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APPARATUS AND ANALYTICAL TECHNIQUE FOR BIOMASS AND MATERIALS

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This research paper presented the concept, design and construction of the device to thermogravimetric measurements and the results of tests carried out on wood pellets. The purpose of this paper is to study the characteristics and thermal degradation behavior of wood pellets for biofuel production via gasification technology and construct the device. The elemental properties of the feedstock were characterized by an elemental analyzer while thermal properties were investigated using thermogravimetric analyzer (TGA). The gasification processes were being carried out at room temperature up to 900°C in the presence of nitrogen and air as gasification agents, gas flowing at 500 ml/min. The investigated parameters are particle sizes and heating rate. The particle size used is in the range of 425 to 500 um. The heating rate applied is 10°C/min. Sample weights were varied from 0.5 to 1 gram, and a stainless - steel tray was used for the test. The further part of the paper contained the results of the tests carried out on wood pellets in the form of thermogravimetric curves. These studies are conducted by looking at opportunities to improve the energy efficiency of the gasification process of biomass.

Keywords: Characterization, Gasification, Thermogravimetric analysis (TGA), Wood pellet, Thermogravimetric measurements

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Introduction. Biomass in the form of biomass energy is one of largest contributor to the world economy. The technology of converting biomass into energy is a right option since it is a renewable type of energy source that could dramatically improve the environment, economy and energy security. Pyrolysis is one of the most promising technologies of biomass utilization, and it is also the first stage of biomass thermochemical conversion. By pyrolysis, biomass is converted into liquid oil, char and gases. In the absence of oxygen, the yields and compositions of pyrolysis products depends on the type of biomass, temperature, heating rates, type of reactors, particles size and co-reactant.

Electricity and thermal energy are two of the primary energy carriers needed in municipal economy. Traditionally these energy carriers are made from fossil fuels. These fuels as non-renewable are going to exhaust. At the moment, the emphasis is placed on increasing the use of renewable energy resources. The gasification process is endothermic and requires the delivery of thermal energy. The source of this energy is the partial combustion of the fuel. Successive stages of gasification of fuel are drying, pyrolysis, partial combustion and gasification of carbon residue. For the purpose of drying and pyrolysis waste heat can be used which will increase the energy efficiency of the conversion process of solid fuel into gas. The drying process takes place at a little more than 100°C. In order to determine the level of the temperature necessary for the process of the partial thermal decomposition, thermogravimetric research of fuels can be used. They will allow to specify for which temperatures and to what extend solid fuel will undergo degassing.

In the case of biomass, which has a high content of volatile substances, pyrolysis process

allows to convert, in considerable part, to transform this solid fuel into gas fuel. The biomass gasification process is also referred as "pyrolysis by partial oxidation". It intends to maximize the gaseous product, and generally takes place between 800 and 1100°C. The product gas contains CO, CO_2, H_2, H_2O, CH_4, N_2 (if air is used), apart from contaminants like small char particles, small amounts of ash and tar. This paper presents thermo-chemical decomposition behavior of wood pellet using thermogravimetric analysis.

Thermogravimetrics. Thermogravimetrics is one of the methods of thermal analysis. It is a method of measuring the change in mass of the analyzed sample of the substance, resulting from the impact of the temperature value on this sample in controlled atmosphere (oxidizing-oxygen, air or neutral – nitrogen, argon). Mass variation is due to the physical and chemical changes occurring as a result of the environmental impact on a sample of the appropriate temperature and atmosphere. Thermogravimetric analysis is indicated as TGA. Due to the versatility of the thermogravimetric analysis and the ability to use it for a variety of materials, it is used in many fields of science and technology.

In the case of studies of fuels in the neutral atmosphere (simulating the process of pyrolysis) mass variation is due to the moisture evaporation and the thermal decomposition of a solid substance as a result of which gases and condensing compounds arise. TGA analysis, by which thermogravimetric curves are obtained, let us estimate the degree of degassing of sample of function of changes of temperature process and the course of process over time.

Thermogravimetric analysis is a very useful technique for determining (1) composition of multicomponent systems, (2) atmosphere effects on materials, (3) reaction kinetics and (4) ash, moisture and volatile contents of materials, being for this reason a very powerful tool in the study of biomass thermal conversion processes, like pyrolysis. Then the TGA mass loss curve was plotted versus the temperature. TGA supplies the range in which maxima thermal degradation of the sample is taken place.

