



Study the Effect of using Different Insulating Jacket on Energy Equivalent of the Bomb Calorimeter

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ABSTRACT

In this research work, effect of outer jacket insulation on energy equivalent of bomb calorimeter has been analyzed. Energy equivalent shows the sum of the heat capacities of the components in the calorimeter, especially the bomb, the bucket and the water in the bucket. Energy equivalents are determined at regular intervals by experimenting with a sample of a standard material with a known heat of combustion. Emphasis is being placed on minimizing thermal losses between bomb calorimeter and surrounding. For that some Saw dust are introduced in the outer jacket of the present calorimeter which was previously designed only for filling with water (water jacket). For assessing the effect and significance of these selected insulations on energy equivalent of bomb calorimeter, experiments and calculation have been done with same fuel (Benzoic Acid). In other words, we want to prove that when heat transfer between system and surrounding is less, energy equivalent of bomb calorimeter would be lesser. Experiments were carried out on bomb calorimeter with four types of jackets which are filled with water (Thermal conductivity – 0.56), glass wool (Thermal conductivity – 0.04), and saw dust (Thermal conductivity – 0.08). Some modification work on existing bomb calorimeter had to be done so that selected insulations can be installed as desired in the apparatus for experiments.

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Introduction

Bomb calorimeter is used to determine the energy of combustion or calorific value of hydrocarbons. The bomb calorimeter consists mainly of the stainless steel bomb, bucket, sample, oxygen, outer jacket and water. Some of other parts are thermometer, stirrer and firing unit.

The Dewar or outer jacket prevents heat flow from the calorimeter to the rest of the universe, *i.e.*,

$$q_{(\text{calorimeter})} = 0$$

Since the bomb is made from stainless steel, the combustion reaction occurs at constant volume and there is no work, *i.e.*,

$$W_{(\text{calorimeter})} = -\delta, p dV = 0$$

Thus, the change in internal energy, ΔU , for the calorimeter is zero

$$\Delta U_{(\text{calorimeter})} = q_{(\text{calorimeter})} + W_{(\text{calorimeter})} = 0$$

In other words, this equation can be described that the bomb calorimeter is isolated from the surrounding.

There are three main parts of a bomb calorimeter: A bomb, which accommodates the sample and oxygen for the combustion reaction. Bucket, which is filled with a measured amount of condensed water. Then the heat extracted by the vapor production is recovered. On the other hand, the Lower Heating Value (LHV) takes into account when the products of combustion contain water vapor and the heat in the water vapor is not recovered. LHV is found by subtracting the heat of vaporization of the water vapor from the HHV.

Measuring the heat of combustion of a sample is then essential to characterize it, in terms of energetic power. For this purpose, in this work we tried three different jackets to

insulate the combustion bomb for making it an ensuring adiabatic combustion calorimeter.

Heating value or calorific value can be measured by burning a known amount of material in a bomb calorimeter and determining the temperature change. For this Benzoic acid has been weighed approximately on an amount of somewhat in excess of 1 gram and compressed it into a pellet. Weighed the pellet on a balance sensitive to about 0.01 gram and adjusted the weight to 1 gram (Tolerance 0.01 gm) by carefully removing some powdered benzoic acid from the pellet by brushing.

The bomb is opened and cleaning of required parts is done. Fuse wire of measured length and weight is taken and the ends of the wire are attached to the holes/slots given in the bomb

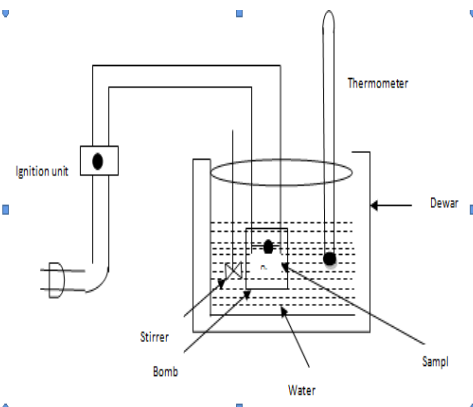


Figure 1. Line diagram of a basic bomb calorimeter.

