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Water Flow Calorimetry, Experimental Runs and Validation Testing for BlackLight Power Inc.

Performed at Rowan University
Glassboro, New Jersey

January 2009 – May 2009

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May 2009

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Executive Summary

The company BlackLight Power (BLP) of Cranbury, NJ has been conducting experiments on the heat generation from materials which demonstrate what they believe is a proprietary process capable of relaxing the hydrogen atom below its normally considered ground state. For some time now, teams of engineering professors and their students at Rowan University have been involved in validating many of the heat experiments performed by BlackLight Power in our own campus laboratory facilities. The project described herein is the most recent continuation of previous calorimetry experiments which were completed over the last few months with the assistance of professors and students from the Chemistry and Biochemistry departments within the University. The findings which will be highlighted in this report confirm the water flow calorimeter can precisely and accurately measure heat from known chemical reactions. The specific reactions examined were the thermal decomposition of ammonium nitrate (NH_4NO_3) to form nitrogen, oxygen and water as well as the reaction of magnesium (Mg) and iron oxide (Fe_2O_3) to form magnesium oxide (MgO). By completing these well known, easily modeled reactions, it was possible to validate the calorimeter's performance when its reactants contained a known quantity of energy.

Rowan University Laboratory Setup

Materials and Equipment

The laboratory setup remained unchanged since the last report of our results, being based around a water flow calorimeter containing a simple test cell. The test cell in which chemical reactants are loaded is housed inside a vacuum chamber around which is wrapped ¼" copper tubing. A water chiller in conjunction with a pump supplies a constant flow of 20°C water to the vacuum chamber tubing. The test cell is surrounded by an electric resistive heater that is powered by a Xantrex XDC 600-10 power supply. A second precision power supply (HP) provides power and a reference voltage to the rest of the instrumentation, such as various thermistors used in the process. Once a test and any reaction inside the cell has completed, helium is introduced into the vacuum chamber to allow for a faster cool-down cycle.

All measurements are recorded with the help of a simple LabView program. Time, cell temperature, inlet and outlet water temperature, water flow rates were all recorded at 5s-10 s interval. Data are then analyzed to calculate the input energy from DC heater and the output energy from the water flow rate and change in inlet and outlet water temperature using a program written in MatLab. Profiles of the change in water temperature, cell temperature and comparing the input and output energies were plotted. This work is done mostly with the help of a simple MATLAB program, which analyzes the many data points and performs all the necessary calculations.

During the course of testing from January 2009 to May 2009, two different cell sizes were used with the 1 kW BLP reactor for chemical control heat experiments. The first was the traditional 1" diameter heavy duty cell used for small exothermic reactions, the second being a new 2" diameter heavy-duty cell used for greater exothermic reactions (Figure 1). As recommended by BLP, the 1" heavy duty cells were used for the magnesium and iron-oxide thermite reaction, while the 2" heavy-duty cells were used for decomposition ammonium nitrate experiment.

Figure 1: Showcasing the 1" (bottom) and 2" (top) heavy duty cell sizes

Calibration and Experimental Run Procedures

Before and after every test or calibration it is crucial to manually measure the water flow rate to ensure it remains relatively constant throughout each run. The heater power supply is briefly turned on with low limits to ensure proper and safe connection with the heater circuit. All valves and lines for the helium and vacuum pump are checked for leaks, and data is recorded until equilibrium of the cell temperature has been reached.

For all the chemical control heat experiments, the chemistry department procured chemicals. The graduate students of the chemistry department in the presence of project team carried out the chemical loading of the cell. In order to improve the conversion rate of the reaction, magnesium and iron oxide were thoroughly mixed during the cell loading.

For all runs, after thermal equilibrium of the cell temperature is reached, the vacuum chamber is pumped down to below 10 mTorr, ensuring that almost none of the supplied and generated heat is released to the calorimeter during the cell heating stage. Once the desired vacuum in the chamber is reached, varying methods of heating are applied

depending on the type of run to be completed. During a calibration run a predetermined amount of energy is supplied to the heater core, which will subsequently be compared to the energy (in terms of heat) measured by the calorimeter. A heat run is completed in almost the same fashion, with the exception that the input energy necessary to start the reaction is unknown. This input energy is recorded as the output voltage and current of the power supply, along with the output energy (again in terms of output heat) measured by the calorimeter. For all types of runs these input and output measurements are compared, providing the researcher with an accurate account of the accuracy of the calorimeter, and the energy produced by the reaction in the cell. The cool-down process, consisting of the infusion of helium into the vacated chamber, speeds up the process of recovering the generated heat, while also reducing observed losses.

