ABSTRACT

Title of Document:	SPONTANEOUS IGNITION OF LINSEED OIL SOAKED COTTON USING THE OVEN BASKET AND CROSSING POINT METHODS.
	Justin T. Worden, M. S., 2011
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The oven basket method coupled with the Jones version of the crossing point method was used to test the following basket sizes with their respectful concentrations of linseed oil soaked cotton, 5cm (33.3%), 5cm (50%), 5cm (75%), 7.5cm (77%), 10cm (80%) with concentrations measured by weight. Some of the samples reached three different stages; ignition, smoldering or constant smoldering and flaming. The activation energies were 42.37 kJ/mol, 27.40 kJ/mol, 16.97 kJ/mol, 15.76 kJ/mol and 11.73 kJ/mol for the 5cm (33.3%), 5cm (50%), 7.5% (77%) and 10cm (80%) basket sizes. It was concluded that as the concentration and the basket size increased the activation energy decreased. The P and M values along with the reaction rate per unit volume were also calculated. The time to ignition increased as the oven temperature that each sample was tested at decreased and as oven temperatures approached ambient the time to ignition significantly increased topping 5.5 hours for the 5cm (75%) basket size.

SPONTANEOUS IGNITION OF LINSEED OIL SOAKED COTTON USING THE OVEN BASKET AND CROSSING POINT METHODS.

By

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Nomenclature

A	Pre-exponential Factor
c	Specific Heat
E	Activation Energy
h	Coefficient of Heat Transfer at Surface
k	Thermal Diffusivity
Q	Heat of Reaction
R	Universal Gas Constant
r	Characteristic Length
T_o	Oven Temperature
T_c	Center Temperature of the Sample
T_i	Initial Temperature
α	Biot Number
ρ	Density
λ	Thermal Conductivity
δ	Frank-Kamenetskii Number or Damkohler Number
δ_c	Critical Frank-Kamenetskii Number or Critical Damkohler Number
τ	Dimensionless Time
θ	Dimensionless Temperature Rise

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1. Introduction

Spontaneous ignition has played a key role in many fires throughout history and because of this many tests have been performed on materials that spontaneously ignite to try to understand their tendencies. There are many ways to go about testing spontaneous materials but many of them can be very complicated to prepare and may take a very considerable amount of time and materials.

The main goal of this research was to help fire investigators better assess an easier approach to testing spontaneously igniting materials. This was done by investigating one of the most common methodologies for testing spontaneous material, the standard oven test or the oven basket method. This method uses common laboratory items, oven, thermocouples, data acquisition system, computer, and easily made items, stainless steel mesh baskets, for its testing along with the material that needs to be tested, which also makes this one of the more available ways to test spontaneous ignition. To go along with the easier methodology an easier way of analyzing the collected data was also pursued to again simplify this process for fire investigators. The typical way to use the oven basket method and analyze its data has been the Frank-Kamenetskii method, which involves finding a critical temperature for several different basket sizes. To find numerous critical temperatures an extremely excessive number of tests must be run, requiring ample amounts of time, materials and ultimately money to achieve the desired results. The crossing point method, which was used in this research, can achieve the desired results, theoretically, using a very minimal number of tests thus saving on time, materials and money. This method also uses the oven basket method in such a way that is less tedious and easier to perform overall.

By using these two methods for testing spontaneous ignition fire investigators could test a wider variety of materials with less time and effort than needed in the past. This could prove to be very beneficial in advancing the methods used in fire protection to prevent spontaneous ignition from occurring. Potentially, with new and different precautions being enforced the safety of people and property will benefit.

2. Background

2.1 Spontaneous Ignition, Frank-Kamenetskii Theory and Linseed Oil Soaked Cotton

2.1.1 Spontaneous Ignition

For a chemical reaction to be deemed exothermic a release of energy must be present. Any substance, by itself or in the presence of air, which possesses this exothermicity is inclined to an unstable condition in which its core temperature can increase significantly due to this chemical reaction. This possibly unstable reaction can be caused by either decomposition or oxidation of a solid, liquid or gas substance. Fertilizer piles, hay stacks, compost piles, saw dust piles, etc. are all common examples of decomposition cases. During the decomposition of the substance that makes up the pile heat is generated. Factors such as externally added heat, moisture within and surrounding the pile and microorganisms can to contribute to the decomposition. Other factors such as pile size, rate of diffusion of oxygen and the substance that makes up the pile also contribute to the heat generation in the pile.

Linseed oil applied to cotton rags is a common example of an oxidation reaction. In the oxidation reaction case, the heat generation from the reaction is accelerated by most of the same factors as the decomposition reaction; externally added heat, moisture, pile size, substance that makes up the pile, etc., but the heat is generated by a different process.

If the heat generated by either of these reactions is unable to escape from the pile it will start to build upon itself. The added heat that does not escape from the pile will accelerate the reaction even more until the temperature inside the pile starts to elevate significantly possibly initiating smoldering and deemed spontaneous ignition. If enough oxygen is present a sustained smoldering may occur, which may transition to flaming. The spontaneous ignition that has occurred is also referred to as thermal run-away or thermal explosion because the temperature inside the pile is said to run-away because of how quickly it increases at ignition.

2.1.2 Frank-Kamenetskii Theory

The theoretical basis behind the analysis is simple and ignores many factors talked about earlier such as the diffusion of oxygen, moisture, non-conduction heating within the substance, mixtures with competing reactions and transient effects. However, the theory, first put forth by Frank-Kamenetskii in 1938, does allow for a means of determining the conditions needed for spontaneous ignition and a means of obtaining data to correlate results over a range of material size and temperature. Because many factors are neglected in this theory its results can only be used as a guide for interpreting the spontaneous ignition in practice. Furthermore, it is the only tool that can be applied without the consideration of complex factors that may not stand the test of accuracy in their modeling.

There are three general configurations considered as representative of most realistic scenarios:

1. A substance of a particular shape exposed uniformly to surroundings with linear (Newtonian) cooling to a constant temperature.

2. A substance heated by a hot surface and with Newtonian cooling on its remaining surface.

3. A substance that is initially hot relative to its surroundings.

An example for scenario 1 would be a pile of linseed oil soaked cotton rags. Scenario 2 would be represented by a layer of dust on a hot surface with an example for scenario 3 being a pile of hot laundry just removed from a dryer.

These scenarios are very general and their application to real life scenarios is not likely to be perfect. These factors and their effects ignored by the theory are responsible for cautionary considerations in making predictions. However, this approach will give reasonable results based on the data that is provided. One important consideration should be taken into account when working with materials that melt. The data obtained at temperatures above the melting point would not conform to exposure conditions below the melting point. When the material is at a high temperature it would lose its integrity by melting but at low temperatures the outside of the material would remain solid.

The basis of this theory and its results are taken from Bowes (1) and his notation will be used. The basic theory is due to Frank-Kamenetskii (1938), and considers a zeroth order exothermic reaction with high activation energy (E) combined with pure conduction heat transfer in the material. The conduction equation with a uniform energy generation rate due to an exothermic chemical reaction is seen in Eq. 1.

$$\rho c \frac{dT}{dt} = \nabla \cdot \lambda \nabla T + \dot{q}''' \qquad \text{(Eq. 1)}$$

The generation term is given by the Arrhenius equation seen in Eq. 2.

$$\dot{q}''' = Q\rho A e^{-E/RT} \qquad \text{(Eq. 2)}$$

The factor A can depend on temperature and the concentration of the reactants. When the reactants are depleted A goes to zero as there is no more energy generation. A plot of the generation term with temperature is shown in Figure 2.1.

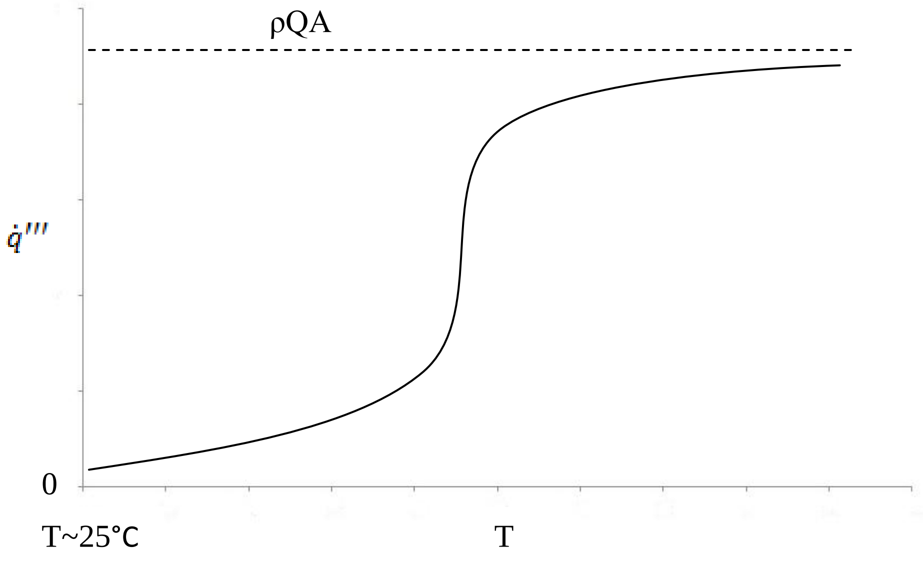


Figure 2.1: Generation Term vs. Temperature

It reaches a maximum of ρQA as T → ∞ and the values of $\dot{q}'''$ can range as low as 10^{-15} W/cm^3 at normal ambient temperature to 10^{10} W/cm^3 at flame temperatures. If A drops reactants are depleted, the curve in Figure 2.1 collapses down to the x-axis.

In Eq. 1, as long as the generation term is greater than the conduction term, the temperature will increase in the material. Even if a steady condition is reached the internal temperatures of the material will be higher than its surroundings, as the generation must equal the conduction loss.

For a pile in uniform surroundings the center temperature would be highest. Bowes (1) considers such a case and sketches the center temperature (T_o), in his notation, relative to the ambient temperature (T_A). The lower curve (0 – I), in figure, represents steady conditions for this reaction.

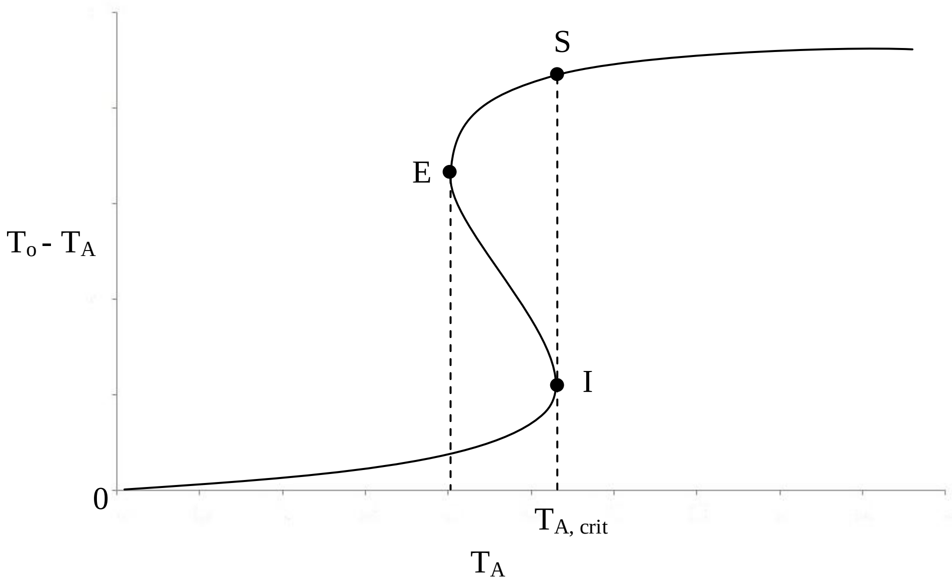


Figure 2.2: Center Temperature Relative to the Ambient Temperature (1)

The upper curve from E represents steady conditions once ignition occurs to a smoldering reaction. The level of the E curve will increase as the diffusion of oxygen also increases. The ambient temperature at I is the temperature needed for spontaneous ignition to smoldering. The new steady state to S involves a jump in the center temperature. The E point is the extinction of the smoldering reaction and the curve I – E is unstable. In reality the depletion of reactants would make the process unsteady. A graph of this is shown in Figure 2.3 where two different outcomes occur for the T_A less than or greater than its critical value at I, $T_{A,\ crit}$.

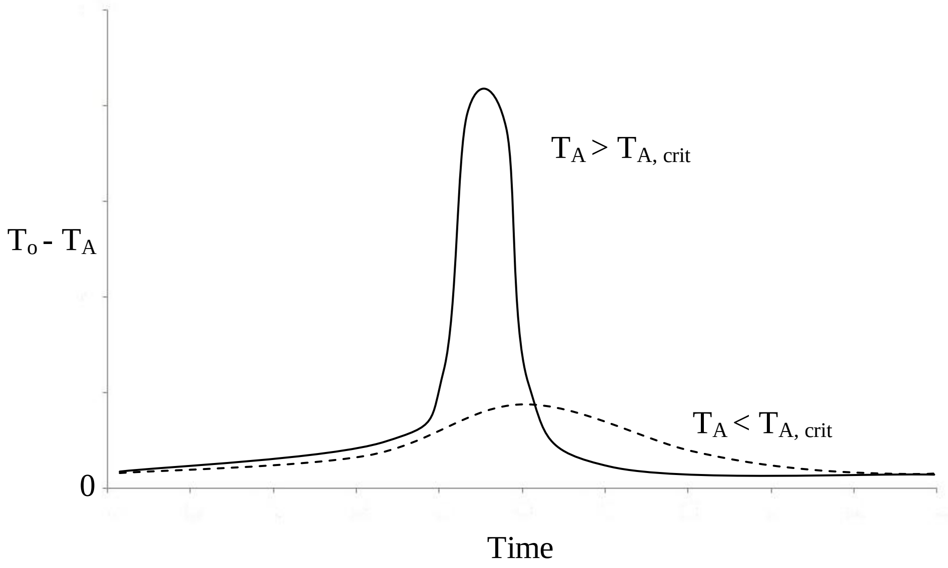


Figure 2.3: Ambient Temperature Above and Below Critical Ambient Temperature

There is a more significant temperature rise above the critical condition for ignition. Alternatively, the size of the pile at a fixed ambient temperature could have a critical condition for this large temperature rise or "ignition". Here the ignition outcome is depicted as steady smoldering but, flow changes in the pile at this jump in temperature could lead to flaming.

With no reaction depletion, two outcomes are possible: (1) a steady solution exists with an increase in the internal ($T_0>T_A$) due to the exothermic reaction, (2) no steady solution exists and the internal temperature will rise significantly. In the approximate theory of F-K to follow, it will become infinite. This is termed a "thermal runaway", or "thermal explosion" in the case of explosive materials, or "thermal ignition" in the case of achieving fire, spontaneous ignition to smoldering or flaming in theory as mentioned in section 2.1.1. Hence, the approach is to examine problem scenarios governed by Eq. 1 and Eq. 2 for which the point of no steady solution is found. This is the “critical condition” that makes the boundary between a stable steady solution and ignition or thermal runaway. In this way the conditions for spontaneous ignition are found.

The basic theory is based on conditions of high activation energy or more specifically

$\in = \frac{RT}{E}$ is small, $\in \ll 1$

by expressing the identity

$$-\frac{E}{RT} \equiv -\frac{E}{RT_A} + \frac{\theta}{1+\epsilon\theta} \qquad \text{(Eq. 3)}$$

where

$$\theta \equiv \left(\frac{E}{RT_A}\right)\left(\frac{T-T_A}{T_A}\right) \qquad \text{(Eq. 4)}$$

and letting $\in \rightarrow 0$, Eq. 1 now turns into Eq. 5.

$$\frac{d^2\theta}{d\tau^2} = \nabla^2\theta + \delta e^{\theta} \qquad \text{(Eq. 5)}$$

Here $\tau = t/(r^2/\ k)$, $k = \lambda/\rho c$. The theory uses three geometric shapes. In one-dimension, the steady equation for a slab of half-width r (j = 0), and infinite cylinder of radius r (j = 1), and a sphere of radius r (j = 2) is shown in Eq. 6.

$$\frac{d^2}{dZ^2} + \frac{j}{Z}\frac{\partial\theta}{\partial Z} + \delta e^{\theta} = 0 \qquad \text{(Eq. 6)}$$

The dimensions are shown in Figure 2.4 with the dimensionless length coordinate being Z = x/r.

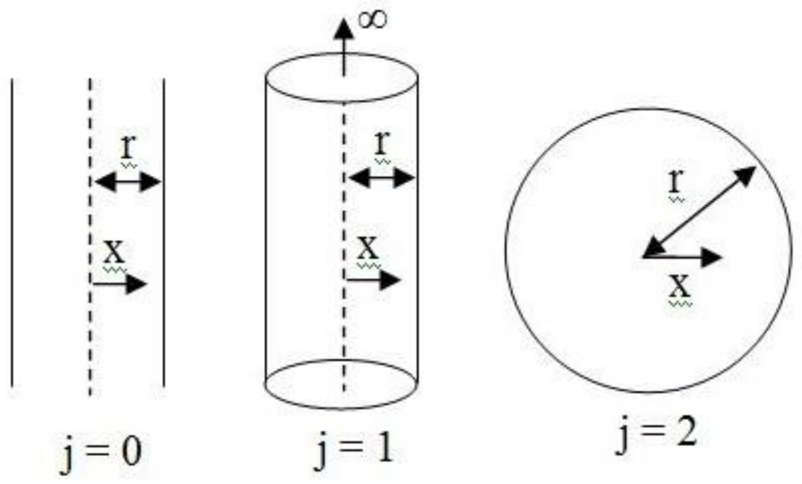


Figure 2.4: Dimensions for a Slab, Cylinder and Sphere

The parameter δ is a Damkohler number (dimensionless). Given in terms of the reference T_A:

$$\delta = \left(\frac{E}{RT_A}\right)\left(\frac{r^2\rho AQ}{\lambda T_A}\right)e^{-\left(\frac{E}{RT_A}\right)} \quad \text{(Eq. 7)}$$

The terms in parenthesis are also dimensionless. $\frac{E}{RT_A}$ is typically (E ~ 100 [kJ/mol]) about $\frac{10^4}{T_A}$. For fire conditions of $T_A \sim 10^3$, $\in \sim 0.1$. This can be thought of as the activation temperature to the system temperature. The second parenthesis represents the energy by chemical reaction to the heat conducted. It too is large for combustion substances, ~ 10^4, as δ is usually of order 1.

A solution for θ will depend on δ. As δ increases, caused by an increase in r or T_A, a value will be reached where a steady solution is not possible. This is the critical value, δ_c. Eq. 6 can be solved for different scenarios, and the δ_c can be determined. Then if $\delta \geq \delta_c$ for the actual state of the substance, ignition is said to occur. The scenario of a cold pile in a hot environment will be examined and the results found in the literature will be presented.

Consider uniform material in a given geometric shape exposed to the environment. Also consider Newtonian heat transfer at the surface, i.e Eq. 8a and Eq. 8b.

$$-\lambda \frac{\partial T}{\partial x} = h(T - T_A) \qquad \text{(Eq. 8a)}$$

or

$$-\frac{d\theta}{dz} = \alpha\theta \quad at\ z = 1 \qquad \text{(Eq. 8b)}$$

The heat transfer coefficient can be a combination of convection and linear radiant heating from the surroundings. The Biot number, α = hr/λ. For the unsteady problem where the initial temperature Ti < TA, the temperature response is illustrated for a symmetric slab of half–width, r, in Figure 2.5. This case is like the “oven basket method”, where cubes of material are inserted into a hot oven, to find critical temperature.

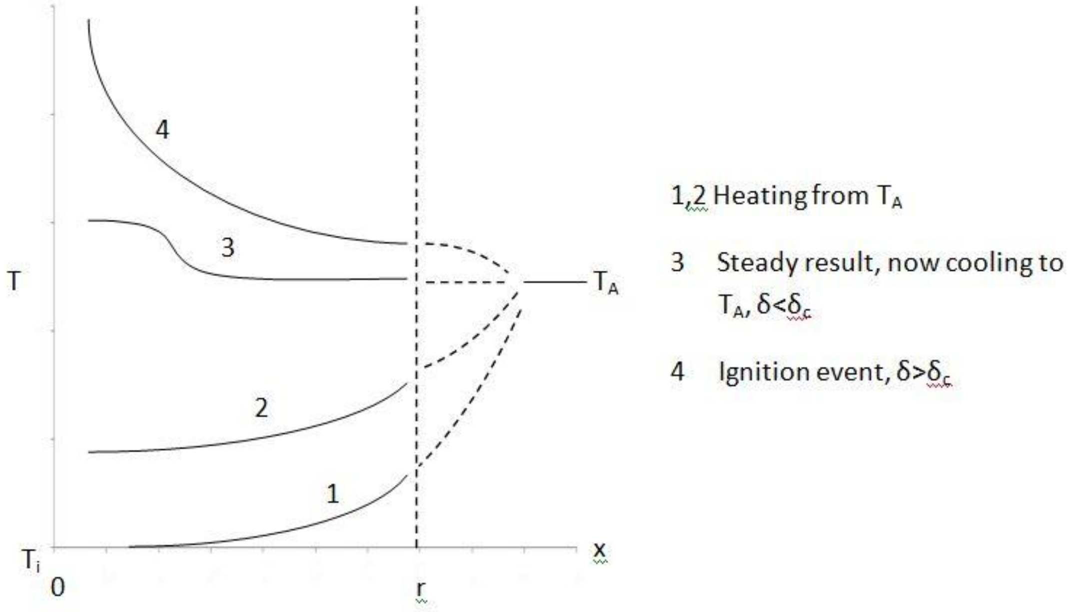


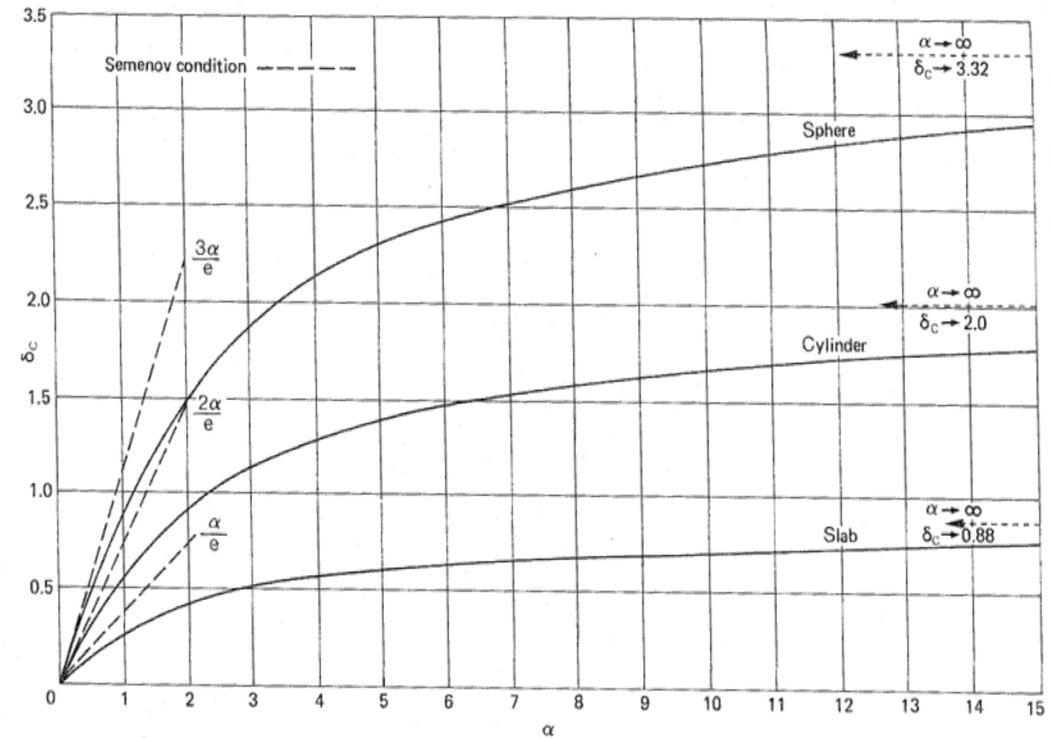
Figure 2.5: Temperature Response for Symmetric Slab

A solution to Eq. 6 with boundary conditions Eq. 8 has been given, by Thomas (1958), for the symmetric slab. The result shows that δc depends on α, such that

$\alpha \to 0, \quad \delta c = \frac{\alpha}{e}$

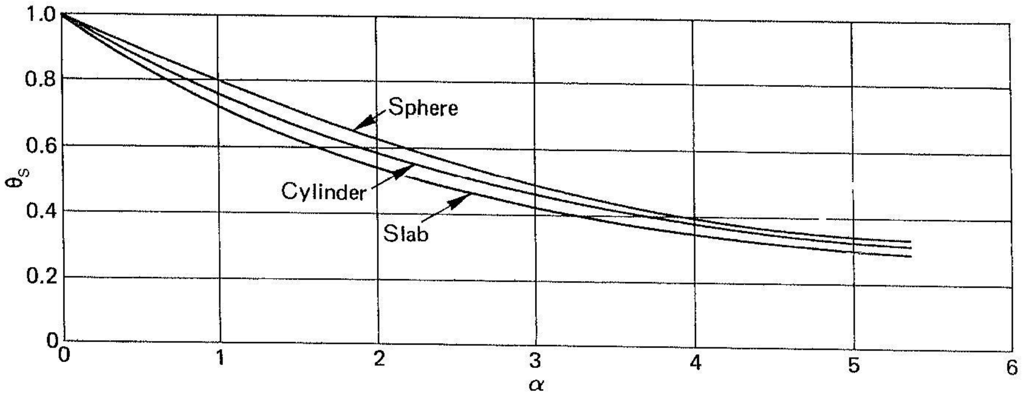
$\alpha \to \infty, \quad \delta_c = 0.88$

Complete results are given for the slab, cylinder, and sphere in Figure 2.6, and θ_s (θ at $z = 1$) and θ_0 (θ at $z = 0$) are given in Figure 2.7 and Figure 2.8, respectively. To implement these results, for a given geometry, the properties must be known, the size (r) and ambient temperature (T_A) must be specified, and α must be computed from heat transfer principles.



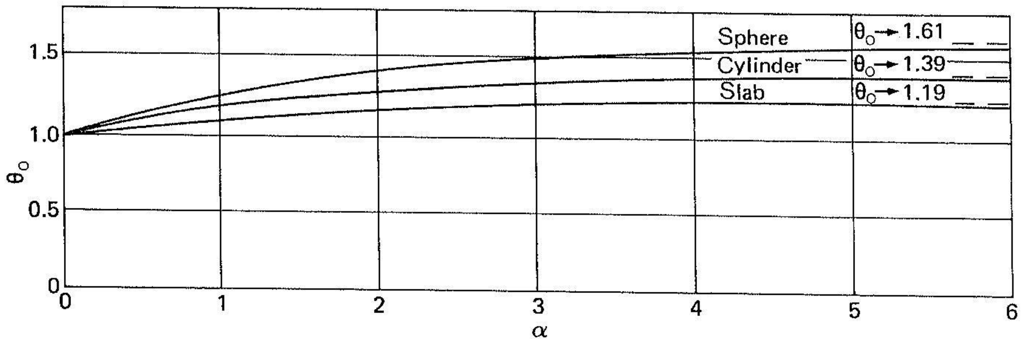
δ_c as a function of α for a sphere, a cylinder and a slab

Figure 2.6: Critical Damkohler Values for the Range of α, (1)



θ_s as a function of α for a sphere, a cylinder and a slab

Figure 2.7: θ_s Values for the Range of α, (1)



θ_O as a function of α for a sphere, a cylinder and a slab

Figure 2.8: θ_o Values for the Range of α, (1)

Table 1 gives values for a more complete list of geometries for $\alpha \rightarrow \infty$.

Body	**∂c(r)**	**θ_0**
Infinite Plane Slab, thickness 2r	0.878	1.119
Infinite cylinder, radius r	2.000	1.386
Sphere, radius r	3.322	1.610
Cube, side 2r	2.569	≈1.89

Infinite square rod side 2r	1.700	≈1.49
Short Cylinder, radius r, height 2r	2.764	≈1.78

Table 1: Critical Values for $\alpha \rightarrow \infty$, Body with Surface at T_A, (1)

2.1.3 Linseed Oil Soaked Cotton Research

Throughout the years it has been prevalent that the spontaneous ignition of oily rags has been a reoccurring cause of fires all over the world. These fires have not only caused hundreds of dollars in property damage, they have also put countless number of people's lives in danger. Numerous studies have been done on linseed oil soaked cotton using several different methods and configurations to understand and recognize its reactivity, and have helped in fire investigation and overall public knowledge.

Gross and Robertson (2) tested spherical piles of numerous materials including linseed oil soaked cotton. The linseed oil was applied to the cotton using a ratio of 1 part oil to 6 parts of cotton by weight or 16.66% concentration. To test their materials they used an adiabatic furnace with a multi-junction thermocouple and additional guard heaters to minimize heat loss. They determined the activation energy of their linseed oil sample to be 88 kJ/mol and their M to be 49.6, to be discussed in section 2.2, taken from Babrauskas converted table(3).

Khattab et al(4) used a differential thermal analysis (DTA), which is very similar to differential scanning calorimetry (DSC), to test their samples. To prepare their samples they immersed 20g of cotton in linseed oil then they wrung them out using a manually operated wringer and then left them to dry at room temperature. They tested four different sample concentrations, 0%, 2%, 10% and 20% of linseed oil to cotton, at six different oxygen volume concentrations. Their uncontaminated cotton had the highest activation energies while the 20% concentration samples had the lowest activation energies.

Taradoire (5) tested many different size samples including samples that involved 25g of cotton and 75g of cotton. It was found that the most favorable results came when 75g of cotton were soaked with 75g of linseed oil, a 50% sample concentration. These samples and ignited between 1hr and 6hrs.

Thompson (6) used a modified Mackey test to examine a combination of different oils including raw linseed oil. He states that the original Mackey test calls for 7g of cotton and 14g of oil to be tested but the samples he used in the modified Mackey test included 15g of cotton with 15g of linseed oil and 30g of cotton with 30g of linseed oil. He found that the linseed oil was the most hazardous substance compared to the other oils tested.

Radford (7) applied boiled raw and refined linseed oil to three different types of material, cotton rags, white waste and colored waste. The waste was lightly packed into cardboard boxes with air vents cut in the sides and top and placed on a metal shelf in a cabinet with heat lamps and smoke detectors installed. Of the 31 tests that were carried out, 13 of the samples went to flaming and 18 did not. During one of his test involving 3.5oz of colored waste soaked with 4oz of boiled linseed the smoke detector gave a signal at 90 minutes and the mass flamed at 125 minutes.

DeHaan (8) filled waste containers with linseed oil soaked cotton rags and achieved a brown discoloration and an acrid odor after 1hr with an internal temperature of 250°C happening after 3hrs and flaming happening 4hrs later.

Other sources found in Bowes (1) and Babrauskas (3) include Kissling who from an initial temperature of 23.5°C achieved ignition from 50g of cotton wool soaked with 100g of linseed oil. Gamble, who also used lightly packed cotton waste soaked with linseed oil into cardboard boxes, achieved ignition and a temperature rise from 21°C to 226°C in 6.25 hrs.

2.2 Oven Basket Method

Originally the oven basket method was used to provide an easier and less demanding way of finding critical temperatures of materials. This would then be used in conjunction with Frank-Kamenetskii's method of analysis to find the thermal parameters P and M. With the crossing point method being the means of analysis the oven basket method is used to reach a slightly different outcome of finding crossing points rather than critical temperatures.

The materials to use the oven basket method are fairly simple and straight forward. The oven can be an ordinary laboratory oven with an interior between .5m and 1m. A circulation fan must be installed on the inside of the oven to ensure forced convection throughout the oven. When using the F-K method of analysis good flow conditions are essential to make the biot number very large. The oven temperature should be controllable within at least ±1°C and go up to at least 200°C. A hanging rod would also be recommended inside the oven to provide a place for the samples to be hung from during the test.

The baskets used to contain the sample during the test should be open-topped baskets made of 60-mesh 0.25mm openings stainless steel, which is subject to the substance being tested. Sizes should include but are not limited to 2.5cm, 5.0cm, 10.0cm and 20.0cm. Depending on the substance being tested the largest and smallest sized baskets may be difficult to prepare which may result in using additional basket sizes other than what is recommended.

For temperature measurement a chromel/alumel (type K) thermocouple of 32-36 swg (0.27-0.19mm) should be placed in the center of the sample or in the middle of the side surface when using the Chen crossing point method, which will be further talked about in section 2.3.2. The thermocouples should be connected to a multi-channel data acquisition system which is connected to a computer to read and store the data from the tests.

The sample is to be prepared at room temperature which should be a cool dry environment. To be sure a consistent packing density is common throughout the prepared samples a light tamping or tapping should be applied during sample preparation. When layering is needed to prepare a sample it would also be recommended that the number of layers be counted. This will also aid in the packing density being consistent.

After preparation is complete the sample is suspended in a preheated oven and the temperature is recorded continuously throughout the duration of the test. At the beginning of the test the sample will start to heat from the reaction inside and the heat of the oven. The sample will then self heat to the point of ignition or self heat to a temperature (T_m) above the oven temperature (T_∞) and then come back down to a stable temperature slightly above the oven temperature as explained about in section 2.1.2 and shown in Figure 2.9.

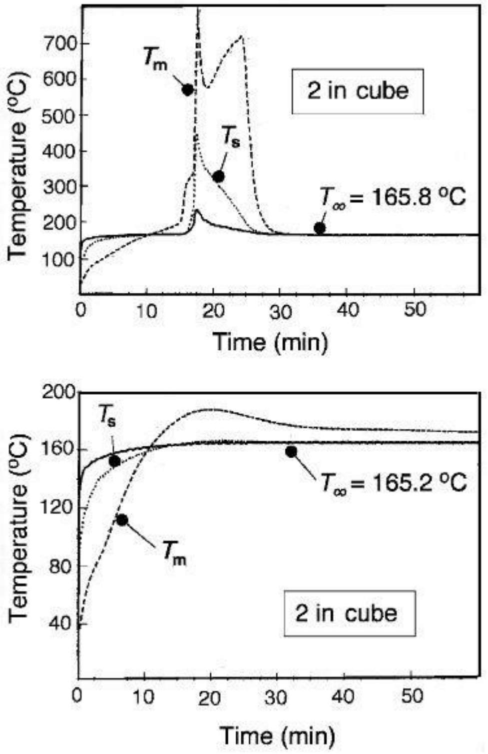


Figure 2.9: Top Graph Showing Super-Critical Case and Bottom Graph Showing Sub-Critical Case, from Quintiere (9)

After the sample has extinguished then a new sample is prepared using new material and a different oven temperature is tested. This process is done until the desired amount of data is achieved.

When using the F-K method for analysis, samples are tested at different oven temperatures until sub-critical and super-critical oven temperatures are both achieved. The temperature difference between oven temperatures may initially be substantial but is narrowed by testing temperatures slightly below and above the super-critical and sub-critical temperatures. Eventually, this process will zone in on a critical oven temperature at which the sample ignites. With the corresponding critical oven temperature, $T_{o,}$ the equation for δ can now be equated to δ_c for the cube in logarithmic form seen in Eq. 9.

$$\ln\left[\left(\frac{T_A}{r}\right)^2\right] = M - P/T_A \qquad \text{(Eq. 9)}$$

where

$$P = \frac{E}{R} \qquad \text{(Eq. 10)}$$

$$M = \ln\left(\frac{EQ\rho A}{R\lambda}\right) \qquad \text{(Eq. 11)}$$

The units for e^M are K^2/mm^2 and P has units of K. By plotting the $\ln\left[\left(\frac{T_A}{r}\right)^2\right]$ against ($1/T_A$) for a set of data, the slope, P, and the intercept, M, can be found. These will only yield good results as long as the chemical rate follows a zeroth order Arrhenius behavior. From this small scale data it is possible to extrapolate to larger scale conditions. This is the best way to quantitatively evaluate whether ignition is possible, i.e. $\delta \geq \delta_c$. However, factors can affect the accuracy of extrapolation. These include the effects of melting, moisture, and maintaining the same material. Never the less, material values for P and M can be compiled for tested materials to provide a framework for assessing their potential for spontaneous ignition.

When using the crossing point method for analysis the oven basket method is utilized in a different way. Instead of finding critical temperatures for each basket size, as required by the F-K method, the different basket sizes are tested over a range of oven temperatures. For example, for this research the 5cm (50% concentration) basket was tested at six different oven temperatures which was enough data to correctly apply the crossing point method. A time vs. temperature curve, as seen in the top half of Figure 2.9, resulted from each test this is necessary to find the crossing points needed to for the crossing point method, talked about further in section 2.3.

2.3 Crossing Point Method

Jones (10) and Chen (11) propose an alternative to the Frank-Kamenetskii method. It starts out with the same conduction equation that the Frank-Kamenetskii theory started out with seen in Eq. 1. From there the data produced by the oven basket method is examined. To analyze the data the center temperature of the sample is looked at and the point at which it crosses the oven temperature (Jones) or the surface temperature (Chen) is located, shown in Figure 2.10.

