

IMPROVING MEASUREMENT ACCURACY OF BIOMASS HEAT OF COMBUSTION USING AN OXIGEN BOMB CALORIMETER

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Abstract: *The paper describes an experimental system developed for measurement of the heat of combustion of a sample of solid wood fuel. The system is set up to use an Parr type Oxygen Bomb Calorimeter together with a temperature sensor. A data acquisition system is used to accurately record temperature versus time response before, during and after the combustion of the fuel sample. The data acquisition system includes an analog to digital converter (METRIX 3283 precision measurement system with optical/RS232 connection) a standard software package (SX-DMM-Chauvin Arnaux) to obtain the measurement under program control.*

Keywords: *heat of combustion, wooden biomass, thermodynamic properties measurements.*

1. Introduction

The heat of combustion of a fuel is the amount of heat released by the complete combustion of a unit weight of fuel and it is expressed in J/kg or other similar specific energy units. It can be experimentally determined by combustion of an accurately weighed sample of fuel in a oxygen bomb calorimeter. In this experimental system, an Parr type Oxygen Bomb Calorimeter (OBC) has been used. The temperature rise, ΔT [°C], resulting from the heat released by combustion of the biomass fuel sample is determined from the temperature-time response of the calorimeter. An experimental procedure is followed to determine ΔT , which is taking into account for non-adiabatic condition of the calorimeter. Several corrections are also applied for an accurate measurement of the heat of combustion of sample. The paper describes the hardware and software

system used for PC-based data acquisition and post-processing procedure of collected temperature-time data.

2. Theory

The net heat of combustion, H_g , of the solid fuel sample is determined from the energy balance relation that states: the heat released by the fuel sample within the OBC is equal to the heat absorbed in the calorimeter. Combustion of the fuel sample is found in a temperature rise of the bucket containing a measured amount of water in which the Oxygen Bomb(OB) is completely submerged. The energy balance relation is expressed by the following equation[2,3]:

$$m_{sf} H_g = K_w (\Delta T) - q_1 - q_2 - q_3 - q_4 \quad (1)$$

where m_{sf} = weight of the solid fuel sample, K_w water equivalent of the

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calorimeter, q_1 correction for heat of formation of nitric acid (HNO_3), q_2 correction for heat of formation of sulfuric acid (H_2SO_4), q_3 correction for heat of combustion of fuse wire and q_4 correction for heat of combustion of benzoic acid when is used as bending material of sample. Finally, with all above values known the heat of combustion, H_g , is determined from Eq.1.

3. Experimental Setup

The experimental setup (Figure 1) consists of the following components:

- the oxygen bomb with a cover which contains charging (inlet) and discharging (outlet) valves and ignition terminals. This cover can be secured onto the oxygen bomb with a screw cap after the fuel sample and fuse wire are assembled inside.

- calorimeter bucket; it is a stainless steel bucket which contains a carefully measured amount of water in which the oxygen bomb containing the fuel sample is submerged.

- calorimeter jacket; the double-wall jacket provides thermal insulation to minimize heat transfer to or from the calorimeter bucket which is placed inside the jacket during experiment. By supplying suitable mixture of hot and cold water into the jacket wall, its temperature can very closely follow the rise in bucket temperature during the experiment and maintain a nearly adiabatic condition. In the experiment, the outer jacket temperature remained nearly constant to provide a non-adiabatic condition.

- cover assembly; there is the temperature sensor and a motor driven stirrer which can be lowered into the calorimeter bucket. The PT-100 sensor records the temperature of the bucket (@the bath temperature). An electric motor operates a stirrer to ensure uniform temperature within the bucket.

- ignition system; the cover of the OB contains ignition terminals which are connected to the fuse wire in order to initiate combustion of the fuel sample inside the OB. The lead wires from the ignition system are connected to the ignition terminals of the OB after it is submerged in water in the calorimeter bucket. The circuit contains an ignition switch and an indicator light. In order to initiate combustion (fire the bomb), the switch is pressed and held down until the indicator light goes out.