Results and Discussion

Calibration Results

A total of 15 successful calibration runs were completed during the spring 2009 semester. Calibration runs were performed frequently between heat runs to ensure energy coupling efficiency of the calorimeter ranged in the high 98-99% . Table 1 gives the data for these calibrations, all of which having a high coupling efficiency. Figures 2, 3 and 4 below represent the energy profile, cell temperature and delta temperature respectively for a calibration run with an input energy of 120 kJ.

**Table 1: Data recorded from calibration runs**

Run	Date	Input	Output (kJ)	ΔkJ	% Difference	Coupling
1	1/21/2009	150.57	152.43	1.86	1.23	1.012
2	1/27/2009	120.16	119.75	-0.41	0.34	0.997
3	1/29/2009	200.72	200.30	-0.42	0.21	0.998
4	1/30/2009	150.32	148.37	-1.95	1.31	0.987
5	2/2/2009	180.22	179.06	-1.16	0.65	0.994
6	2/5/2009	200.21	199.93	-0.27	0.14	0.999
7	2/6/2009	94.03	94.72	0.70	0.74	1.007
8	2/9/2009	120.41	121.53	1.13	0.93	1.009
9	2/10/2009	149.96	152.49	2.53	1.67	1.017
10	2/13/2009	150.32	152.41	2.09	1.38	1.014
11	2/24/2009	49.67	49.99	0.33	0.52	1.007
12	2/27/2009	99.59	99.42	-0.18	0.18	0.998
13	3/23/2009	99.91	99.80	-0.11	0.11	0.999
14	4/7/2009	198.63	194.76	-3.87	1.97	0.981
15	4/16/2009	109.87	109.42	-0.46	0.42	0.996
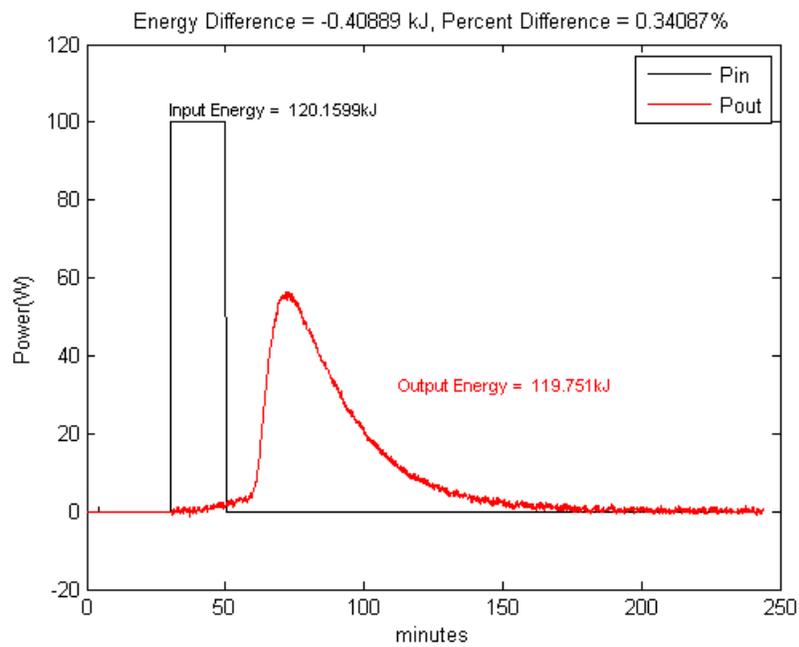
Figure 2: Input energy vs. output energy
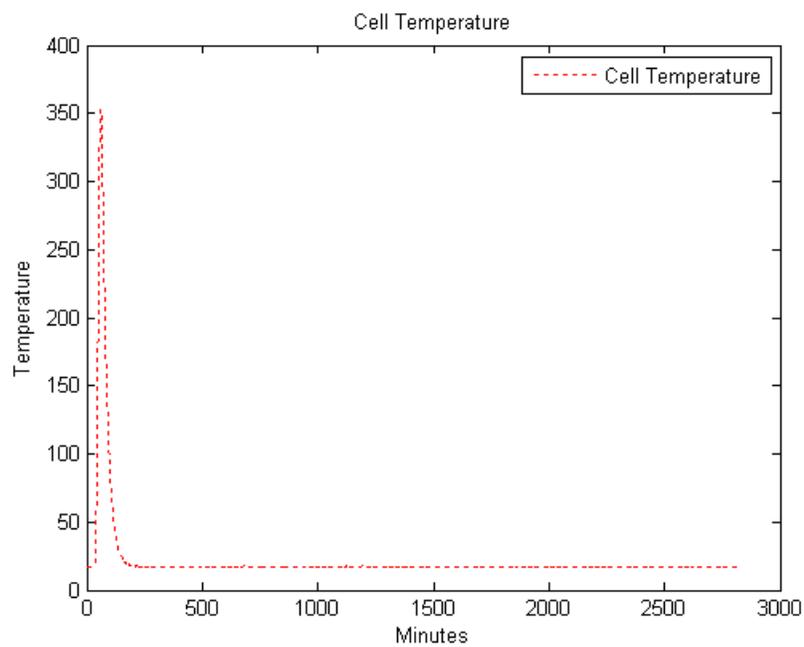
Figure 3: Cell temperature
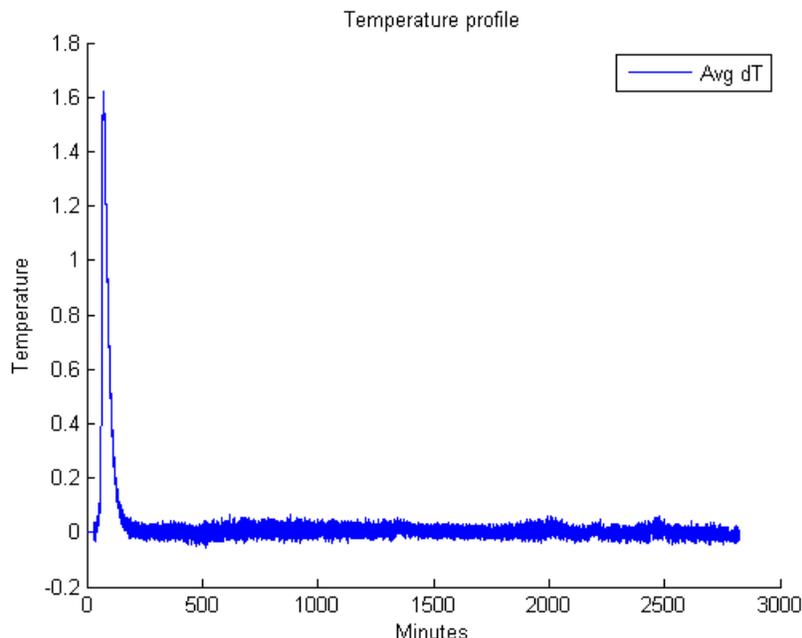
Figure 4: Change in Water Coolant Temperature