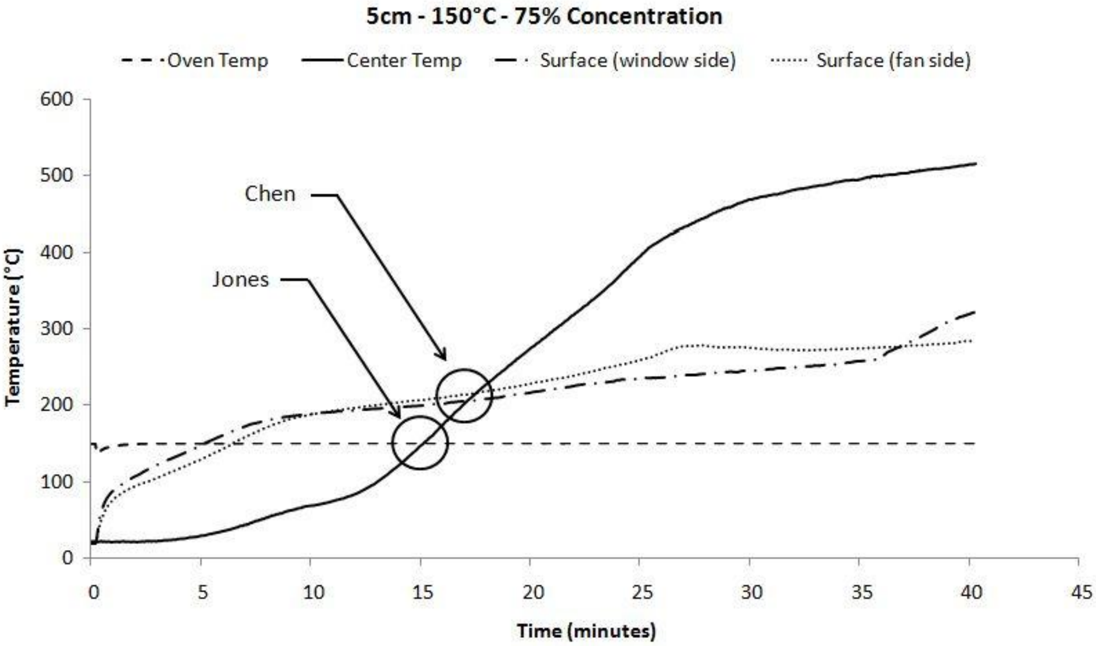


Figure 2.10: Location of Both Chen and Jones Crossing Points

The point at which the temperatures cross is deemed the crossing point. At this point, for the Jones method, the oven temperature is the same as the center temperature of the sample and for the Chen method, the center temperature is the same as the surface temperature of the sample. This means in both cases the temperature gradient (∇T) is zero and the conduction term in Eq. 1 goes away leaving Eq. 12.

$$\rho c \frac{dT}{dt} = Q\rho A e^{-E/RT} \qquad \text{(Eq. 12)}$$

With the crossing point located the slope of the center temperature, in both methods, is collected which can easily be done using Excel, Matlab or by hand. Figure 2.11 shows an example of a crossing point slope being found using Excel.

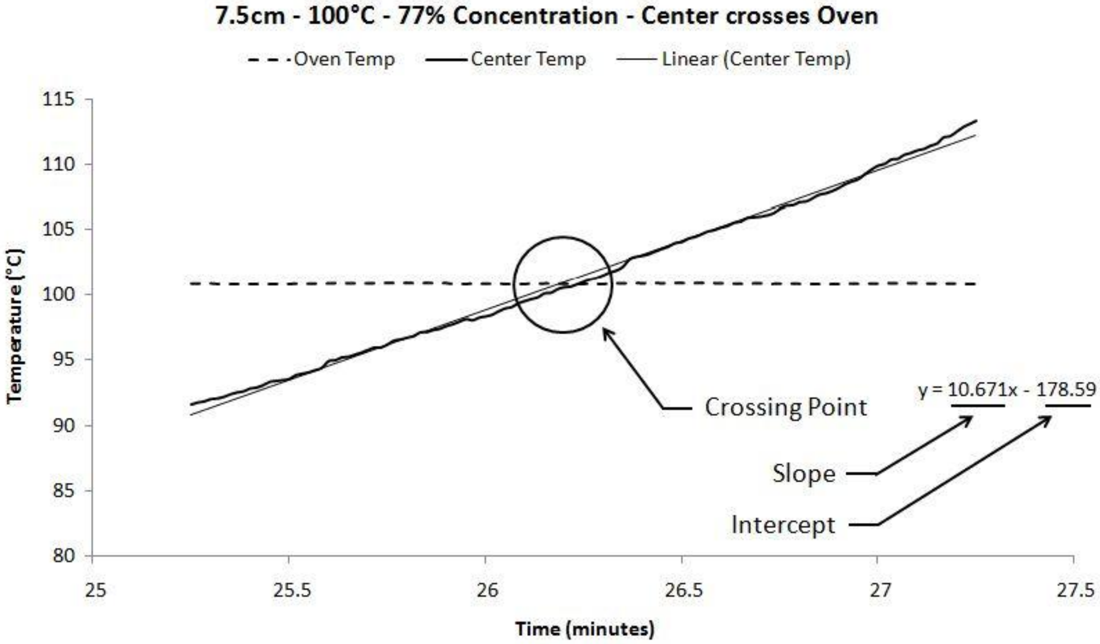


Figure 2.11: Finding the Slope of a Jones Method Crossing Point Using Excel

An array of crossing points is obtained after going through the rest of the whole set of data produced by the oven basket method. The crossing point slopes are then plotted with the inverse of the oven temperatures, $1/T_o$, plotted on the x-axis and the natural log of the crossing point slopes, $\ln(dT_c/dt)$, plotted on the y-axis as seen in Figure

2.12. Sometimes 1000/T_o will be used on the x-axis when working with larger oven temperatures.

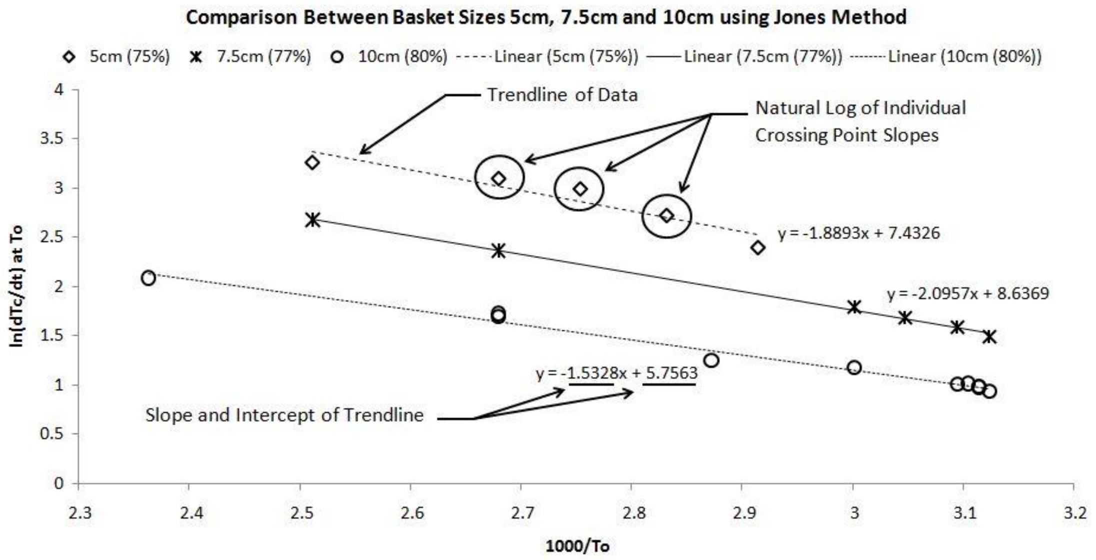


Figure 2.12: Plot of ln(dT_c/dt) vs. 1000/T_o with Labels

Once the data is plotted a trend line is fitted to each set of data also shown in Figure 2.12. Again, the slope of the trend line is found but the intercept is also needed this time. The trend line that is fitted to each set of data is represented by the relationship seen in Eq. 13.

$$\ln\left(\frac{dT_O}{dt}\right) = \ln\left(\frac{QA}{c}\right) - \frac{E}{R}\frac{1}{T_O} \tag{Eq. 13}$$

where the slope of the line is represented by –E/R and ln(QA/c) is the y-axis intercept. The slope and intercept found by using Excel or other methods is used in the determining of E and QA/c. To find E, the slope from the trend line (-E/R) is multiplied by the negative universal gas constant, -R. To find QA/c, the intercept from the trend line ln(QA/c) is taken to an exponential. From here c can be substituted for in Eq. 13 using Eq. 14.

$$c = \lambda / \rho k \qquad \text{(Eq. 14)}$$

which gives Eq. 15

$$\ln\left(\frac{dT_0}{dt}\right) = \ln\left(\frac{QA\rho k}{\lambda}\right) - \frac{E}{R}\frac{1}{T_0} \qquad \text{(Eq. 15)}$$

Finally, P and M can be found using Eq. 10 and Eq. 11 in conjunction with the slope (-E/R) and the intercept in Eq. 15 ($\ln(QA\rho k/\lambda)$).

3. Instruments, Testing Materials, Safety Precautions and Procedure

3.1 Instruments

3.1.1 Oven and Oven Modifications

The oven used for testing was a Memmert UFE500 115V Forced Air Controlled Convection Oven seen in Figure 3.1.

Figure 3.1: The Memmert UFE500 Forced Controlled Convection Oven

The dimensions of the inside of the oven were 56cm wide by 48cm tall by 40cm deep with an approximately 15cm diameter fan on the back wall, shown in Figure 3.2.

Figure 3.2: Inside of Oven Showing Thermocouple Placement

The fan positioned on the back wall of the oven blew out across the back wall, not straight forward. There was a display and dial on the front that controlled the oven settings including the temperature, fan speed, maximum temperature, time, etc. shown in Figure 3.3.

Figure 3.3: Front Display of Oven with On/Off Switch and Adjustment Dial

There was also a window on the front of the oven door which helped in viewing the sample during a test. The temperature of the oven was accurate to ±.5°C and during all of the tests the fan speed was set to the highest setting to assure forced convection.

Four ungrounded 1/16” diameter stainless steel sheath type K thermocouples were installed in each of the four corners of the oven. Each thermocouple was positioned 4” from the left or right nearest wall and 4” from either the top or the bottom walls of the oven. They extended into the oven 8” from the back wall which left the tip of the thermocouples at the center of the oven depth shown in Figure 3.2. These thermocouples were used to make sure the temperature was the same throughout the oven at all times and that the temperature did not differ between the reading on the front display of the oven and the actual temperature inside the oven.

There was also a hanging rod installed above the fan which gave each sample a place to hang during its test, shown in Figure 3.4.

Figure 3.4: Hanging Rod Installed Above Fan in Oven

The hanging rod had a notch near the end of it which was used to make sure each sample hung in the same location each time, which was approximately the middle of the oven. This eliminated the need for a shelf for the sample to sit on during its tests which would have possibly introduced conduction to the sample from the shelf and tainted the results of the test.

3.1.2 Materials

The baskets were made from 60 mesh .25mm opening stainless steel. That posed to be a very good material for holding the contents of the sample, even as the cotton turned to ash, throughout the whole duration of the test. There were three different sized baskets used for testing; 5cm, 7.5cm and 10cm. These baskets all had tiny holes near the top where a small metal hanging wire could be inserted and secured so the baskets could hang safely inside the oven.

The linseed oil that was used for testing was Klean-Strip Boiled Linseed Oil seen in Figure 3.5.

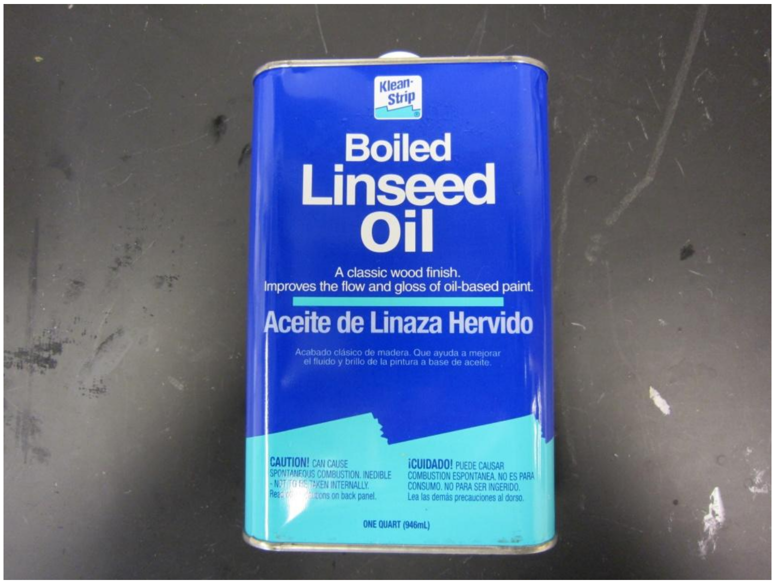


Figure 3.5: Klean-Strip Boiled Linseed Oil Used for Testing

One quart resealable containers were used to keep the linseed oil from oxidizing and losing its potency.

The cotton used for the experiments was 100% cotton batting, shown in Figure 3.6.

Figure 3.6: 100% Cotton Batting Roll

The 100 yard roll of cotton was 46” wide and made it easy to cut into strips for testing.

3.1.3 Data Acquisition

The three thermocouples used during almost all of the tests were all 36 AWG (.127mm dia) Type K glass insulated thermocouples. These thermocouples were positioned in the same specific spots for each test which will be further discussed later in section 3.3.

All of the thermocouples, from the oven and the sample, were connected to a Fluke 2645A NetDAQ system which was also connected to a computer through an Ethernet cable, seen in Figure 3.7.

Figure 3.7: Fluke 2645A NetDAQ System Connected to the Computer

This system collected and stored the data for each test throughout the testing process. Measurements for all tests were taken at one second intervals.

3.2 Safety Precautions

Throughout the testing process, scenarios arose where it was concluded that a special action should be taken to control the scenario before it became hazardous to the lab area. One main hazardous scenario was discovered that could be very dangerous and even life threatening if it arose during the actual running of the tests. During testing, the sample could possibly give off enough vapors, at some point between smoldering and flaming, to potentially cause an explosion inside the oven. With this in mind a safety system was installed to control the environment inside the oven, shown in Figure 3.8.

Figure 3.8: Safety System

The safety system was controlled by the center temperature of the sample and the parts it controlled included an outlet where the oven would be plugged into, a solenoid which would control the flow of nitrogen to the oven and a switch. When the center temperature reached its specified temperature the switch would operate and the two safety measures would execute. First, the safety system would shut off power to the oven thus removing heat from the interior of the oven which slowed down the reaction and created a less likely environment for auto ignition. The oven would stay off until the green button on the top of the safety system was pressed. Secondly, the solenoid opened thus purging the oven with cool nitrogen to try to slow down the reaction by taking the place of oxygen and cool down the interior of the oven. The line for the nitrogen was run through an exhaust hole in the back of the oven seen in Figure 3.9.

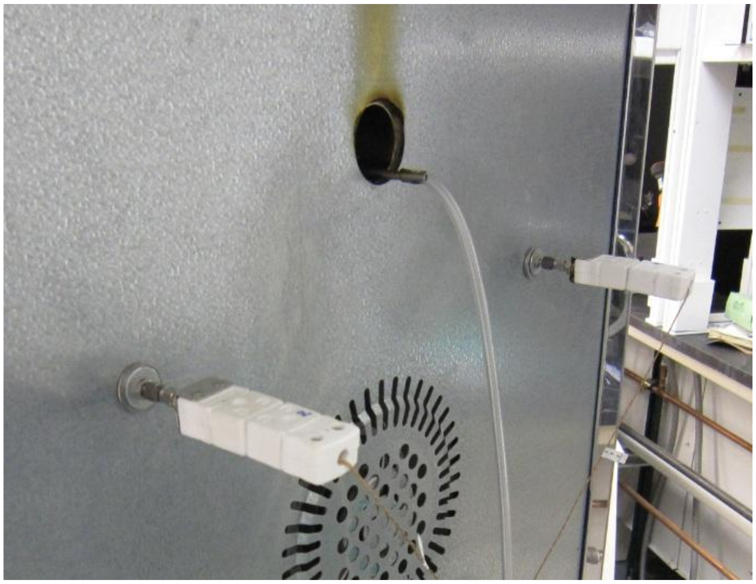


Figure 3.9: Nitrogen Line Running Through Exhaust Port Hole

This safety system's operation was very important because an explosion inside the lab could happen.

The second main safety precaution involved the containment of all leftover linseed oil saturated materials. A metal garbage can with a metal lid was filled half way full of water and acted as a safe containment vessel to discard any materials containing linseed oil. While preparing samples there were many rags, towels, gloves etc. used to help contain and clean the linseed oil from the lab area. If not properly disposed of, the trash could very easily be saturated or ignited by the linseed oil soaked rags, towels, gloves etc. This posed another very large hazard that needed to be dealt with before any testing started.

The last main safety precaution was the installation of a portable hood over the exhaust port of the oven. The portable exhaust hood, seen in Figure 3.10, was a must to export the smoke coming out of the oven so the lab area did not fill with the smoke.

Figure 3.10: Portable Hood Positioned Over the Open Oven Door

It also had to be portable so it could be moved over the top of the oven door when the door needed to be opened.

3.3 Procedure

The oven was turned on first so it could start its preheating process and reach the specified temperature before the sample was prepared. The preheating process was by far the longest out of the whole procedure which is why it was sometimes done well in advance to make sure it was ready. With the oven on and preheating, the strips were then measured and cut out of the roll of the 100% cotton fabric. Squares, of the specified size to fit the basket being tested were then measured, marked and cut out of the cotton strips, shown in Figure 3.11 and Figure 3.12.

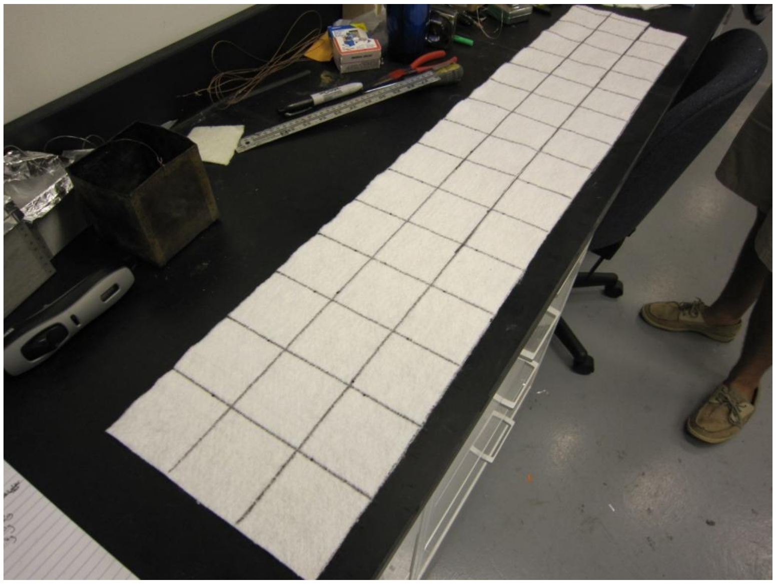


Figure 3.11: Squares Measured and Marked on a Strip of Cotton

Figure 3.12: Cut Out Squares Stored in Bin

Linseed oil was then applied to the specified amount of squares by one out of two different methods.

The first method of linseed oil application, which was used for the 5cm (75% concentration), 7.5cm (77% concentration) and 10cm (80% concentration) tests, was a saturate and wring method. This method involved of a pan of linseed oil and the squares being laid flat in the pan which totally saturated the squares, seen in Figure 3.13. The squares were then wrung out one by one and weighed afterwards to confirm the correct concentration.

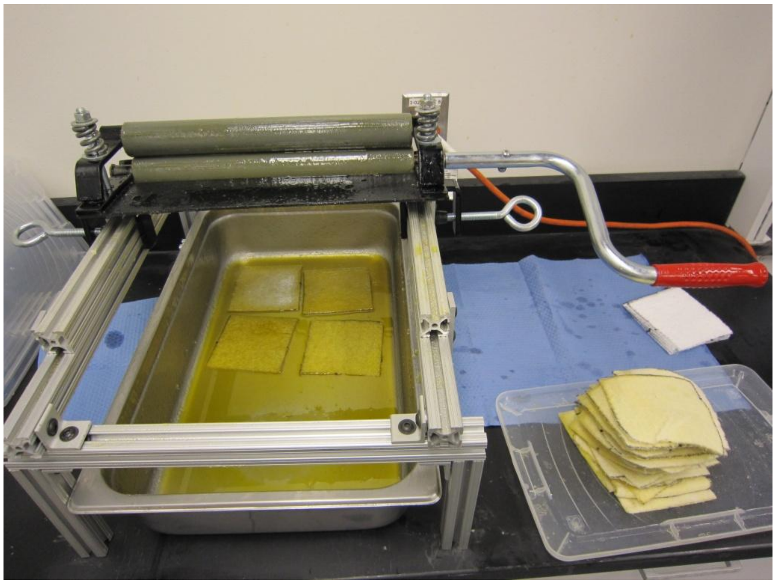


Figure 3.13: Saturating the Squares in a Pan of Linseed Oil

The second method of application used an oil sprayer that would evenly spray the linseed oil onto the squares one by one, shown in Figure 3.14.

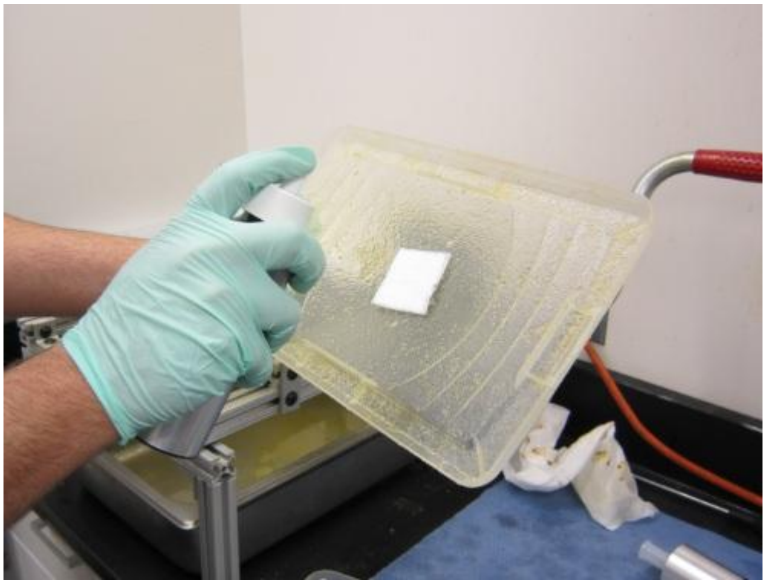


Figure 3.14: Spraying Linseed Oil on a Cotton Square

This method was used for the 5cm (50% concentration) and 5cm (33.3% concentration) tests. The sprayer was a reusable oil sprayer that used a hand pump to create pressure. Using the sprayer for application made it very easy to test different linseed oil to cotton concentrations. Again, each square was weighed to assure the correct linseed oil to cotton concentration was met shown in Figure 3.15.

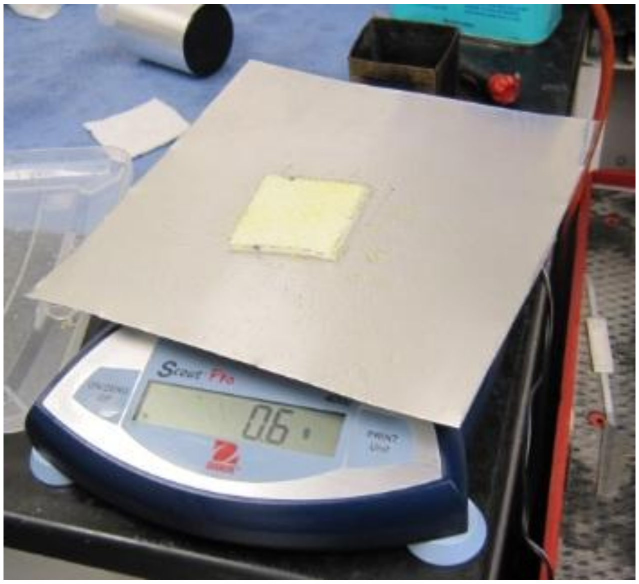


Figure 3.15: Individual Square Being Weighed After Linseed Oil Application

This application and weighing process was done for the whole stack of squares involved in the test then the whole linseed oil soaked cotton stack was weighed one last time to double check the concentration.

Once the application process was completed, the squares were placed into the metal basket one by one until it was half full. They were placed in the basket while the linseed oil was still wet. When half full, the three 36 AWG Type K thermocouples were inserted into the basket, one in the center of the sample and the other two on opposing surfaces as seen in Figure 3.16.

Figure 3.16: Thermocouple Placement In the Center and On the Surface of the Sample

With the thermocouples in place the rest of the squares were placed on top till the basket was full and the sample took the shape of a cube. The hanging wire was then securely attached to the top of the basket, shown in Figure 3.17, and the sample was ready to be placed in the oven.

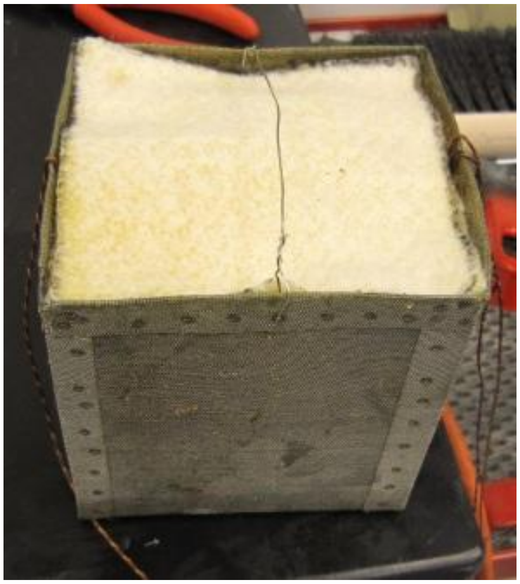


Figure 3.17: Prepared Sample Ready to be Placed in Oven

Immediately after the NetDAQ system started collecting data the sample was placed in the oven by opening and shutting the door very quickly so minimal heat would escape. If a lot of heat were to escape then the results of the test may be skewed. The sample was left in the oven either until the desired data had been received or, for longer tests, over night.

4. Results and Discussion

4.1 Overview

For this research, a method to easily test spontaneous material was explored. An oven was instrumented so the oven basket method could be performed on linseed oil soaked rags. The hazardous combination of linseed oil and cotton was picked due to its popularity for spontaneously igniting and the demand for more information on its tendencies. To safely run these tests a safety system was built and installed to control the internal conditions of the oven.

Originally, the Frank-Kamenetskii method of analysis was to be coupled with the oven basket method to produce critical temperatures and eventually the parameters P and M talked about in section 2.2. During the trial and error process of pinpointing a critical temperature for the 5cm (75%), 7.5cm (77%) and 10cm (80%) baskets a critical temperature was not found because every trial reaching a super-critical state or “igniting”, to be further discussed in section 4.2. The lack of ability to produce a sub-critical test was due to the high concentration of linseed oil contained in the samples. This left the crossing point method (Jones) to be used to analyze the data from the oven basket method instead.

To use this method the oven basket method had to be tested in a slightly different manor. Instead of looking for critical temperatures, as called for by the F-K method, each basket was tested at a range of oven temperatures. From this data, the point at which the center temperature (T_c) and the oven temperature (T_o) crossed, which is referred to as the crossing point, was found for each test, which will be further discussed in section 4.4. From these crossing points the crossing point method of analysis was applied to find the activation energy (E) and QA which eventually would lead to the calculation of P and M, originally sought by the F-K method. The crossing point (Jones) method resulted in some very nice data and also compared very well to data from other sources which will be shown further in section 4.4. This method coupled with the oven basket method provided for a very quick and easy approach in testing spontaneous material and is recommended for testing other spontaneous materials in the future.

4.2 Raw Data

When investigating the test results of the different basket sizes and concentrations, with no suppression system activated, three different states were reached, ignition, smoldering and flaming, seen in Figure 4.1.

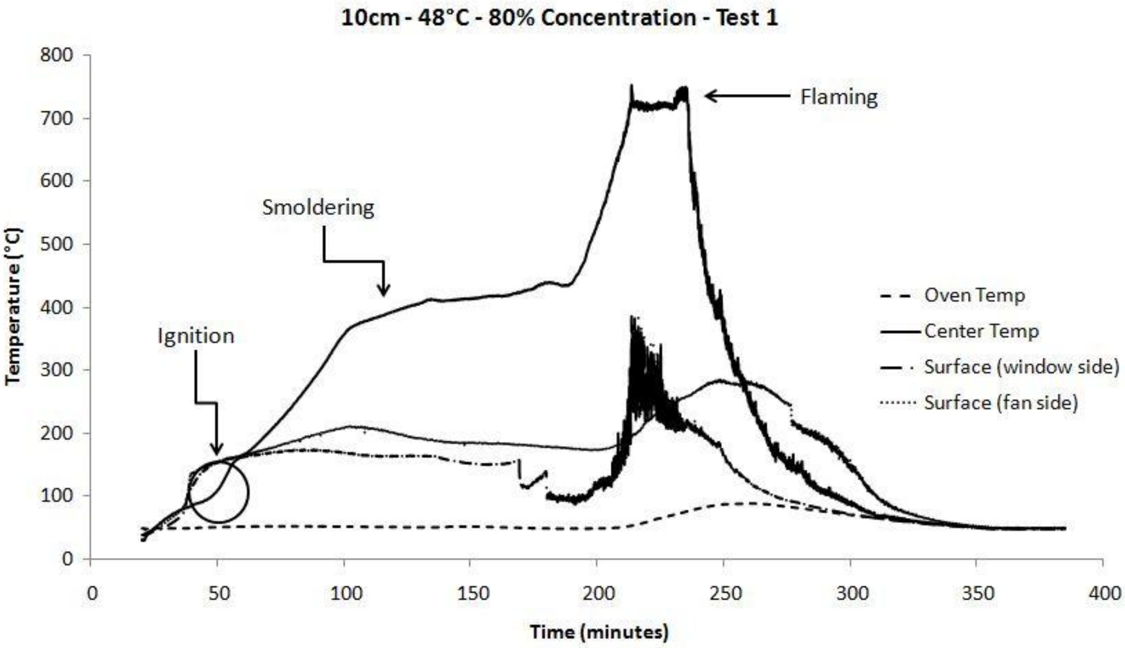


Figure 4.1: The Three States of a Typical Test

The first state reached was the ignition state which clarified whether this sample was a run-away case or not. In Figure 4.1, ignition happened at approximately 100°C shown by the sharp increase in the center temperature. This is when the reaction happening inside the sample has run-away and keeps building on itself thus making the reaction happen faster and faster. The heat generated from the reaction causes the center temperature of the sample to increase very quickly till a new state is reached. The 100°C temperature at which ignition happened was not the same with every test due to changing sample sizes, linseed oil concentrations and oven temperatures.

The smoldering state was the second state reached which in Figure 4.1 started at approximately 100 minutes and lasted approximately 90 minutes while ranging in temperature from 375°C to 425°C. During this state, the reaction in the center part of the sample reached a high enough temperature where the cotton started to char as seen in Figure 4.2.

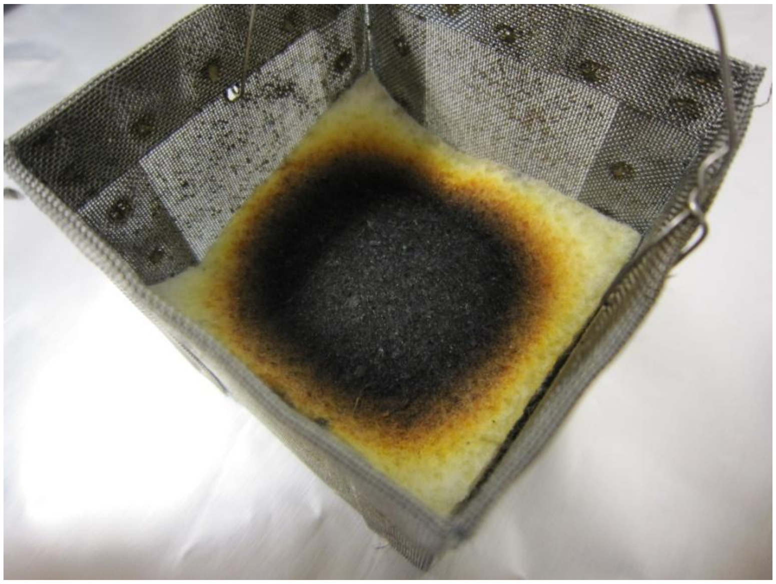


Figure 4.2: Charred Center of a Sample

In some cases, a sample would have a very prolonged charring state, as seen in Figure 4.1 lasting approximately 90 minutes at about 400°C, which created a void in the center of the sample as seen in Figure 4.3, Figure 4.4 and Figure 4.5.

Figure 4.3: A Void Created Around the Center Thermocouple

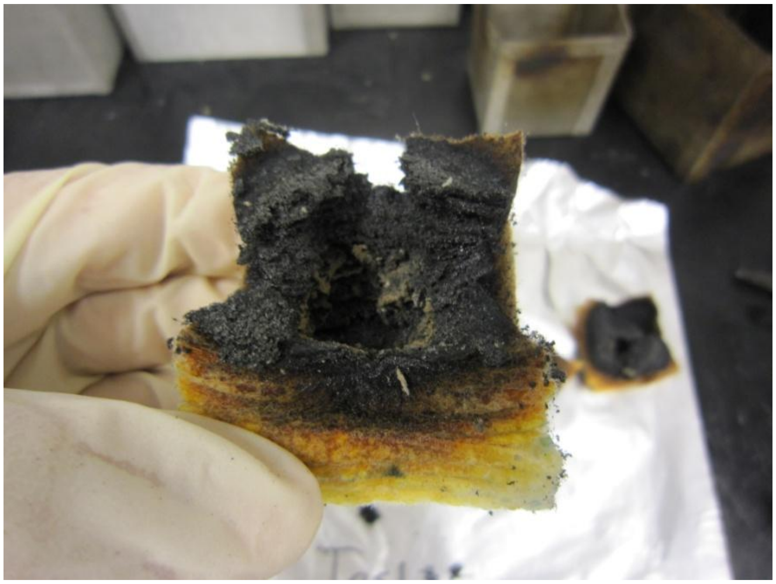


Figure 4.4: Showing a Void in the Center of a Sample

Figure 4.5: A Very large Void Created Around the Center Thermocouple

The voids were circular in shape and ranged in size from a couple centimeters to approximately 10cm in diameter. Also during the smoldering stage, the oven completely filled with smoke making it so the sample was not visible through the window on the front of the oven. The smoke mainly had a white/grey color and also had a very strong odor.

The final state that occurred was the flaming state. This was shown as huge spike in the center temperature data happening right after the smoldering stage. In Figure 4.1, the flaming state lasted approximately 30 minutes at around 725°C. Flaming occurs when there is enough oxygen present inside the sample and the heat produced by the run-away reaction and smoldering is great enough to ignite the flammable vapors given off by the cotton. Although flaming was reached in Figure 4.1 it was not reached in a majority of the samples mainly because the smaller samples ran out of cotton to burn during the test and the safety system was activated most of the time, which shut down the system before the flaming state could be reached.

At approximately 240 minutes the flaming state started to extinguish and the center temperature of the almost totally charred sample began to decrease. With not much substance left of the sample the temperature of the sample decreased all the way to oven temperature where it stayed for the remainder of the test.

4.3 Critical Ignition Temperature Testing

Originally the Frank-Kamenetskii method was to be used to analyze the data produced by the oven basket method. This meant the oven basket method would be used to find a critical temperature for each of the different basket sizes as talked about in section 2.2. When the process of pinpointing critical temperatures began the 5cm (75%), 7.5cm (77%) and 10cm (80%) were all tested first. At the beginning of testing, higher oven temperatures were used and every sample tested for all three baskets went to ignition. To try to get a sub-critical test a decrease in the oven temperatures was carried out until an ambient oven temperature (~20°C) was finally tested in which the samples still went to ignition, shown in Figure 4.6, Figure 4.7 and Figure 4.8.

