

## Some Results of Preliminary Studies to Create an Industrial Reactor for Oxidative Torrefaction of Biowaste

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The torrefaction process of biowaste types, such as a mixture of chicken manure and wood sawdust, wood sawdust and sunflower husk, is proposed to be carried out in a fluidized bed, which is formed by the crushed biowaste, blown from the flue gases leaving the boiler. The preliminary studies of the oxidative torrefaction process showed that the time of biomass particles in such reactor may not exceed 5 minutes. In order to ensure the indicated time of the biomass particles in the oxidation torrefaction reactor, it is proposed to place a number of vertical partitions installed at a distance of 50 mm from each other in a fluidized bed. These partitions provide a loop-like movement of biowaste material from the place of loading into the reactor to the place of unloading. As studies on a cold apparatus showed, these partitions can provide the necessary time in the reactor and a set degree of biowaste treatment. For a reactor with a diameter of 1 m, the presence of such a package of partitions provides the time of biomass particles in a fluidized bed for 5 minutes with a reactor productivity of 300 kg/h in the initial biomass or 150 kg/h in the resulted biochar.

### 1. Introduction

Biomass is a CO<sub>2</sub> neutral and widely available fuel. However, its widespread application is limited due to the fact that the initial biomass has a lower calorific value, higher humidity and lower bulk density, than fossil fuels. Torrefaction is a method of low-temperature (200 - 300 °C) treatment in an oxygen-free environment that can reduce the biomass moisture, increase the hydrophobic properties of biomass, increase the calorific value of biomass, make the biomass more suitable for further thermal processing through pyrolysis, gasification, and burning (Chen et al., 2015).

At the same time, torrefaction is an energy-consuming process, and the use of an inert gas (nitrogen) makes this process even more expensive.

It is more economical to use not inert gas, but flue gas from a boiler or furnace (Mei et al., 2015, Uemura Y et al., 2017). The oxygen concentration in the flue gas can be between 6 and 14 vol. % (Uemura Y. et al., 2017). In oxidative torrefaction of biomass, in addition to reactions associated with the removal of part of the volatile substances and oxygen, exothermic reactions are also observed. These reactions contribute to the reduction of energy consumption for the torrefaction process. During the oxidative torrefaction, the time required for torrefaction is also reduced. In order to obtain the same mass loss as with non-oxidative torrefaction, lower temperatures are required for oxidative torrefaction (Chen W.-H., et al., 2014).

Unfortunately, oxidative torrefaction is implemented only in laboratory set-ups, including fluidized bed reactors (Wang Z., et al., 2018). In this research we studied the process of oxidative torrefaction of sawdust in a fluidized bed of glass balls with a diameter of 1.0 mm at a temperature of 240 - 300 °C and the oxygen concentration in the gas mixture, which was supplied to the reactor, from 0 to 9 vol. %. The experiments have shown that at oxidative torrefaction it is possible to increase the calorific value of sawdust to 22 MJ/kg (in the absence of oxygen, the higher calorific value did not exceed 20 MJ/kg), reduce the hemicellulose content to 8.1% versus 14.5% in the absence of oxygen, and reduce the volatile content substances up to 56% versus 70.38%, increase the content of fixed carbon to 43% versus 29%. So, the oxidative torrefaction provides biofuels more suitable for co-burning with coal than non-oxidative torrefaction.

On the other hand, in a fluidized bed it is difficult to ensure a uniform processing of raw materials. In paper (Wang Z., et al., 2018), sawdust was continuously introduced into a torrefaction reactor. As the sawdust was processed, the sawdust lost weight, was removed from the fluidized bed by a gas stream and separated from the gas stream in a cyclone. It turned out that after 50 minutes of continuous work of the experimental setup, not all sawdust could leave the fluidized bed of glass balls, which led to an increase in the bed height by 10-30%. Sawdust, taken from a fluidized bed after continuous oxidative torrefaction for 50 minutes at a temperature of 300 °C, contained 56% of volatiles and 43% of fixed carbon, and selected from a cyclone, contained 78% of volatiles and 22% of fixed carbon. So, the degree of heterogeneity of the biomass processing in the experiments (Wang Z., et al., 2018) was 39 - 95%.