Unfortunately, commercially available apparatus for thermogravimetric analysis is expensive. Due to the high price of devices an attempt was undertaken to build a thermogravimetric snap to a precision scale for the purpose of equipment of the Laboratory of Fuel and Propellant Engineering Department, Myanmar Aerospace Engineering University.

The project aims to minimize the construction costs of the system. The premise is to allow the stand to perform a thermogravimetric analysis of fuels for laboratory and classroom purposes. The further part of this research paper presents the concept, design and construction of the device to thermogravimetric measurements and the results of tests carried out on wood pellets.

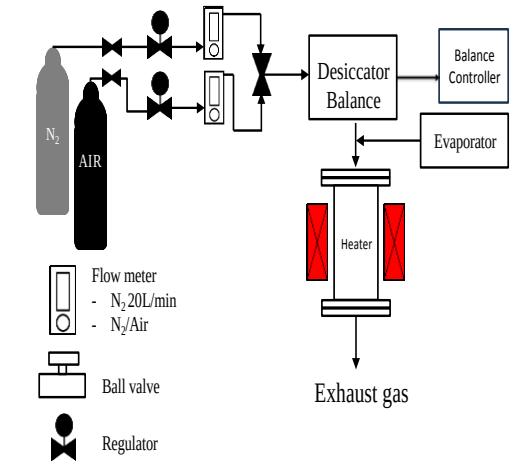
Experimental Section. Biomass Feedstock. Wood pellet was used as a feedstock in this experiment. The feedstock was first milled and sieved into smaller particle size of 425 um. The characteristics of wood pellet were carried out. The ultimate analysis was carried out using elemental analyzer (JMA-Auto Sampler 1000) which determines the amount of C, H, O, N and S in the feedstock. Carbon Sulphur Nitrogen Hydrogen percentage are determined by elemental analyzer. By subtracting these values from a total of 100 percent we can get the oxygen percentage. Table 1 showed the ultimate analysis of wood pellet.

The proximate analysis was conducted using thermogravimetry analyzer (my construction) to analyze moisture content, volatile matter (VM), fixed carbon (FC) and ash content in wood pellet.

Table 1. Ultimate analysis of Wood Pellet (wt% dry basis)

Sample	**C**	**H**	**O**	**N**	**S**
Wood Pellet	51.7	6.1	41.8	0.2	0.2

Experimental Setup. The experiments were conducted using a reactor in a batch process at atmospheric pressure. The height and inside diameter of the reactor were 400 and 52.7 mm respectively. Fig. 1 showed the experimental apparatus. This unit consists of the nitrogen bottle, the air bottle, desiccator, semi micro balance, small computer, petit logger, reactor, sample cup with the sample on it and thermocouple to control the sample temperature, heater, thermocouple to control the heater temperature, regulators, gas flow meters to supply gases such as nitrogen and air and evaporation device, control system with integrated power switch and two temperature regulators type PID.



(a)

(b)

Fig. 1 (a) Schematic diagram of experimental apparatus; (b) Overall construction of experimental apparatus

A schema of designed measuring system (thermobalance) is showed in figure 1. At the bottom of the desiccator was made PTFE tube to the reactor and delivery of nitrogen to the heating chamber. Nitrogen is used to displace oxygen from the heating chamber while simulating the process of pyrolysis. The key element of thermobalance is the heating chamber. The basis for the construction of the heating chamber is stainless steel pipe SUTSP 50A – interior size Ø52.7x400mm with heating element 1200W (100V). For temperature control PID type controller was used.

Nominal temperature of the heating chamber is 1100°C (maximum 1200°C). For temperature measurement K-type thermocouple was used. A sample tray is made of stainless steel with a diameter of 36 mm and thermocouple 0.1 mm. Thermocouple is placed below the sample tray.

Experimental procedures. Thermogravimetric Analysis (TGA) is an essential laboratory tool used for obtaining weight loss of the biomass sample versus temperature or time. In this experiment, the weight of the wood pellet sample is determined as a function of time and temperature as it is subjected to a controlled temperature program.TGA was carried out in the presence of nitrogen at the flowing rate of 500 ml/min. Wood pellet samples between 0.5 and 1.0 g were gasified to a maximum temperature of 900°C. The sample was first heated to 110°C and kept at that temperature for 30 minutes to remove any moisture. After that, the samples were individually heated at 10°C/min until 900°C.The temperature is held room temperature to 107˚C for 10 min, then kept 107 ˚C for 1hr. And then 107˚C to 900˚C for 1hr and 20 min, then kept 900˚C for 7 min, then reduced to 815˚C.Air is introduced at 900˚C after 7 min until the oxidation reaction is complete and no further weight loss is observed. The experiment was repeated for each weight.