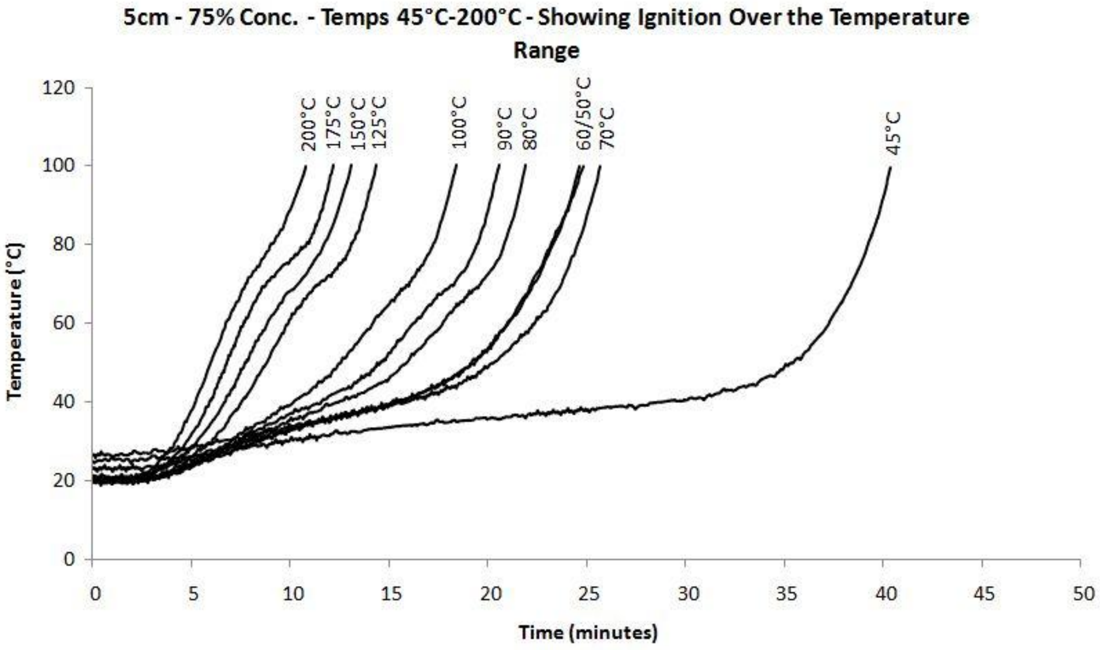


Figure 4.6: Showing Ignition for the 5cm (75%) Basket over the Temperature Range of 45°C - 200°C

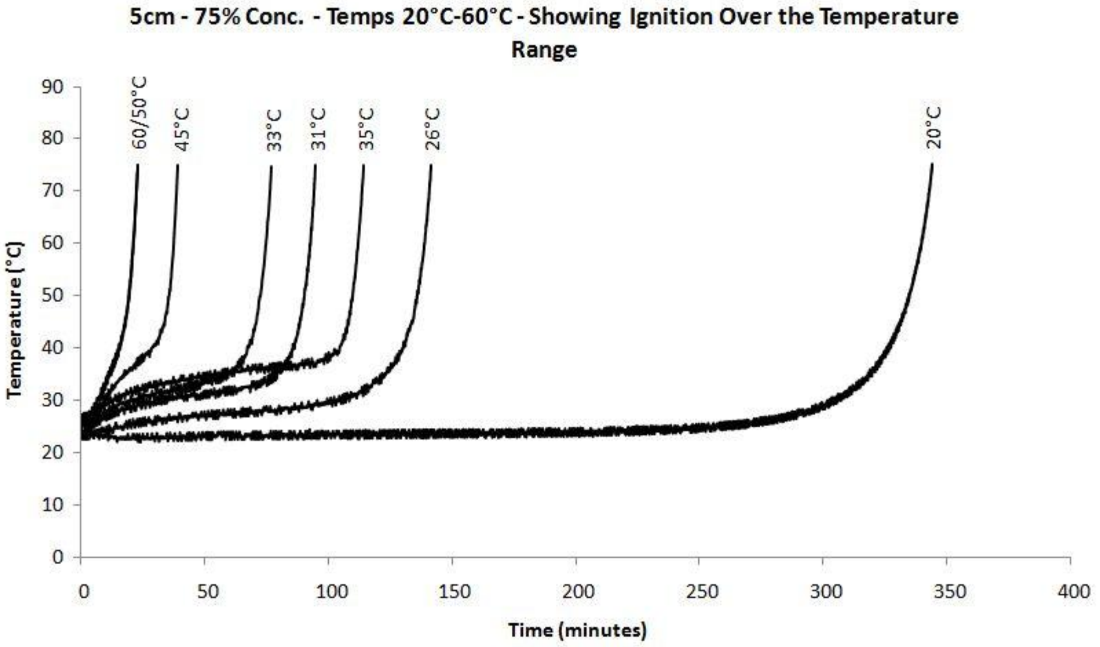


Figure 4.7: Showing Ignition for the 5cm (75%) Basket over the Temperature Range of 20°C - 60°C

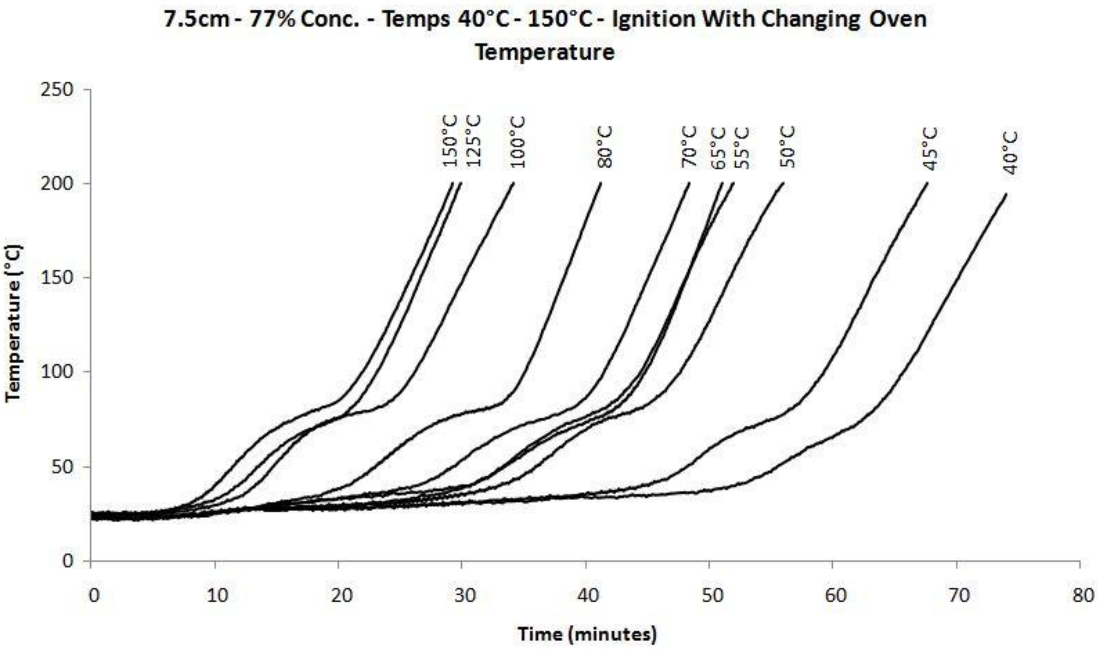


Figure 4.8: Showing Ignition for the 7.5cm (77%) Basket over the Temperature Range of 40°C - 150°C

Each of the figures shows the area where ignition occurred during their test run. With a sub-critical test unable to be achieved, even at ambient temperatures, the F-K method could not be used on these basket sizes at their linseed oil concentrations. The only way to get a sub-critical test with those basket sizes and concentrations would be to run a test at lower than ambient oven temperatures. This would require a refrigerated room or some apparatus that would create the same oven environment at lower than ambient temperatures, which was not available. Since the conditions to get results for the Frank-Kamenetskii method were not available other options had to be considered, for example, changing the concentration of linseed oil.

Later on in the testing stages the linseed oil concentration was changed to 33.3% and 50% of oil by weight for the 5cm basket size. A critical temperature for the 5cm (33.3%) basket size was pursued but was not found as seen in Figure 4.9.

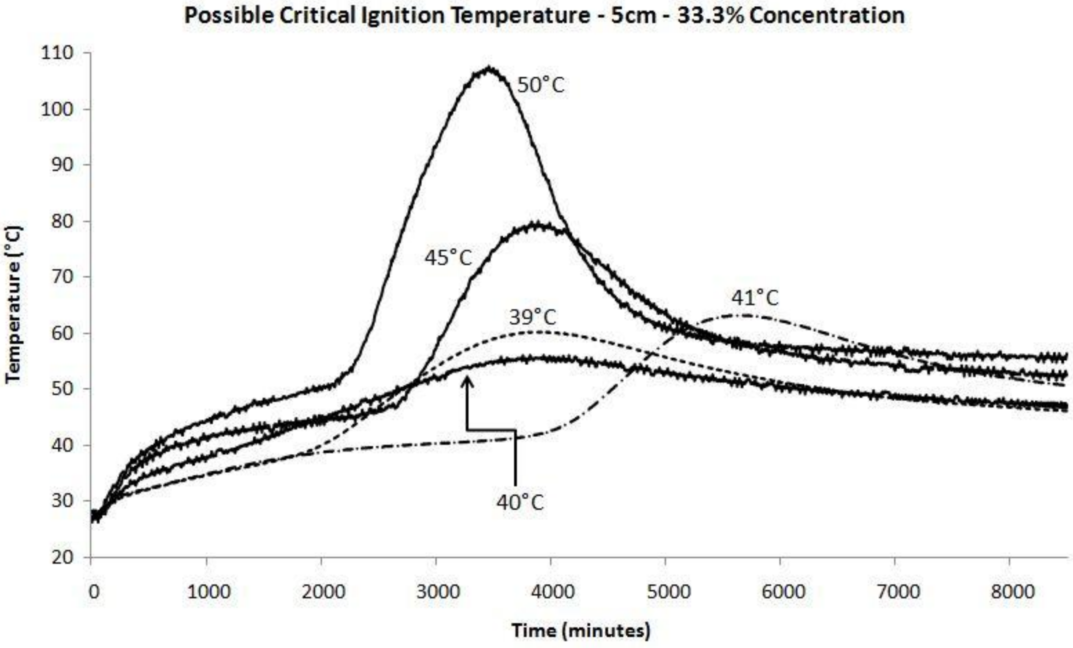


Figure 4.9: Possible Critical Ignition Temperature for the 5cm (33.3%) Basket Size

Oven tests began around 50°C and were decreased untill a sub-critical test was found at an oven temperature of 40°C. Even though it is hard to see, Figure 4.9 shows the 40°C test not having a sharp increase in temperature confirming that it is a sub-critical test. With no ignition at 40°C and ignition at 41°C an oven temperature of 39°C was tested to see if another sub-critical test would result. When looking at the 39°C test in Figure 4.9 at around 2000 minutes the center temperature of the sample starts to increase rapidly, not common of a sub-critical test, thus resulting in ignition. With the sub-critical test at 40°C it is believed that the critical temperature for that sample is very close to being achieved but additional testing would be needed to exactly pinpoint the critical temperature of that basket size and concentration.

With the lack of critical temperatures for any of the basket sizes the Frank-Kamenetskii method of analysis was not able to be used. Because of this other means of analysis needed to be resorted to namely, the crossing point method.

4.4 Crossing Point Method (Jones)

4.4.1 Crossing Points

The crossing point method was used as a means of analysis because no critical temperatures were found for the Frank-Kamenetskii method. Originally, both the Chen and Jones method, as talked about in section 2.3, were going to be applied to all of the data but after examining the data given from the oven basket method it seemed as if the thermocouples placed on the surface of the sample were giving strange readings. Due to these outcomes only the Jones method was used for the final analysis.

To start out using the crossing point method the whole scheme of testing with the oven basket method had to be reassessed. As talked about at the end of section 2.2, when it was decided that the crossing point method was going to be used for analysis the oven basket method had to be used in a different way than it was originally used by the F-K method. Critical temperatures, as required by the F-K method, were not explored but instead the different basket sizes were exposed to a range of oven temperatures. For example, the 5cm (33.3%) basket size was exposed to six different oven temperatures ranging from 60°C to 150°C. The data that was collected from these tests was recordings of the center, surface and oven temperatures throughout the duration of the test. An example is shown in Figure 4.1.

After all the basket sizes were tested at their respective oven temperatures the raw data produced was examined using the Jones method. To help understand this method an example will be explained at each step of the process, the first example is shown in Figure 4.10.

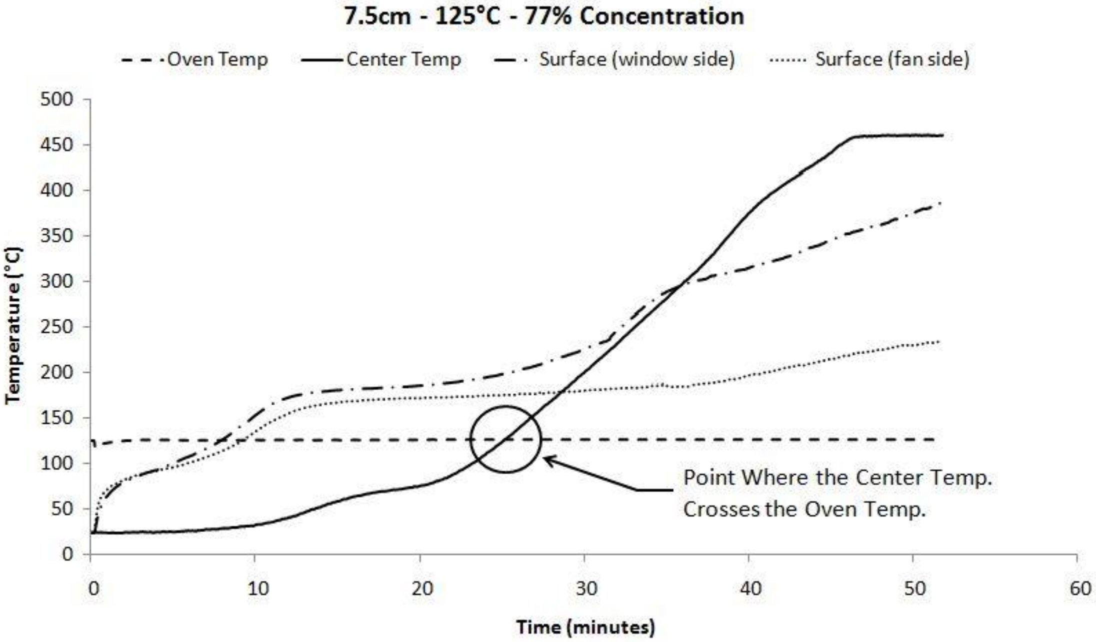


Figure 4.10: Point where the Center Temperature Crosses the Oven Temperature for the 7.5cm (77%) Basket Size

The center temperature of each data set was located and inspected until the point at which the center temperature crossed the oven temperature was found, seen in Figure 4.10. This was deemed the crossing point of this test. The next step was to find the slope of the center temperature at the crossing point location. First, a graph of the crossing point was made which zoomed in on the intersection. A trend line was then fitted to the center temperature data, seen in Figure 4.11.

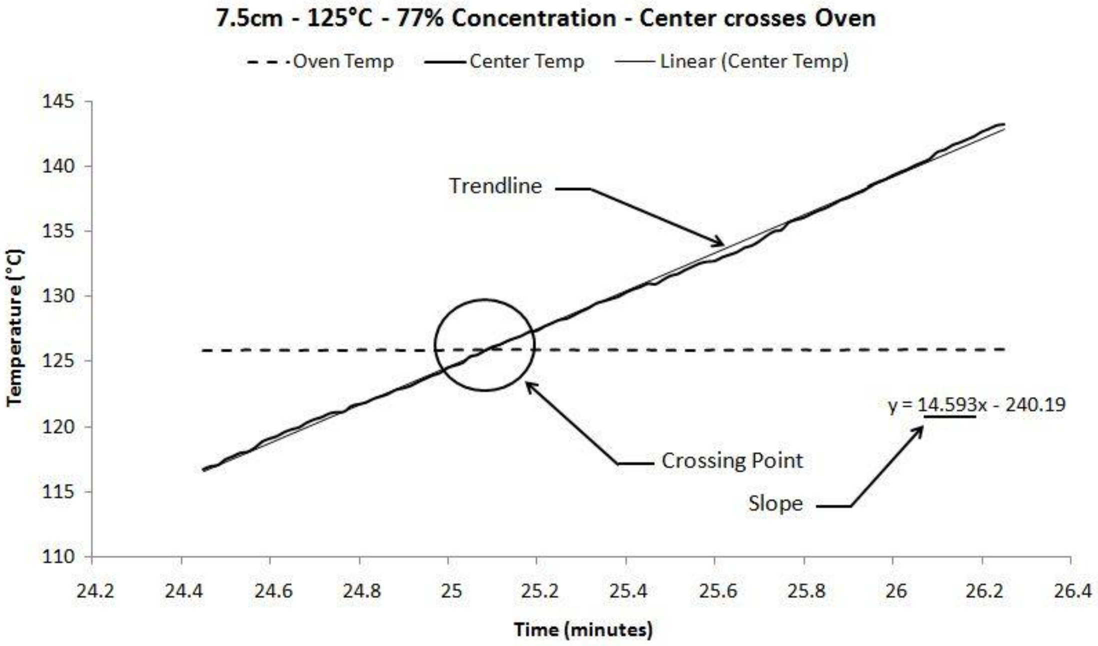


Figure 4.11: Showing the Trend line Fitted to the Center Temperature and Showing the Slope

With a trend line fit to the center temperature data the equation for the trend line was shown and the slope of the line was recorded. This was done for all basket sizes at every oven temperature tested.

With every slope recorded, a small calculation was made to the slopes and their respective oven temperature. The natural log was taken of the slope, ln(dT_c/dt), and the oven temperature was first converted to Kelven, by adding 273.15, and then the inverse was taken, $1/T_o$. The values for the 5cm (50%) are shown in Table 2.

5cm (50%)				
Oven Temps (°C)	Oven Temps (K)	$1000/T_o$ (K^{-1})	Crossing Point Slopes (K/s)	ln(dT_c/dt)
160	433.15	2.31	59.64	4.09
145	418.15	2.39	51.45	3.94
125	398.15	2.51	49.69	3.91
100	373.15	2.68	27.89	3.33
90	363.15	2.75	17.13	2.84
80	353.15	2.83	9.52	2.25

Table 2: Showing the Calculations Done to the Oven Temperature and the Crossing Point Slope

The inverse of the oven temperature was multiplied by. The next step involved the new numbers being graphed with 1000/T_o on the x-axis and ln(dT_c/dt) on the y-axis, seen in Figure 4.12.

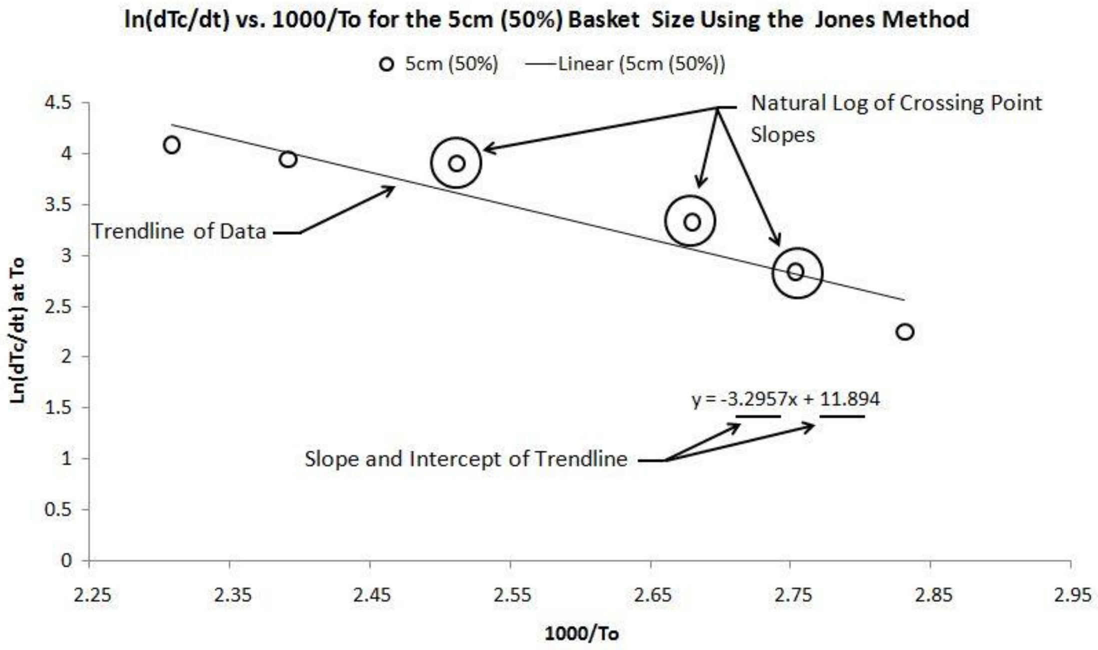


Figure 4.12: The Graph of ln(dT_c/dt) vs. 1000/T_o for 5cm (50%) Basket Size

Just as in Figure 4.11, a trend line was fit to this data but this time the slope and intercept were both recorded. This trend line is represented by Eq. 16, where the slope of the trend line is equal to –E/R and the intercept is equal to ln(QA/c).

$$\ln\left(\frac{dT_O}{dt}\right) = \ln\left(\frac{QA}{c}\right) - \frac{E}{R}\frac{1000}{T_O} \qquad \text{(Eq. 16)}$$

When the rest of the data was graphed a group of outliers showed up. In Figure 4.13, the data for the 5cm (75%), 7.5cm (77%) and 10cm (80%) baskets did not have the same straight line shape as seen in Figure 4.12.

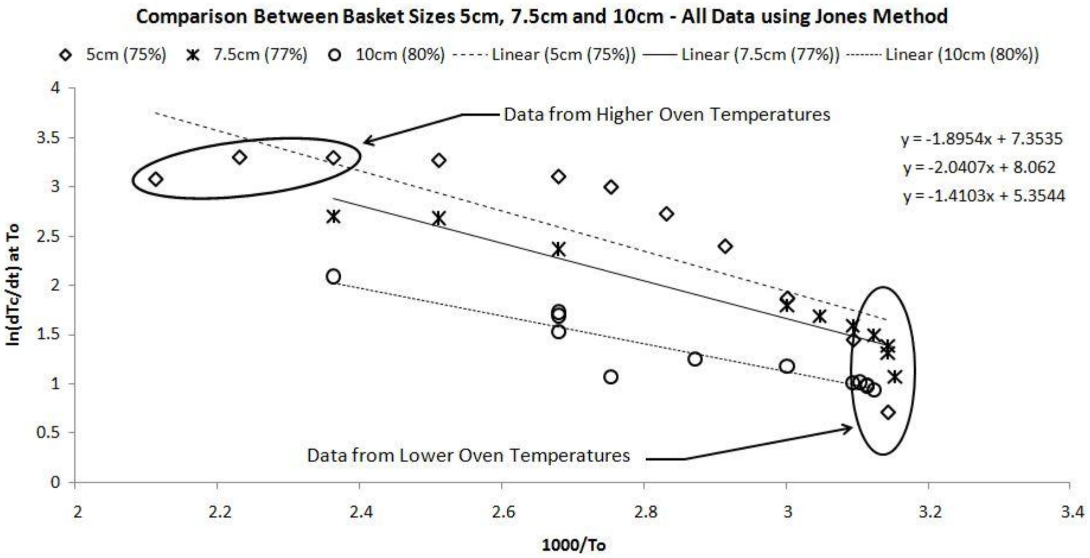


Figure 4.13: The Graph of ln(dT_c/dt) vs. 1000/T_o Comparing the 5cm (75%), 7.5cm (77%) and 10cm (80%) Basket Sizes

The tests using extreme oven temperatures, high and low, produced data that is not what is expected. The data using the higher oven temperatures seems to plateau from approximately 2.1 to 2.4. At the other end, when using lower oven temperatures the data seems to drop off or sharply decrease around 3.1. Another graph was made excluding the outlying data points seen in Figure 4.14.

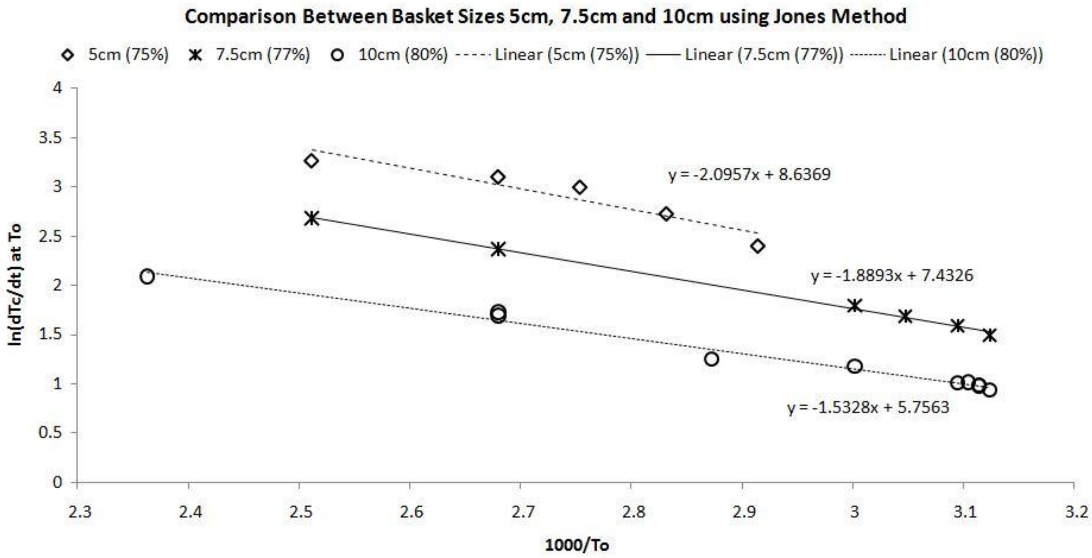


Figure 4.14: The Graph of ln(dT_c/dt) vs. 1000/T_o of the 5cm (75%), 7.5cm (77%) and 10cm (80%) Basket Sizes without the Outliers

With the outliers not included the data forms a much straighter line as expected. When the slopes were compared from Figure 4.13 to Figure 4.14 to see how much of a difference the outlying points made, there was not much difference found. In Figure 4.13, the slopes were -2.0407, -1.8954 and -1.4103 for the 5cm (75%), 7.5cm (77%) and 10cm (80%) baskets. In Figure 4.14, the slopes were -2.0957, -1.8893 and -1.5328 for the 5cm (75%), 7.5cm (77%) and 10cm (80%) baskets. With the 5cm and 7.5cm basket slopes only having an approximate .01 difference and the 10cm basket only having an approximate .1 difference there is no real reason to exclude the outlying data. Resulting from this, the slopes produced by using all of the trials, seen in Figure 4.13, for the 5cm (75%), 7.5cm (77%) and 10cm (80%) baskets were used in the rest of the calculations.

4.4.2 Activation Energy

After finding all of the slopes from the ln(dT_c/dt) vs 1000/T_o graphs another small calculation was done to produce their activation energies. From Eq. 16 the slope is equal to –E/R and to find the activation energy the slope was multiplied by -1000/R. Usually it would be multiplied by -1/R but 1000/T_o was used on the x-axis to make the numbers larger as stated above. The values can be seen in Table 3.

Size (cm)	Slope	Activation Energy, E (kJ/mol)
10 (80%)	-1.4103	11.73
7.5 (77%)	-1.8954	15.76
5 (75%)	-2.0407	16.97
5(50%)	-3.2957	27.40
5(33.3%)	-5.0965	42.37

Table 3: Calculation of Activation Energy using the Slope from Figure 4.13

These values were then plotted with concentration seen in Figure 4.15.

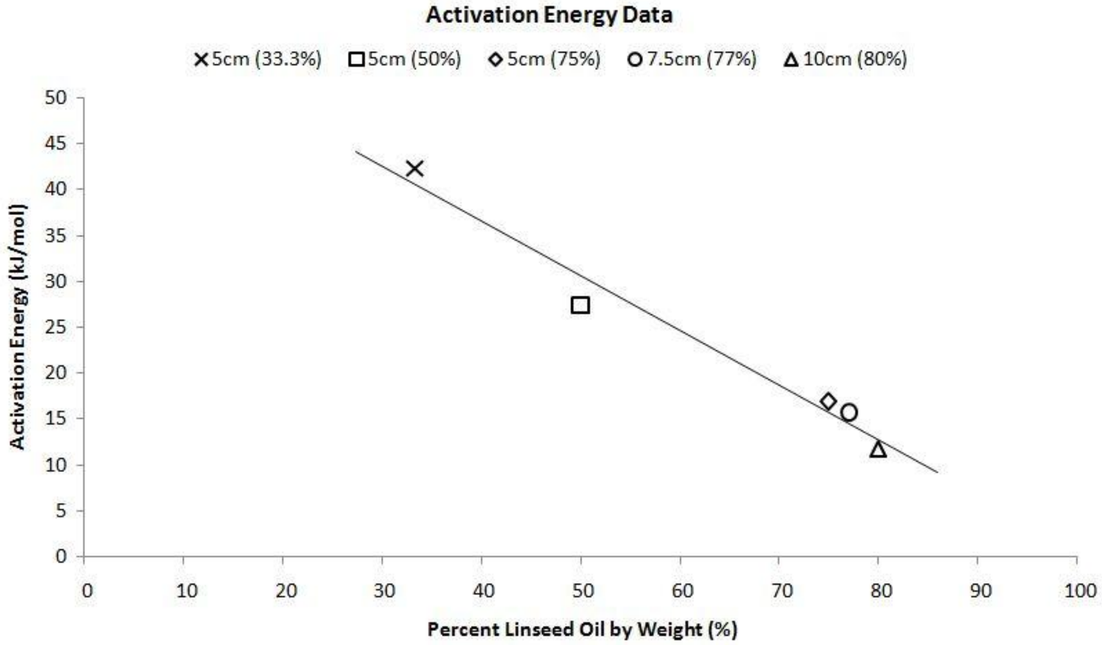


Figure 4.15: Activation Energy for All Different Sized Baskets and Concentrations

Figure 4.15 shows that as the concentration of linseed oil and the size of the sample both increase the activation energy decreases. With the higher concentration samples having lower activation energy this means the samples need less energy from the heat of the oven to activate the reaction inside the sample. By requiring less energy to activate the reaction it also requires less energy to produce a run-away scenario. The samples that have a lower concentration have a higher activation energy. This means the lower concentration samples require more energy from the heat of the oven to activate the reaction inside. A comparison between the results shown in Figure 4.15, the data from Gross and Robertson (2) and the data from Khattab et al (4) is shown in Figure 4.16.

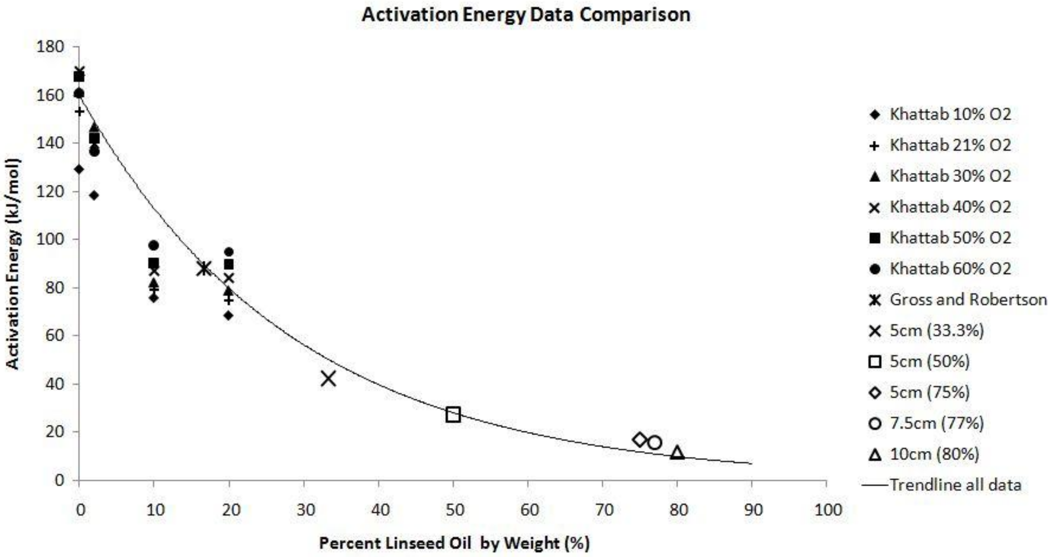


Figure 4.16: Comparing Activation Energy from Testing, Khattab et al (4) and Gross and Robertson (2)

The data from Khattab et al (4) begins to show how the concentration of linseed oil contained in the sample effects the resulting activation energy. It is seen in their data that as the concentration of the sample goes to zero the activation energy reaches a peak value between 130 and 170 kJ/mol. As they increase the linseed oil loading from 0% to 20% the activation energy starts to decrease and reaches a value between 65 and 95 kJ/mol. The data from Khattab et al shows a range of activation energies because during their testing they also varied the oxygen concentration, as stated in section 2.1.3. The data from Gross and Robertson (2) reinforces the numbers that Khattab et al found at their lower concentration range. Gross and Robertson found a 16.6% concentration to have an activation energy of 88 kJ/mol, as talked about in section 2.1.3. When the data from this research is graphed with the data from Khattab and Gross the decreasing trend started by Khattab and Gross is completed, shown in Figure 4.16. All of the data together shows very nicely that as the concentration of linseed oil is increased in a sample of cotton the energy needed to activate the reaction inside the sample becomes less and less. This means that as the concentration of linseed oil increases the less energy is needed for the sample to go to ignition also. This is exactly why concentrations of linseed oil that would be seen in real world applications, probably around 75% of linseed oil by weight of cotton, are so dangerous.

4.4.3 Calculation of P and M

To calculate the parameters P and M the slopes and intercepts collected from the $\ln(dT_c/dt)$ vs $1000/T_o$ graphs, for example Figure 4.12, were needed. P was calculated first. P was calculated using Eq. 10 which is exactly the slope from the $\ln(dT_c/dt)$ vs $1000/T_o$ graph but, since $1000/T_o$ was used the slope had to be multiplied by 1000 to be correct. The values for P can be seen in Table 4 and Figure 4.17. Next, M was calculated using Eq. 11 and the approximated thermal properties listed in Table 5. The values for M can also be seen in Table 4 and Figure 4.18.

Basket Size and Concentration	**P**	**M**
Gross and Robertson 16.6%	10584.56	49.6
5cm - 33.3%	5096.50	39.50
5cm - 50%	3295.70	34.46
5cm - 75%	2040.70	30.98
7.5cm - 77%	1895.40	30.15
10cm - 80%	1410.30	27.91

Table 4: The Values of P and M for All Basket Sizes and Gross and Robertson (2)

For units for P and M see section 2.2. The values for thermal conductivity (λ) were extrapolated by using .06 W/mk for uncontaminated cotton and .147 W/mk (12) for linseed oil. The extrapolation also took into account the ratio of linseed oil to cotton by weight in each sample shown in Eq. 17.

$$\lambda_{mix} = Y_{cotton}\lambda_{cotton} + Y_{linseed}\lambda_{linseed} \qquad \text{(Eq. 17)}$$

The values for density were calculated from the recorded weights of the samples during preparation as talked about in section 3.3. For example, the weight of a sample for the 5cm (33.3%) basket weighed approximately 12g. This gives 12g per 5cm^3 which was then converted to units of kg/m^3. Specific heat (c) values were also extrapolated using 1300 J/kg-K for uncontaminated cotton and 1796 J/kg-K for linseed oil (12) while also taking into account the ratio of linseed oil to cotton in each sample. The equation used for this is shown in Eq. 18.

$$c_{mix} = Y_{cotton}c_{cotton} + Y_{linseed}c_{linseed} \qquad \text{(Eq. 18)}$$

The thermal diffusivity was calculated using Eq. 19.

$$k = \lambda / \rho c \qquad \text{(Eq. 19)}$$

Basket Size and Concentration	**E (kJ/mol)**	**QA (J/s-kg)**	**λ (J/s-m-k)**	**ρ (kg/m3)**	**c (J/kg-K)**	**k (m2/s)**
10cm - 80%	11.73	3.59E+05	0.130	339	1696.80	2.25E-07
7.5cm - 77%	15.76	2.63E+06	0.127	317	1681.92	2.38E-07
5cm - 75%	16.97	5.30E+06	0.125	329	1672.00	2.28E-07
5cm - 50%	27.40	2.27E+08	0.104	128	1548.00	5.22E-07
5cm - 33.3%	42.37	2.60E+10	0.089	96	1465.24	6.33E-07
Gross and Robertson 16.6%	88	4.7E+13	0.046	309	1400.00	1.06E-07

Table 5: Spontaneous Ignition Properties Used in the Calculation of M and Compared to Gross and Robertson's Data (2)

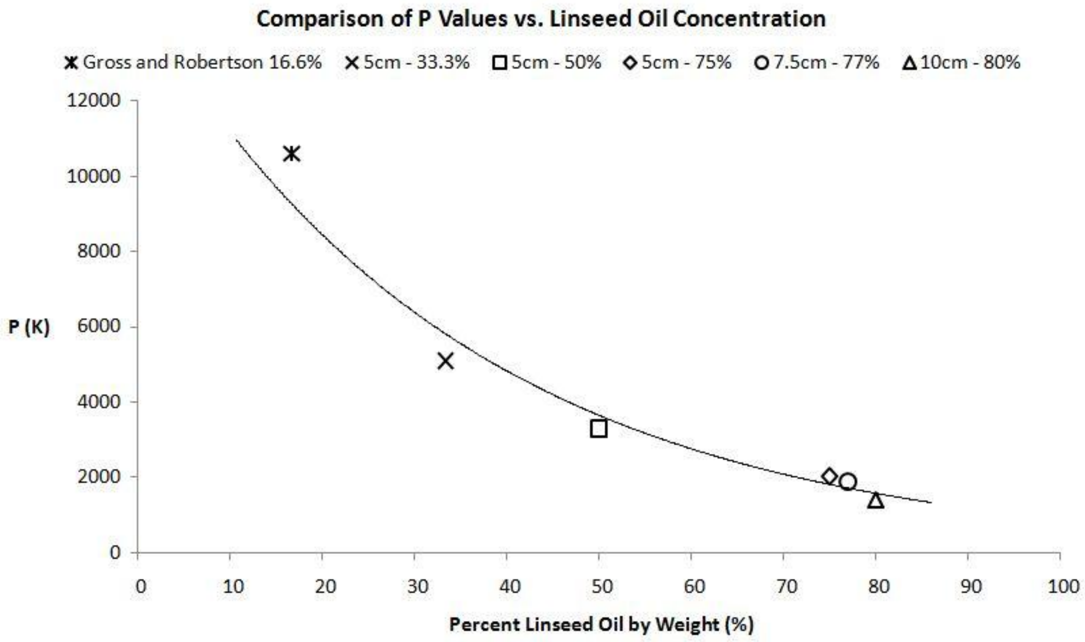


Figure 4.17: Graph of values of P vs. Concentration of Linseed Oil

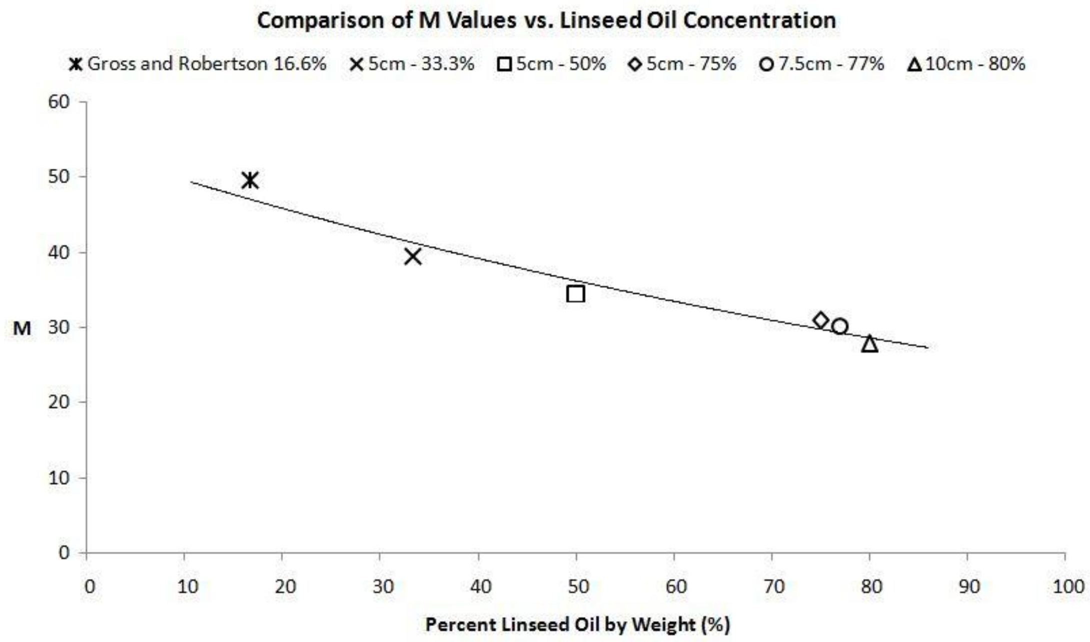


Figure 4.18: Graph of values of M vs. Concentration of Linseed Oil

With P equaling –E/R the P values, shown in Figure 4.17, should decrease with increasing concentration and basket size just as the activation energy did in Figure 4.16, which they do. This makes sense since the activation energy is the driving parameter in P with R being a constant. Since the activation energy is also included in the equation for M the values for M should also decrease with increasing sample concentration and basket size, which they do. The values for M don’t exactly match the same decreasing trend as the P values because of the other factors taken into account when calculating M, thermal conductivity, density, specific heat, QA. Graphs of these thermal properties with linseed oil concentration are shown in Figure 4.19, Figure 4.20, Figure 4.21, Figure 4.22 and Figure 4.23.