According to a contract with the Ministry of Science and Higher Education of Russia, we have developed a set of equipment for generating thermal and electric energy from biowaste (a mixture of chicken manure and sawdust, sunflower husk, wood waste and others). The complex includes a 2 MW boiler equipped with a fluidized bed furnace in which biowaste is burned. In the boiler, a high-temperature coolant is heated up to 160 °C, which is supplied to a turbogenerator manufactured by ZUCCATO ENERGIA S.r.l., (Italy) with the organic Rankine cycle. The turbogenerator generates 250 kW of electrical energy. The flue gases behind the boiler contain 6% oxygen and have a temperature of 350 °C. It is proposed to use these gases for oxidative torrefaction biowaste. Based on our own research experience in the area of torrefaction of various biowaste (Isemin R., et al., 2018), including in a fluidized bed (Isemin R., 2019), and drawing on the experience of designing plants for heat treatment of biomass in a fluidized bed ( Isemin R., et al., 2016) we chose a fluidized bed oxidative torrefaction method for biowaste processing.

In order to create an industrial reactor, the laboratory studies of the oxidative torrefaction process in a fluidized bed were carried out, as well as the studies of the motion of particles in a "cold" fluidized bed apparatus. The results of these studies are presented in this paper.

## 2. Experimental Set – Up.

### 2.1 Oxidative torrefaction set-up

The studies of the oxidative torrefaction process were carried out on a set-up, a diagram of which is shown in Figure 1.

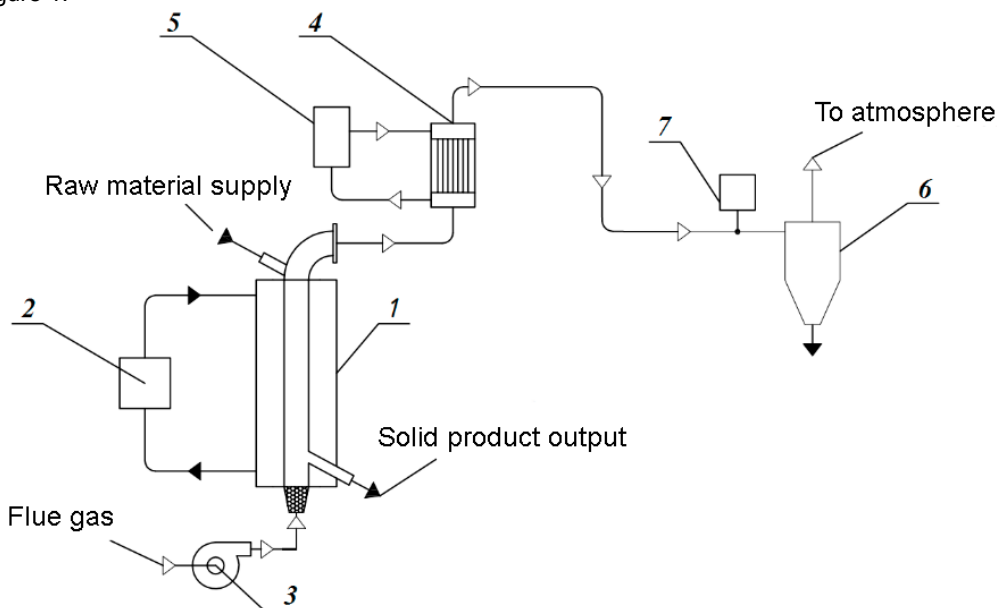


Figure 1: Scheme of experimental setup for studying the process of oxidative torrefaction of biowaste

The experimental setup for studying the process of oxidative torrefaction of biowaste in a fluidized bed consisted of a reactor 1 with a diameter of 108 mm and a height of 1000 mm. The reactor had a jacket, into which a liquid high-temperature coolant with a temperature of 300 °C was supplied, heated in an electric boiler 2. The biomass bed in reactor 1 was converted into a fluidized state by flue gases, which were supplied to the lower part of reactor 1 using a smoke exhauster 3 from the boiler (not shown in the diagram), in which biofuel is burned. The gases, leaving the reactor 1, were cooled in the heat exchanger 4 by water circulating with a pump (not shown in the diagram) between the heat exchanger 4 and the water storage tank 5.

