1}, pointing to a fluidization change, whereas E_{wb2} identified the change of the bulk dynamics at t = 9 min. Concerning E_{wb2}, Figure 9-b, the energy values decreased up to a constant value, indicating that part of the bed behind the cap-clinker was still fluidized. This effect was also detected by the *S*-test analysis shown in Figure 8. Regarding the long term phenomena, which is encoded in the energy of E_{wb1} (Figure 9-c), a peak permitted also to detect the bed defluidization, although the values of E_{wb1} returned to the control zone as the test progressed in time. Such a result, together with the high energy values of E_{wb2} in comparison to the low values of E_{wb2} after defluidization in the sepiolite bed (Figure 6-b), suggests that the bottom of the bed was still fluidized after the formation of the cap-clinker agglomerate at the bed surface. The fast fluidization phenomena, which is identified with E_{wb3}, presents two different patterns. In the first part, the energy decreased after the biomass feeding until the bed defluidization (t = 8 min). This energy decrease was produced by the progressive clogging of the bed surface, which made difficult the eruption of bubbles on the bed surface. Since the energy of E_{wb3} is related to the fast fluidization phenomena and the finer structures of the bubbling fluidization [19, 26], the modification of the bubble eruptions over the bed surface changes the frequency spectra from multiple bubble to slug-like regimes, and thus, modifies the energy of E_{wb3}. In the second part, the energy increased after the bed defluidization up to a constant value during the rest of the test. Such an energy change is explained by the formation of channels throughout the cap-clinker, which tend to increase the energy contained in the higher frequencies of the power spectrum (E_{wb3}) [26].

Figure 9. Wide band energy analysis during a biomass gasification process in a silica sand bed (U/U_{mf} = 6).

The energy of E_{wb3} at the end of each experiment was further analyzed as a function of the air velocity in order to check whether or not this clogging effect and subsequent channels formation was common for all the silica sand tests. Pictures and further details of the agglomerates can be seen in [30]. Figure 10 shows the energy of E_{wb3} at the end of each experiment in the silica sand bed, calculated as the mean value of E_{wb3} during the last minute of the test. A progressive energy increase of E_{wb3} with the air velocity is observed in Figure 10. This result is caused by the different width and consistency of the agglomerate formed at the bed surface. For lower velocities, the cap-clinker was easily broken by small channels, and thus, the energy of E_{wb3} present lower values. As the air velocity was increased, greater cap-clinkers were formed and bigger channels appeared through the agglomerate, which increases the energy of E_{wb3}.

Figure 10. Influence of the air velocity on the values of E_{wb3} at the end of the gasification process in a silica sand bed.

- Defluidization time

The defluidization time is calculated using both the *S*-test and the wide band energy analysis in order to compare the current analyses with the previous study of Serrano et al. [30], where the defluidization time was determined by means of the analysis of the pressure fluctuations in the time and frequency domains. In that work, the standard deviation was used to determine the defluidization time of the gasification tests. The defluidization time was determined when the standard deviation values were below a threshold, which was fixed to 50% of the standard deviation values before to the biomass feeding. Figure 11 shows the results reported in the previous work [30] based on the standard deviation of the pressure signals, together with the times estimated using the analysis techniques proposed in this work.

The standard deviation and the wide band energy analysis show similar defluidization times for silica sand bed, whereas the *S*-test presents higher defluidization times (Figure 11-a). However, the time resolution of the *S*-values, which is 1 min, should be considered. Longer defluidization times were obtained for the sepiolite tests in Figure 11-b due to the different behavior of the fuel particles. Similarly to the silica sand results, the standard deviation and the wide band energy analyses present similar results for the sepiolite bed, while the *S*-test shows higher defluidization times.

Figure 11. Defluidization time during a biomass gasification process in a fluidized bed using as bed material: (a) silica sand, and (b) sepiolite.

4. Conclusions

The gasification behaviour of jetsam and flotsam fuel particles have been studied in detail by analyzing pressure fluctuation signals, using both the *S*-test and the wide band energy analysis. The monitoring techniques showed their capability to determine the defluidization time, and, in the case of the wide band energy, a detailed description of the agglomeration mechanisms occurring in each bed.

The jetsam behaviour of fuel particles in a sepiolite bed formed endogenous bubbles within the dense bed. This effect was clearly detected by the energy distribution of the power spectrum at the lower frequencies. Similarly, the wide band energy method identified the bed defluidization by a significant reduction of the energy of E_{wb2}, which indicates a deterioration of the bulk dynamics for jetsam fuel particles.

In contrast to the sepiolite bed, flat plate shape agglomerates located at the top of the bed were formed when operating in a silica sand bed, because of the flotsam behaviour of the biomass particles. In this case, the defluidization process was faster than when using sepiolite due to the higher concentration of alkali compounds at the top of the bed, enhanced by the low axial mixing within the bed. According to the energy of the high frequencies (E_{wb3}), the bubble eruption rate was progressively reduced as the cap-clinker agglomerate was formed on the bed surface. However, the bed was still fluidized under the cap-clinker, which is pointed by the energy values of E_{wb2} and E_{wb1}. As the superficial gas velocity increased, greater cap-clinkers were formed and bigger channels appeared through the agglomerate, which produced the increase of E_{wb3}.

Future works should study the cap-clinker formation and its breakage for flotsam fuel particles in larger facilities. This agglomerate is usually found in the bed surface, and thus, could attenuate the bubble coalescence, changing the pressure fluctuations signals.

Acknowledgements

The authors would like to express their gratitude to the financial support of the Spanish Ministry of Economy and Competiveness from project ENE2014-54942-R.

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