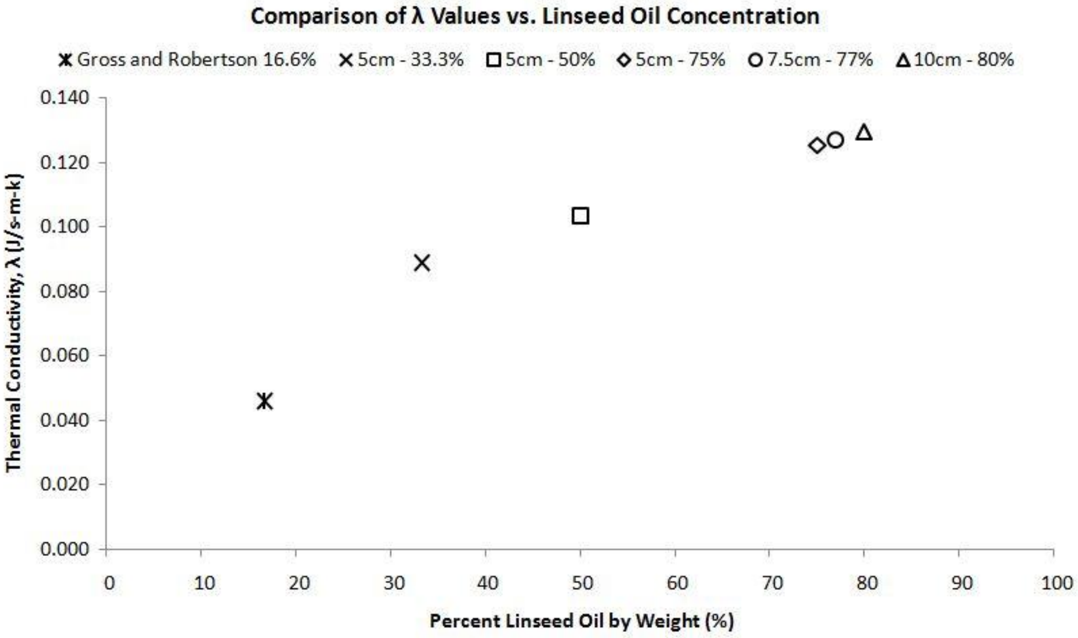


Figure 4.19: Graph of Thermal Conductivity with Linseed Oil Concentration

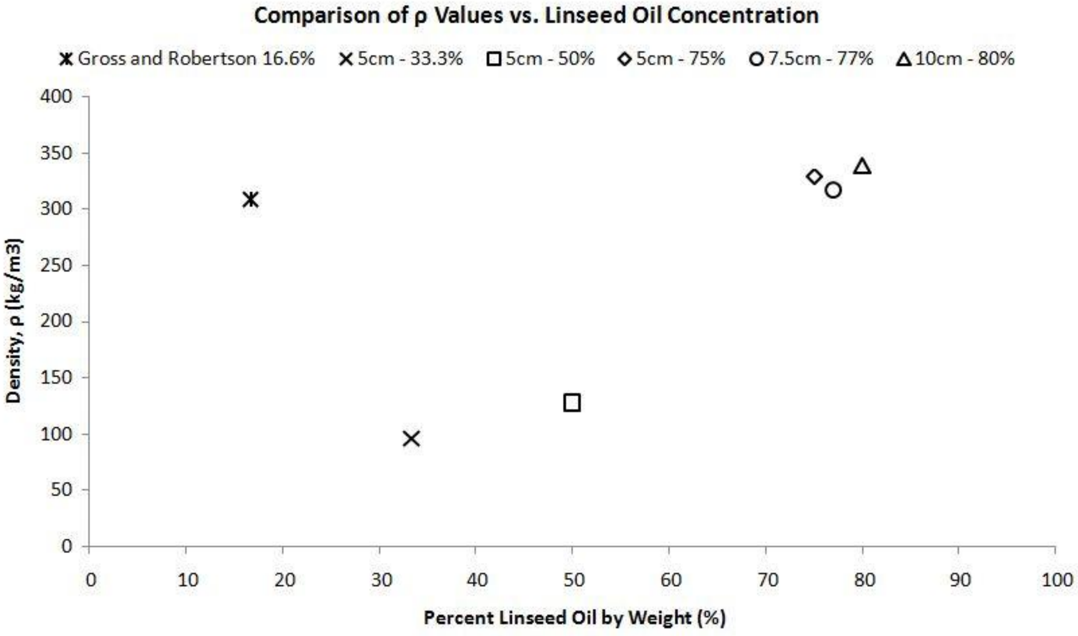


Figure 4.20: Graph of Density with Linseed Oil Concentration

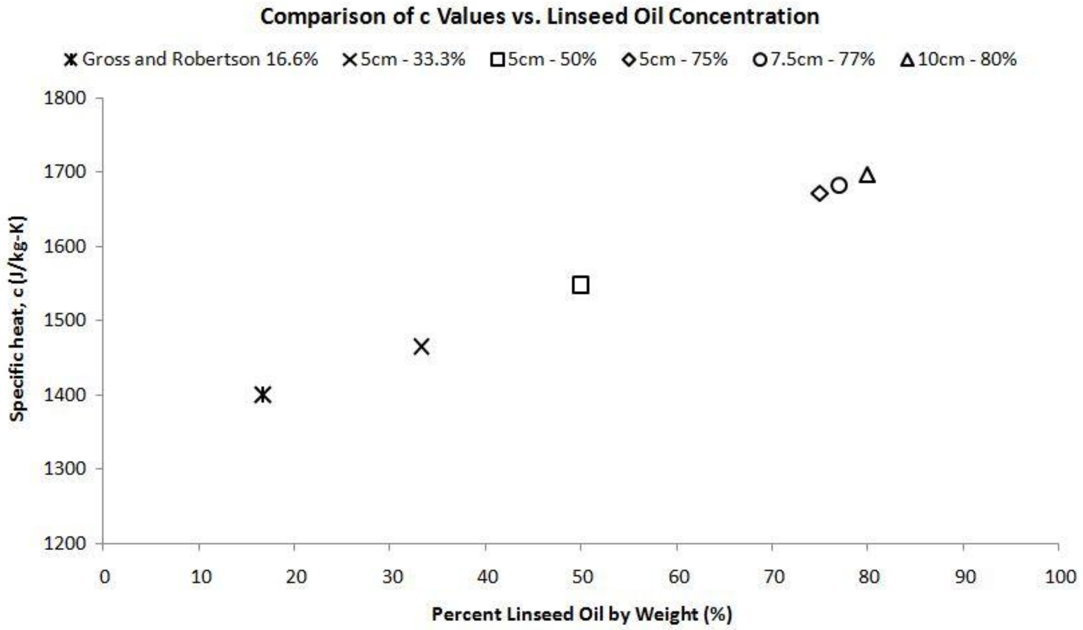


Figure 4.21: Graph of Specific Heat with Linseed Oil Concentration

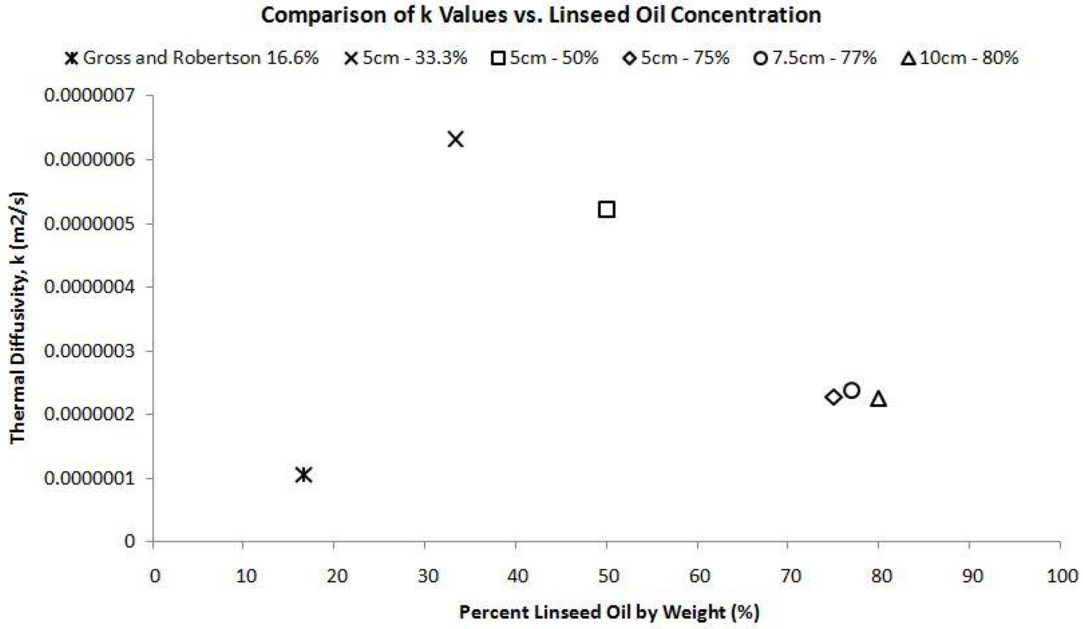


Figure 4.22: Graph of Thermal Diffusivity with Linseed Oil Concentration

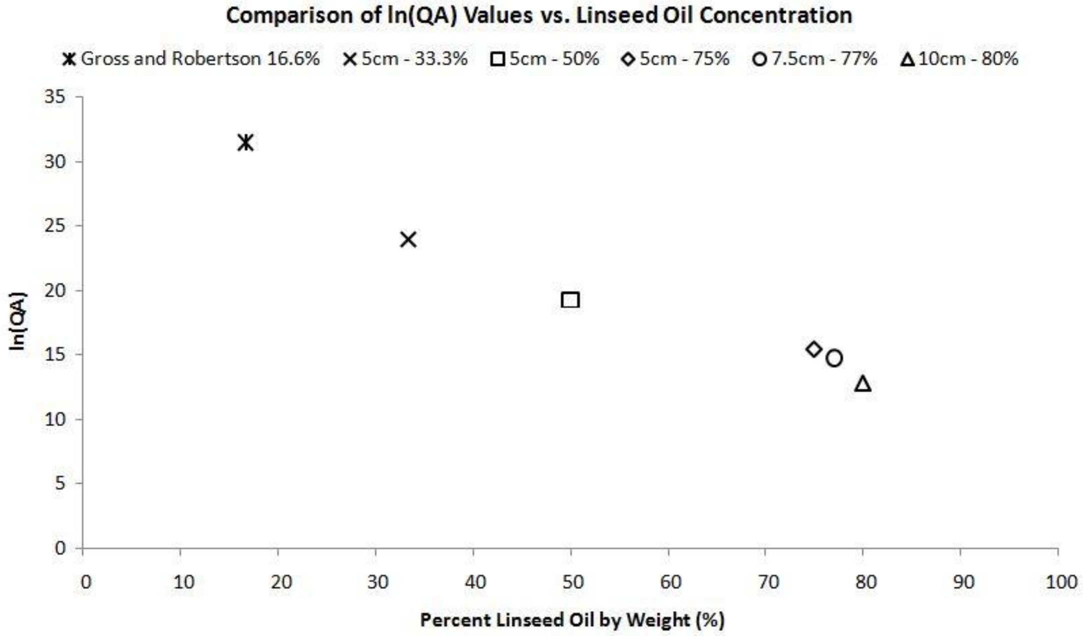


Figure 4.23: Graph of ln(QA) with Linseed Oil Concentration

When investigating the graph of thermal conductivity, Figure 4.19, Density, Figure 4.20, and Specific Heat, Figure 4.21, it makes sense that they all increase with the concentration of linseed oil. The thermal conductivity and specific heat of linseed oil are both higher than that of cotton, shown above, so it makes sense that these values increase as the sample concentration increases. Linseed oil is also much denser than cotton and again it makes sense when the sample density increases with increasing linseed oil concentrations. In Figure 4.20, it is shown that the density for the Gross and Roberson is in the same range as the 5cm (75%), 7.5cm (77%) and 10cm (80%) basket sizes. This doesn’t mean that the densities used the 5cm (33.3%) and 5cm (50%) basket sizes are wrong it just means that a higher packing density was used for their testing. Figure 4.22 shows the thermal diffusivity which was calculated from Eq. 19 using the three parameters, thermal conductivity, density and specific heat. As the concentration of linseed oil increases the thermal diffusivity decreases meaning that it becomes harder for heat to diffuse into the sample as the concentration of linseed oil in the sample increases. This makes sense and plays a big part in activating the reaction inside the sample. Figure 4.23 shows that as the concentration of linseed oil increases, ln(QA) decreases. These were the y-axis intercept values from the ln(dT_c/dt) vs 1000/T_o graphs.

4.4.4 Rate of Reaction per Unit Volume

Using these parameters the rate of reaction per unit volume, $\dot{Q}'''$, from Eq. 2 can also be calculated for an individual test run, as seen in Figure 4.24.

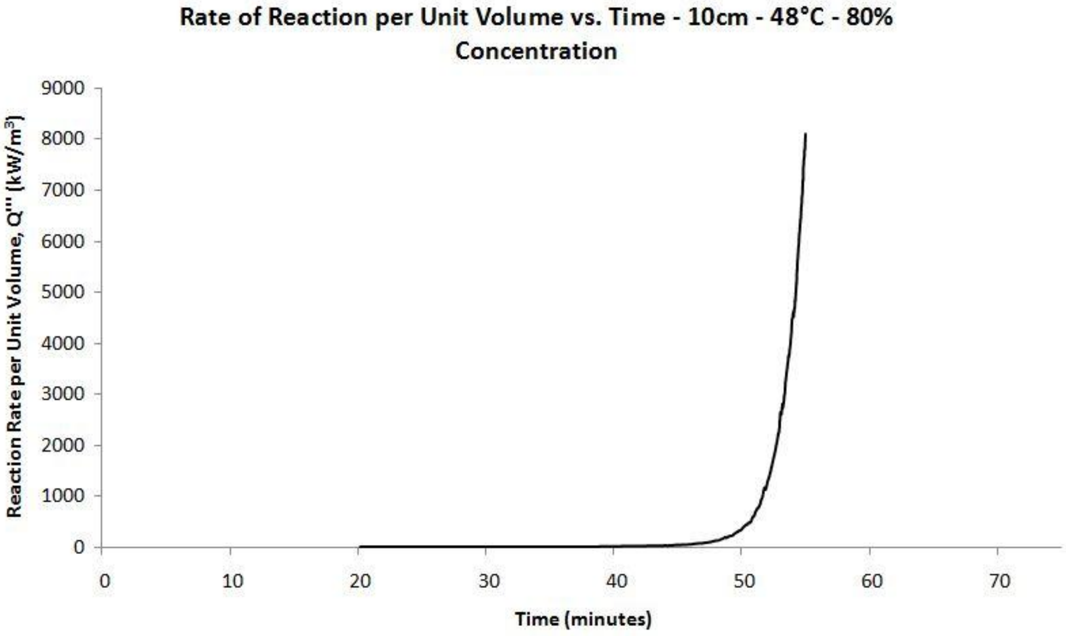


Figure 4.24: Reaction Rate per Unit Volume for 10cm (80%) Basket with 48°C Oven Temperature

Figure 4.24 show the reaction rate per unit volume for the 10cm (80%) basket size tested at an oven temperature of 48°C with time. The reaction rate is calculated from the start of the test till just after ignition. This time period is the only time when the calculated properties are correct, after this time period the properties change resulting from ignition. When comparing the reaction rate per unit volume to the time temperature curve, seen in Figure 4.1, it is evident that the reaction rate directly applies to the center temperature of the substance. As the center temperature starts to increase after the sample is placed in the oven the rate of reaction also starts to increase. At 50 minutes the rate of reaction spikes which is the exact moment that ignition occurs in the sample. This can also be seen in Figure 4.1 when the center temperature increases significantly at the ignition point.

4.5 Time to Ignition

When looking at all the tests from Figure 4.6, Figure 4.7 and Figure 4.8 along with some additional data for the 10cm (80%) basket size, the ignition time for each test run was found. Ignition was determined to be when the center temperature of each sample started to increase 10°C per minute for the 5cm (75%), 7.5% (77%) and 10cm (80%) basket sizes and 20°C per minute for the 5cm (33.3%) and 5cm (50%) basket sizes. When the sample reached this rate of increase it was determined that the reaction inside the sample achieved run-away conditions. After compiling the results the different basket sizes were compared, shown in Figure 4.25 and Figure 4.26.

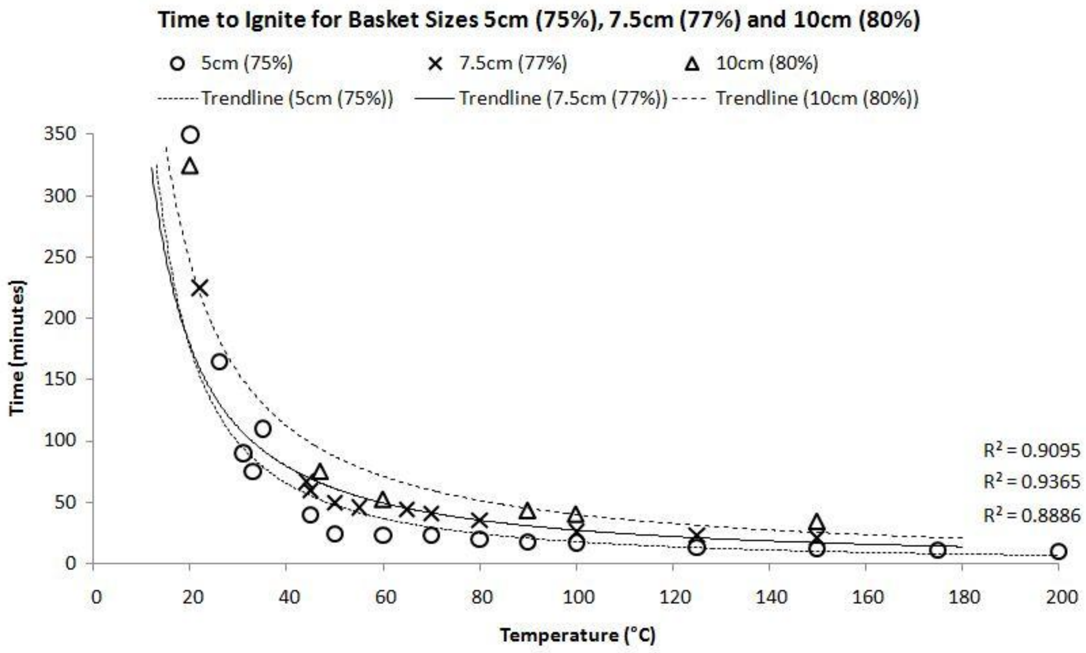


Figure 4.25: Time to Reach Ignition for 5cm (75%), 7.5cm (77%) and 10cm (80%) Basket Sizes

Figure 4.25 shows a decreasing trend in time as the oven temperature increases. This means that as the oven temperature the sample is exposed to increases, the time it takes the sample to reach ignition decreases, which is expected. As the oven temperatures reach ambient temperature (20°C) the time it takes samples to go to ignition significantly increases, reaching a time of over 5.5hrs. It is also shown in Figure 4.25 that as the basket size and concentration are decreased the time to ignition also decreases. This phenomenon happens very distinctively at 100°C where the 5cm (75%) reaches ignition before the other two basket sizes. This is most likely due to the fact that as the sample gets larger the thermal diffusivity decreases, as talked about in section 4.4.3. This means it takes longer for the heat produced by the oven to heat up the sample and effect the reaction happening inside the sample. Another factor that could be effecting the time of ignition could be the time it takes the oxygen to filter into the sample to feed the reaction. As the basket size increases it would take longer for oxygen, like the heat from the oven, to filter into the sample thus resulting in the larger samples taking a longer time to reach ignition. Figure 4.26 shows the ignition times for the different concentrations of the 5cm basket.

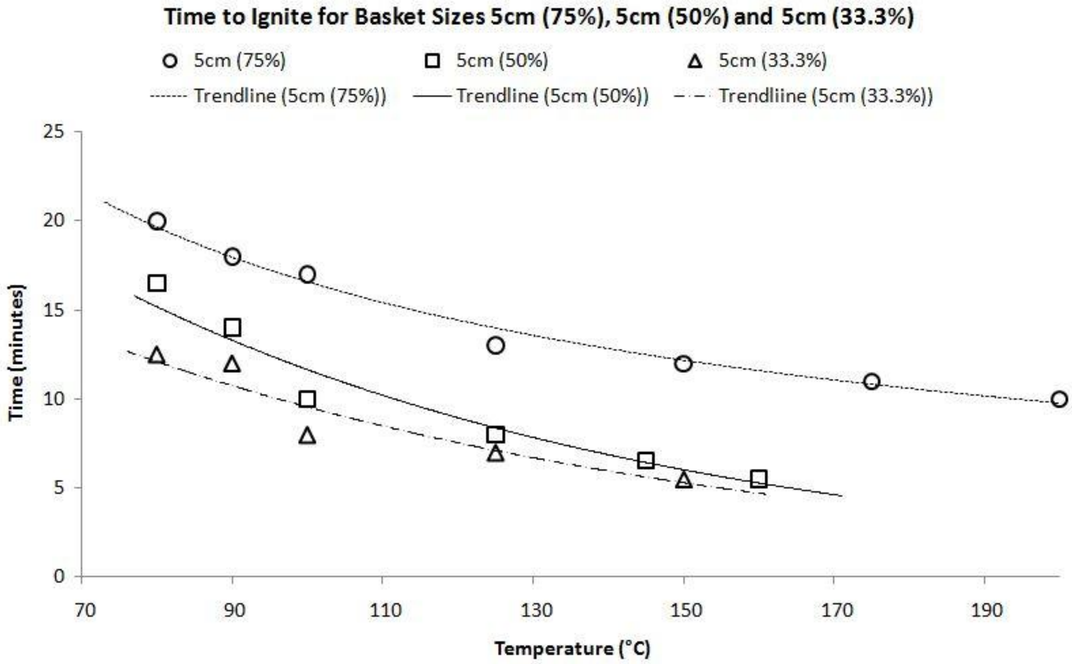


Figure 4.26: Time to Ignite for 5cm (75%), 5cm (50%) and 5cm (33.3%)

Again, as the oven temperature the sample is exposed to increases the time it takes to reach ignition decreases. Figure 4.26 also shows that as the concentration of the sample decreases the time it takes for the sample to go to ignition decreases. This is due to the less concentrated samples having a lower density and higher thermal diffusivity. With a higher thermal diffusivity the sample will be affected by the heat of the oven quicker which will affect the reaction inside the sample earlier in the testing. With further testing the optimal concentration could be found that would provide the quickest ignition times.

There is also an induction period theory in Bowes (1) which can be used to estimate the time to ignition for a sample having the geometry of a slab, cylinder or a sphere. There are also different factors that are accounted for by his theory including the effect of moisture and reactant consumption. This theory was not pursued but future analysis could be done using the acquired data.

5. Conclusions

This research was conducted to help fire investigators find an easier way to test and analyze spontaneous material. Originally the Frank-Kamenetskii was to be coupled with the oven basket method to test linseed oil soaked cotton. An oven was instrumented and a safety system was set in place to control the oven conditions throughout the testing stages. After it was concluded that the F-K method would not be able to be used the crossing point method was resorted to. Both the Chen and Jones crossing point methods were originally used but due to complications in the testing of the data the final results were done using only the Jones crossing point method. The results given from the Jones crossing point method were very favorable and fit nicely with results from other sources. The Jones method coupled with the oven basket method was very easy to use and is recommended to be used in future testing of spontaneous material.

After investigating the raw data produced by the oven basket method there were three different stages that were reached during a typical test run. The first stage reached was the ignition stage which resulted when the reaction inside the sample ran away thus producing a significant rise in the center temperature over a short period of time. The second stage was a smoldering stage when the inside of the sample started to smolder which could be sustained for a long period of time if the appropriate amount of oxygen in the center of the sample was present. The final stage was a flaming stage which was an elevated temperature stage where flaming occurred if enough fuel, oxygen and heat were present. After the flaming stage was finished the center temperature of the sample decreased to a final temperature at approximately the oven temperature.

To use the Frank-Kamenetskii method critical temperature were pursued using the oven basket method. After testing the 5cm (75%), 7.5cm (77%) and 10cm (80%) basket sizes it was concluded that every oven temperature tested led to ignition, including ambient temperature (20°C). Later on in the testing stage the concentration of the 5cm basket was reduced to 33.3% and 50% where a critical temperature was pursued when testing the 5cm (33.3%) basket size. A sub-critical test was found at an oven temperature of 40°C but it was eventually concluded that further testing needed to be done to exactly pinpoint a critical temperature. With no critical temperatures found the crossing point method was resorted to.

When applying the Jones version of the crossing point method it was very easy to examine the raw data produced by the oven basket method to find the crossing points and their slopes. After completing the analysis of the crossing point method the activation energy was found for all of the basket sizes. The activation energies were 42.37 kJ/mol, 27.40 kJ/mol, 16.97 kJ/mol, 15.76 kJ/mol and 11.73 kJ/mol for the 5cm (33.3%), 5cm (50%), 7.5% (77%) and 10cm (80%) basket sizes. It was concluded that as the concentration of the linseed oil increased in the sample the activation energy of the sample decreased. When the results from Khattab et al (4) and Gross and Roberston (2) was graphed with the data from this research a very nice decreasing trend was found showing further that as the concentration of linseed oil in the sample increase the activation energy decreased.

The calculation of P and M for all basket sizes also found favorable results seen in Table 4. Both the P and M values decreased as the concentration of the linseed oil in the sample increased. This trend was the same as the trend for the activation energy, which was expected since the activation energy was included in the calculation of both parameters. The trend for the M parameter was not exactly the same as the trend found for the P parameter. It was concluded that the difference was due to the other properties, thermal conductivity, density, QA, that were included in the calculation of M. The P and M values agreed very nicely with the data from Gross and Robertson. When the two sets of data were graphed together the Gross data appeared in the expected area based on the trend of the research data.

The properties that were needed to calculate M were also used to calculate the rate of reaction per unit area, $\dot{Q}'''$. This was done for the 10cm (80%) basket size with an oven temperature of 48°C. The reaction rate was calculated from the start of the test to just after ignition. After ignition the calculated properties would change but during the specified region of the test they would remain as calculated. The reaction rate increased slowly as the center temperature of the sample also increased. When the sample reached ignition the reaction rate spiked just as the center temperature. This gave a good picture of what was exactly happening in the center of the sample up to ignition.

Lastly, the time to ignition was looked at closer for all three basket sizes. Ignition was determined when the center temperature of each sample started to increase 10°C per minute for the 5cm (75%), 7.5% (77%) and 10cm (80%) basket sizes and 20°C per minute for the 5cm (33.3%) and 5cm (50%) basket sizes. When the ignition times for all of the basket sizes were found they were graphed with oven temperature. It was concluded that as the oven temperature of the sample increased as the time to ignition decreased. Also, as the oven temperature got very close to ambient temperature (20°C) the time to ignition increased significantly to where the 5cm (75%) basket took over 5.5hrs to ignite. It was also concluded that as the concentration and basket size increased the time to ignition also increased. This was due to the effect the increased linseed oil concentration and basket size had on the thermal diffusivity and density of the sample. As the linseed oil concentration and basket size both increased the thermal diffusivity decreased and the density increased thus making it harder for the heat produced by the oven to effect the reaction happening inside the sample. This led to the larger more concentrated samples having a longer ignition time.

Understanding that the oven basket method coupled with the crossing point method is easy, cheap and effective will only expand the knowledge of spontaneous materials and their tendencies. The method for using both the oven basket method and the crossing point method described in this research is an effective, accurate, understandable way of testing and analyzing spontaneous ignition results. It is understood that linseed oil soaked rags, like any other spontaneous material, are a dangerous combination when not dealt with properly and with the help of these methods future products like this will be able to be tested while saving materials, time, energy and ultimately money.