It is important to note the difference in the time-span of these figures. When calculating the output of a given run, the equipment is allowed to cool down for several hours. This is crucial in determining the offset due to thermocouple inconsistencies, later to be added or subtracted from the all data. The cool-down period is not incorporated into the final projections for output energy, as its value consists only of environmental factors such as ambient temperatures. Figure 2 represents the entire span used in the calculation of output energy, whereas Figures 3 and 4 plot the entire set of recorded data.

Experimental Results

Example results from control heat runs, three using the reactants Mg and Fe_2O_3 and one using the reactant NH_4NO_3 are listed in Table 2. Figure 5, 6 and 7 represent the energy profile, cell temperature, and change in water temperature recorded during run 3, the second control heat run with Mg and Fe_2O_3.

Table 2: Recorded Heat run data for $MgFe_2O_3$ and NH_4NO_3

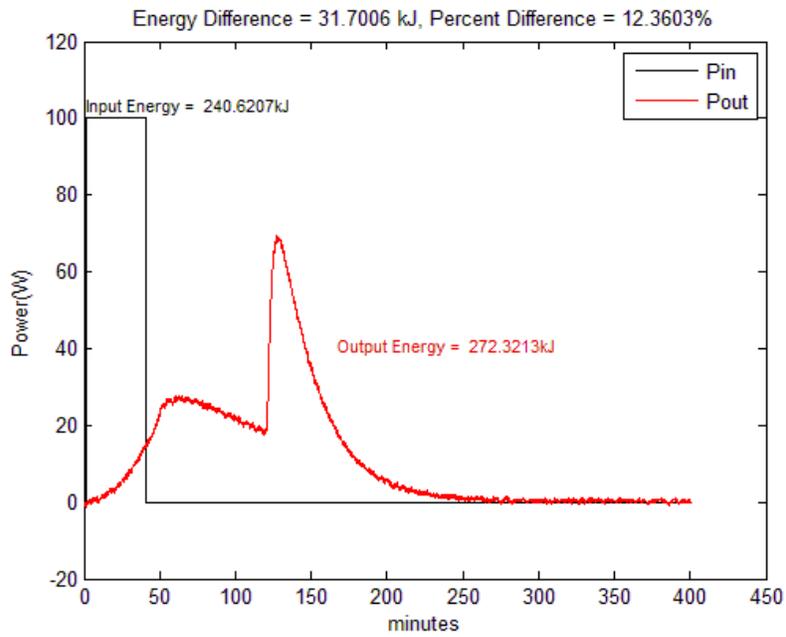| Run	Date	Chemicals	Cell Size	Input (kJ)	Output (kJ)	ΔkJ	Theoretical
1	3/12/2009	NH_4NO_3	2"	443.95	481.65	37.691	41.2
2	4/9/2009	$MgFe_2O_3$	1"	240.62	271.83	31.70	32.75
3	4/12/2009	$MgFe_2O_3$	1"	227.51	256.46	28.95	32.75
4	4/22/2009	$MgFe_2O_3$	1"	177.83	210.21	32.37	32.75

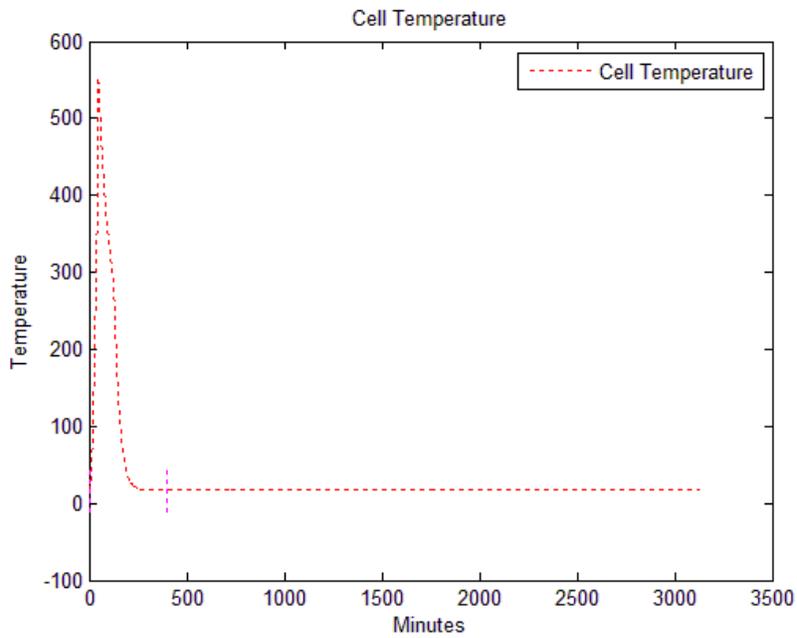**Figure 5: Heat run energy profile of Magnesium Iron Oxide on 4/9/09**