The formed condensate was removed from the system, and non-condensing gaseous torrefaction products were released into the atmosphere after purification in cyclone 6.

Before the experiments began, the set-up was cleaned with flue gases, which were cooled in the heat exchanger 4, and, after removing moisture from the gases, they were exposed to the chemical analysis for the content of  $CO$ ,  $CO_2$ ,  $H_2$  and  $CH_4$  using Vario Plus Industrial flow-type gas analyzer (SYNGAS) 7. The torrefaction process of crushed sunflower husks was studied, which had the following fractional content: the proportion of particles larger than 5 mm - 0.02%, the proportion of particles from 2 to 5 mm in size - 6.48%, from 1 to 2 mm - 30.84%, from 0, 4 to 1.0 mm - 50.54%, from 0.2 to 0.4 mm - 9.72%, from 0.09 to 0.2 mm - 2.4%. The true density of husk particles was  $520 \text{ kg/m}^3$ . The minimum fluidization velocity of husk particles was 0.3 m/s. The working gas velocity at the inlet to the reactor was 0.45 m/s.

The mass of crushed particles of sunflower husk before and after torrefaction was determined using Acom JW-1 laboratory scales, husk moisture was measured using Ohaus MB45 moisture analyzer, and ash and volatiles were determined by a thermogravimetric analysis using NETSCH STA 409 PC/PG thermal analyzer. The elemental composition of husks before and after heat treatment was determined on Elementar Vario Macro Cube elemental analyzer. The value of the higher calorific value of the husk before and after treatment was determined using BKS-2X calorimeter.

During the experiment, a portion of biomass in the amount of 218 g was periodically loaded into reactor 1, which created a bed of particles of 100 mm height. The temperature of the flue gases supplied to the reactor 1 was 350 - 400 °C. The temperature inside the fluidized bed of crushed husk particles was 300 °C.

## 2.2 "Cold" apparatus for movement test

In order to simulate the movement of biomass particles in a fluidized bed reactor and to obtain the necessary data for calculating an industrial reactor, the "cold" apparatus was created, the scheme of which is shown in Figure 2.

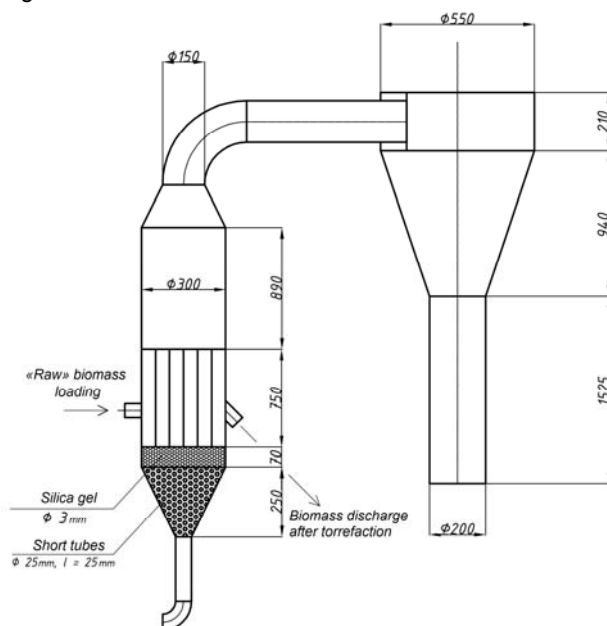


Figure 2: Scheme of "cold" apparatus

The "cold" apparatus was a cylindrical apparatus with a diameter of 300 mm and a height of 1640 mm. The apparatus relied on an air distribution grid made in the form of a layer of silica gel particles with a diameter of 3 mm, clutched between two grids. A block of vertical partitions (Figure 3) was placed inside the fluidized bed apparatus, which ensured a loop-like movement of biomass particles from the loading point to the discharge point.

In order to study the time of husk particles in such apparatus, the husk particles colored with a water-soluble dye were introduced into the bed of uncolored husk particles. After drying of the dye, these particles did not differ in mass and size from other particles of the bed. The experiments were carried out at an air velocity related to the cross section of an empty apparatus equal to 0.45 m/s. 500 g/min of colored particles were continuously introduced into the bed of uncolored particles. (30 kg/h).