6. Appendix

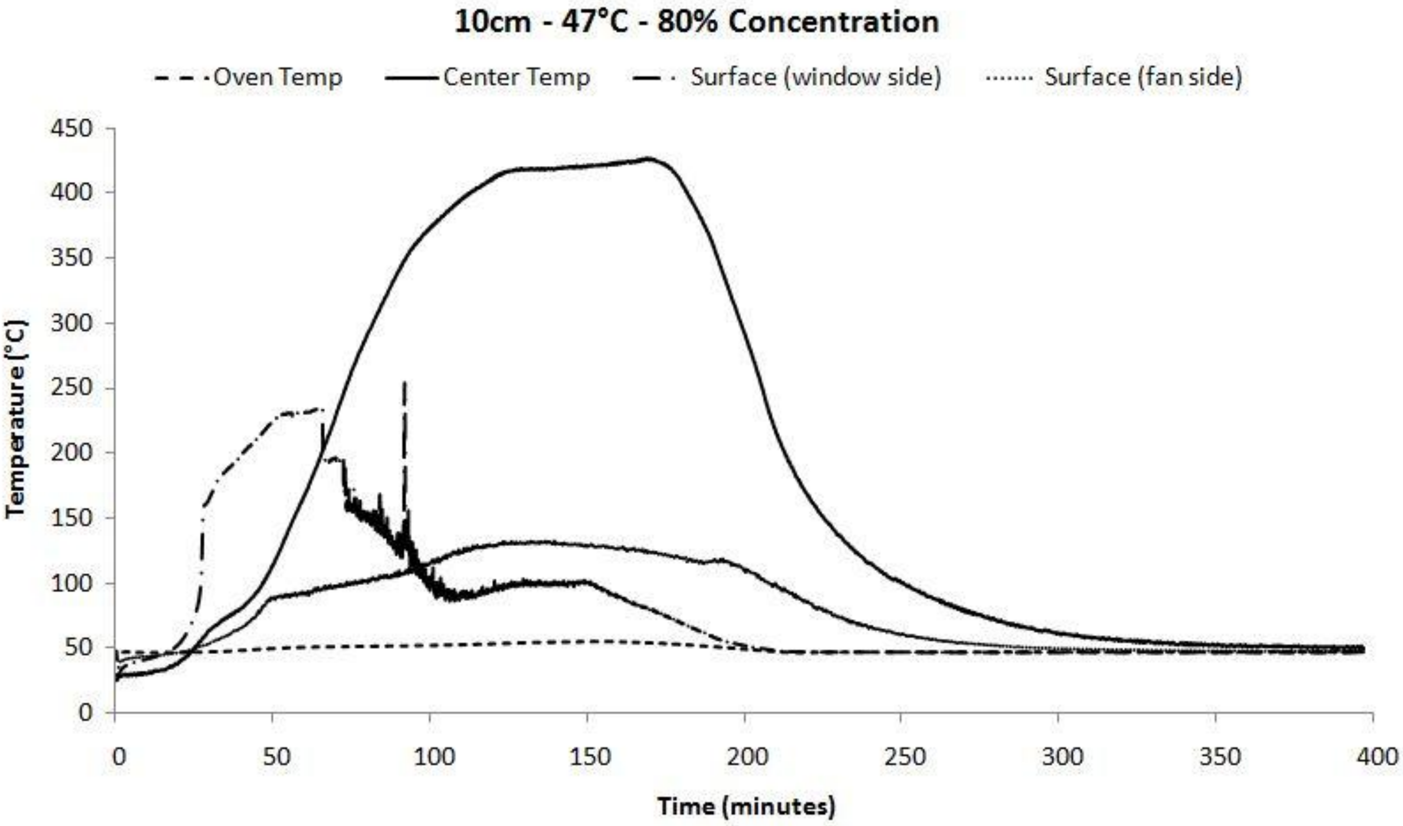


Figure 6.1: 10cm - 47°C – 80% Concentration

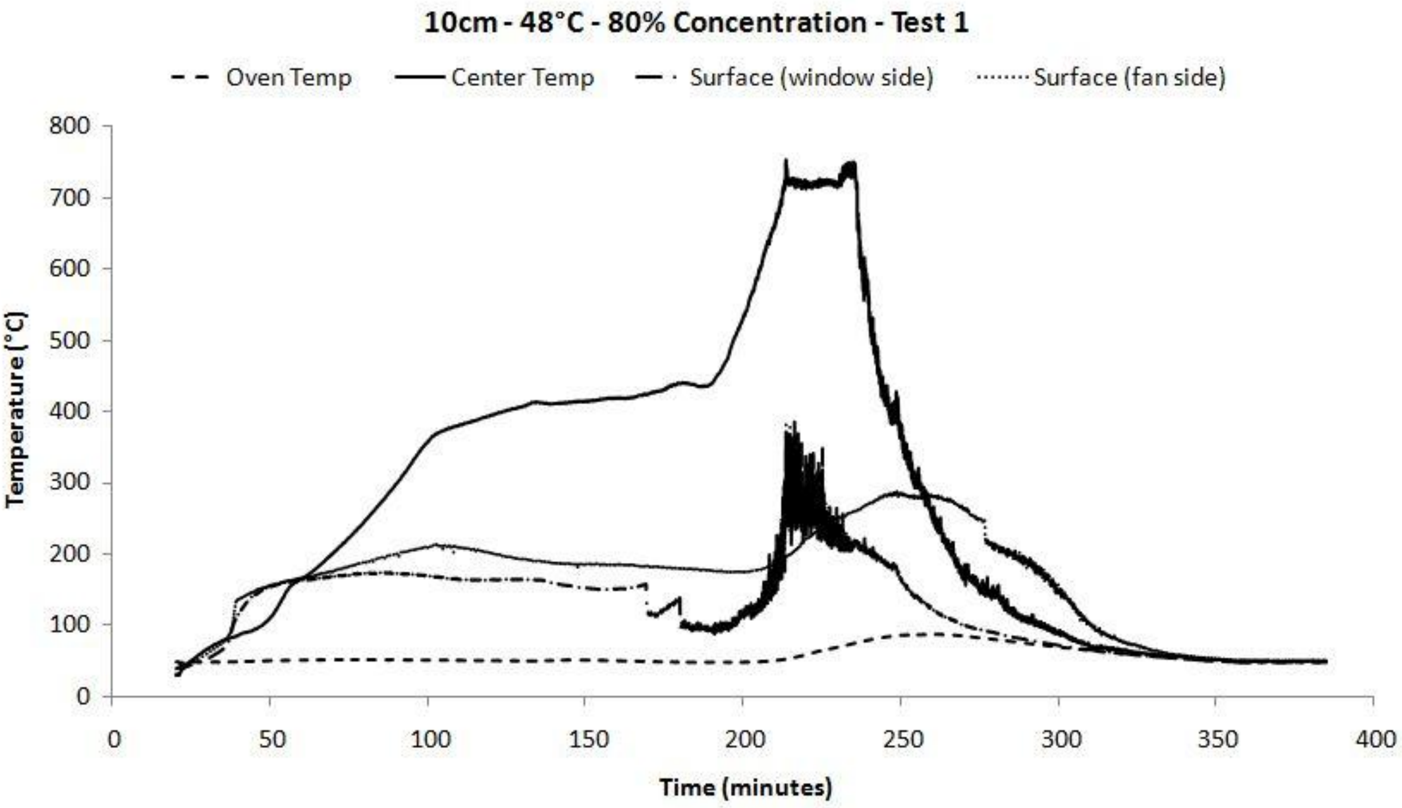


Figure 6.2: 10cm - 48°C – 80% Concentration – Test 1

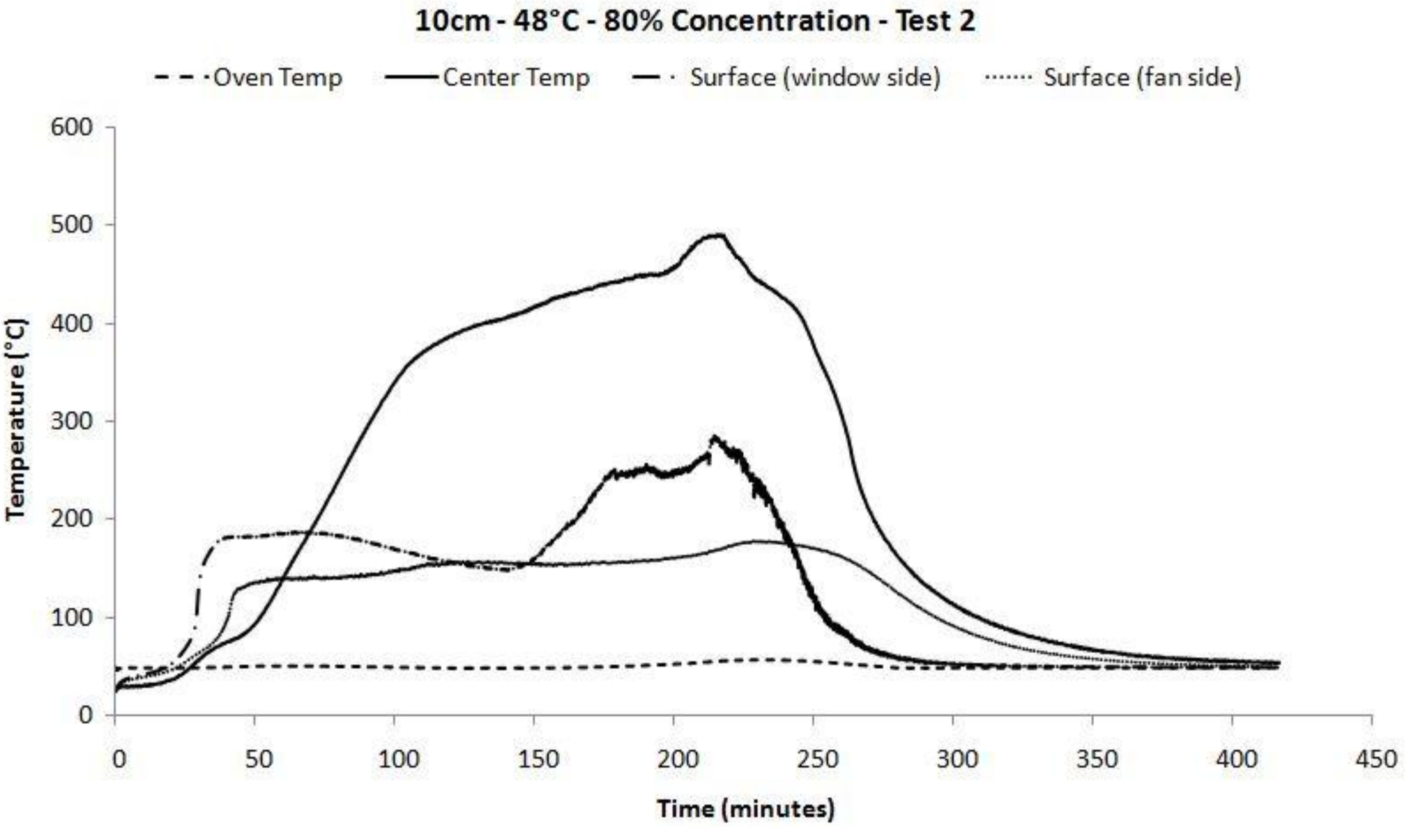


Figure 6.3: 10cm - 48°C – 80% Concentration – Test 2

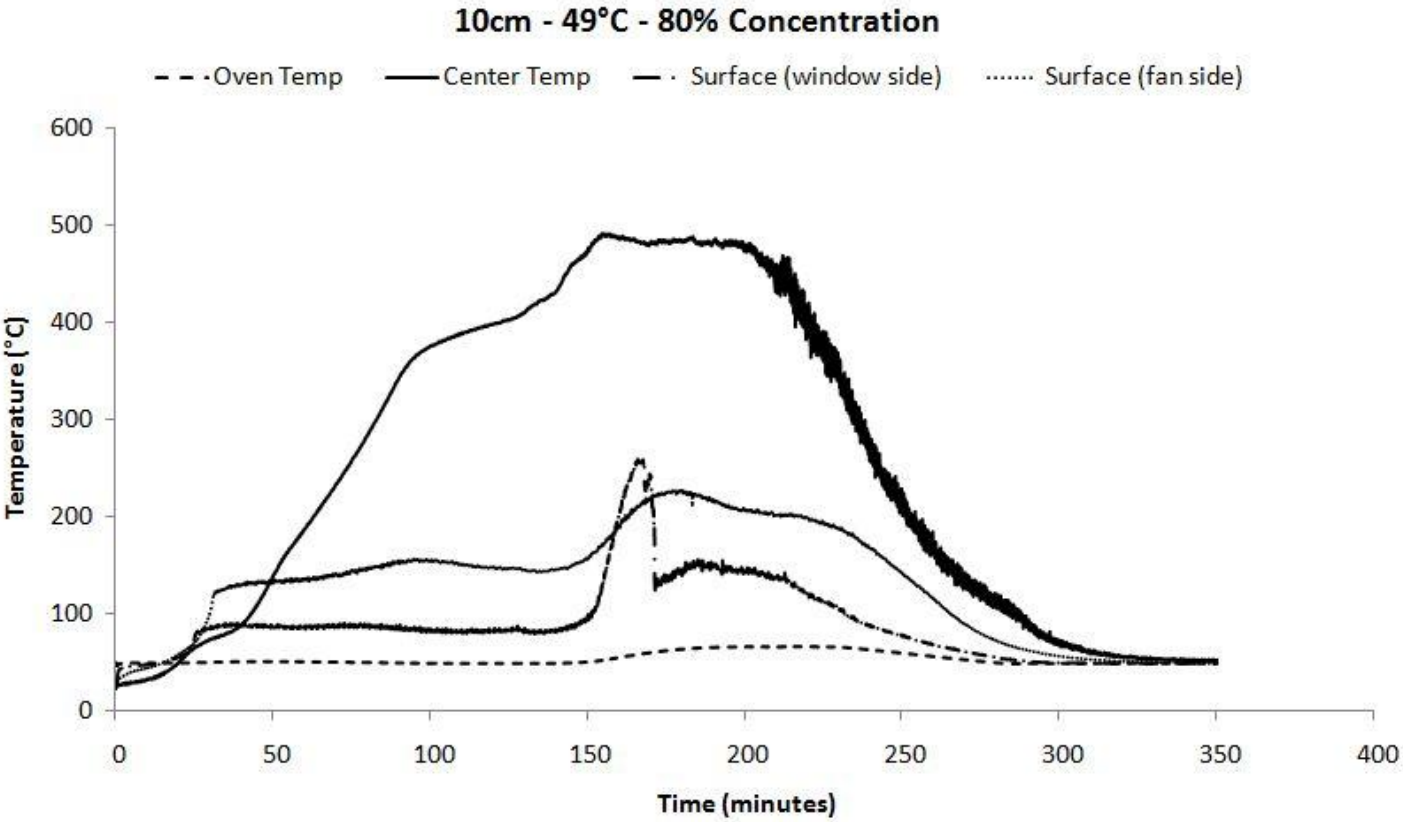


Figure 6.4: 10cm - 49°C – 80% Concentration

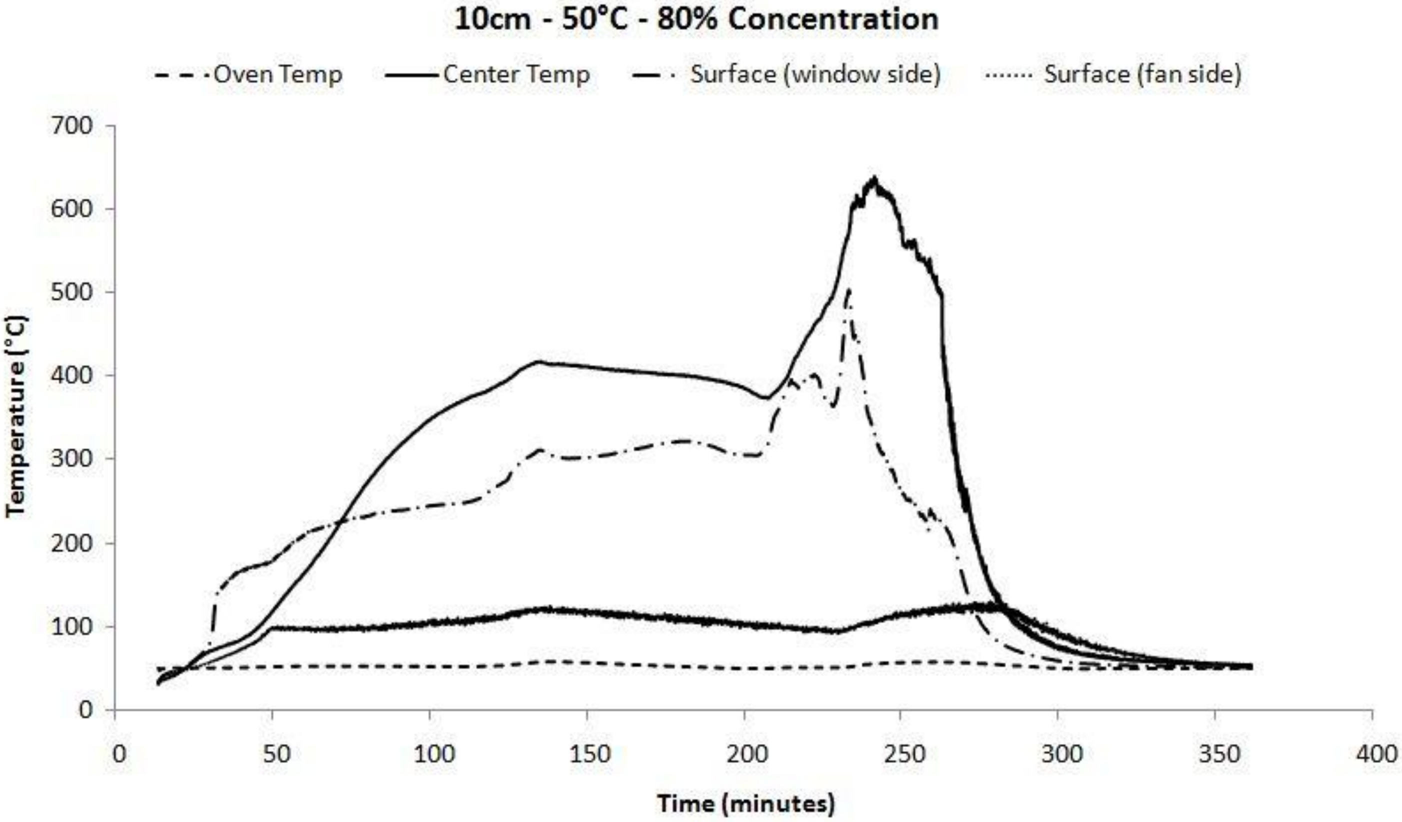


Figure 6.5: 10cm - 50°C – 80% Concentration

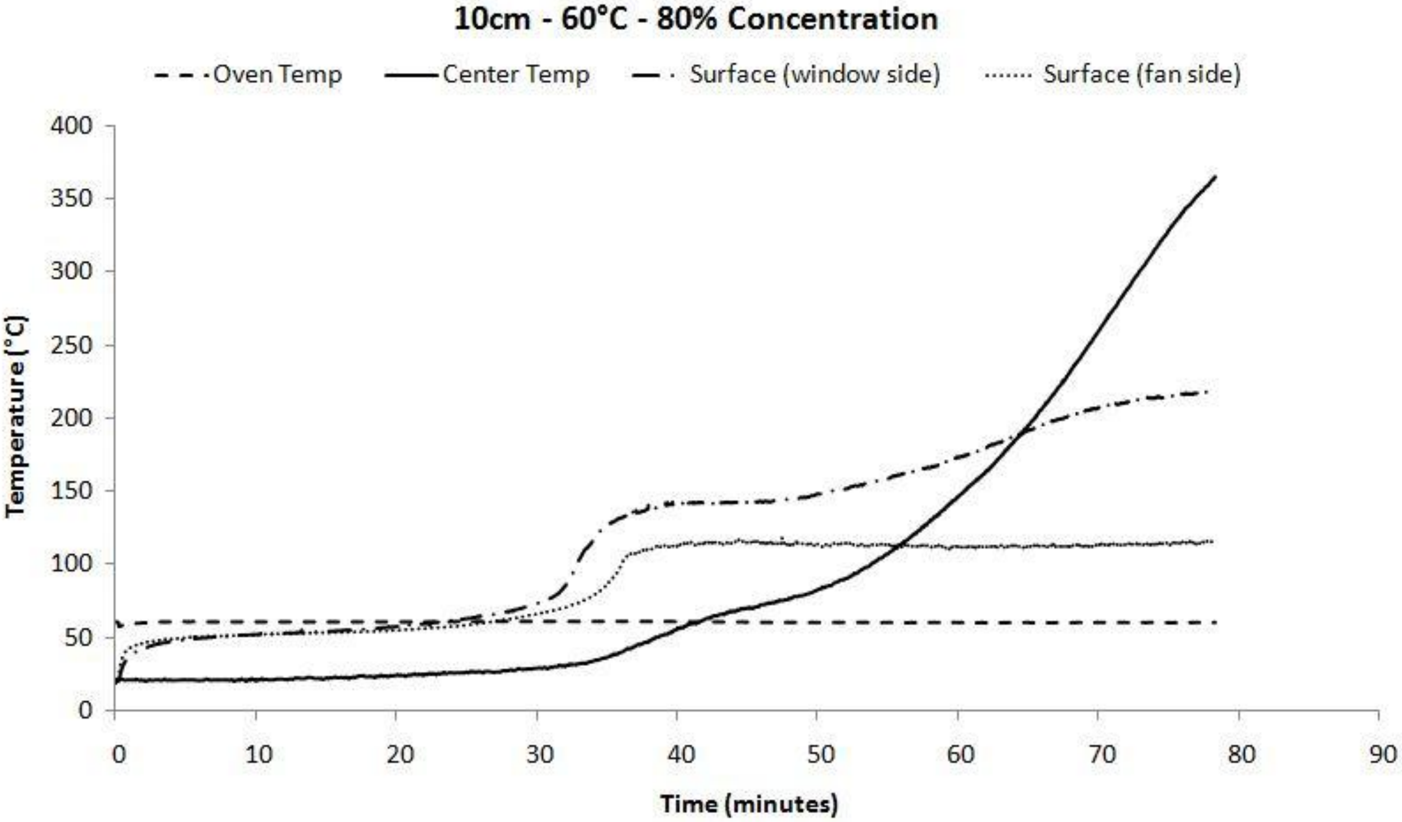


Figure 6.6: 10cm - 60°C – 80% Concentration

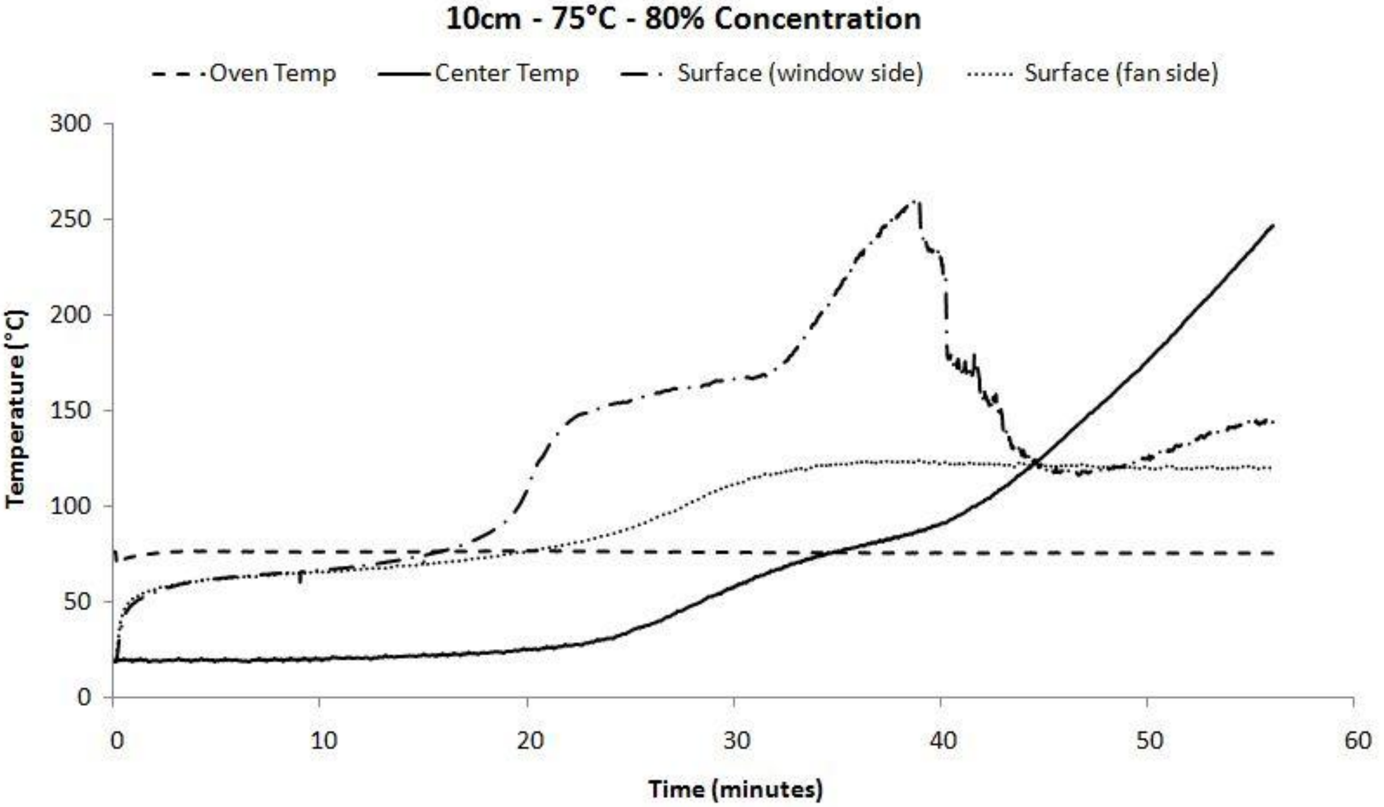


Figure 6.7: 10cm - 75°C – 80% Concentration

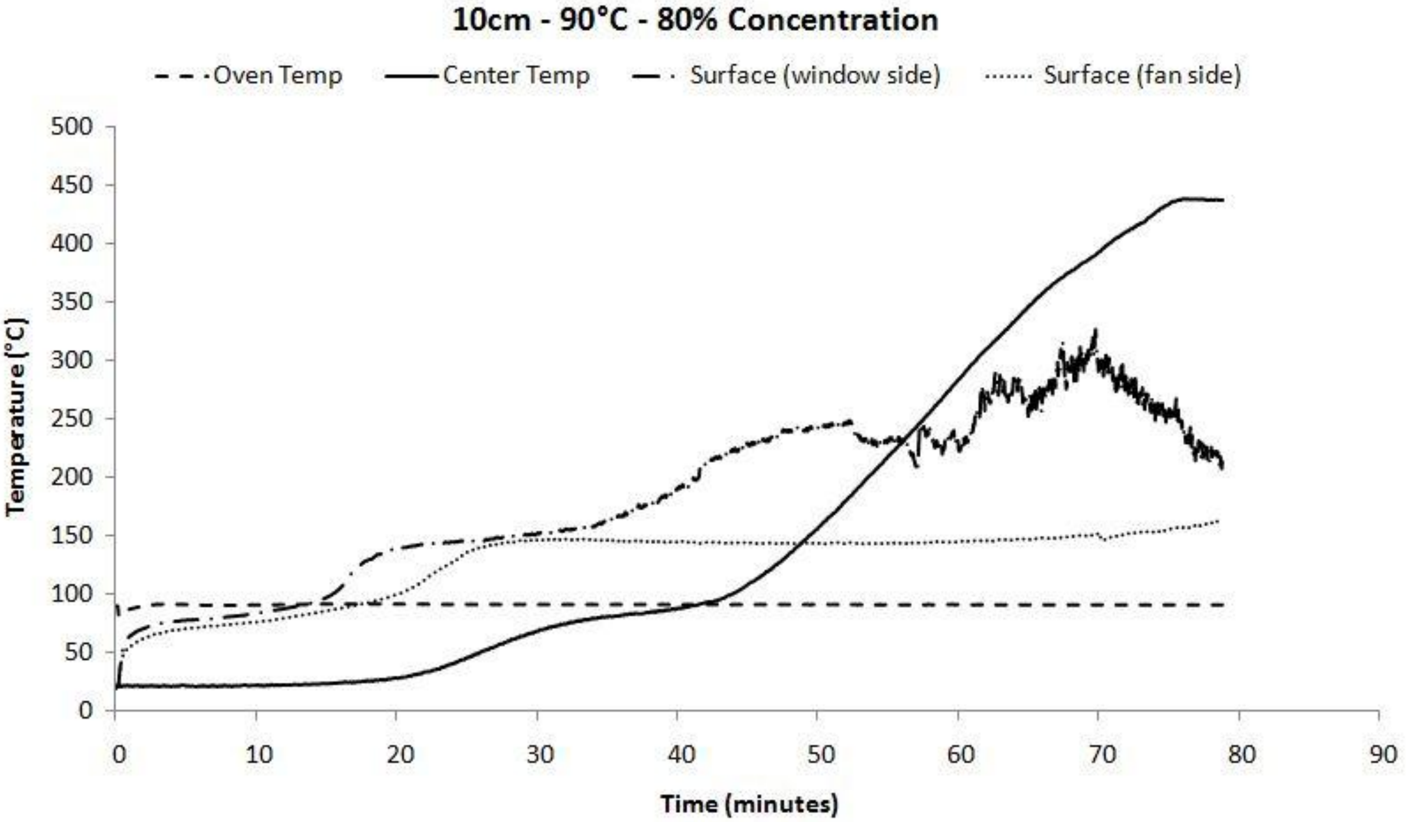


Figure 6.8: 10cm - 90°C – 80% Concentration

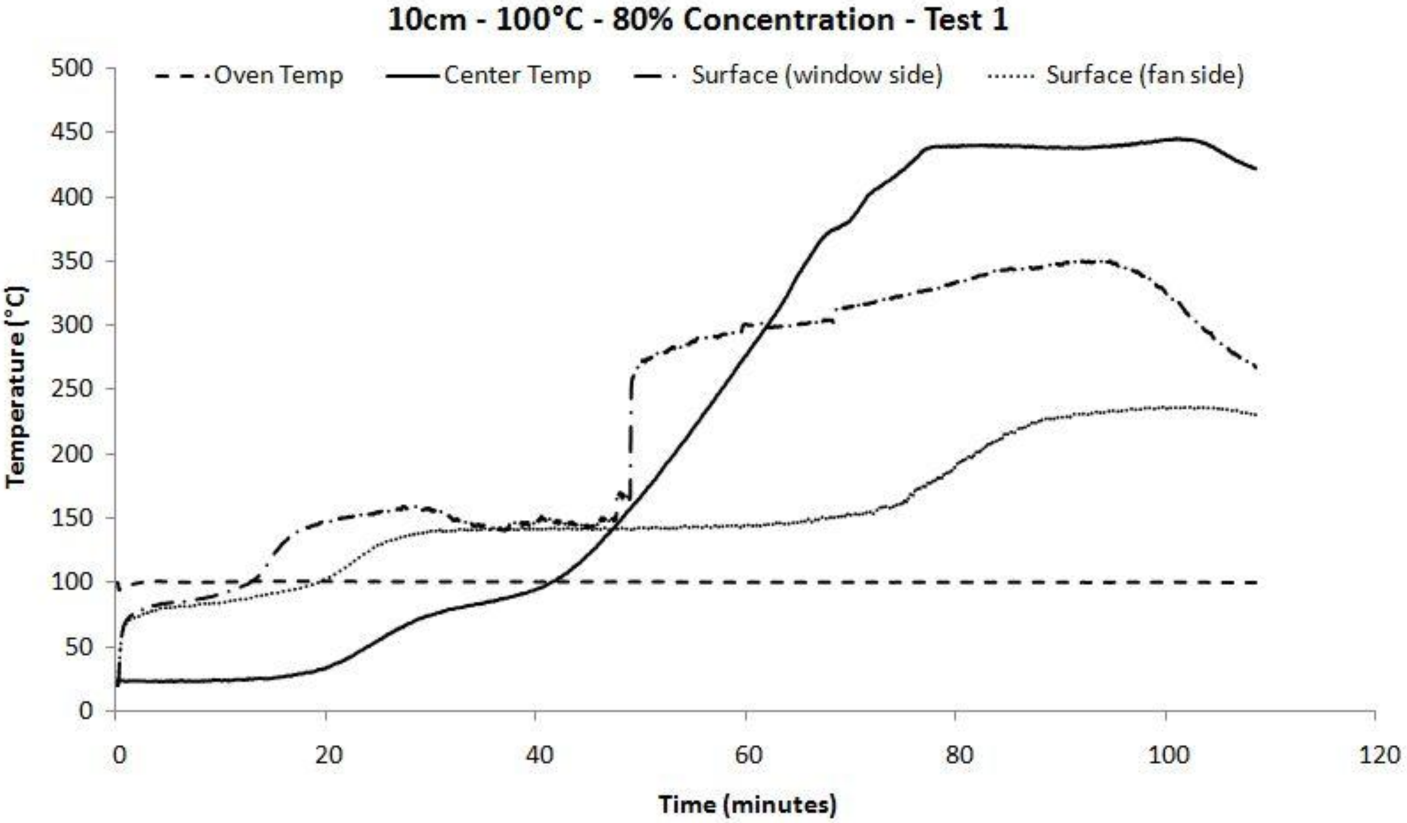


Figure 6.9: 10cm - 100°C – 80% Concentration – Test 1

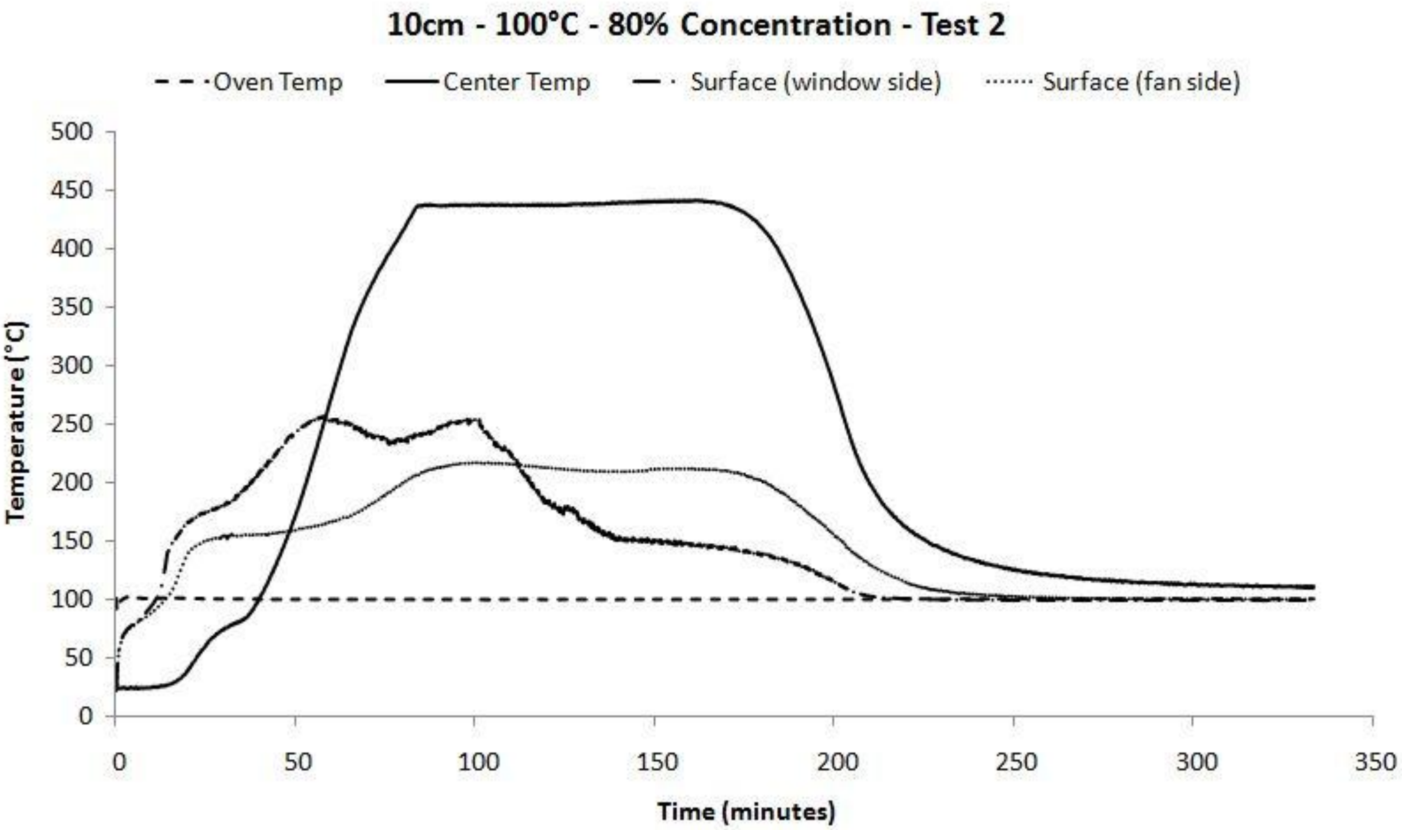


Figure 6.10: 10cm - 100°C – 80% Concentration – Test 2

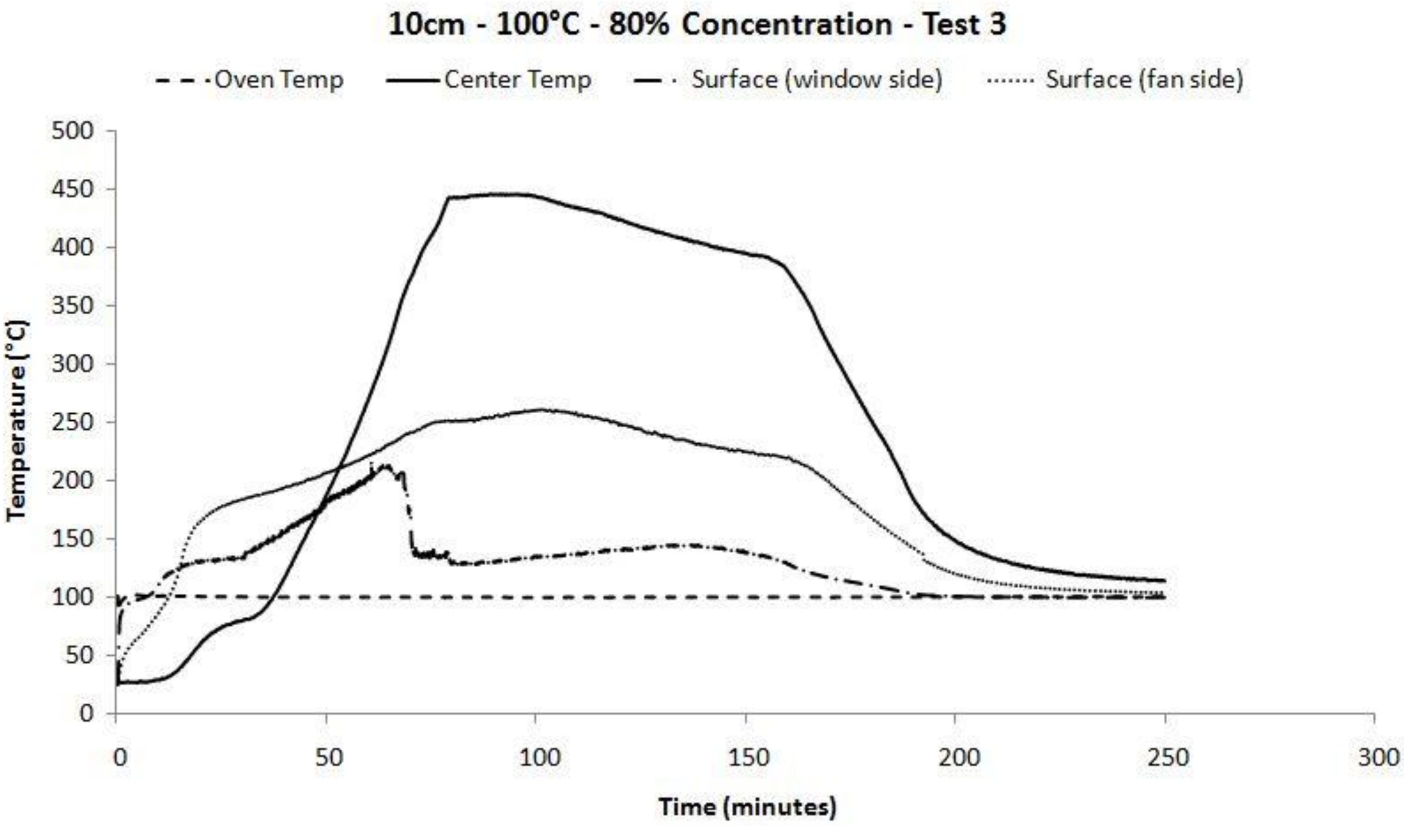


