Design and Validation of a Continuous Flow Calorimeter

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Introduction

Calorimeters are typically very expensive products ranging in the thousands of pounds [1]. Through completion of a third-year project and the continuation of the research over the summer a low-cost, high sensitivity continuous flow reaction calorimeter was designed and built. The device uses a concept of an isothermal power compensated spatially resolved reaction calorimetry in continuous flow. A Peltier element cools the reactor coiled around a heat exchanger (figure 1). The Peltier element compensates for the reaction heat to keeps the reactor isothermal, so by measuring the electrical power it is possible to determine the reaction heat. This design could be used to measure milliwatt changes in thermal power from chemical reactions. By producing a micro-calorimeter there are economic and safety advantages as chemical consumption is low [2]. Utilizing computer aided design (Solidworks, Fusion 360) it was possible to simulate the thermal distribution of the brass heat exchanger (figure 2).

Figure 1: Basic Principle of the Calorimeter Device

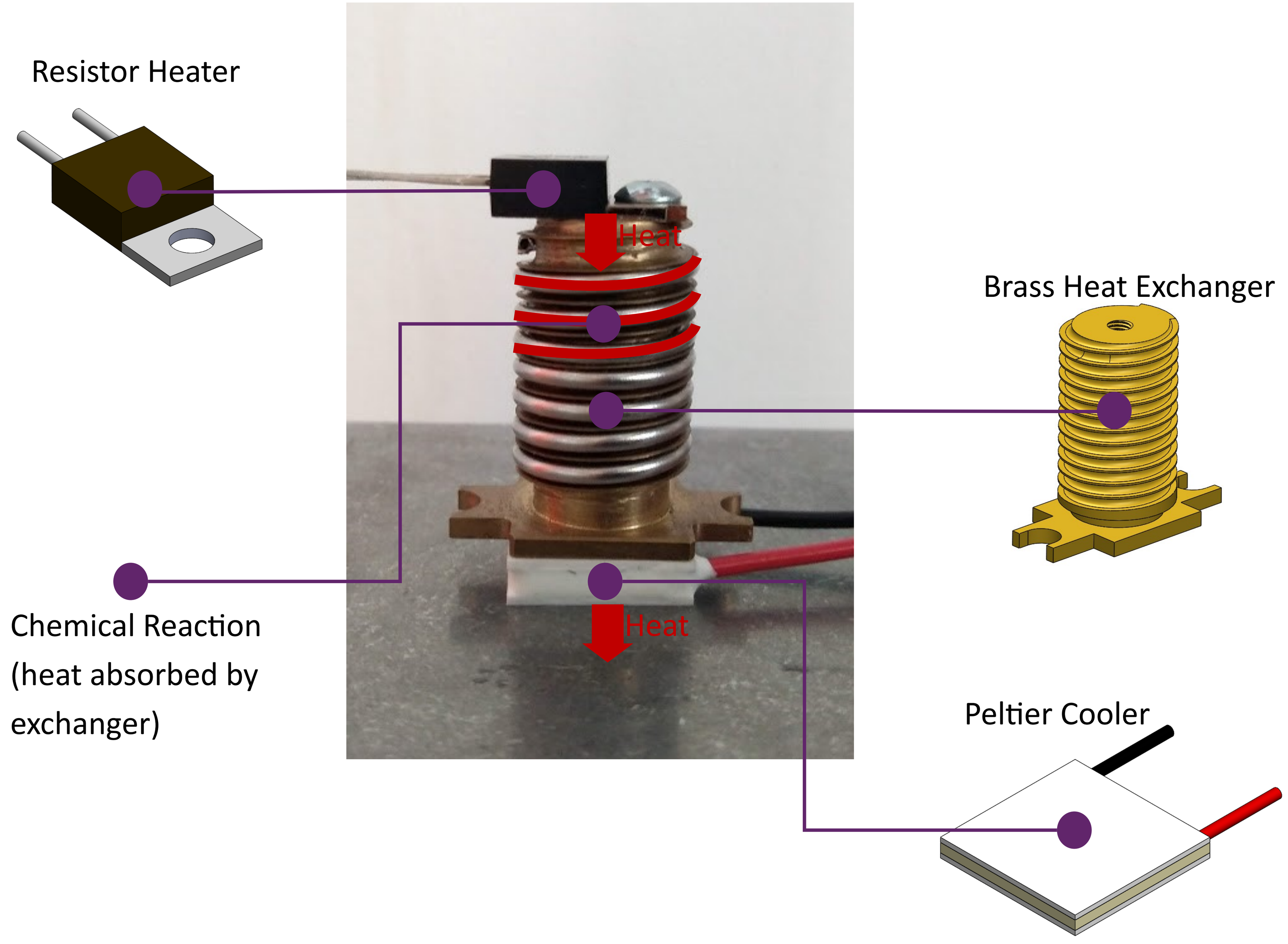


Figure 2: Thermal Distribution - Infrared Image and Solidworks Simulation

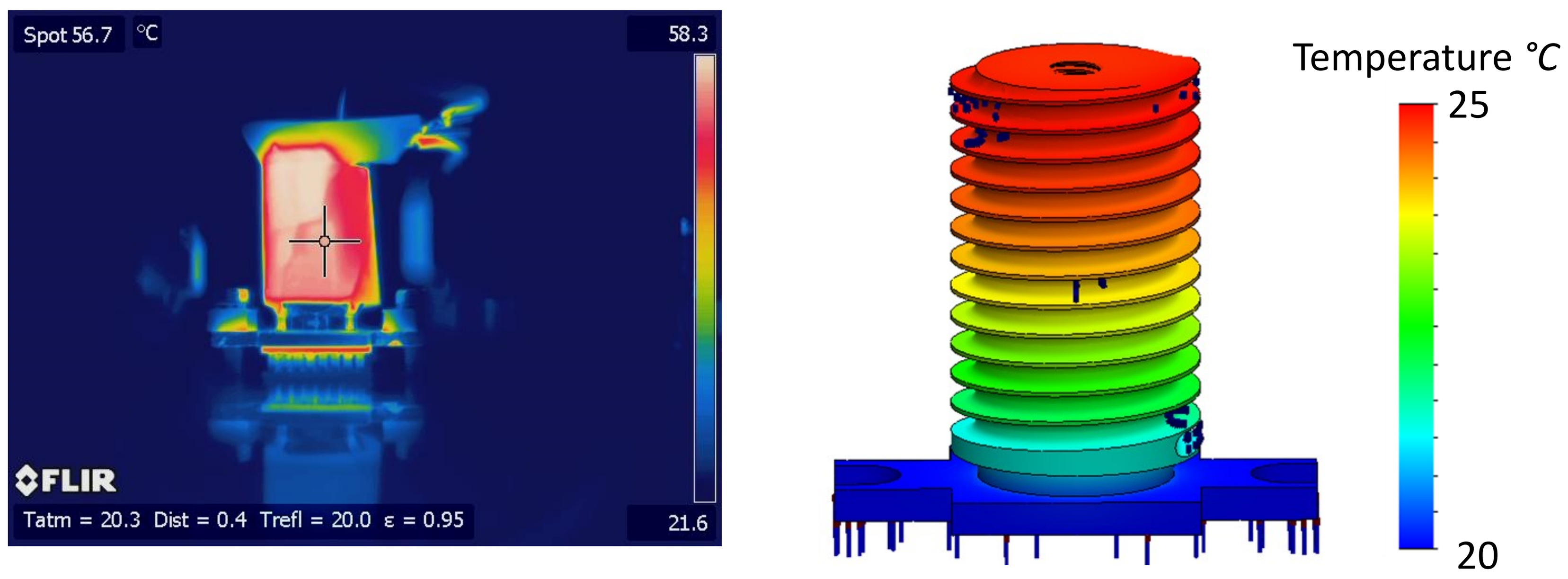
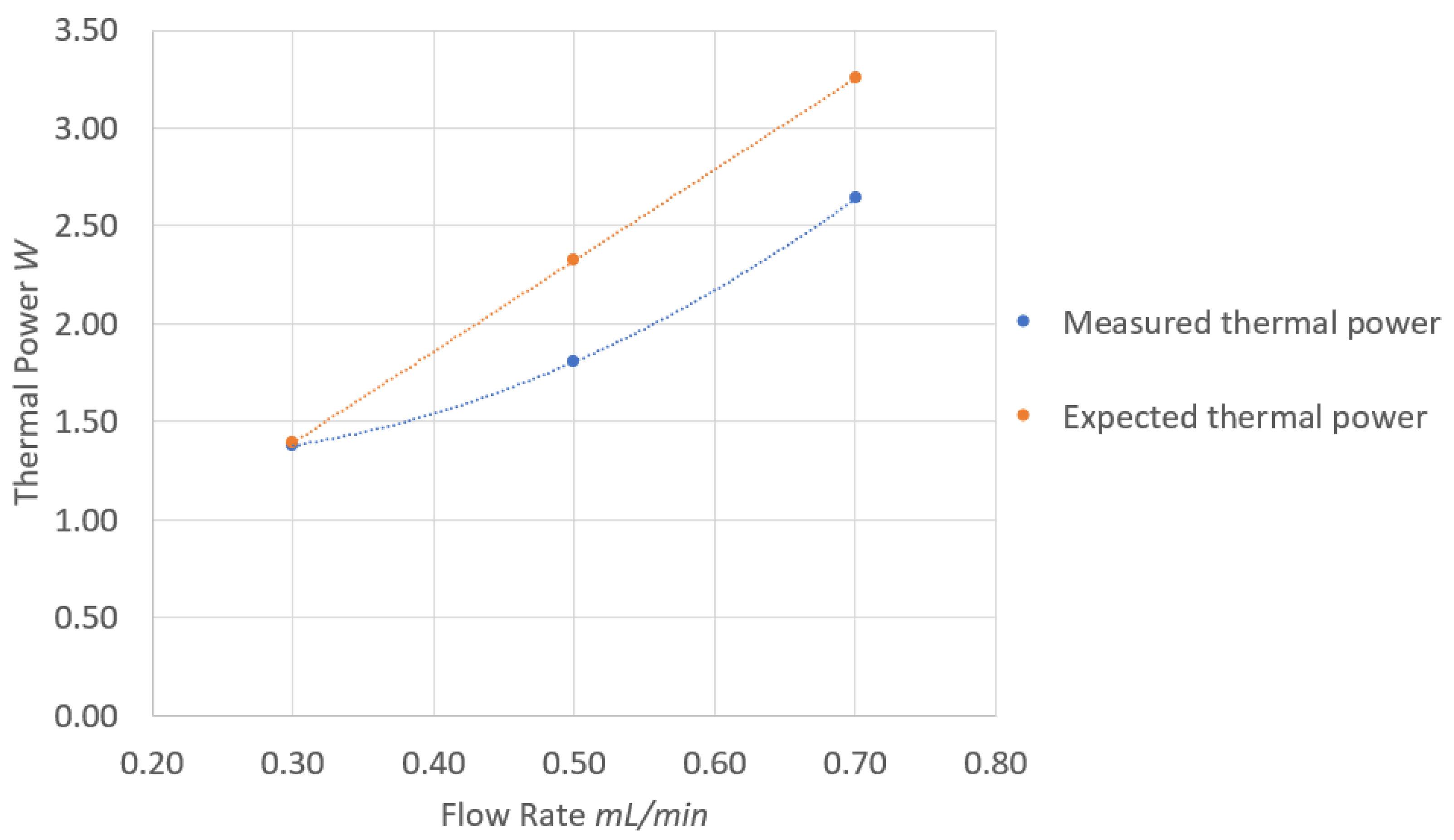