Results and Discussion. Blank Test. In blank test, without sample, air is passed at 500ml/min, and the temperature is raised up to 1000˚C at heating rate of 10˚C/min. By this

blank test, the general condition of the apparatus can be known. The TGA curve can drift slightly as the temperature is increased. When noise appears in the TG curve, the possible cause may include contact between sample tray and reactor. The result of the blank test showed in Fig. 2.

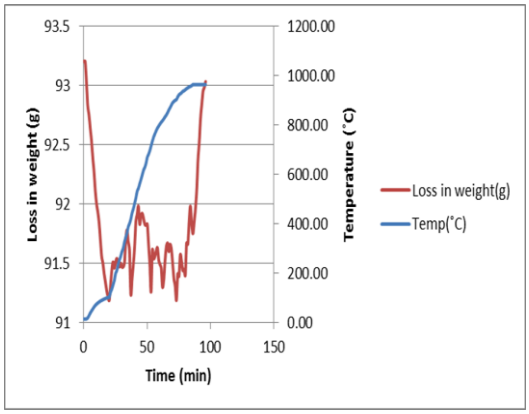


Fig. 2. Blank test

Test Results. After placing the sample in the sample tray of the heating chamber it is washed out with inert gas (N_2) in order to remove the oxygen. The presence of oxygen in the air would cause that, instead of the pyrolysis process, combustion process would occur. Then the process of heating of the sample begins to the assumed temperature controlled by PID-type controller. In the course of the study the change of the mass of the sample is recorded and the temperature value. The results of the technical analysis of these fuel was presented in table 2. Thermobalance tests were carried out by heating the weighed sample of fuel to the required temperature by keeping on this temperature for some period of time. In the course of the research the change of temperature and the mass of the sample were recorded. The results of the research in the form of thermogravimetric curves are shown in fig. 3–5.

Table. 2. Proximate analysis of Wood Pellet (wt %), Comparison with Literature Data and Actual Data

Wood Pellet	Literature Data	Actual Data
Moisture	5.07	2.18
Volatile Matter	84.82	8.62
Fixed Carbon	14.88	81.66
Ash	0.30	7.54