Figure 6.11: 10cm - 100°C – 80% Concentration – Test 3

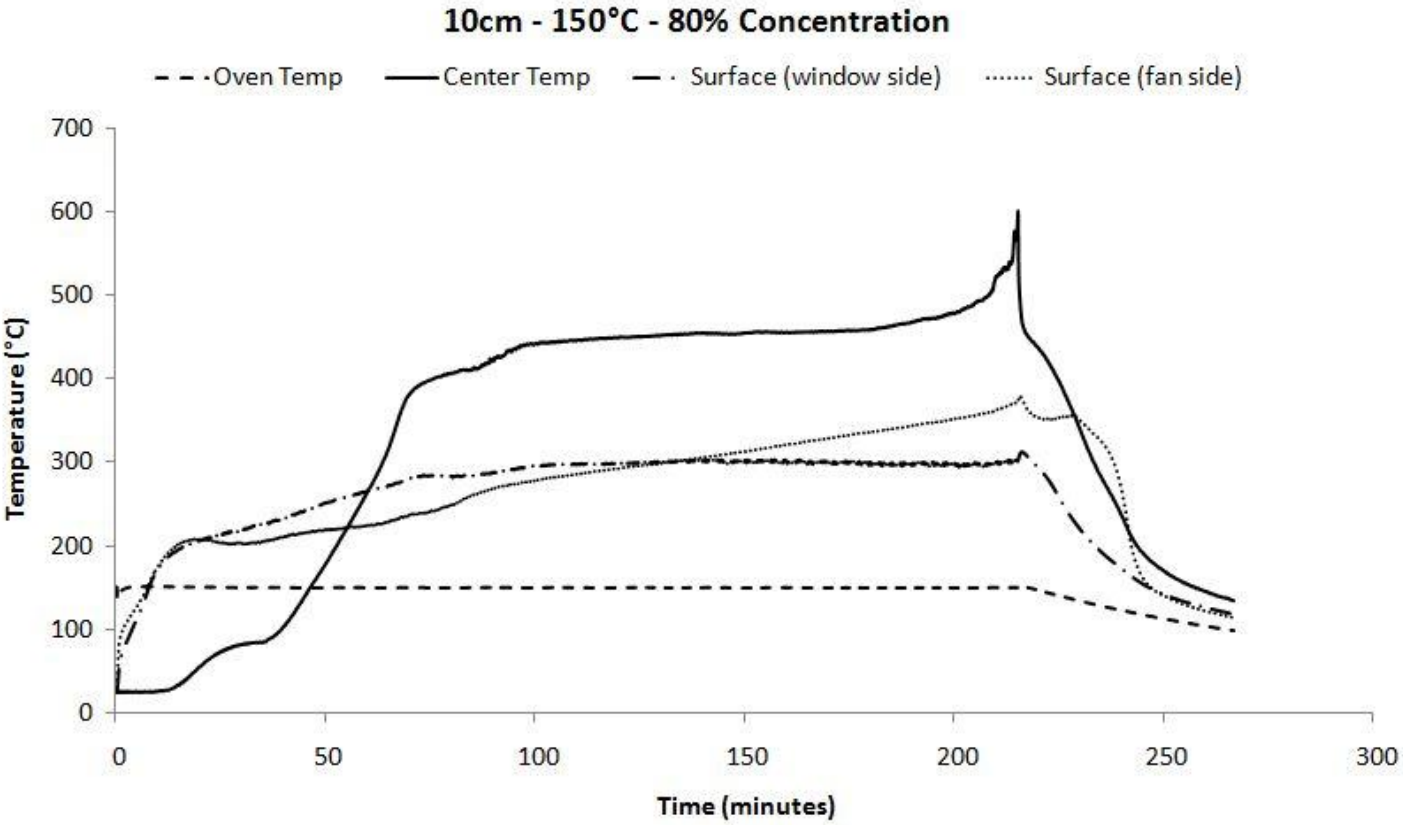


Figure 6.12: 10cm - 150°C – 80% Concentration

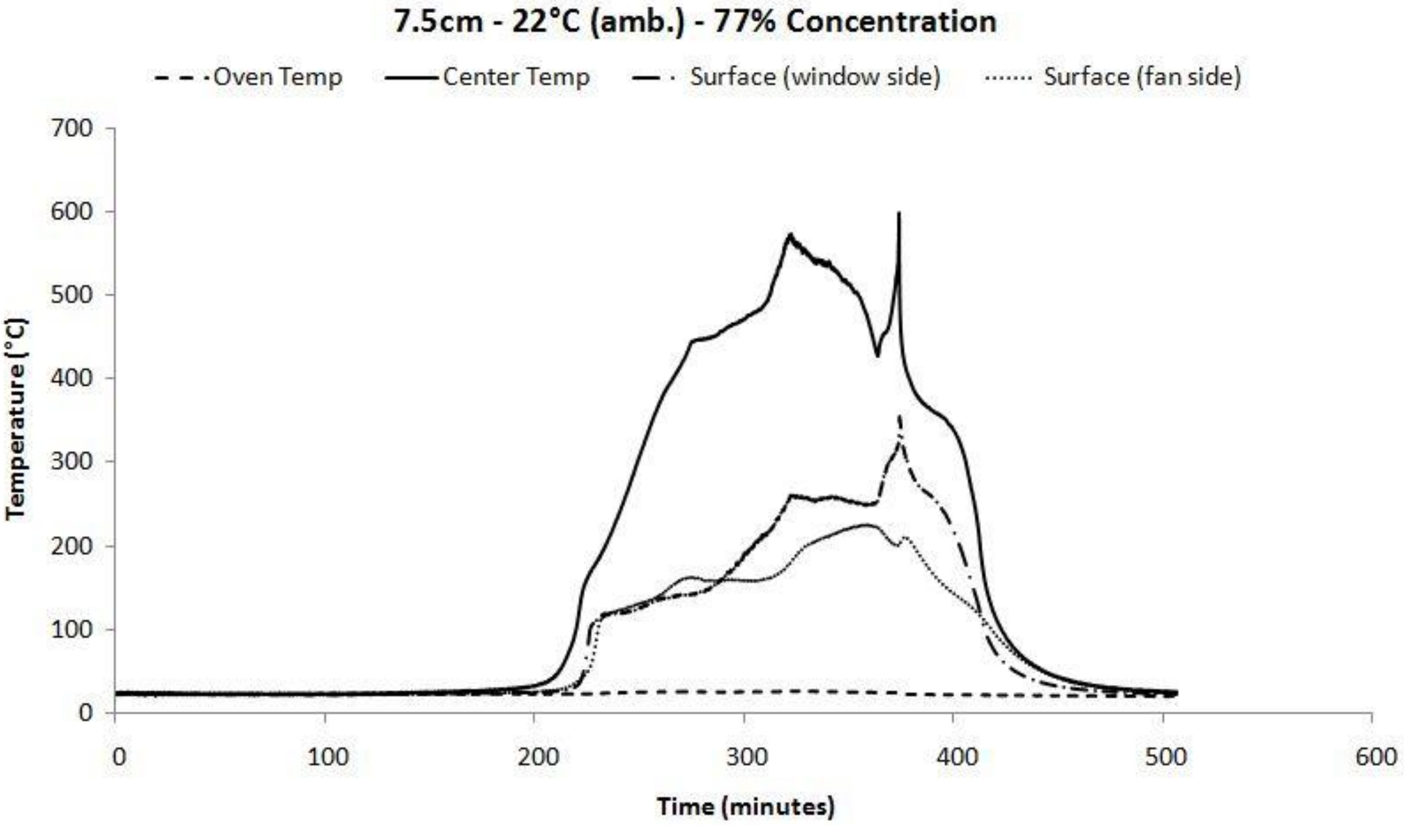


Figure 6.13: 7.5cm - 22°C – 77% Concentration

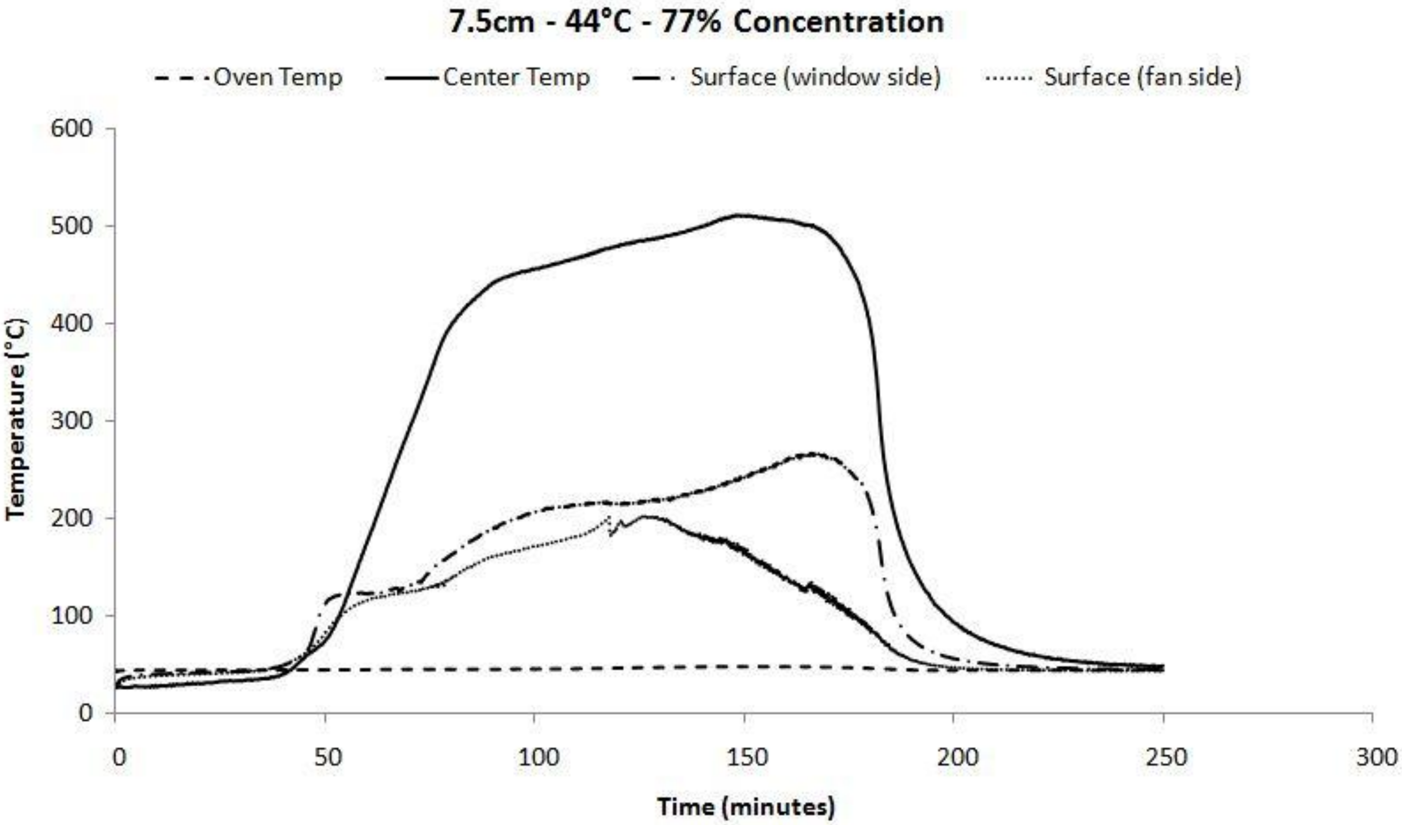


Figure 6.14: 7.5cm - 44°C – 77% Concentration

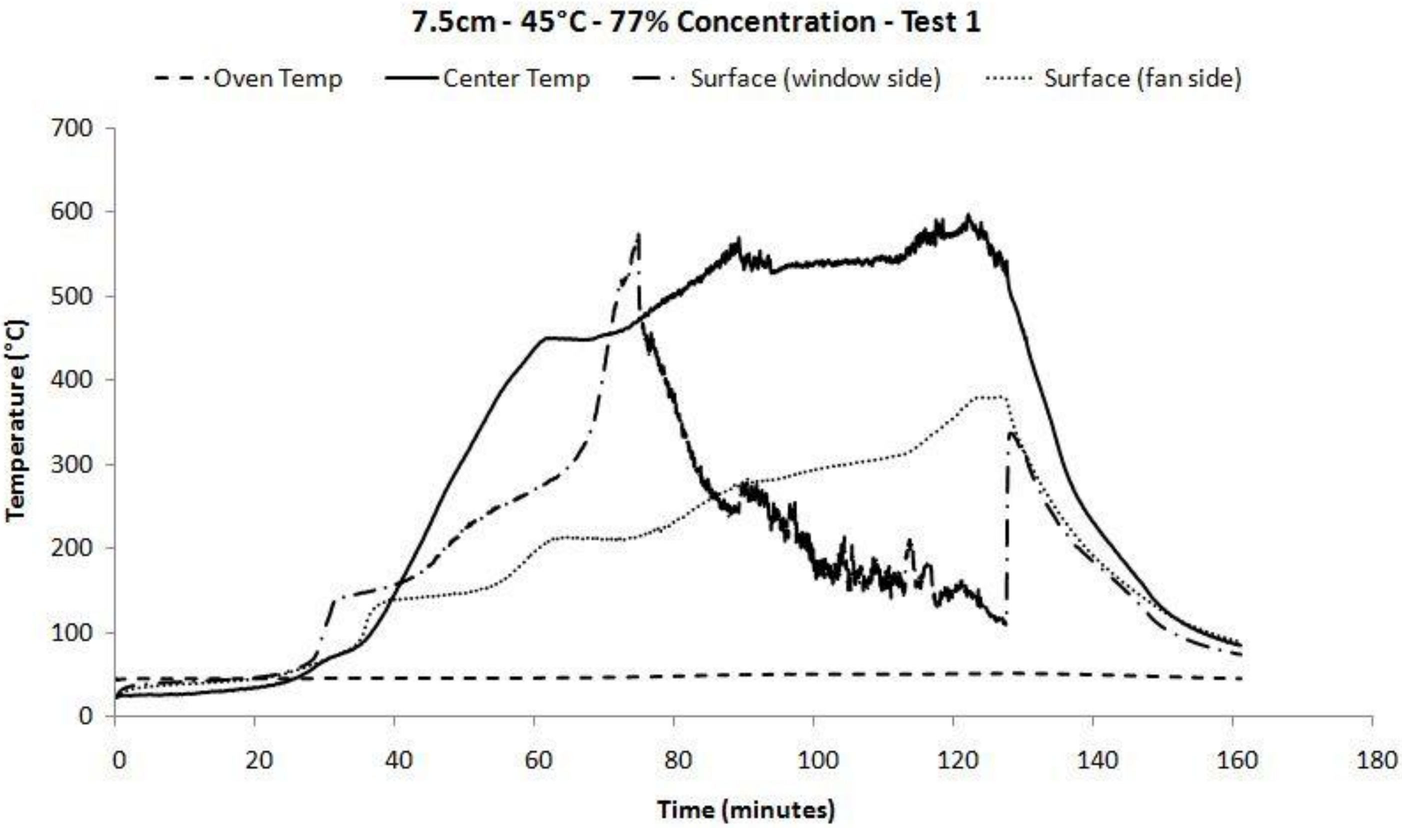


Figure 6.15: 7.5cm - 45°C – 77% Concentration – Test 1

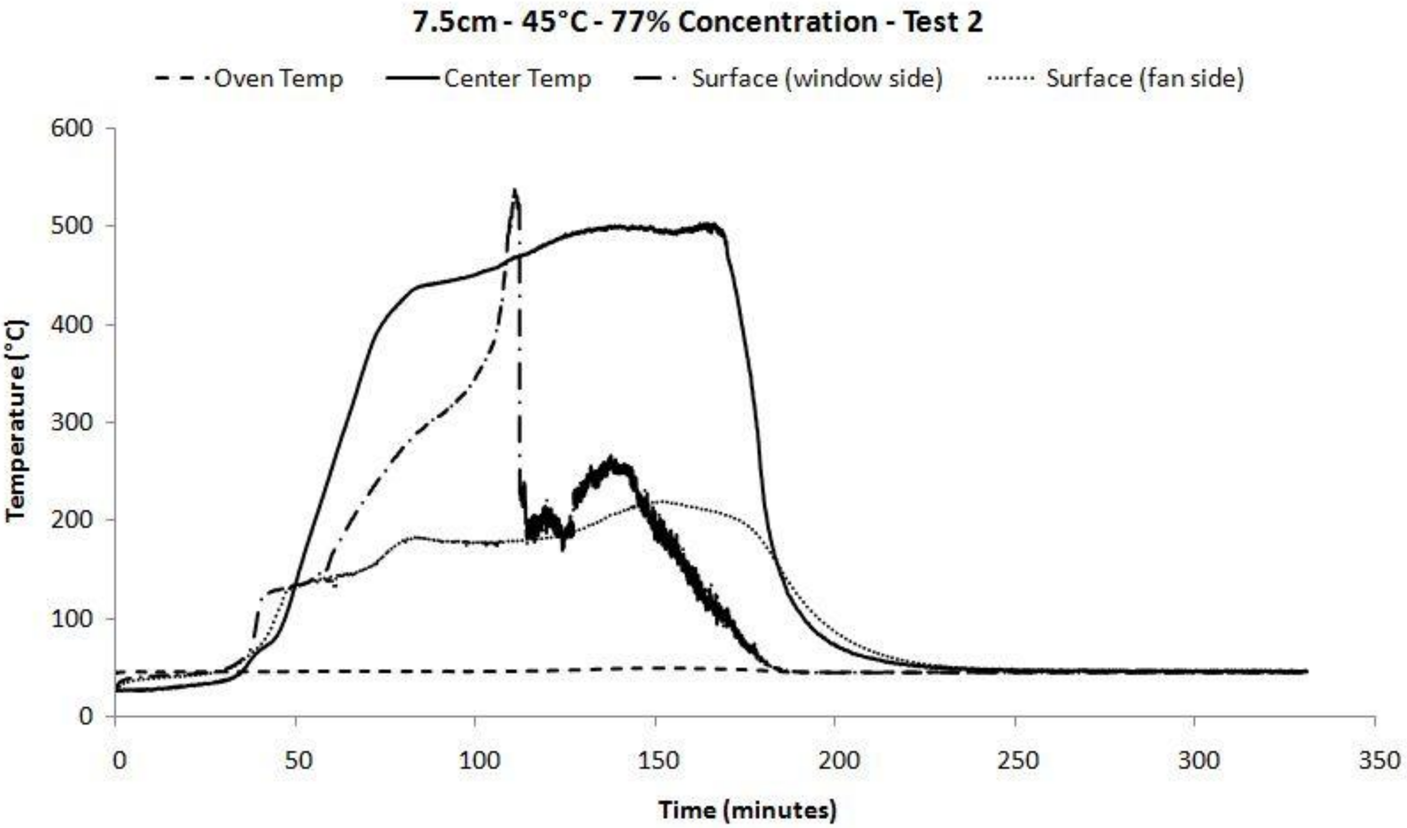


Figure 6.16: 7.5cm - 45°C – 77% Concentration – Test 2

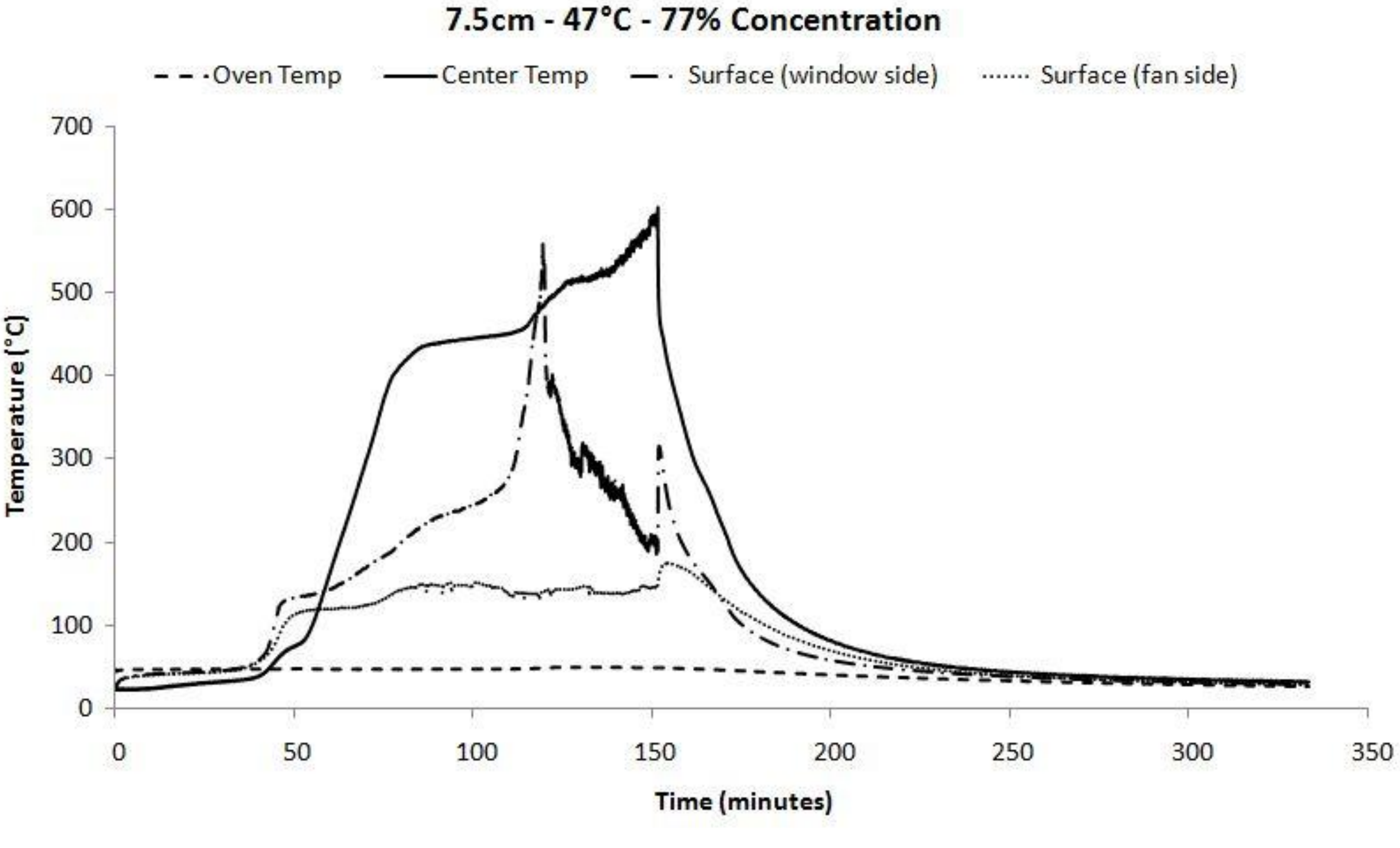


Figure 6.17: 7.5cm - 47°C – 77% Concentration

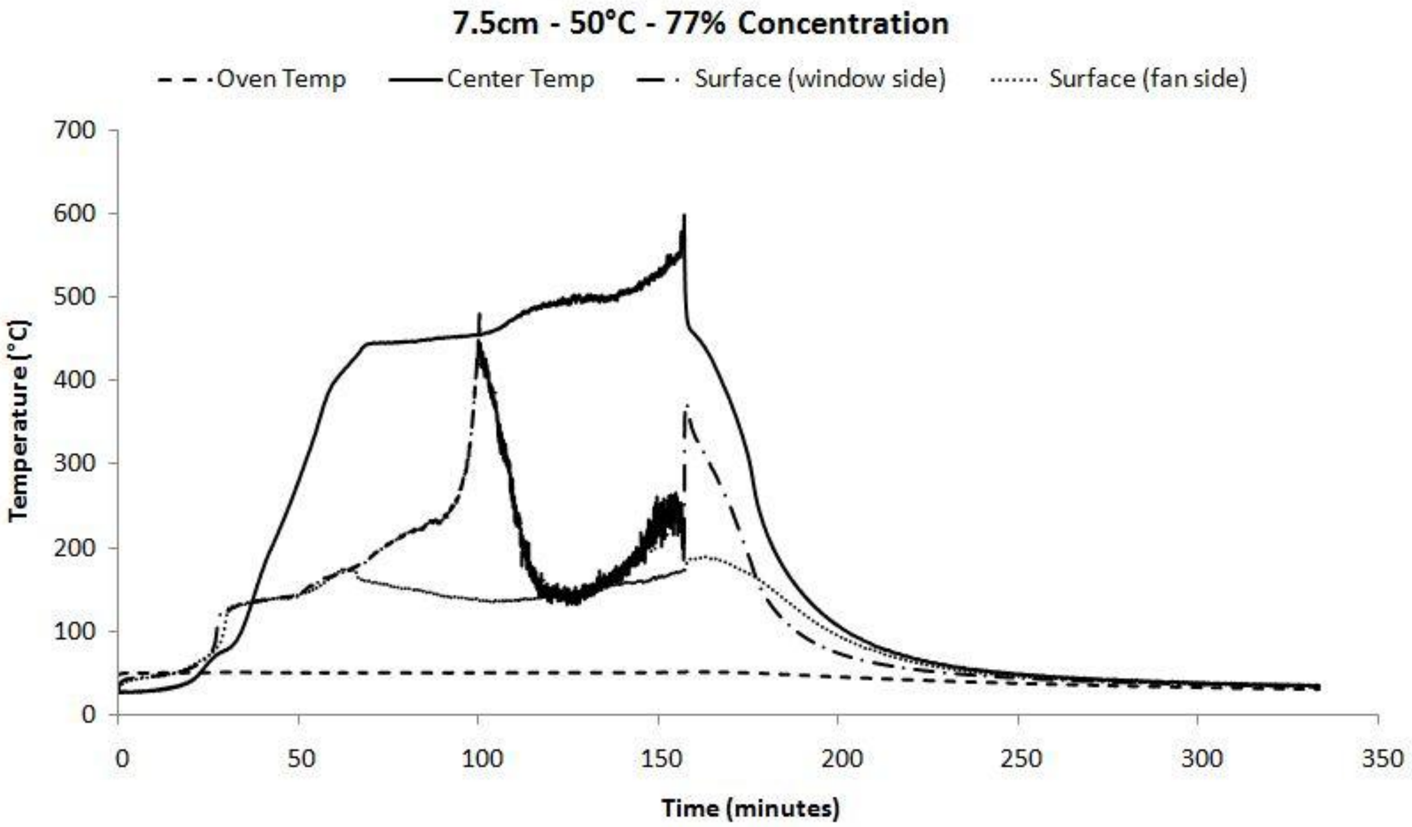


Figure 6.18: 7.5cm - 50°C – 77% Concentration

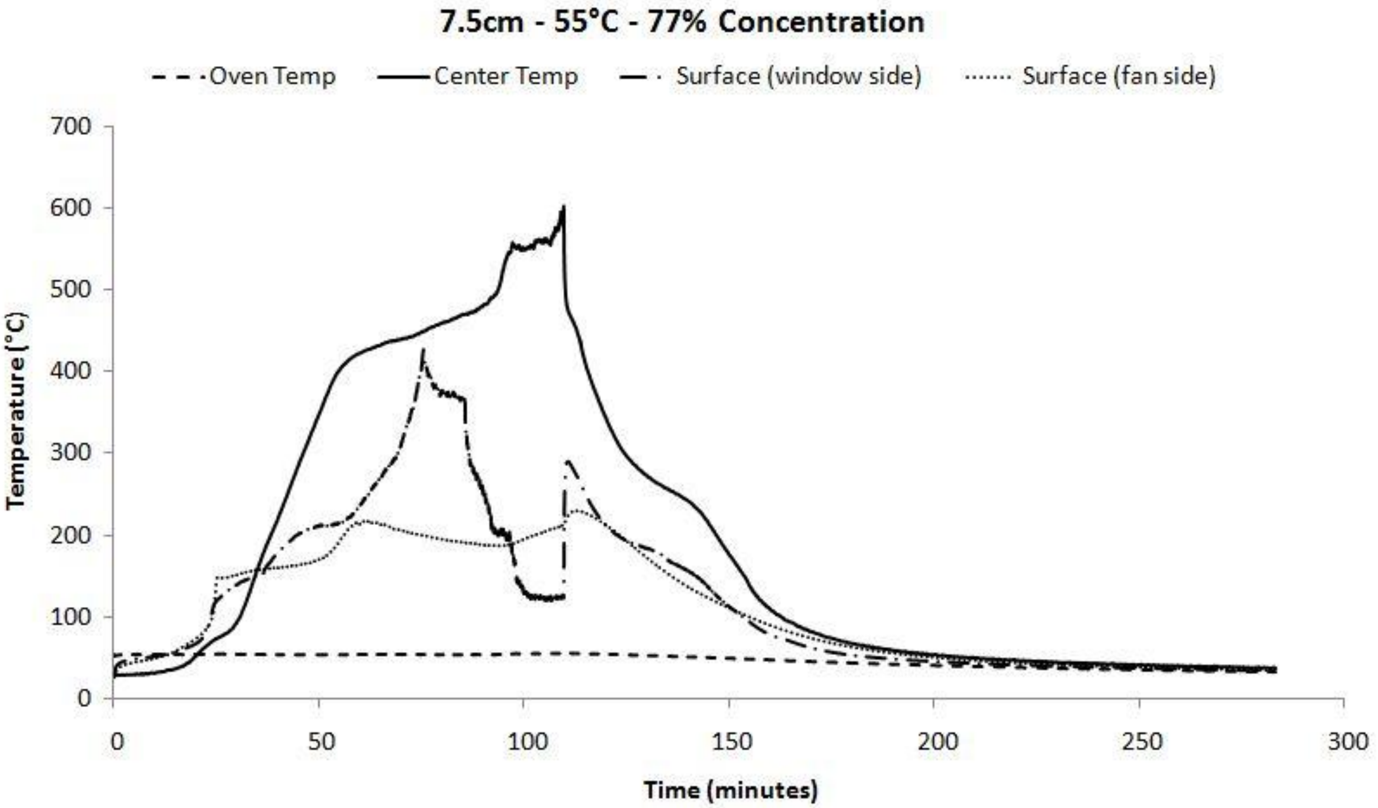


Figure 6.19: 7.5cm - 55°C – 77% Concentration

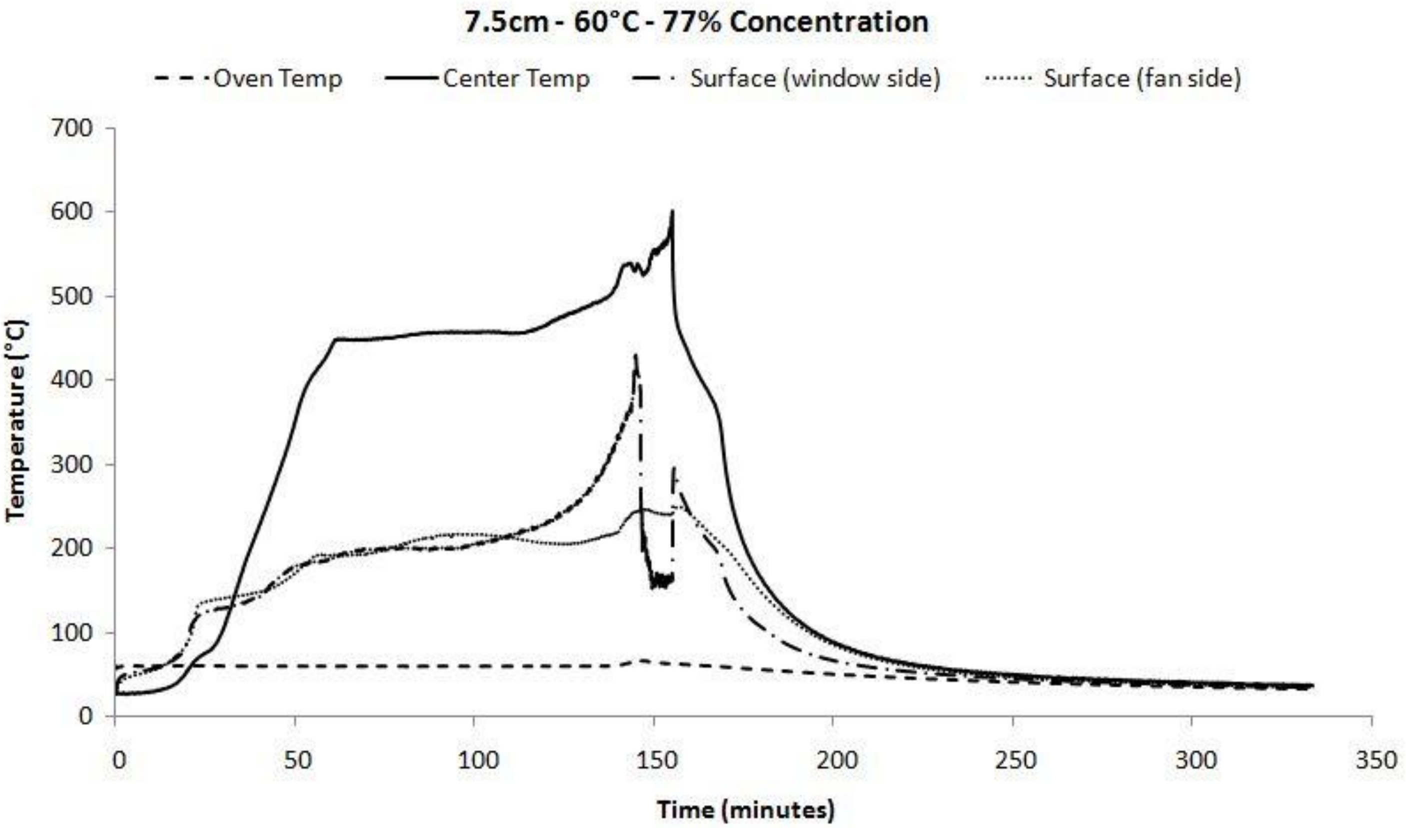


Figure 6.20: 7.5cm - 60°C – 77% Concentration

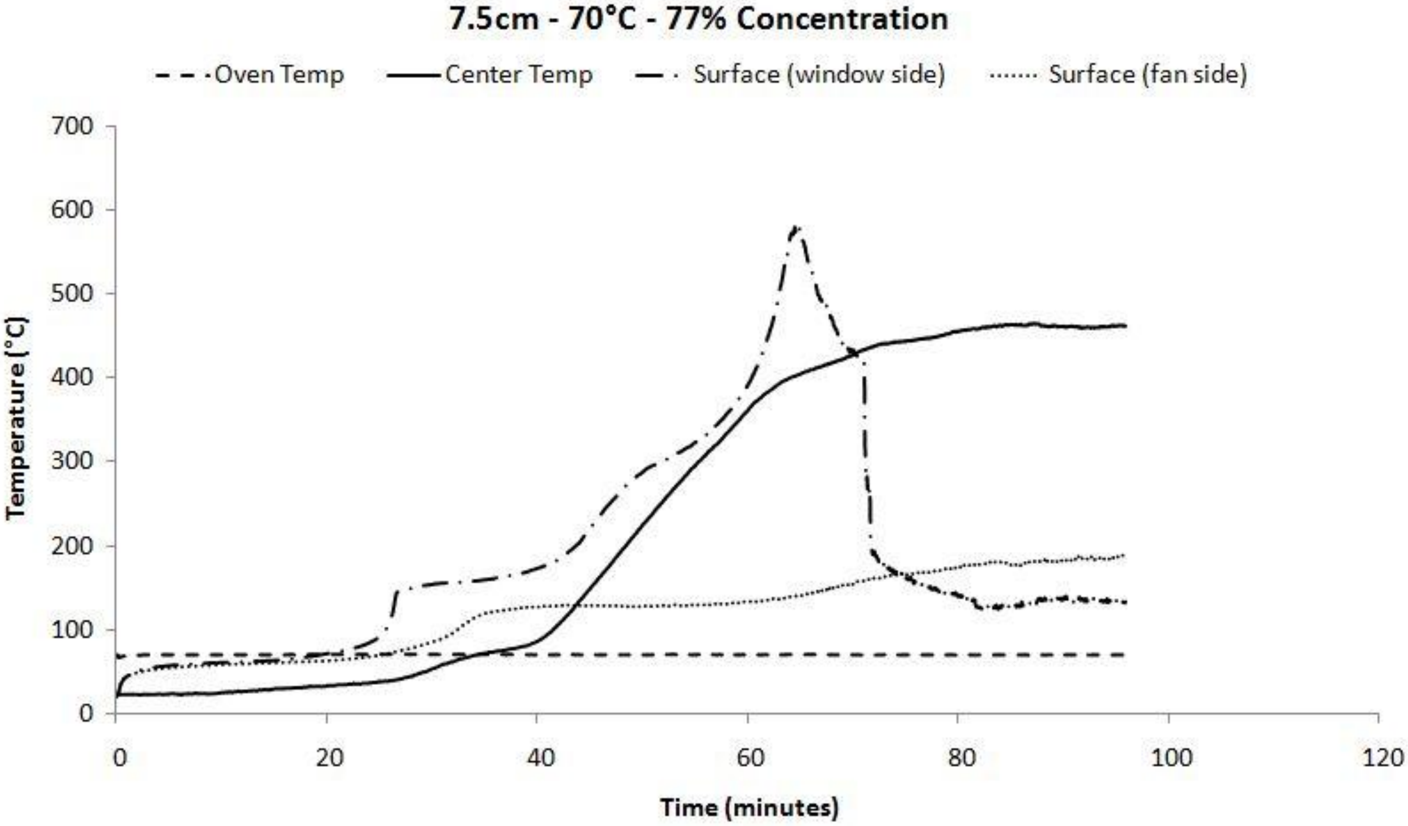


Figure 6.21: 7.5cm - 70°C – 77% Concentration