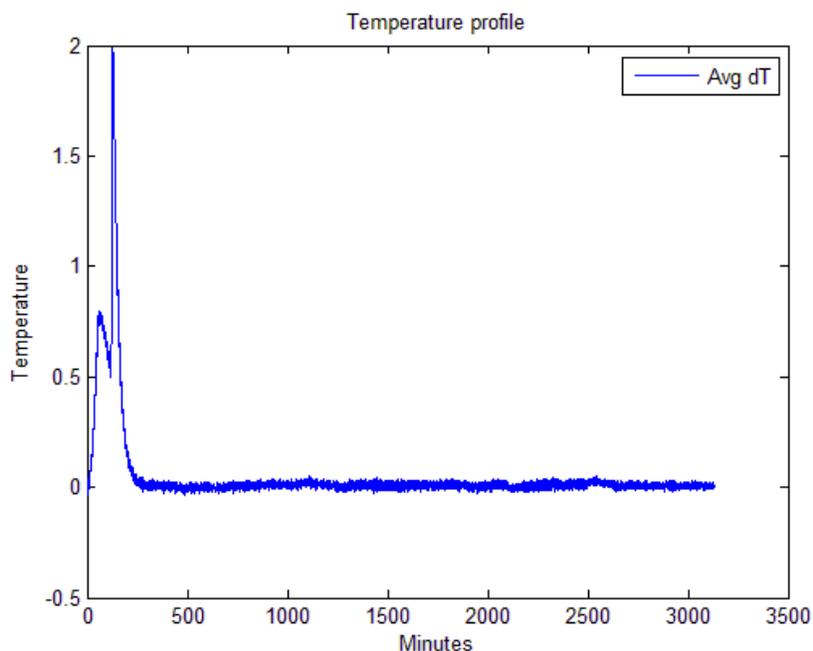**Figure 6: Cell temperature profile of heat run for MgFe$_2$O$_3$ on 4/9/09**

Figure 7: Change in water coolant temperature of heat run for $MgFe_2O_3$ on 4/9/09

Background calculations

Two exothermic reactions were considered for testing in the experimental calorimeter set up as chemical control heat runs. The reactions were (1) thermal decomposition of ammonium nitrate (NH_4NO_3) and (2) the reaction of iron oxide (Fe_2O_3) and magnesium (Mg). The theoretical heats of reaction provided below are under an assumption of complete conversion.

$$2NH_4NO_3 \rightarrow 2N_2 + O_2 + 4H_2O \tag{1}$$

$$Fe_2O_3 + 3Mg \rightarrow 3MgO + 2Fe \tag{2}$$
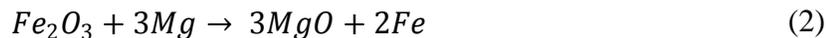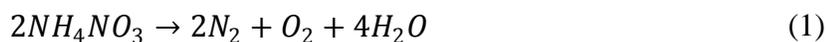
Reaction 1 is highly exothermic, ΔH_{rxn}=-412 kJ/mol, and occurs at temperatures above 300°C. However, there is a side reaction that occurs during the thermal decomposition of ammonium nitrate.

$$NH_4NO_3 \rightarrow N_2O + 2H_2O$$
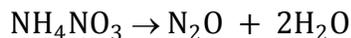
The side reaction is favored at lower temperatures (200-250°C) and constitutes a net heat of reaction of -124.4kJ/mol. Therefore, this reaction is only a minor part of the overall mechanism and can be avoided by heating cell to a temperature where reaction 1 would be dominant. The second reaction used for the experimental procedure was the reduction of iron oxide by magnesium. Reaction 2 is also an exothermic reaction with a ΔH_{rxn}=-980.6 kJ/mol. No significant side reactions were found for this process.

The quantities of reactants used by the team for heat runs during the semester were identical to those used by BLP in the supplemental materials provided. Reaction 1

required 16 g of ammonium nitrate, which yielded a theoretical energy output of 41.2 kJ. The quantities of reactants used for reaction 2 were 5.34 g and 2.43 g of iron (III) oxide and magnesium respectively. The energy released by reaction 2, using reactant quantities above, was calculated to be 32.75 kJ. These calculations represent the maximum theoretical amount of energy being generated. In order to ensure the completion of the reaction, chemical cells were heated to a significantly higher temperature. Still full completion of the reaction is very difficult to achieve and this is evident from the calorimetric measurements which are off by a few kJ.

Conclusions

From the experimental results obtained, it can be concluded that the calorimeter system is both accurate and precise at measuring the heat evolved from a thermal decomposition of known reactants. The accuracy of the calorimeter was established with heater calibration runs, since DC current and voltage measurements and hence input power and energy measurement can be performed more accurately than chemical reactions. It was possible to provide a measure of the calorimeter accuracy using the energy generated by a known chemical reaction. All in all, the calibration runs proved that the calorimeter is accurate within one percent of the power input by a resistive heater, and the chemical control heat runs gave strong indication that there are no significant differences between heat loss mechanisms. The Rowan University team is confident that the calorimeter is very accurate, and will continue to complete calibration, control and heat runs to verify BlackLight Power's claims.