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Figure 2. Outer shell of bomb calorimeter jacket.

Fuel sample has been palletized with the help of pallet and then small measured mass of it is taken in the crucible (about 1 gm). The crucible is kept in the crucible holder of the bomb such that the fuse wires passes through the fuel. The lid of the bomb then fastened manually. After that with the oxygen intake valve not more than 2 Mpa of oxygen is supplied to the bomb. Some amount of oxygen is purged through the bomb to remove the air already present and then the oxygen supply is cut off. Oxygen pressure also helps in sealing of the bomb. Then fill the bucket with water (approximately 2000 ml) and check the water temperature. Then weighed the bucket plus water. After that prepared bomb is then placed on tripod in the water filled bucket which insures its minimum contact with the bucket. The bucket with water is then placed over a tripod at the calorimeter jacket base. Lid of the calorimeter is kept in place with stirrer and temperature sensing device which were also adjusted with the calorimeter lid. The jacket is kept empty for now. All the required electrical connections are made. Lead wires connect the fuse wire inside bomb with the controller. Stirrer is placed in the bucket water for maintaining throughout even water temperature.



Figure 3. Pelletizing machine.



Figure 4. Benzoic acid is being weighed in digital weighing machine.

Temperature sensor is then inserted in the bucket water such that there is adequate space between stirrer, thermometer, bomb and lead wires. Now the apparatus is fully assembled and prepared for experiment.

Stirrer is turn on and temperature of the water is noted every minute and if temperature remains constant for 3 consecutive readings then that reading is noted down (T_{initial}) now we press fire button for almost 3 seconds and temperature of bucket water is observed. After the ignition, temperature rises rapidly to the maximum, and temperature readings are to be noted down (T_{final}) when no further increase in temperature is observed.

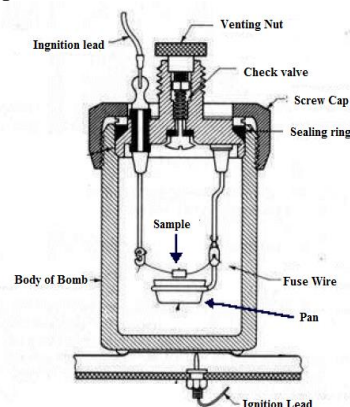


Figure 5. Construction of a bomb.

After some time, temperature starts to decrease. This decrement in temperature is noted down in every 1 minute interval. Now the stirrer got turn off, lead wires are got removed and thermometer/sensor is taken out. Bomb is removed from the bucket and the residual gases of the bomb are discharged. The bomb is then opened, if any amount of fuel is present in the crucible that means combustion of fuel was not completed and we have to weigh the remaining fuel. If there is no fuel found in the crucible that means combustion is completed and if any residual fuse wires are found then get them collected and weighed. Heat loss to the surroundings can be calculated by use of a cooling correction curve, or, as in this experiment, prevented by use of a insulated jacket (Water, Glass wool and saw dust) around the calorimeter, maintained at the same temperature as the calorimeter itself which ensures the adiabaticity of apparatus.

Bomb calorimetry provides desired data in a very direct way that's why it is used widely in chemistry; it is still used in the fuel and food industries. In the field of food industries its results are sometime criticized because our body does not convert the food into the same products as those formed by combustion in a calorimeter. Other, more subtle, factors may also be significant.



Figure 6. Temperature sensing device



Figure 7. Controller unit



Figure 8. Assembly of bomb calorimeter.

e) Experimental procedure

All the required electrical connections are made. Lead wires connect the fuse wire inside bomb with the controller. Stirrer is placed in the bucket water for maintaining the even water temperature. Temperature sensor is then inserted in the bucket water such that there is proper space between stirrer, thermometer, bomb and lead wires. Now the system is fully prepared for experiment except the outer jacket, which is prepared as describe further in the next section of the paper. now stirrer is turned on and we press fire button for almost 3 seconds and temperature of bucket water is observed which is termed as T_{initial} . After the ignition, temperature rises rapidly to the maximum, and temperature readings are to be noted down in every minute and if three consecutive readings are same that means experiment is complete when no further increase in temperature is observed. After some time, temperature starts to decrease. This decrement in temperature is noted down in every 1 minute interval. Now the stirrer got turn off, lead wires are got removed and thermometer/sensor is taken out. Bomb is removed from the bucket and the residual gases of the bomb are discharged. The bomb is then opened, If any amount of fuel is present in the crucible that means combustion of fuel was not completed and we have to weigh the remaining fuel. If there is no fuel found in the crucible that means combustion is completed and if any residual fuse wires are found then get them collected and weighed.