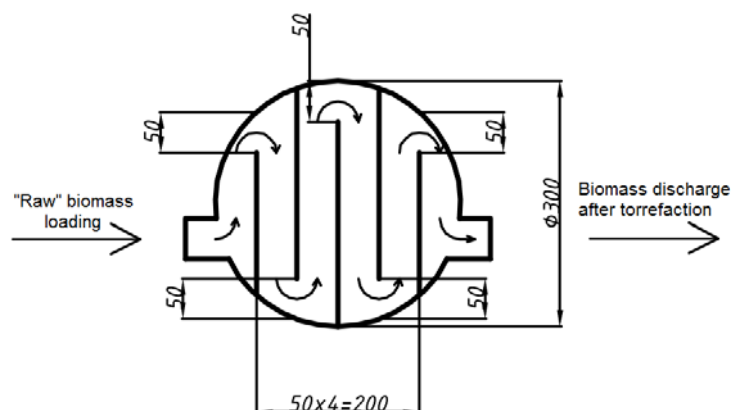


Figure 3: Scheme of package of vertical partitions

At the exit from the apparatus, every 30 seconds a mixture was selected consisting of colored and uncolored particles. The sample volume was 40 ml. Each experiment was repeated 5 times.

Then, the samples were processed according to the method described in (Wang Z., et al., 2018). For this, each sample was scattered on a sheet of paper with a thin layer and photographed. Next, the photos were processed using a computer program that read each pixel of the image from left to right and from top to bottom. The program calculated colored and uncolored pixels. Thus, the proportion of colored particles in this mixture was determined. This made it possible to define the time after which the colored particles introduced into the fluidized bed reach the particle discharge unit from the bed.

### 3. Result and Discussion

The initial biomass sample had a moisture content of 10.6%, an ash content of 3.7%, and a lower calorific value of 16 MJ/kg. As follows from the thermogravimetric analysis of husk samples (Figure 4 a, b, c), husk samples that underwent a torrefaction within 5 minutes have almost the same characteristics as husk samples after a torrefaction for 7.5 minutes, i.e. the heat treatment time of biowaste cannot exceed 5 minutes. After a torrefaction for 5 minutes, husk samples had a moisture content of 3.2%, an ash content of 6.1% and a lower calorific value of 20.7 MJ/kg.

Figure 5 shows the curve change during the experiment of the concentration of colored particles in samples taken at the exit of the "cold" apparatus. As can be seen from Figure 5, 2.5 minutes after the start of loading the colored particles into the "cold" apparatus, the proportion of colored particles in the sample taken at the exit of the apparatus does not exceed 15%. This means that at a given speed of discharge of particles from the reactor (30 kg/h) and a given speed of air (0.45 m/s), it is guaranteed that 85% of the particles loaded into the reactor will be in the reactor for at least 2.5 minutes. 5 minutes after the start of loading the colored particles into the reactor, it can be guaranteed that 65% of the particles will be in the apparatus for 5 minutes, while the remaining particles in the apparatus will be below 5 minutes.

7.5 minutes after the start of colored particles loading into the reactor, only 40% of the particles will be in the reactor for at least 7.5 minutes.

Thus, the proposed reactor design for oxidative torrefaction ensures that for a given processing time of 5 minutes 35% of the particles will be less than the specified processing time. This is significantly smaller than in the reactor, the design of which was proposed in paper (Wang Z., et al., 2018).

By increasing the diameter of the reactor, it is possible to increase the time of the biomass particles in the reactor or to increase the productivity of the reactor. If the time, spent by the particles in the reactor, is limited to 5 minutes, then the discharge rate can be increased to 150 kg/h. In a fluidized bed at a process temperature of around 300 °C, the mass loss of the sample is about 50% (Wang Z., 2017). Therefore, the productivity of a reactor with a diameter of 1.5 m for an oxidizing torrefaction in a fluidized bed can be 150 kg/h for the finished product or 300 kg/h for the initial biomass.