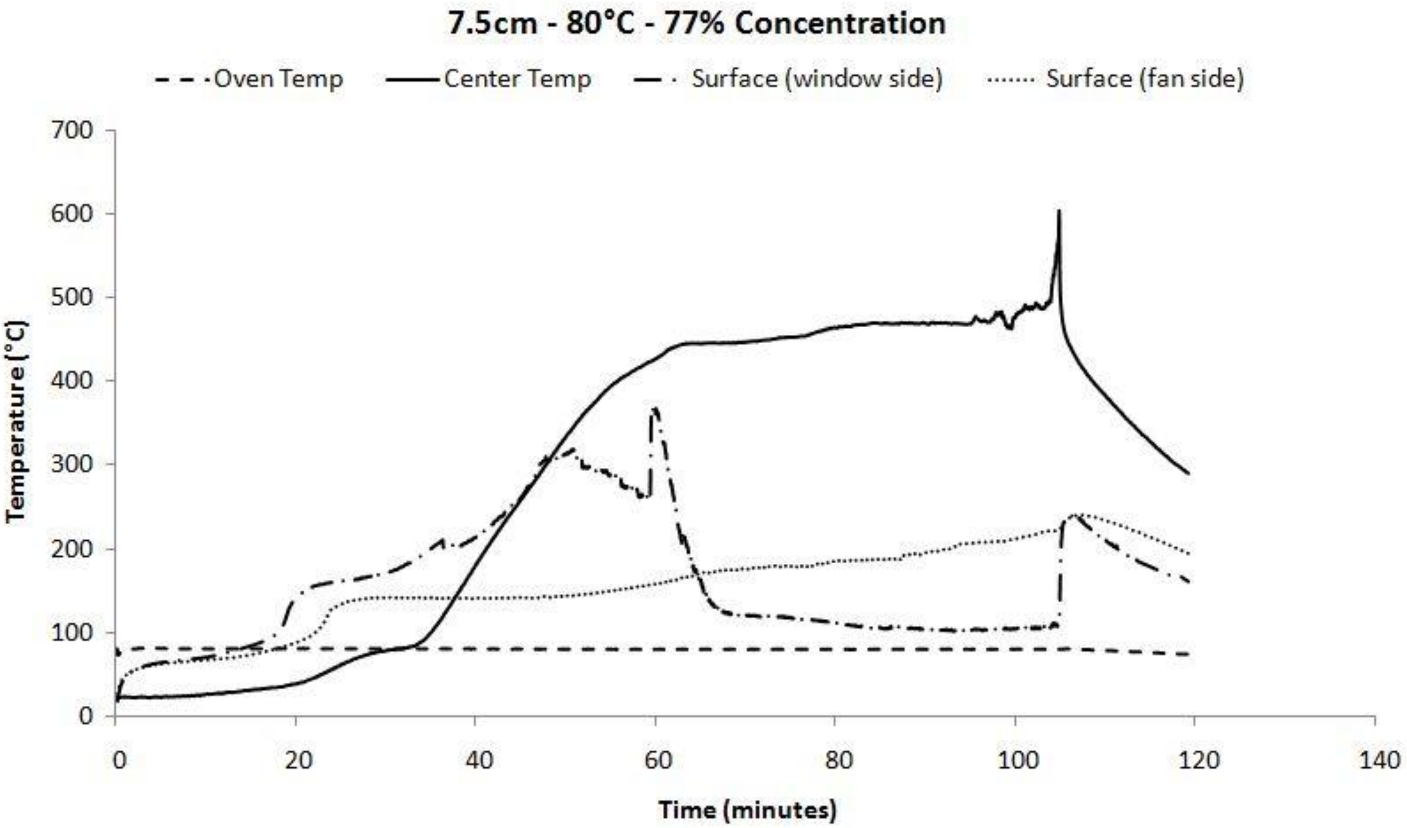


Figure 6.22: 7.5cm - 80°C – 77% Concentration

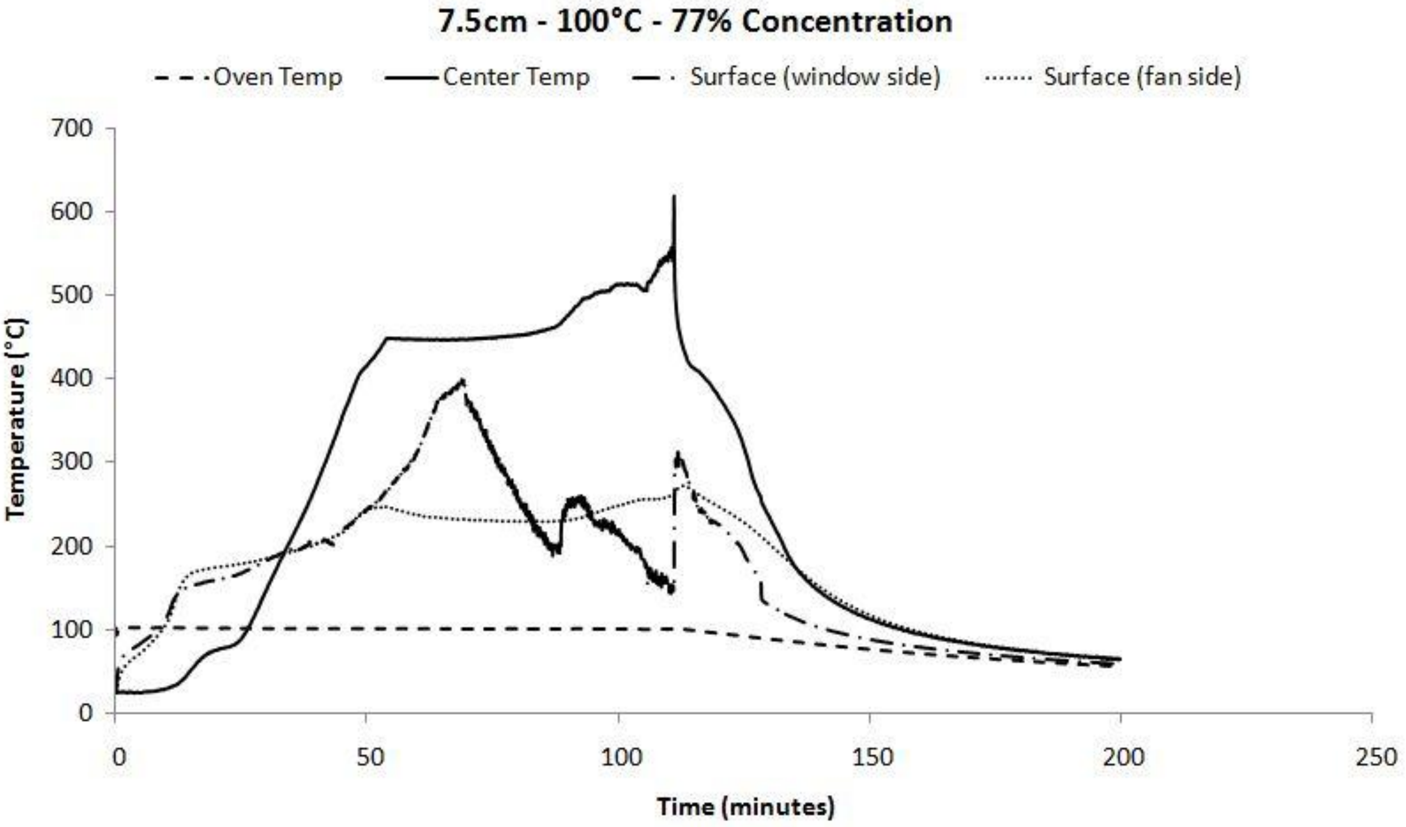


Figure 6.23: 7.5cm - 100°C – 77% Concentration

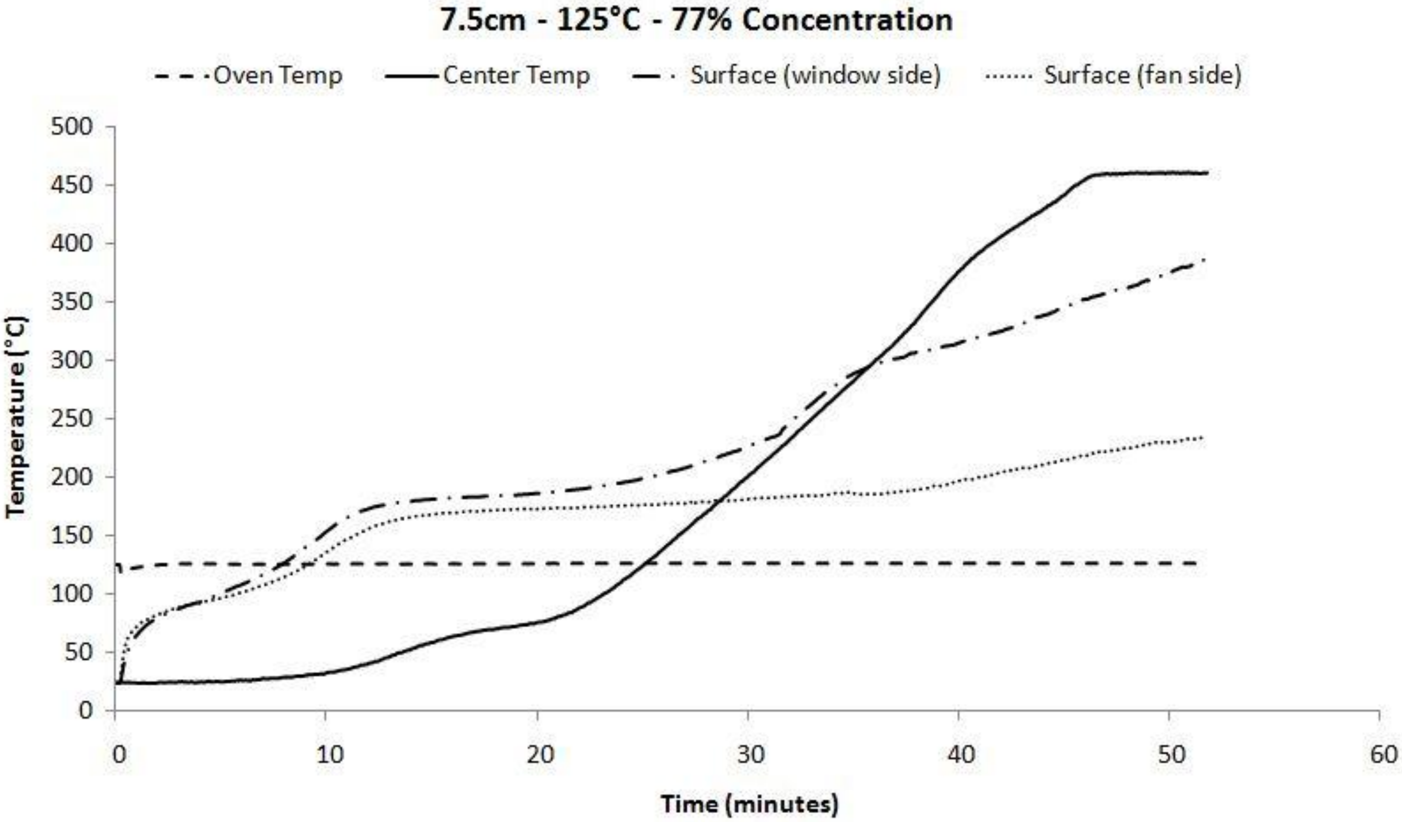


Figure 6.24: 7.5cm - 125°C – 77% Concentration

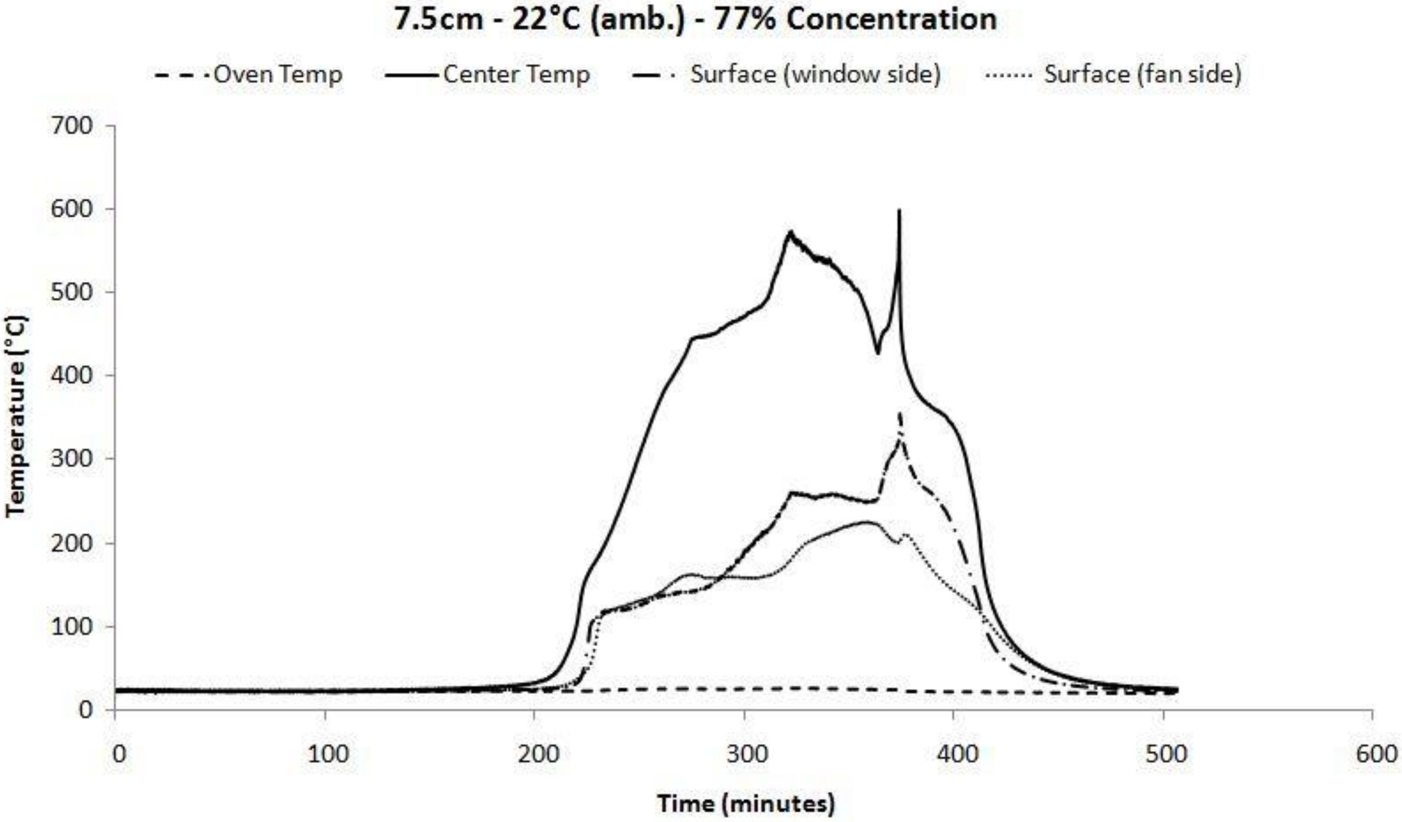


Figure 6.25: 7.5cm - 22°C – 77% Concentration

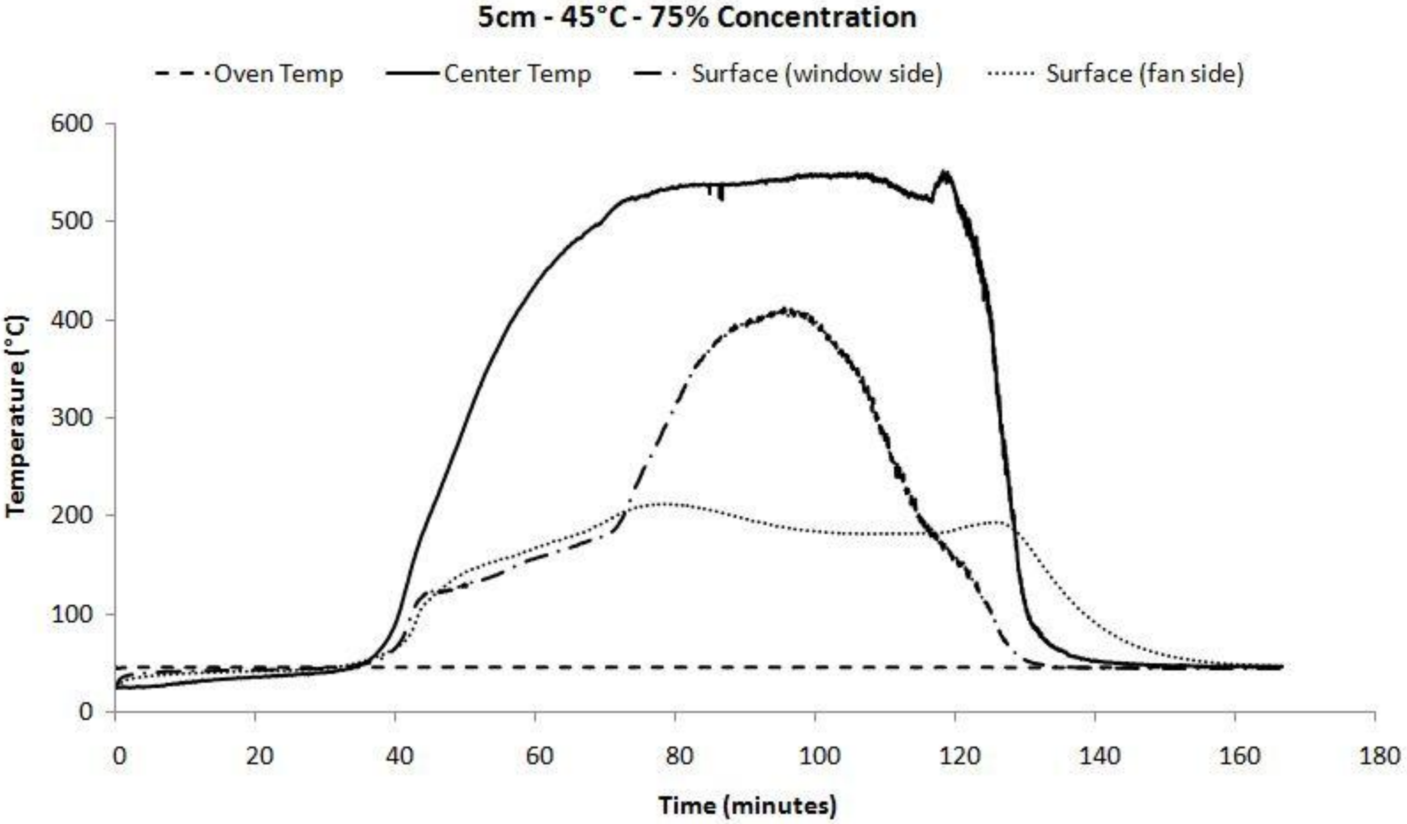


Figure 6.26: 5cm - 45°C – 75% Concentration

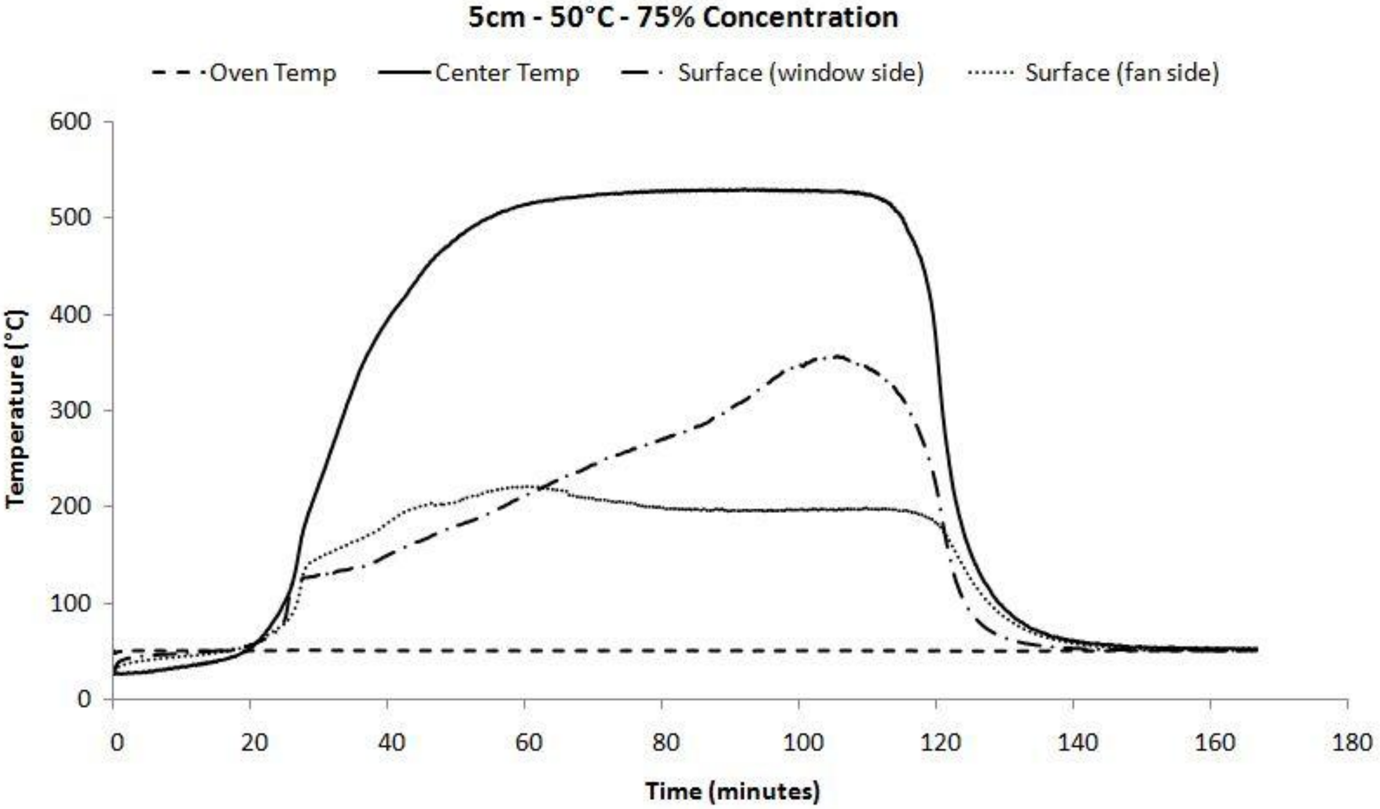


Figure 6.27: 5cm - 50°C – 75% Concentration

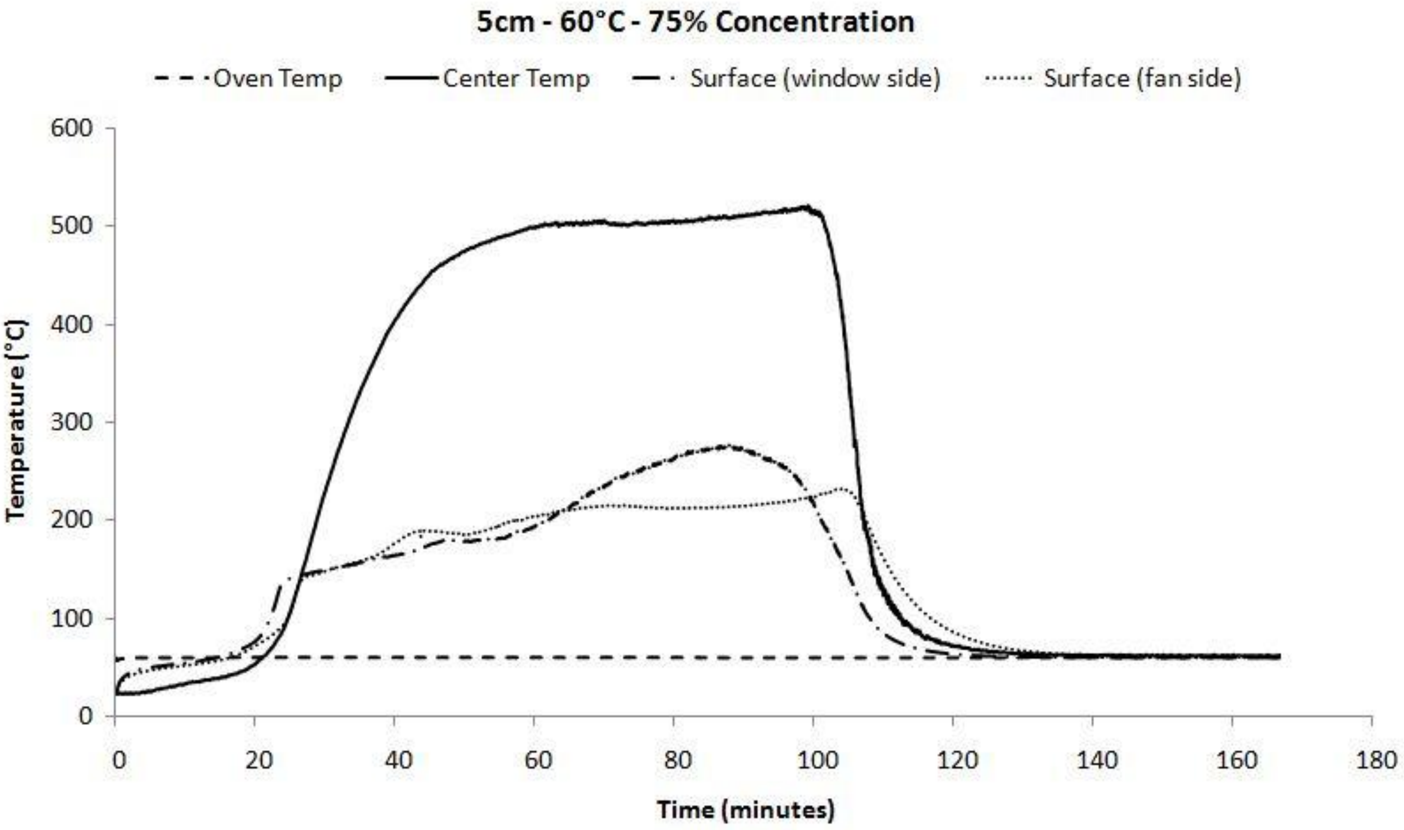


Figure 6.28: 5cm - 60°C – 75% Concentration

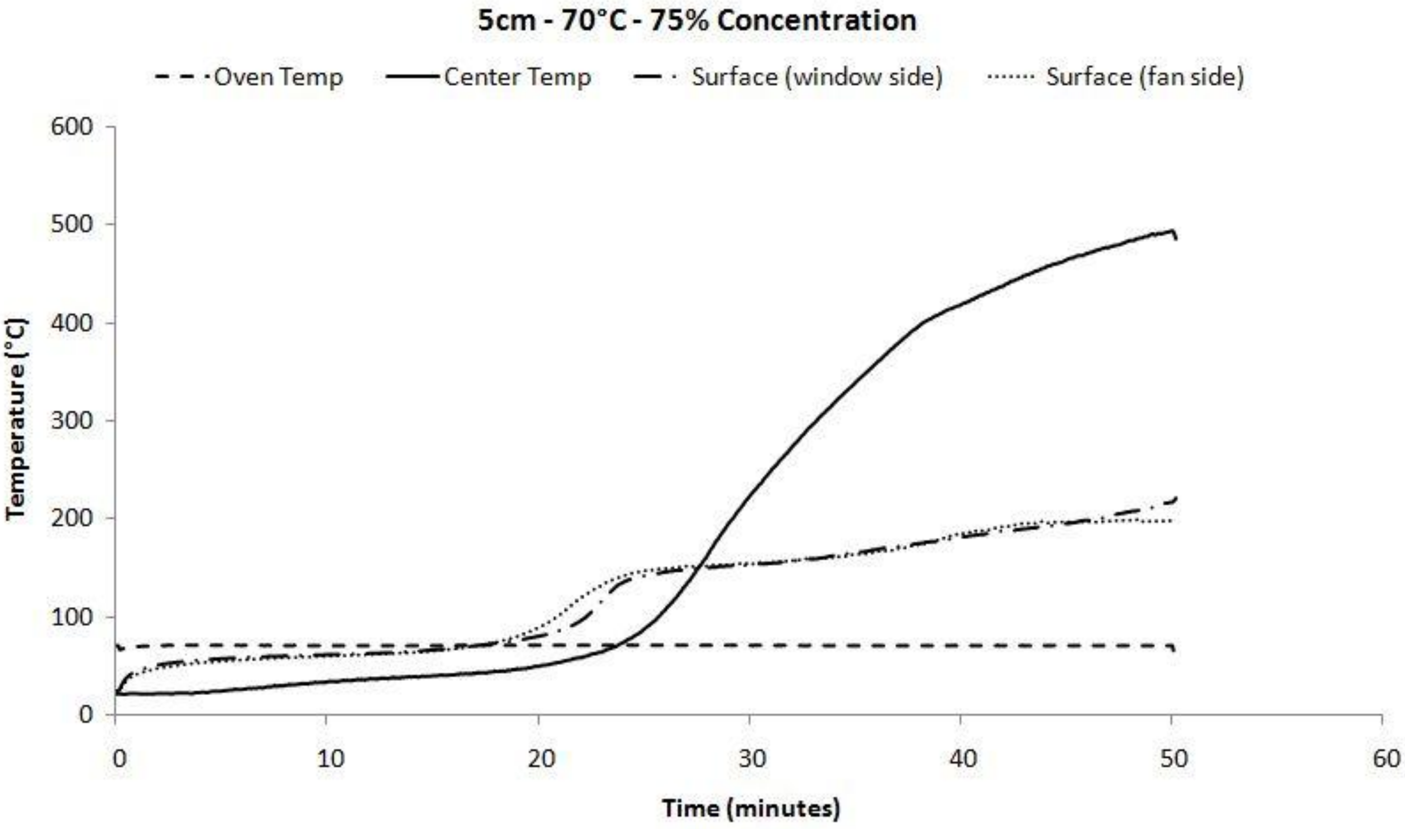


Figure 6.29: 5cm - 70°C – 75% Concentration

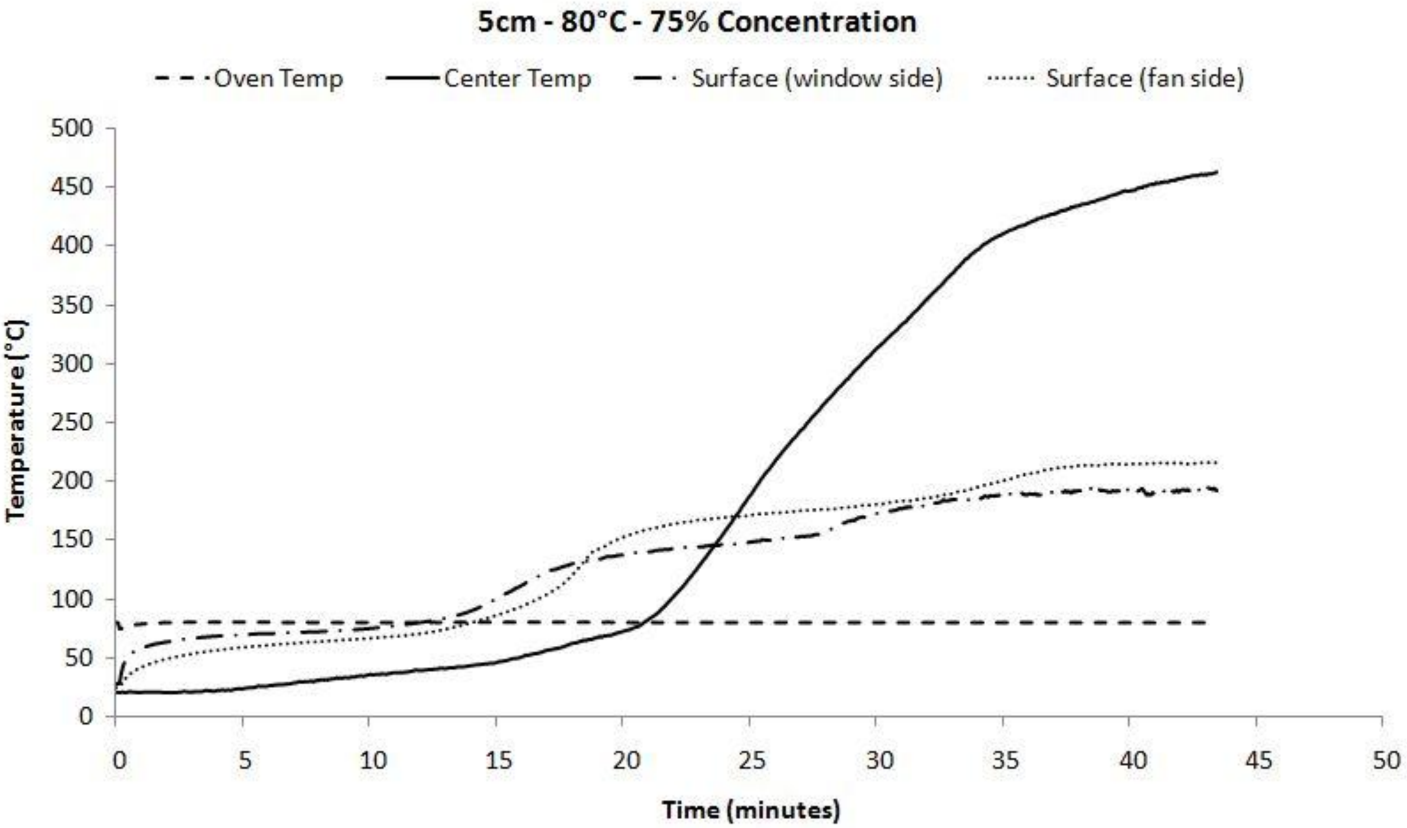


Figure 6.30: 5cm - 80°C – 75% Concentration

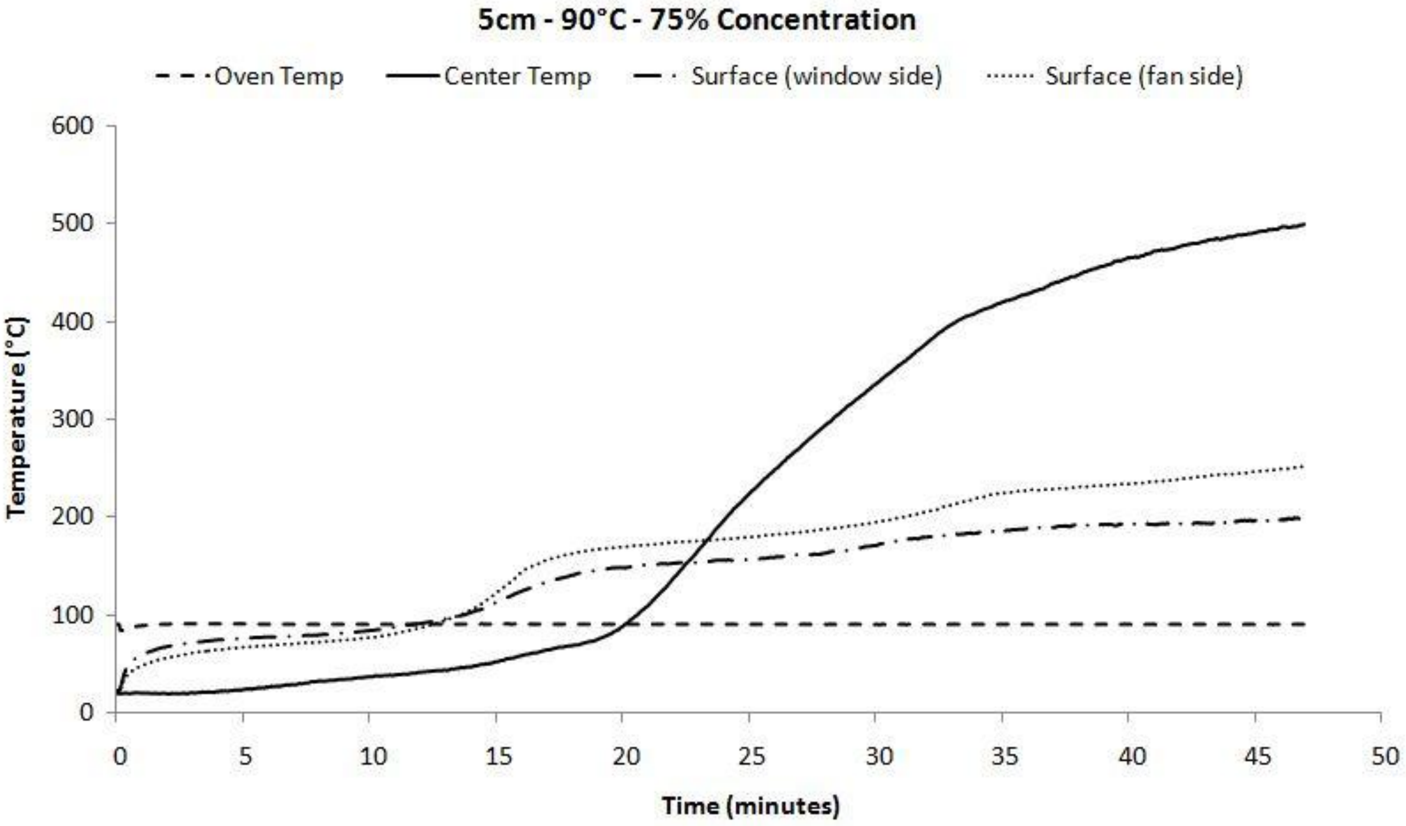


Figure 6.31: 5cm - 90°C – 75% Concentration

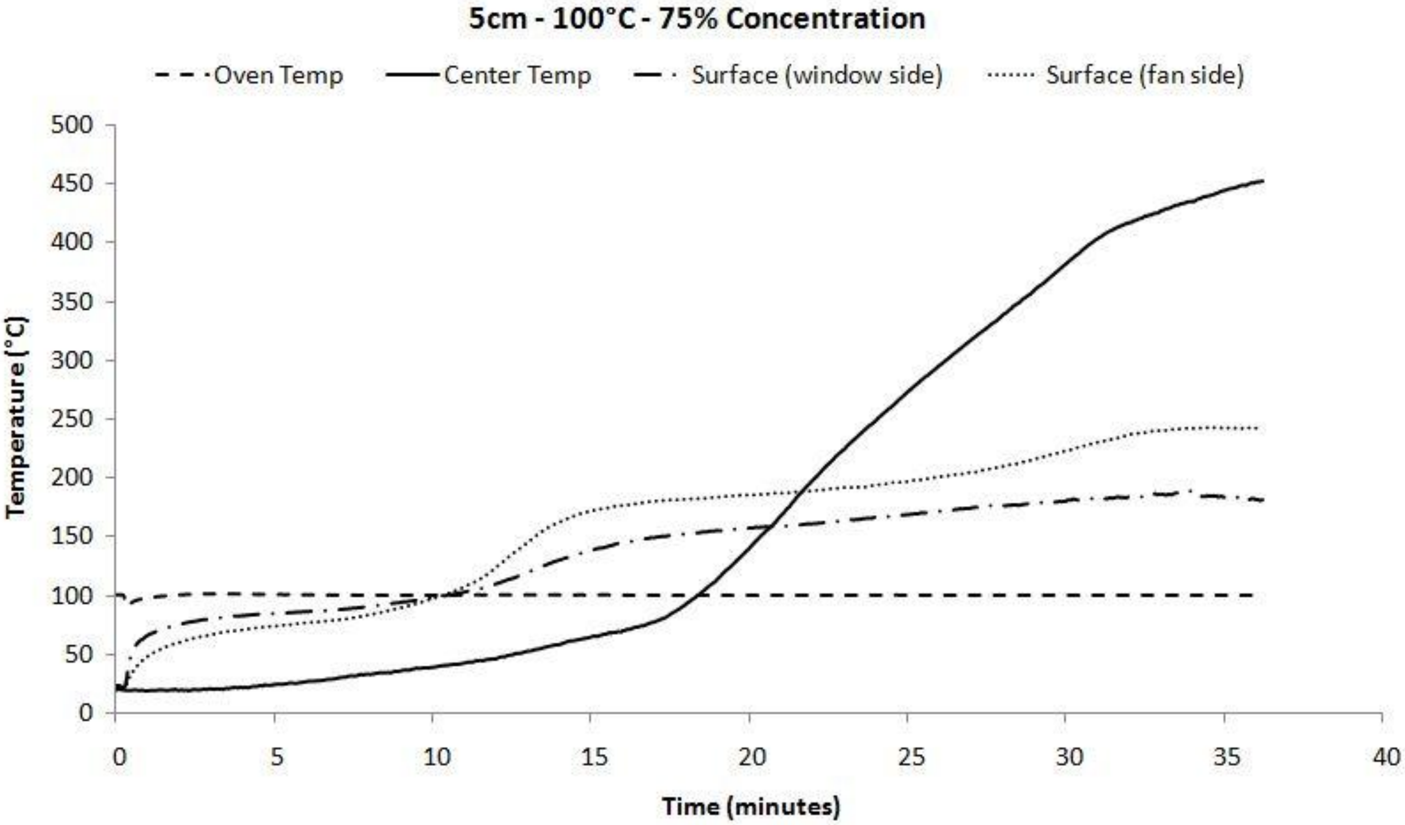


Figure 6.32: 5cm - 100°C – 75% Concentration