Table 1. List of thermal insulating materials used in experiments.

S. no.	Insulating material	Thermal conductivity (k)
1	Water	0.56
2	Glass wool	0.04
3	Saw Dust	0.008

Experiment I

In our first experiment entire apparatus is insulated with water jacket:

With the same apparatus discussed above and following the general procedure experiment was started. In the previous section, it was stated that the jacket was kept empty, so for this case jacket was filled with water. Having the apparatus discussed above and following the general procedure with water jacket we observed the temperature readings while using benzoic acid as a fuel. We did not do any modification in existing bomb calorimeter for this experiment. We took 1 gram (approx) of benzoic acid for each experiment and experimented three times with water jacket and took average readings of temperature on each point of time.

Factors affecting efficiency of bomb-calorimeter

a) Incomplete combustion

If the inside of the bomb is sooty that means combustion of the fuel was incomplete and the experiment and results are no longer usable. Incomplete combustion generally takes place when the fuel is in powdered form. That's why such materials are compressed into pellets. In the case of volatile fuels, probability of incomplete combustion is increased because types of fuels are kept in glass bulbs for preventing them from evaporation and these glass bulbs generally break during the experiments and large amount of carbon is formed. This may be due to metal oxides formed in combustion of the fuse wire, or to inclusion of a small amount of carbon in the glass while it is molten.

b) Oxidation of crucible and fittings

Sometimes oxidation of the crucible and its support also takes place which can be avoided by platinum crucibles and fittings.

c) Reaction of acids with bomb material

Accuracy of the bomb calorimeter also gets affected by reaction of acids formed in combustion with the material of the bomb. But it can be minimize by using suitable corrosion-resistant material for the bomb. Bombs provided with gold or platinum linings have also been found to be satisfactory,

d) Combustible impurities in oxygen

Very severe errors may be occurred in the results, if admitted oxygen has some combustible pollutants. Oxygen may have enough H_2 to cause serious errors. For avoiding this situation, it can be passed over CuO at $500^\circ C$

Experimental set up & Methodology

Summarized directions are given for bomb calorimetric experiment with benzoic acid with different types of insulating jackets, to determine the cooling correction factor for the apparatus". Following these instruction there will be a brief discussion of the differences in procedure in an experiment to determine the cooling correction factor of bomb calorimeter in each case.

These procedure are followed step by step before performing experiments:

- Preparation and Weighing of Sample of Benzoic Acid
- Preparation of bomb
- Preparation of Bucket
- Assembly of bomb calorimeter

Table 2. Results of Experiment I

Experiment no.(Water jacket)	T
1	2.2
2	2.4
3	2.1

$$\Delta T = T_{\text{initial}} - T_{\text{final}}$$

Average temperature difference is 2.23 °C

Experiment II

In our second experiment entire apparatus is insulated with glass wool jacket:

We slightly modified our existing bomb calorimeter for this experiment so that glass wool can be inserted in the jacket instead of water. Second experiment took place, where entire water from the outer jacket was removed and jacket got filled with glass wool. We observed the temperature readings while using benzoic acid as a fuel following the general procedure. We took 1 gram (approx) of benzoic acid for each experiment and experimented 3 times with water jacket and took average readings of temperature on each point of time and plotted graph with those readings.

Table 3. Results of Experiment II

Experiment no. (Water jacket)	ΔT
1	2.3
2	2.6
3	2.4

$$\Delta T = T_{\text{initial}} - T_{\text{final}}$$

Average temperature difference is 2.43 °C



Figure 9. Jacket of bomb calorimeter filled with glass wool.