In the equipment complex that we are creating, it is proposed to install three reactors with a diameter of 1.5 m, each of which will have a capacity of 300 kg/h in the initial biomass and 150 kg/h in the resulted biochar. Tests of the constructed equipment complex are planned for the end of 2020.

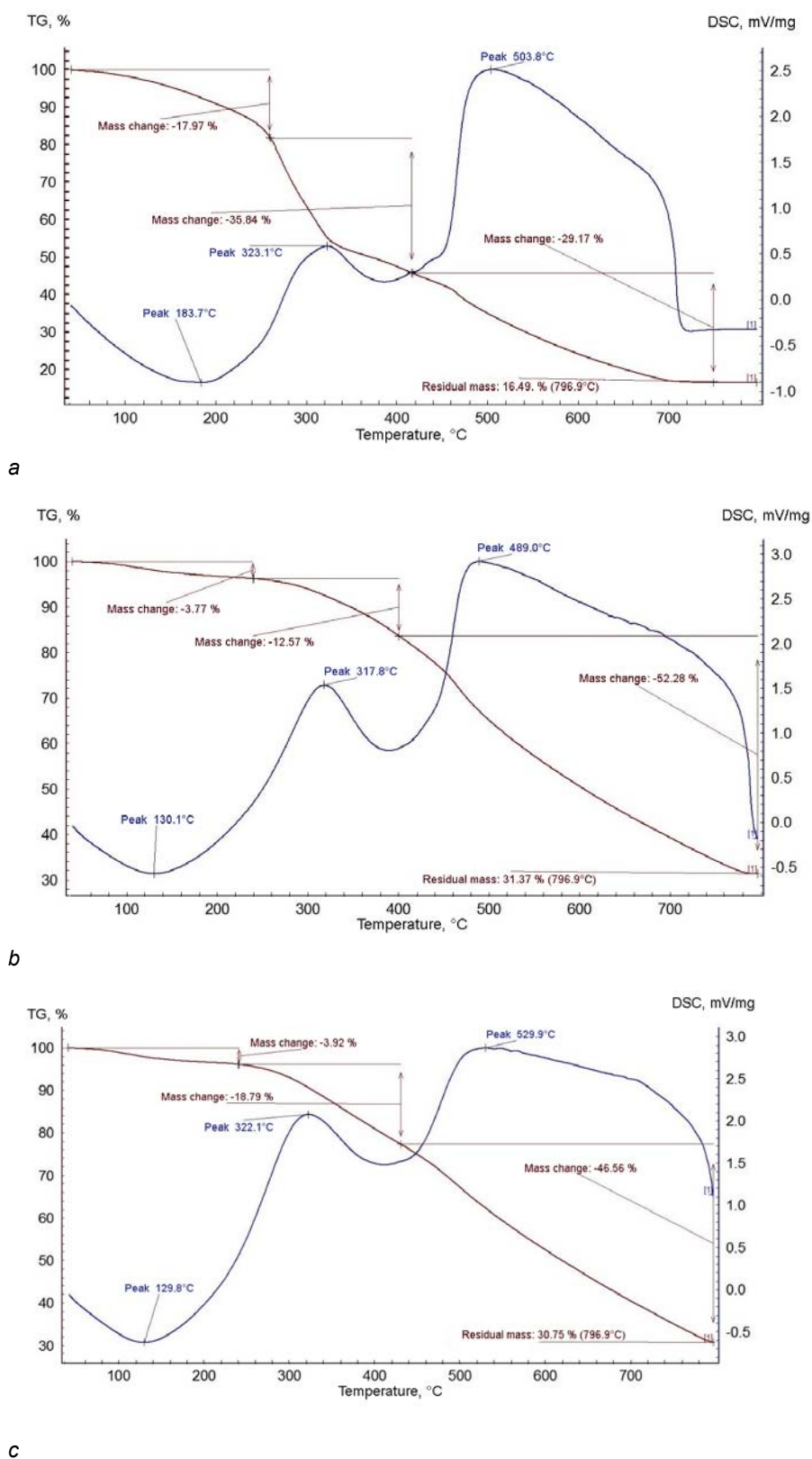


Figure 4: The results of thermogravimetric analysis of sunflower husk samples (a - initial husk, b - after torrefaction for 5 minutes, c - after torrefaction for 7.5 minutes)