- accessories for this experimental system includes fuel sample, pellet press, precision weighing balance (resolution 0.0001 mg), fuse wire, titration system, oxygen tank and charging system. Hardware and software used for data acquisition and data analysis in this experiment are described below.

4. Experimental Procedure

The experimental process consists of the steps recommended and outlined in detail in the Instruction Manual[2] of OBC and it is briefly described here.

4.1. Preparations

(a) Fuel Sample and Fuse Wire: A sample of solid biomass in powder form is pressed to form a pellet in a pellet press. The mass of the fuel sample should be about 1 gram (no more than 1.2 gram). The biomass pellet is carefully placed in a combustion sample in an upright position and weighed in a precision electronic balance. After that the sample is placed in a ring attached to the cover assembly through the electrodes to which 10 cm of fuse wire is attached.

(b) Closing the Oxygen Bomb. About 10 ml of distilled water is placed inside the oxygen bomb cylinder. The cover assembly together with the biomass pellet

and fuse wire is then placed on the oxygen bomb cylinder which is closed by hand-tightening the screw cap.

(c) Charging the Oxygen Bomb: The oxygen bomb is filled with oxygen to a pressure of 22-30 atm to ensure complete combustion of the fuel sample. Oxygen from a high pressure tank is transferred, using a charging-reduction valve.

After filling, the charging line is depressurized using the bleed valve and must be disconnected from the oxygen bomb.

(d) Calorimeter Bucket: The calorimeter bucket is filled with 2000 (± 0.5) grams of distilled water at approximately ± 1.50 – 2.00 °C around the room temperature. The bucket is then set into the calorimeter and the oxygen bomb is slowly inserted into the bucket. The ignition lead wires are connected

to the terminal sockets on the bomb head. The cover of the jacket is closed. The thermometer and stirrer assembly is lowered into the calorimeter bucket.

4.2. Operation

The stirrer must be run for about 5 minutes and the data acquisition system is started to collect data for the next 5 minutes as “Pre-Period” following which the fuel is ignited by pressing the ignition switch.

The data collection is continued during the “Rise-Period” where there is a rapid temperature rise and into the “Post-Period” which follows. The temperature-time data are processed, as outlined later (under Data Acquisition System), to obtain the corrected temperature rise, ΔT , for non-adiabatic condition of the calorimeter.

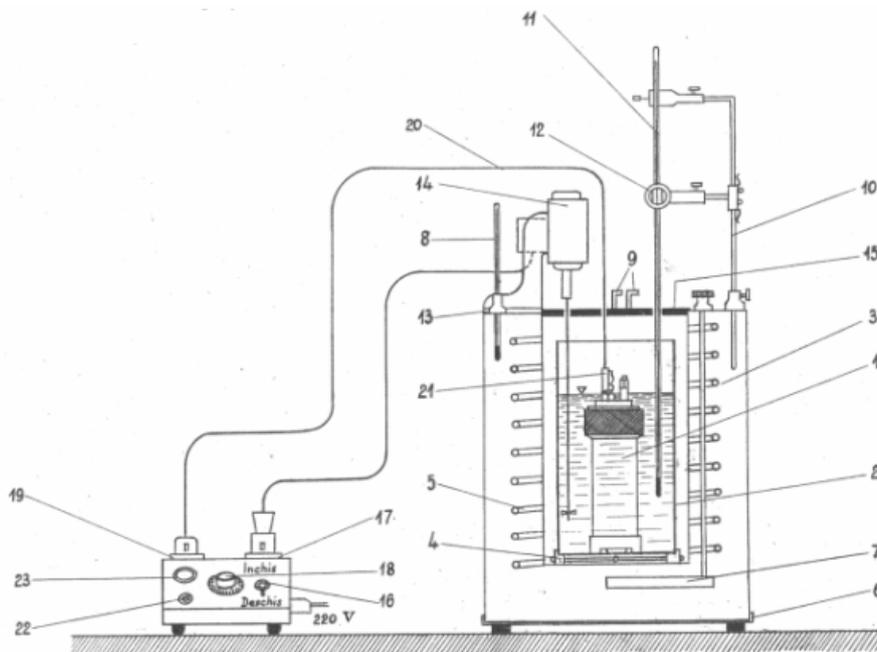


Fig. 1. Oxygen bomb calorimeter: 1-oxygen bomb; 2-bucket; 3-thermostat; 5-heating element; 6-support; 7-stirrer; 8-bath thermometer; 9- connection tubes; 10- thermometer holder; 11-precision thermometer; 12- sight glass; 13-holder; 14-bath stirrer; 15-insulation cover; 16-ignition switch; 17-socket; 18-rheostat; 19.20-main power; cable: 23-ignition lamp