Figure 3: Initial Experiment - Thermal Power from Neutralisation



Design and Validation

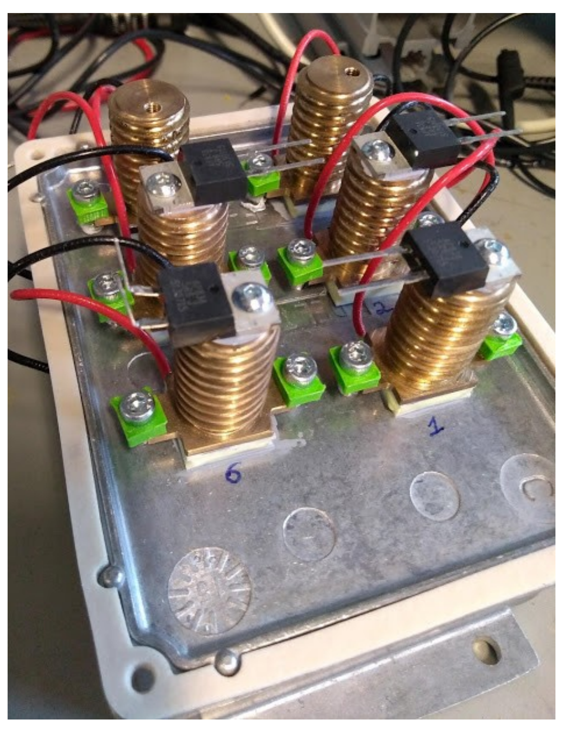
Firstly, from the requirements and specification of the device for example coiled pipe diameter, expected thermal power, temperature distribution, heat transfer coefficient and resonance time, the heat exchanger was designed. Then manufactured using a CNC machine (computer numerical control) because of the mechanical properties of copper it was not an appropriate material and brass was hence used. Initially, to obtain isothermal flow and reduce heat loss to the environment a vacuum chamber was built. To calibrate the calorimeter device the Joule effect was used whereby the heaters fixed to the top of the brass heat exchangers mimic the thermal power release of a chemical reaction. Through mixing Hydrochloric acid HCl and Sodium Hydroxide NaOH in a single reactor unit within the vacuum chamber results were gained for differing flow rates (figure 3).

This approach was not satisfactory for the milliwatt level of precision desired for the device. The new approach adopted consists of using six reactor units thus increasing resonance time, using high precision 16-bit temperature sensors and controlling the Peltier's and heaters to a greater level of precision (figure 4). To ensure isothermal flow, the reactor units are submerged in a temperature-controlled water bath and an Arduino nano is employed along with other hardware to implement proportional-integral-differential (PID) control to accurately compensate for the thermal power released from chemical reactions.

Conclusion

To conclude, from the first calorimeter approach reasonably accurate readings were gained for thermal power at low flow rates (0.02 W from the expected result) however there are many limitations with this design due to only one unit used, error from heat loss and human error involved. Therefore, the results differed from the expected thermal power, especially at the higher flow rates. Through the second approach achieving a higher level of precision, improving the isothermal characteristics and using a PID system to reduce human error very small changes in temperature will be able to be measured. A publication will be prepared from the findings of this calorimeter device.

Figure 4: Final Calorimeter Device Setup



Acknowledgments

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Citations

[1] LabX, "LabX - Calorimeter Listings." [Online]. Available: https://www.labx.com/calorimeter. [Accessed: 27-Oct-2018].

[2] F. Reichmann, S. Millhoff, Y. Jirmann, and N. Kockmann, "Reaction Calorimetry for Exothermic Reactions in Plate-Type Microreactors Using Seebeck Elements," Chem. Eng. Technol., vol. 40, no. 11, pp. 2144–2154, 2017.