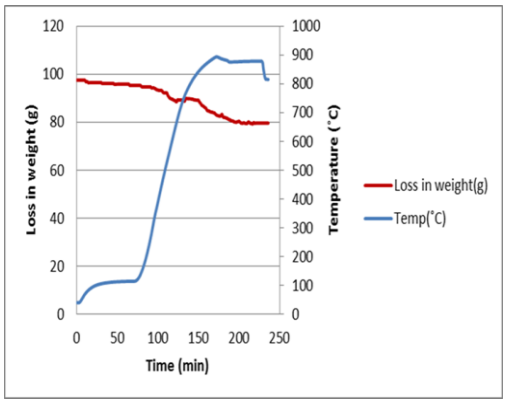


Fig. 3. Thermogravimetric curves for wood pellets used 1 gram

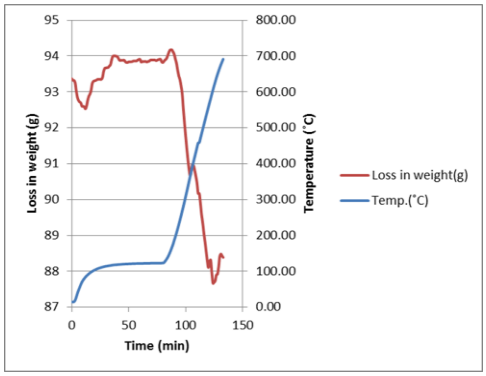


Fig. 4. Thermogravimetric curves for wood pellets used 1 gram

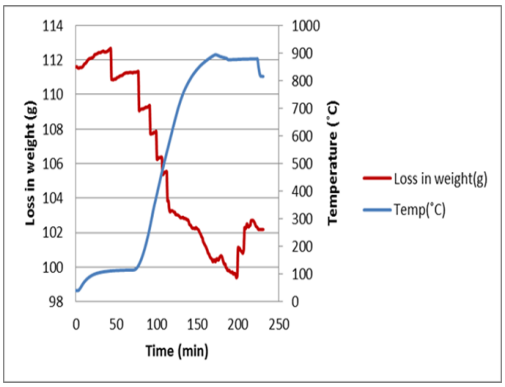


Fig. 5. Thermogravimetric curves for wood pellets used 0.5 gram

Data obtained from thermo-gravimetric analysis were analyzed for any changes in thermo-chemical decomposition behavior. Fig. 3–5 showed TGA analysis plot at different sample weight of wood pellet sample various atmospheric conditions i.e., Nitrogen and Air. The TGA curves heated from room temperature to 900°C at the heating rate of 10°C/min under an inert atmosphere. As shown in the TGA curve (Fig. 5), due to the considered low amount of the sample(0.5g), this process should be repeated several times to obtain a representative pyrolysis behavior. As shown in fig. 3 and 4, 1 gram sample weight has the highest moisture content (2.18%) and 0.5 gram sample weight has the lowest moisture content (0.2778%). Higher moisture content usually contributed to a lower amount of volatile matter since more heat are needed to first volatilize the water before decomposing the biomass. The treatment process results in a higher fixed carbon value in 0.5 g sample.

From the results obtain, sample weight 1g is better than 0.5g because of experimental apparatus by balance. We should use a larger volume sample but time is limited for research. The problems observed they are mainly related to the stability of the process. The stable operation of the process were very thin thermocouple with sample tray, balance and petit LOGGER.

In fig. 3, firstly, 2.18% of weight loss was occurred during the early initiation of the decomposition due to the removal of moisture and water content in wood pellet. Secondly, 8.62% of weight loss occurs due to the release of volatile matter content. Thirdly, 81.66% of weight loss occurs during the gasification of fixed carbon content and the remaining 7.54% are coined as ash residue of the wood pellet. Here in this case the secondary stage of disintegration or decomposition is coined as active pyrolytic zone due to rapid disintegrating or decomposing rate per unit time. In this second stage, the weaker bonds are destroyed, and the inter molecular interaction between them is also affected which results in breaking of those bond. Some of the gaseous molecules are formed due to the lower temperature, and some aliphatic side chains are also damaged. In the third stage, the parent molecular skeleton and many chemical bonds are destroyed due to the presence of very high temperature. This accompanies the formation of smaller molecules because of the decomposition of the larger molecules. They appear in the gaseous phase and even in char remains. The properties of the biomass change indifferently nitrogen atmosphere to that of air atmosphere.

Conclusions. Constructed apparatus allowed to study thermal decomposition of solid fuels and obtained thermogravimetric curves are similar to those that can be performed on commercial thermobalances. The advantage of the device is definitely lower price than of commercial solutions. Our main task is construction of the device (Thermogravimetric analyzer). Preferred option seems to be also a larger size of a chamber and sample tray allowing the analysis of larger samples of fuel. Despite these imperfections it can be considered that building of such a device meets the goal of thermogravimetric testing of fuels. Finally, we concluded that this device will be modified in Myanmar and continued to research it. It is important to perform another literature study or conduct some experiment in the future for the behavior of the test carried out on types of solid fuels in the form of a thermogravimetric curve and apparatus applied to improve the energy efficiency of the gasification process of biomass.

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ПРИБОР И МЕТОДИКА АНАЛИТИЧЕСКОГО ИССЛЕДОВАНИЯ БИОМАССЫ И МАТЕРИАЛОВ

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В данной исследовательской работе представлены концепция, дизайн и конструкция устройства для термогравиметрических измерений, а также результаты испытаний, проведенных на древесных

гранулах. Целью данной работы является изучение характеристик и поведения термического разложения древесных гранул для производства биотоплива по технологии газификации. Элементные свойства сырья характеризовались с помощью элементного анализатора, а термические свойства исследовались с помощью термогравиметрического анализатора (ТГА). Процессы газификации проводились при комнатной температуре до 900°C в присутствии азота и воздуха в качестве газифицирующих агентов, скорость потока газа 500 мл/мин. Исследуемыми параметрами являются размеры частиц и скорость нагрева. Размер используемых частиц находится в диапазоне от 425 до 500 мкм. Применяемая скорость нагрева составляет 10 °C/мин. Вес образцов варьировался от 0,5 до 1 г, для испытаний использовался поднос из нержавеющей стали. Дальнейшая часть статьи содержит результаты испытаний древесных пеллет в виде термогравиметрических кривых. Эти исследования проводятся путем изучения возможностей повышения энергоэффективности процесса газификации биомассы.

Ключевые слова: характеристика, газификация, термогравиметрический анализ (ТГА), древесные гранулы, термогравиметрические измерения.