4.3. Experimental corrections

After the completion of the experiment, the thermometer and stirrer assembly is raised and the cover is lifted to clear the jacket opening. The oxygen bomb is carefully removed from the bucket and secured on the lab bench. The outlet valve of the oxygen bomb is slowly opened to de-pressurize it. The screw cap is then unscrewed and the cover assembly together with combustion capsule and fuse wire is removed. The residue within the bomb is washed with distilled water and collected in a beaker. The volume (c_1 in ml) of the alkali solution used in acid titration is determined. Correction, q_1 , for heat of formation of nitric acid is obtained from [2, 3] $q_1 = c_1 =$ milliliters of alkali solution used in acid titration

Correction, q_2 , for heat of formation of sulfuric acid is obtained from the following

empirical relation: $q_2 = 14 c_2$; $c_2 =$ percentage of sulfur in the fuel sample.

All the remaining fuse wire is also collected and measured to determine the length of wire consumed (c_3) in the experiment. The correction, q_3 , for heat of combustion of the fuse wire is expressed by: $q_3 = 2.5 c_3$ (for OBC Parr type nickel-chromium wire used in this experiment).

5. Data Acquisition System

5.1. Hardware and Software

The hardware system consists of two E-type thermocouples, two PT-100 and an Personal DAQ Board with hardware CJC (cold junction compensation), 2.490V precision voltage reference, J-FET constant current injection to avoid self heating of the PT-100 sensor and RS232 connection.