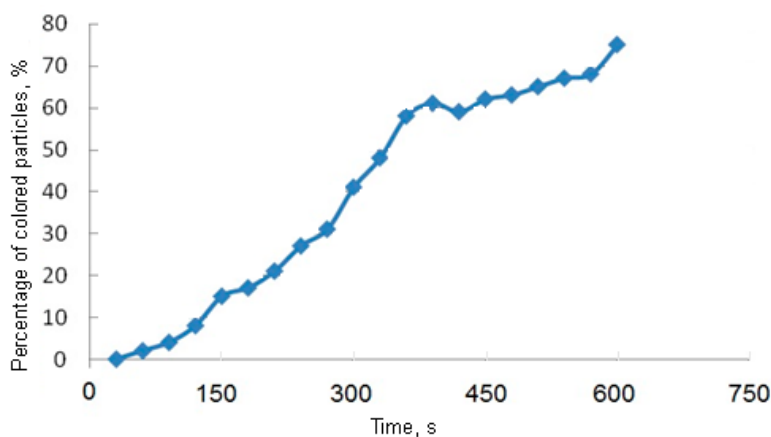


Figure 5: Change curve during the experiment of the concentration of colored particles in samples taken at the exit of "cold" apparatus

#### 4. Conclusions

The use of exhaust flue gases and the short duration of the oxidative torrefaction in the fluidized bed should ensure the competitiveness of the proposed technology for preliminary heat treatment of biomass. The proposed technical solution to ensure a given residence time of biomass particles in a boiling reactor is quite simple. Placement of partitions in the fluidized bed does not increase the hydraulic resistance of the reactor and does not create stagnant zones, which is very important from the point of view of the fire hazard of the reactor. This solution can also be used in the case of using a fluidized bed for conducting other chemical and technological processes.

#### Acknowledgments

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#### References

- Chen W-H, Peng J, Bi XT. A state-of-the-art review of biomass torrefaction, densification and applications. *Renew, 2015, Sust Energy Rev*; 44, 847–866.
- Chen W.-H., Lu K.-M., Lee W.-J., Liu S.-H., Lin T.-C. Non-oxidative and oxidative torrefaction characterization and SEM observations of fibrous and ligneous biomass, 2014, *Applied Energy*, 114, 104 – 113.
- Isemin R., Klimov D., Larina O., Mikhalev A., Zaitchenko V., 2018, Integrated waste treatment system combining biogas technology and pyrolysis, *Chemical Engineering Transactions*, 67, 505-510.
- Isemin, R., Mikhalev, A., Muratova, N., Kogh-Tatarenko, V., Teplitskii, Y., Grebenkov, A. & Pitsukha, E. 2019, Low-temperature pyrolysis of poultry litter for biofuel production, *Chemical Engineering Transactions*, 75, pp. 103-108.
- Isemin, R., Klimov, D., Mikhalev, A., Milovanov, O., Muratova, N. & Zaichenko, V. 2016, Fluidized bed straw pellets combustion with minimal emissions of carbon monoxide, *Chemical Engineering Transactions*, 53, 13-18.
- Mei Y., Liu R., Yang Q., Yang H., Shao J., Draper C., Zhang S. Torrefaction of cedarwood in a pilot scale rotary kiln and the influence of industrial flue gas, 2015, *Bioresour Technol*, 177, 355 – 360.
- Uemura Y., Sellapah V., Trinh T.H., Hassan S., Tanoue K. Torrefaction of empty fruit bunches under biomass combustion gas atmosphere, 2017, *Bioresour Technol*, 243, 107 - 117.
- Wang Z., Li H., Lim C.J., Grace J.R. Oxidative torrefaction of spruce-pine-fir sawdust in a slot-rectangular spouted bed reactor, 2018, 174, 276 – 287.
- Wang Z. Biomass Torrefaction in Slot – Rectangular Spouted Bed, 2017, A Dissertation Submitted in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy in the faculty of graduate and postdoctoral studies (Chemical and Biological Engineering), the University of British Columbia (Vancouver).