Experiment III

In our third experiment entire apparatus is insulated with cotton wool jacket

Third experiment took place, where all the glass wool from the outer jacket was removed and jacket got filled with cotton wool. We observed the temperature readings while using benzoic acid as a fuel following the general procedure. Now we observed the temperature readings while using benzoic acid as a fuel. For this experiment we used our modified bomb calorimeter which was used for previous experiment, we just removed glass wool and filled jacket up with cotton wool.

We took 1 gram (approx) of benzoic acid for the experiment and experimented 3 times with water jacket and took average readings of temperature on each point of time and plotted graph with those readings.

Table 3. Results of Experiment III.

Experiment no. (Water jacket)	ΔT
1	2.2
2	2.4
3	2.3

$$\Delta T = T_{\text{initial}} - T_{\text{final}}$$

Average temperature difference is 2.3 °C



Figure 10. Jacket of bomb calorimeter filled with glass wool



Figure 11. Jacket of bomb calorimeter filled with saw dust.

Result & discussion

In this section results obtained from experiments with different insulating materials have been used to establish relation with heat equivalent of bomb calorimeter in each case (Experiment I, II & III) and then discuss the significance of using these insulating materials in place of water jacket.

Table 4. Average temperature differences of the experiments with different insulation

S. no.	Jacket	$\Delta T_{\text{(Avg)}}$
1	Water jacket	2.23
2	Glass wool jacket	2.43
3	Saw dust jacket	2.3

$$\Delta T = T_{\text{initial}} - T_{\text{final}}$$

Formula used for calculation –

$$EE = \frac{CV \text{ of Benzoic acid} \times \text{Mass of Benzoic acid}}{\text{Rise in temp. of bucket water due to combustion of same mass of benzoic acid}}$$

Where,

Calorific value of benzoic acid = 6325 Kcal / kg.

EE = Energy Equivalent of bomb calorimeter

CV = calorific value

Mass of benzoic acid = 1 gm

Using above formula following results have been observed:

a) Energy equivalent (EE) of bomb calorimeter with water jacket

$$EE_{\text{(water jacket)}} = 2.83 \text{ Kcal / kg}$$

$$\text{Or } EE_{\text{(water jacket)}} = 11840.72 \text{ J / kg}$$

b) Energy equivalent (EE) of bomb calorimeter with glass wool jacket

$$EE_{\text{(glass wool jacket)}} = 2.602 \text{ Kcal / kg}$$

$$\text{Or } EE_{\text{(glass wool jacket)}} = 10886.77 \text{ J / kg}$$

c) Energy equivalent (EE) of bomb calorimeter with saw dust jacket

EE (saw dust jacket) = 2.75 Kcal / kg

Or EE (saw dust jacket) = 11506J / kg

From the above results it can be said that increment in the adiabaticity of bomb calorimeter lowers the energy equivalent of bomb calorimeter. That means better insulation of the bomb calorimeter ensures the adiabatic system and clearly affects the heat equivalent or energy equivalent.

Conclusion

Standardization procedure of the bomb calorimeter produces the energy equivalent of the calorimeter for a specific temperature rise. Before a material with an unknown heat of combustion can be tested in a bomb calorimeter, the energy equivalent (EE or E) or heat capacity of the calorimeter must first be determined. This value represents the heat capacities of different components of the apparatus i.e. metal bomb, bucket stirrer and jacket. Since the system changes slightly with use, energy equivalents are determined empirically at regular intervals by burning a sample of a standard material with a known heat of combustion under controlled and reproducible operating conditions. In other words it can also be defined as number of calories necessary to raise the temperature of the entire calorimeter system by 1°C. From this definition we can suggest that if there is less heat leakage from the system then less calories will be required to raise the temperature which directly affect the energy equivalent of the calorimeter. In other words, energy equivalent of any bomb calorimeter is dependent upon a set of operating conditions also on the adiabaticity of system. If the apparatus is more adiabatic then energy equivalent will be less which is clearly seen from our test results. Now, it can be said from our research work that energy equivalent of any bomb calorimeter also depends upon the adiabaticity of the system.

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