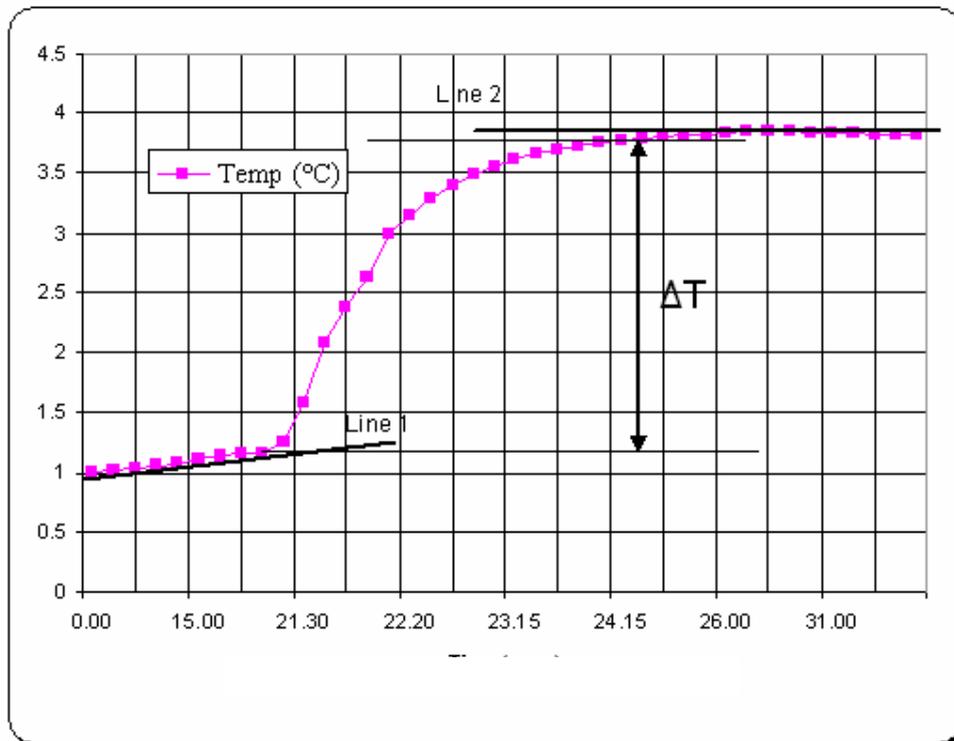


Fig. 2. Temperature rise ($^{\circ}\text{C}$) vs. time

One sensor (thermocouple and PT-100) measures the bucket temperature and the other records the outer jacket temperature. Data acquisition and storage of temperature-time data is carried out using SX-DMM software. Further analysis of the temperature-time response to obtain ΔT is done with software written in MATLAB7 or can be exported as EXCEL file. Final determination of the heat of combustion, H_g , is obtained with MATLAB which prompts the user to input all the corrections and mass of fuel determined earlier in this experiment.

5.2. Data Acquisition Process

This section outlines the working of the data acquisition system. To sample dc voltage signal from the thermocouples which are connected to Personal DAQ Board a 1uV base resolution is used. The thermocouples are connected in differential mode as required setup for reading thermocouples. A sampling rate of 100 kHz is used. The software provides necessary filters for noise rejection and conversion of dc voltage readings into temperature ($^{\circ}\text{C}$).

The original Beckmann mercury thermometer of this apparatus has a certified accuracy and precision of ± 0.01 degree C. However, this thermometer was replaced as reading device with a E-type thermocouples and two PT-100 sensors for computerized measurement of temperature. The E thermocouple has an error limit of ± 0.1 $^{\circ}\text{C}$, precision PT-100 has $\pm 0.01^{\circ}\text{C}$ but, together with the DAQ and the software, it provides a measurement precision of 0.01°C . This is considered acceptable because the measurement of temperature rise (ΔT) of the bucket rather than its temperature (T) is the main objective in this experiment. The temperature rise in this case is only about 2 to 4°C . Once the DAQ system has

obtained individual temperature readings, the data is subjected to a running average to further reduce noise. The sampled temperature is then fed to a filter set to allow frequencies below 0.1 Hz and to reject harmonics of 50Hz frequency. This filter is used to eliminate noise in the derivative signal of the bath temperature.

The temperatures and the derivative are then plotted on monitor screen and also recorded. Included in the system are other functions to allow the time and cold junction compensation to be displayed. The data acquisition process is shown on the monitor screen. This screen allows the user to view the data acquisition process during the experiment and shows the plots of bath (bucket) temperature, its derivative and the jacket temperature as a function of time as well as digital displays of the bath temperature, jacket temperature and elapsed time.

5.3. Data Analysis Software

The data analysis program is written in MATLAB. This is done for several reasons. First, it is a programming language that is taught to as being an available analysis tool on almost all engineering computers and PCs used for a real-time data acquisition. To begin the analysis of the experimental data, the MATLAB file, OBC.m, is opened and executed. The analysis software begins by reading the data file (in ASCII format) from directory where the software and the data-file are stored. It then asks for the parameters of the experiment, such as the mass of the fuel sample (m_f), titration volume (c_1), length of the fuse wire consumed (c_2) and the sulfur content (c_3) in the fuel sample. The program converts the ASCII file into three different arrays to extract the bucket temperature, its derivative and the jacket temperature. The (bath) temperature and the jacket

temperature are plotted on a single graph (Figure 2).

Figure 2 shows the temperature-time plot obtained in this experiment. Rising bucket temperature during 120 sec. (Figure 2) before combustion indicates heat loss due to a lower jacket temperature. Similarly, the decreasing bucket temperature after 300 sec. is due to heat gain from the bucket which is at a higher temperature after combustion. In order to correct for the heat loss/gain during experiment, a procedure is used to determine the correct temperature rise extrapolated at a time when 60% of the total temperature rise is realized. The program prompts the user to select two points (t_i and t_f in Figure 2) on bath temperature curve in Figure 2 to define the nominal initial and final temperatures and determine approximate temperature difference, ΔT , in the calculation process. The user selects the two points on the graph and the software creates a linear fit to the data (lines 1 and 2 in Figure 2). These two fits are used in the calculation of true and accurate temperature rise and plotted on the graph. The approximate initial and final temperatures (T_i and T_f) are used to find the reference temperature. It is defined as the temperature at 60% of the distance between the two temperatures selected earlier on the plot and is calculated using the following equation:

$$T_m = T_i + 60\% (T_f - T_i) \quad (2)$$

A horizontal line representing T_m is drawn on the temperature-time plot. This reference temperature is used in an algorithm to find the time (t_x) at X where the reference temperature intersects the bath temperature data. The algorithm is a simple "for loop" with a comparison enclosed within. The time at which the two plots intersect is passed into the equations fitted to the temperature changes shown by

lines 1 and 2 before and after the ignition due to heat gain and loss respectively as shown in Figure 2. From these two extrapolated temperatures $T_{f_corrected}$ and $T_{i_corrected}$, the correct temperature rise, ΔT , is calculated from the following equation:

$$\Delta T = T_{f_corrected} - T_{i_corrected} \quad (3)$$

The output of this analysis is displayed on the command line in MATLAB. Also, the jacket temperature is curve-fitted to find the average jacket temperature.

5.4. Heat of Combustion (H_g)

At this stage, all the parameters are known; either determined from data analysis (e.g. ΔT) or obtained by other experimental procedures completed in this experiment (e.g. m_f , c_1 , c_2 , and c_3). The corrections q_1 , q_2 , q_3 and q_4 are calculated using their equations (4.3). The manufacturer supplied value of W , the water equivalent of the calorimeter, is used. The calibration of the instrument to get the water equivalent of the calorimeter will be subject of other article. Finally, the heat of combustion of the sample is calculated using Equation 4 which is same as Equation-1 rewritten in an alternate form.

$$H_g = \frac{K_w (\Delta T) - q_1 - q_2 - q_3 - q_4}{m_{sf}} \quad (4)$$

All the calculations are performed within MATLAB program developed for this experiment. Two trial runs were conducted with pellets made from wood pellets as the solid fuel. The results obtained in these tests are presented below in Table-1. The deviation between the values of H_g determined in these trial runs is within about $\pm 5\%$ of each other.

Table 1

Results from the Trial Runs		
Parameters	Test 1	Test 2
m_f = weight of biomass fuel [gram]	1.1385	1.2388
c_1 = volume of alkali solution in acid titration [ml]	6.59	7.01
c_2 = length of wire consumed [cm]	4.0	3.5
C_3 = sulfur content in fuel sample [%]	0(assumed)	0
W= water equivalent of the calorimeter [cal/°C]*	2420	2420
ΔT corrected for non-adiabatic condition [°C]	1.833	1.889
Hg = Heat of Combustion [J/g]	16230	15435
Hg= Heat of Combustion [cal/g]	3876	3687

* Data supplied by after calibration; **1 kcal/kg = 4.1868 kJ/kg

6. Conclusions

A fully computerized data acquisition and analysis system has been developed and implemented for the use with the oxygen bomb calorimeter experiment for solid biomass fuel heat of combustion determination and can be used also for Engineering students in Thermodynamics course.

The use of these two software programs (data acquisition and data analysis programs) allow the fast and accurate analysis of the heat of combustion of a given fuel sample, allowing the student to have a greater insight into this process. The application of the data acquisition and analysis programs of this project is applicable to almost any experiment where several different temperatures are required to be recorded and analyzed..

Examination of the temperature-time plot (Figure 2) shows temperature fluctuations of about ± 0.01 deg C in temperature data recorded during line 1 (before combustion) and line 2 (after combustion).

This is attributed to the fluctuations in the reference temperature used in the DAQ Board for cold junction compensation. Although the effect of this fluctuation on ΔT determined by the extrapolation technique used in this experiment has a second order influence.

The accuracy of temperature measurement can be improved by using a stable and constant temperature ice-point

or by using fast sensors like semiconductors or high precision resistance temperature detector (RTD-1000). Given the available data acquisition system's analog voltage capabilities, a precision RTD-1000 would be best recommended in this situation.

Acknowledgements

The work was performed under a program of research funded by the CNCSIS Romania, code CNCSIS 6GR/2007, no. 945/2007.

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