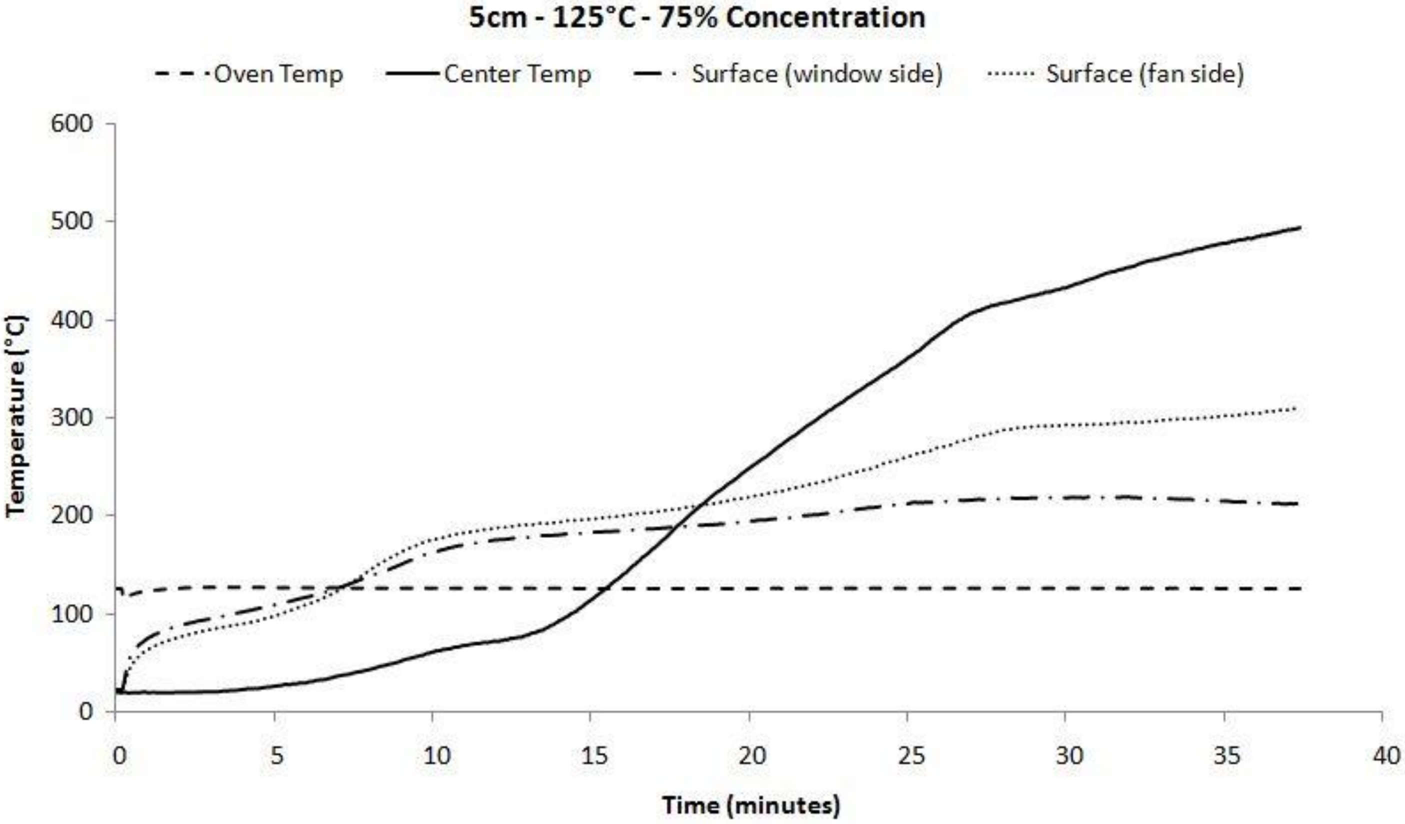


Figure 6.33: 5cm - 125°C – 75% Concentration

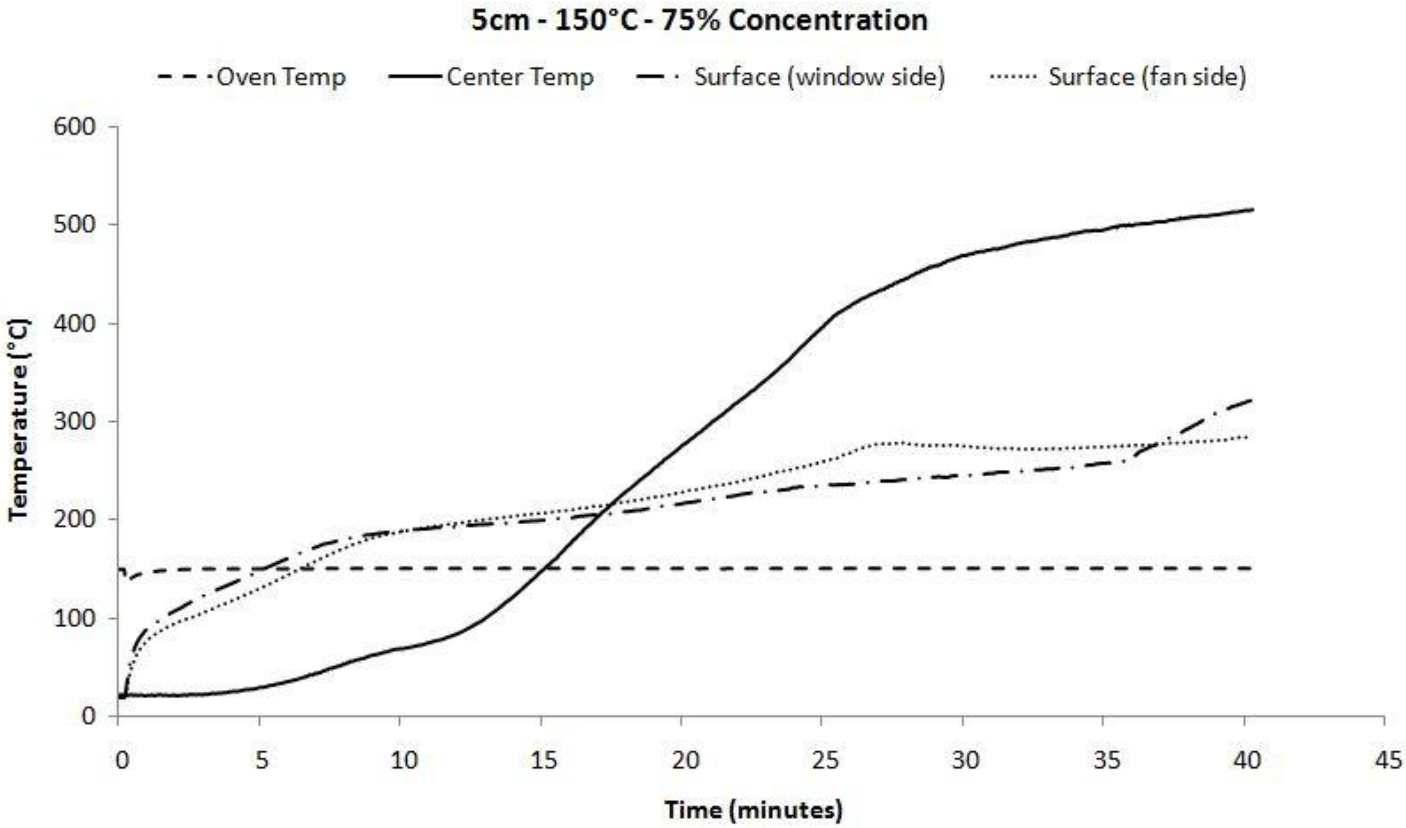


Figure 6.34: 5cm - 150°C – 75% Concentration

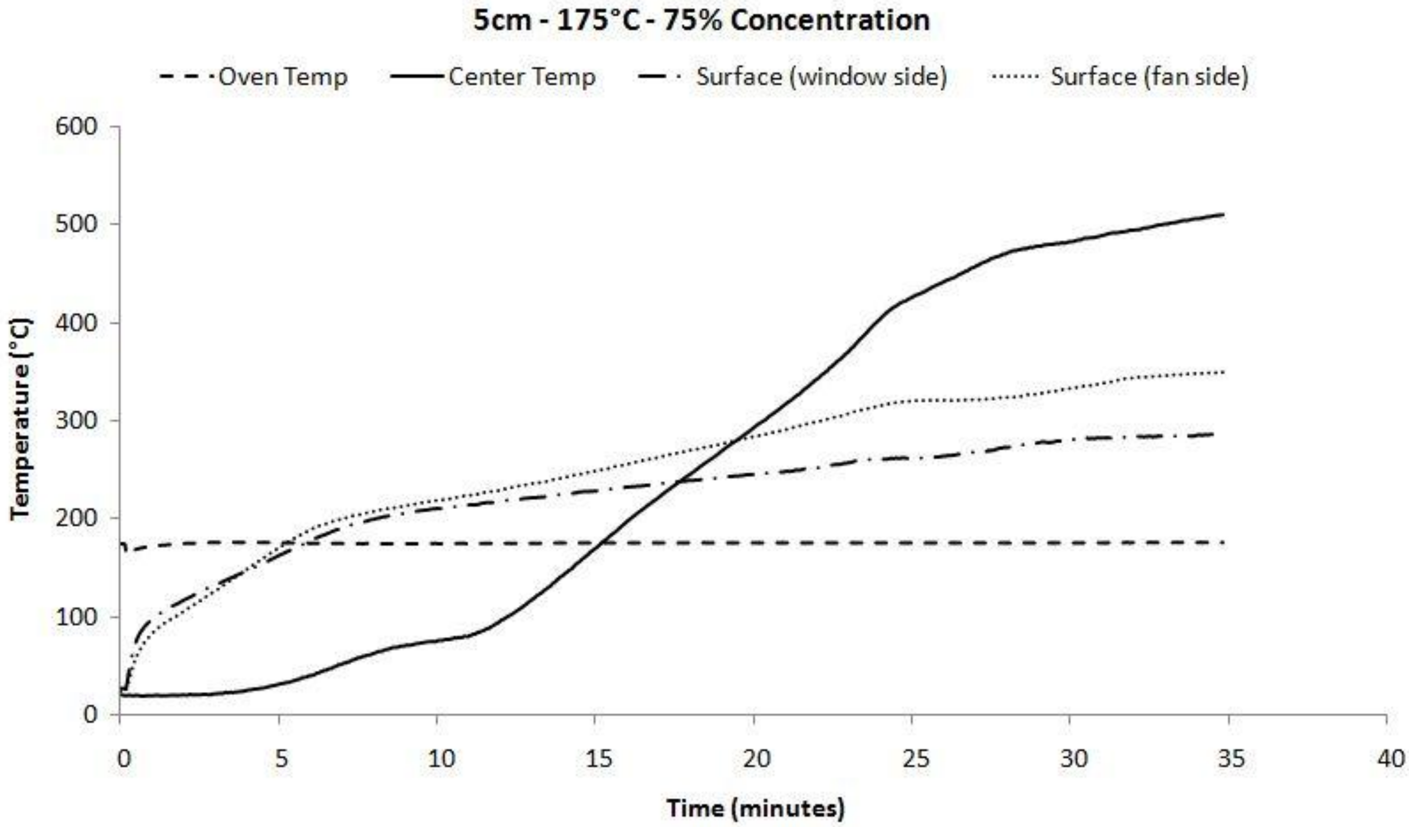


Figure 6.35: 5cm - 175°C – 75% Concentration

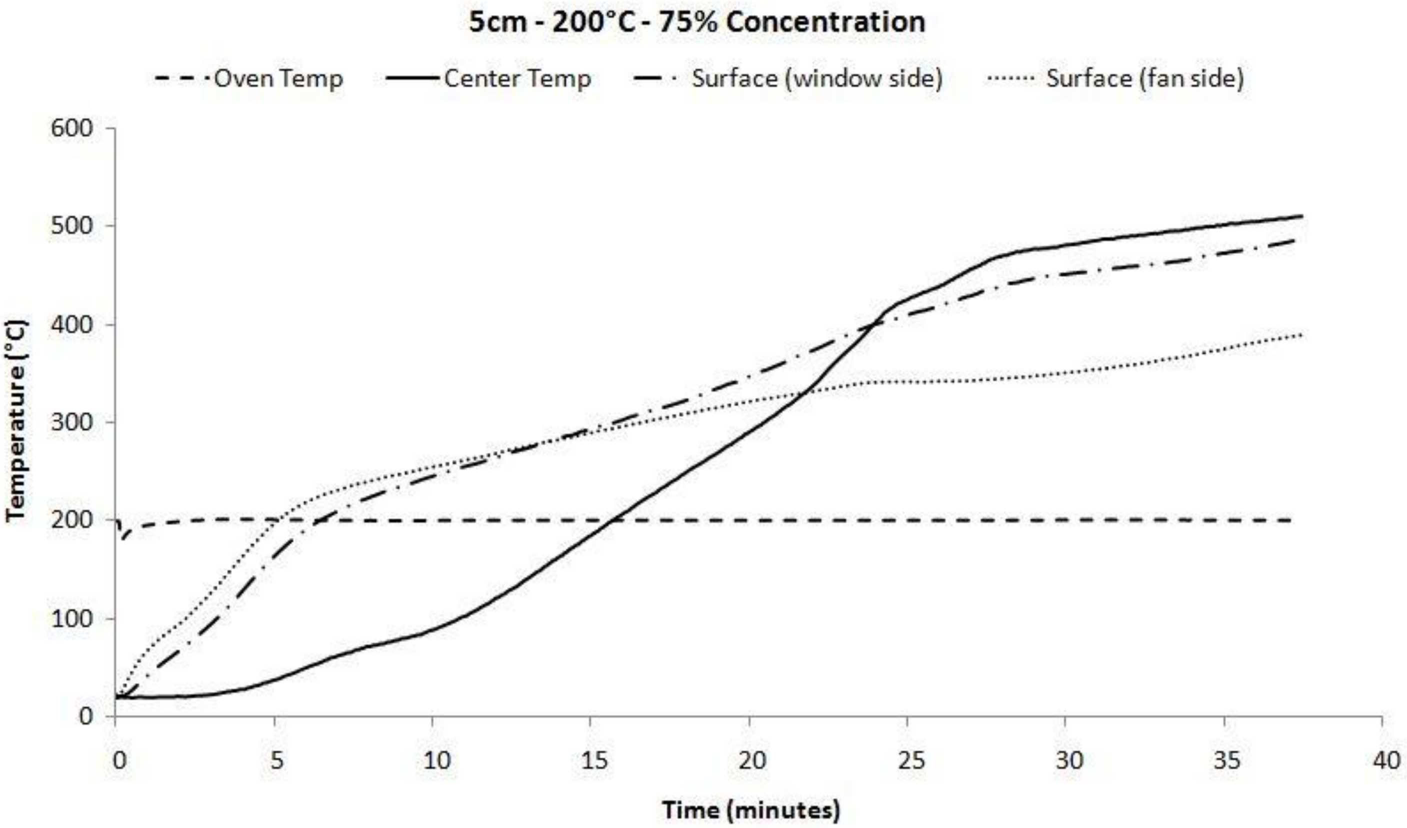


Figure 6.36: 5cm - 200°C – 75% Concentration

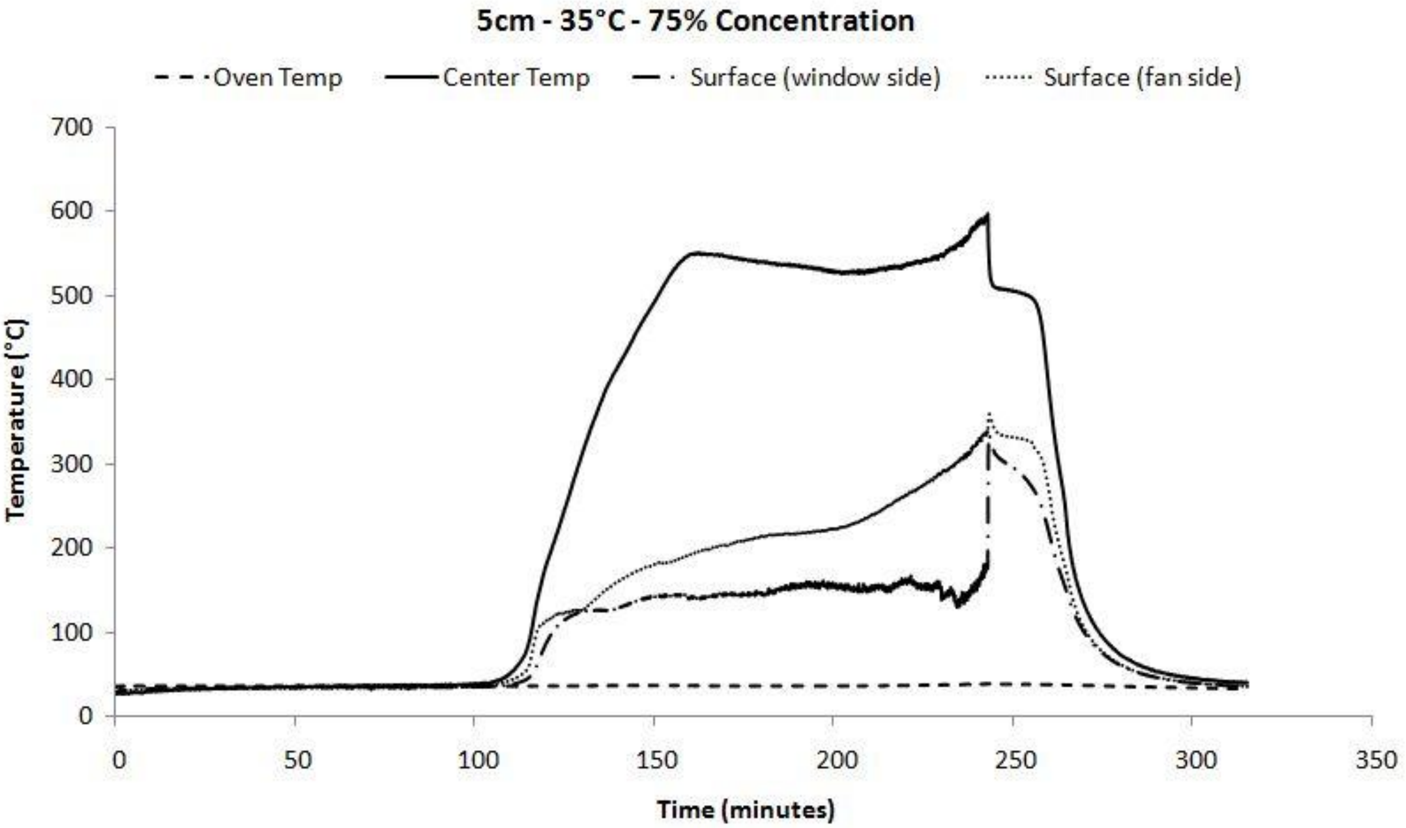


Figure 6.37: 5cm - 35°C – 75% Concentration

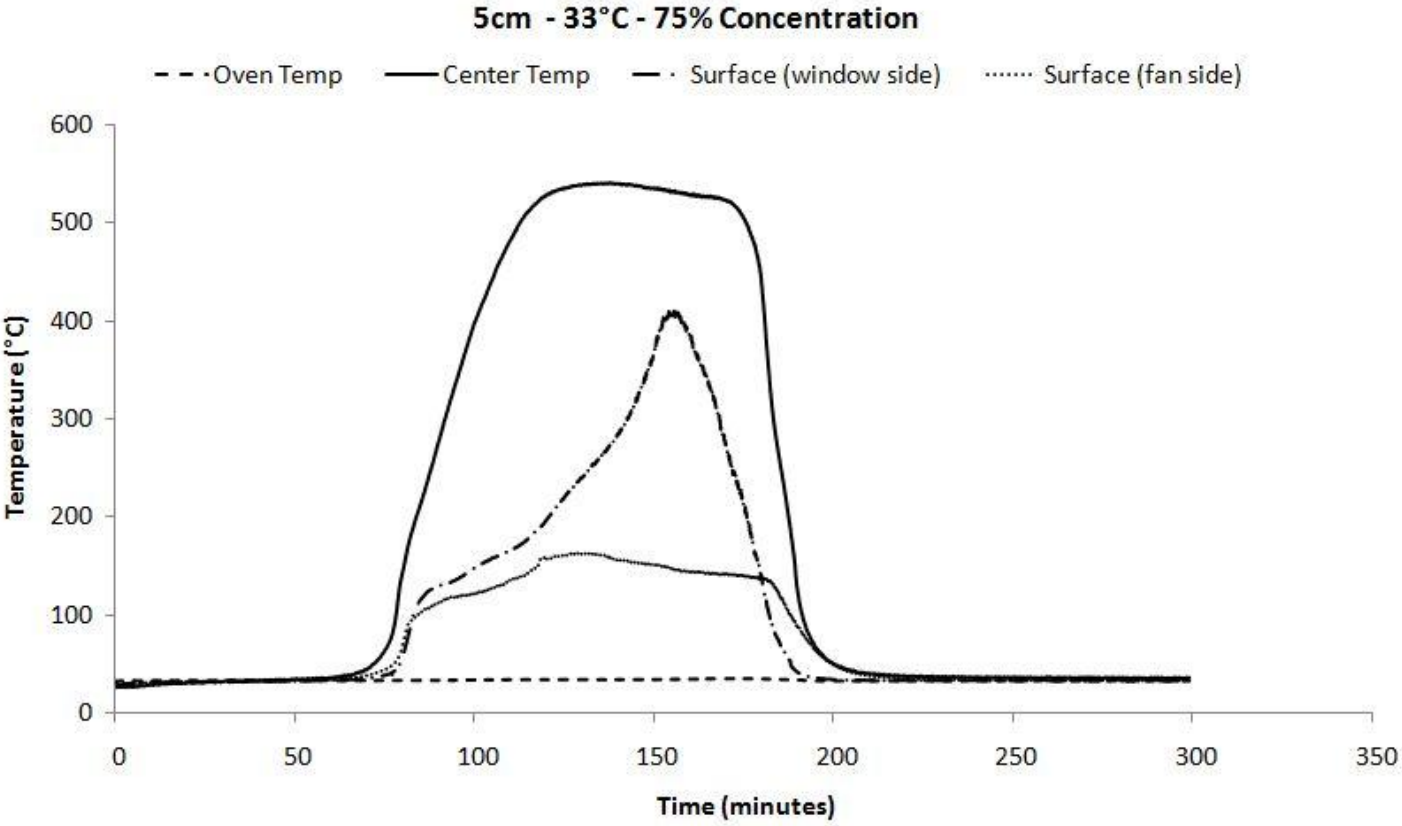


Figure 6.38: 5cm - 33°C – 75% Concentration

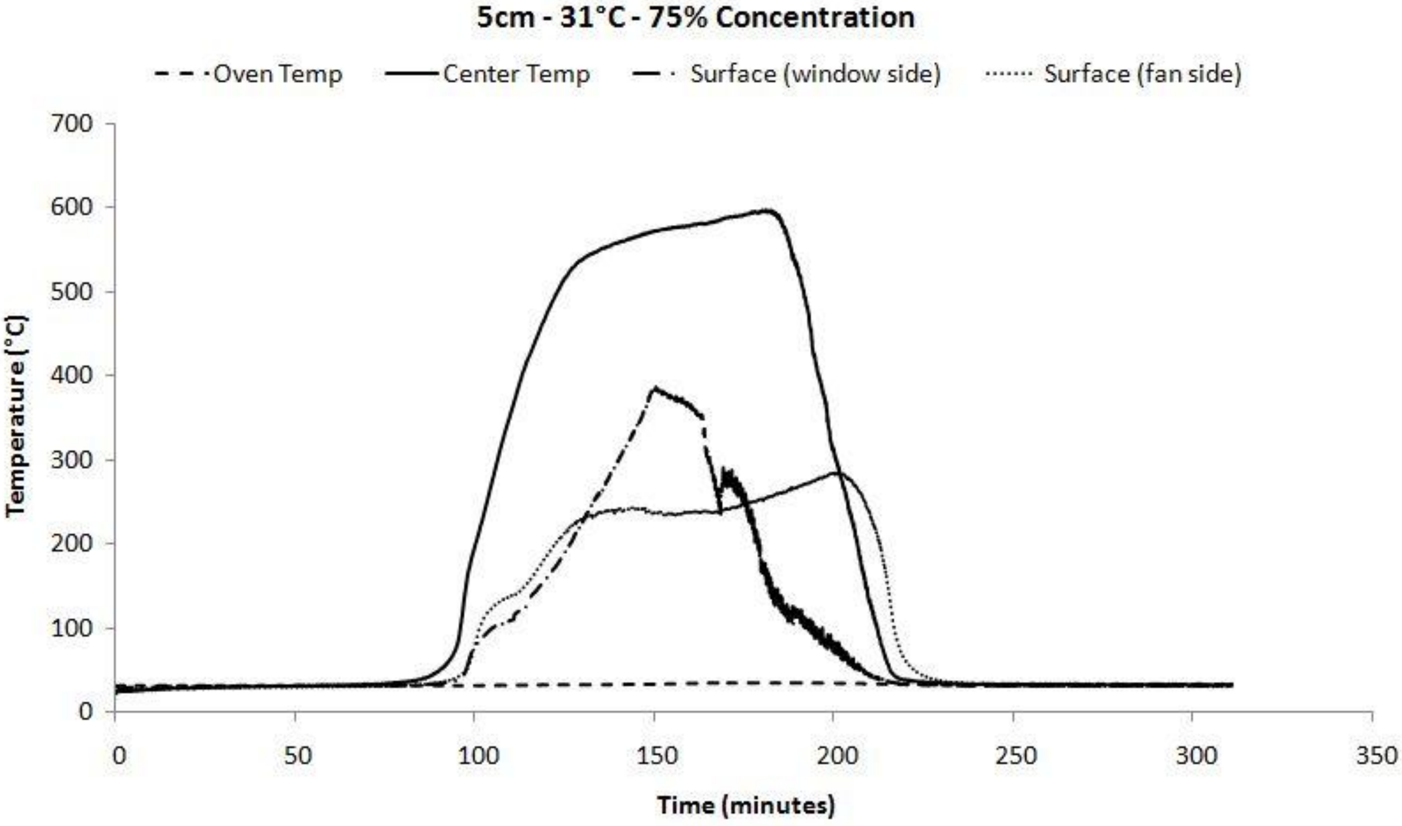


Figure 6.39: 5cm - 31°C – 75% Concentration

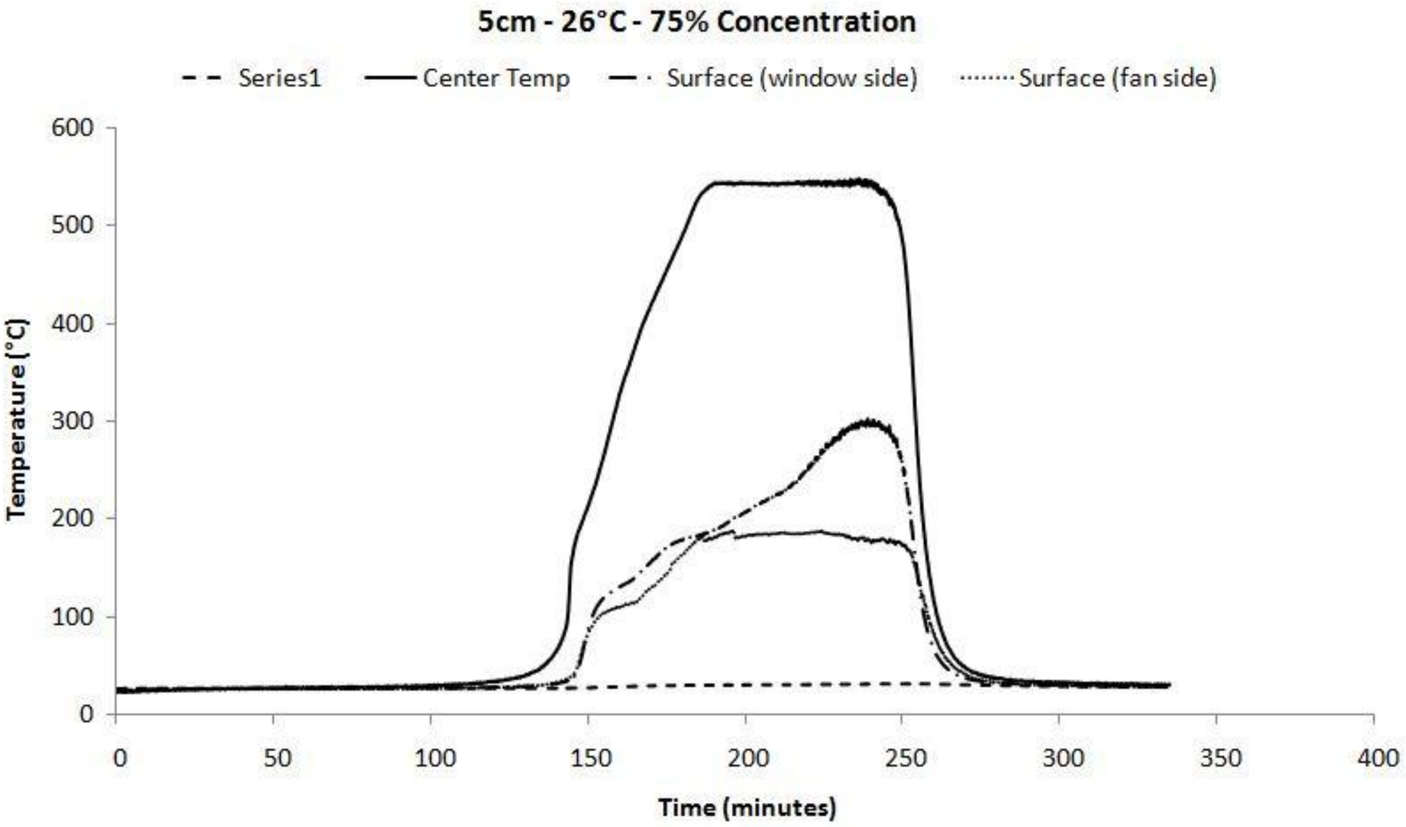


Figure 6.40: 5cm - 26°C – 75% Concentration

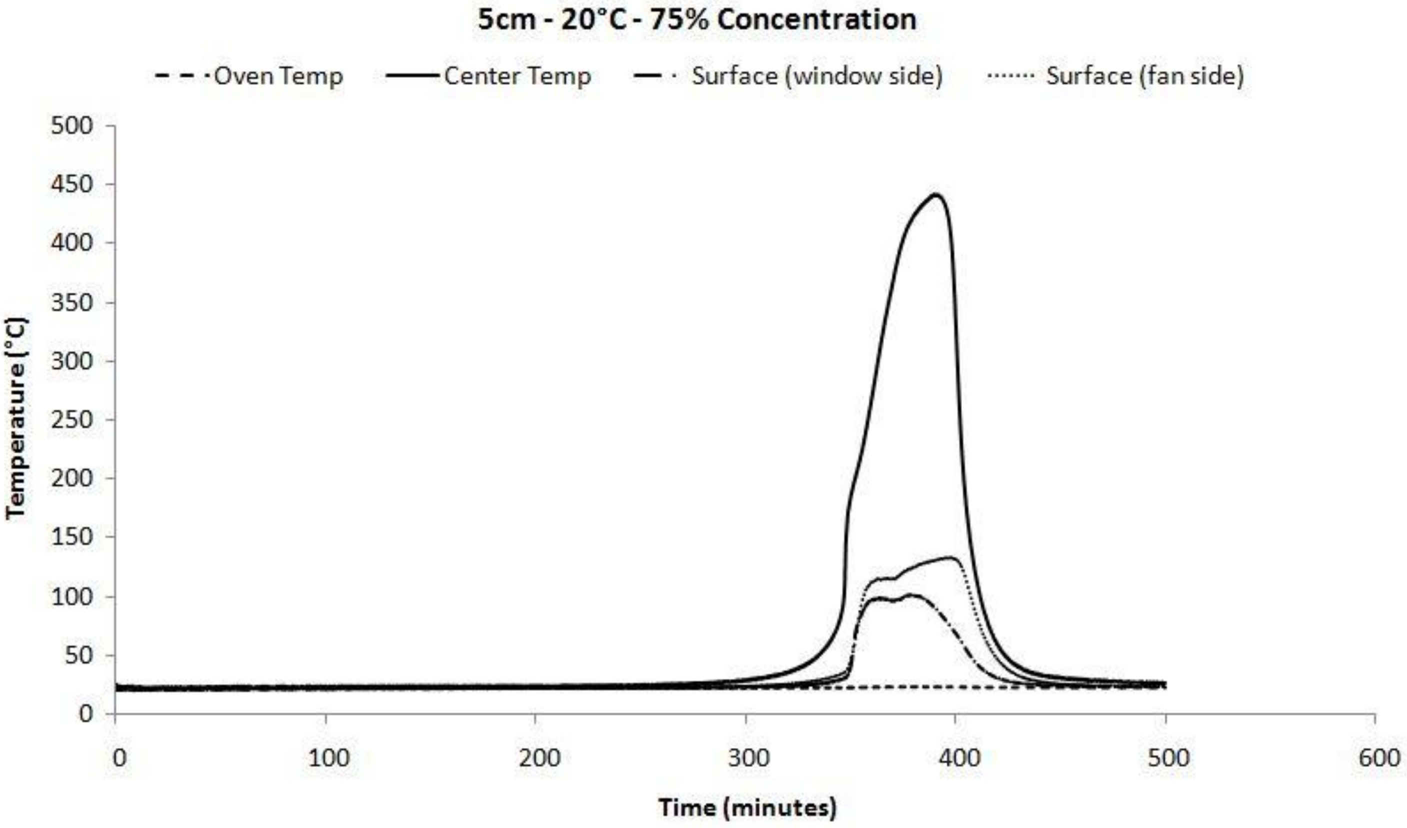


Figure 6.41: 5cm - 20°C – 75% Concentration

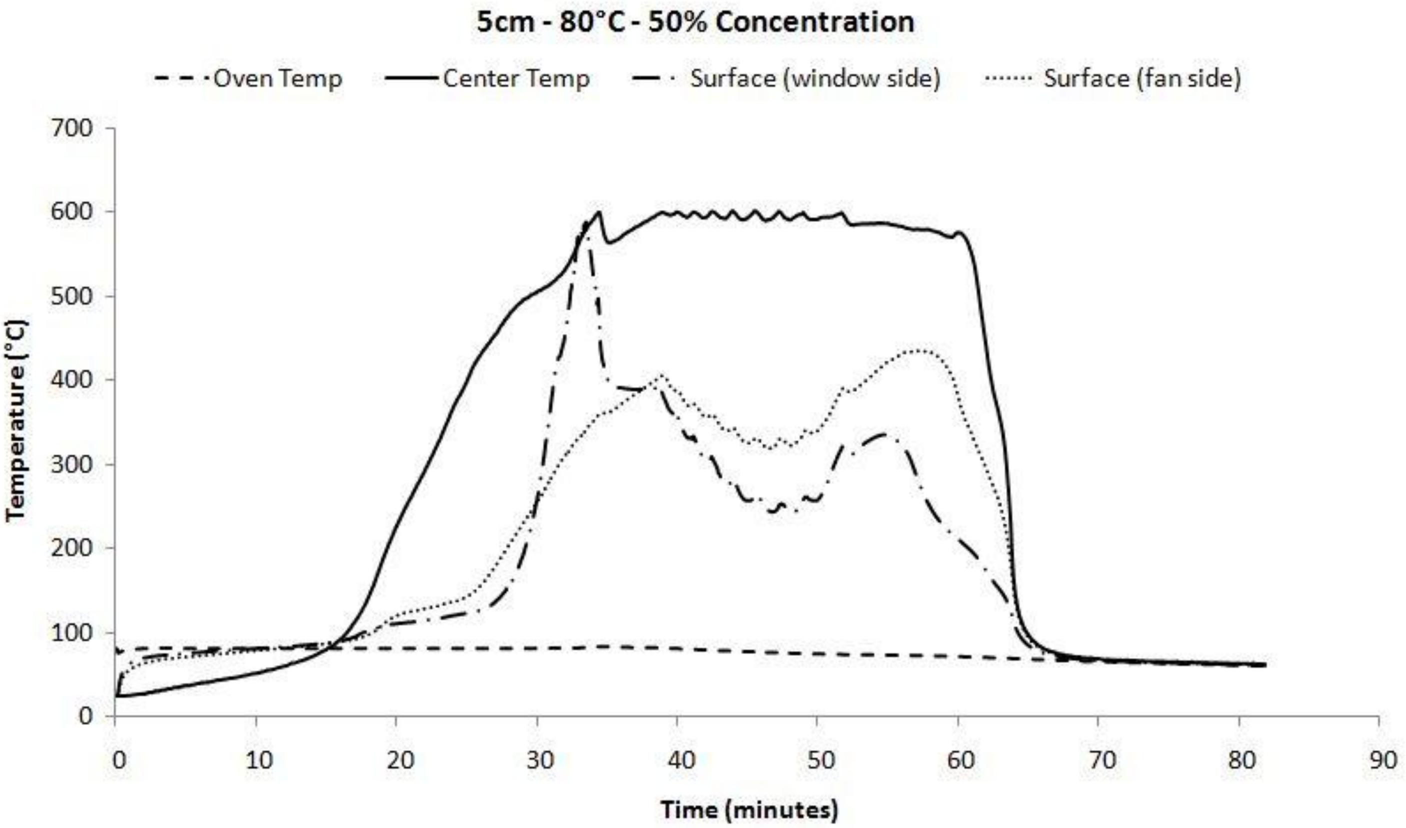


Figure 6.42: 5cm - 80°C – 50% Concentration

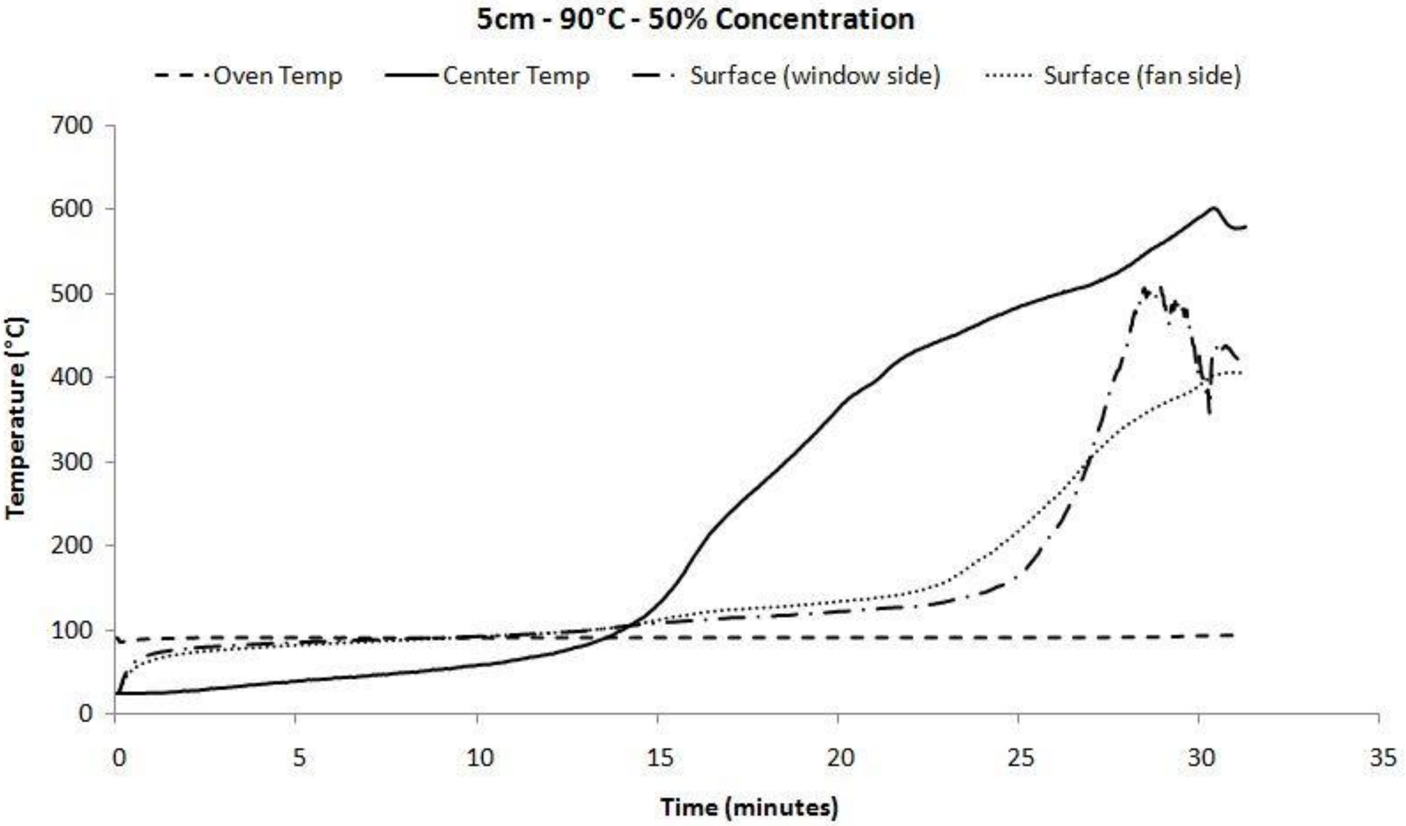


Figure 6.43: 5cm - 90°C – 50% Concentration

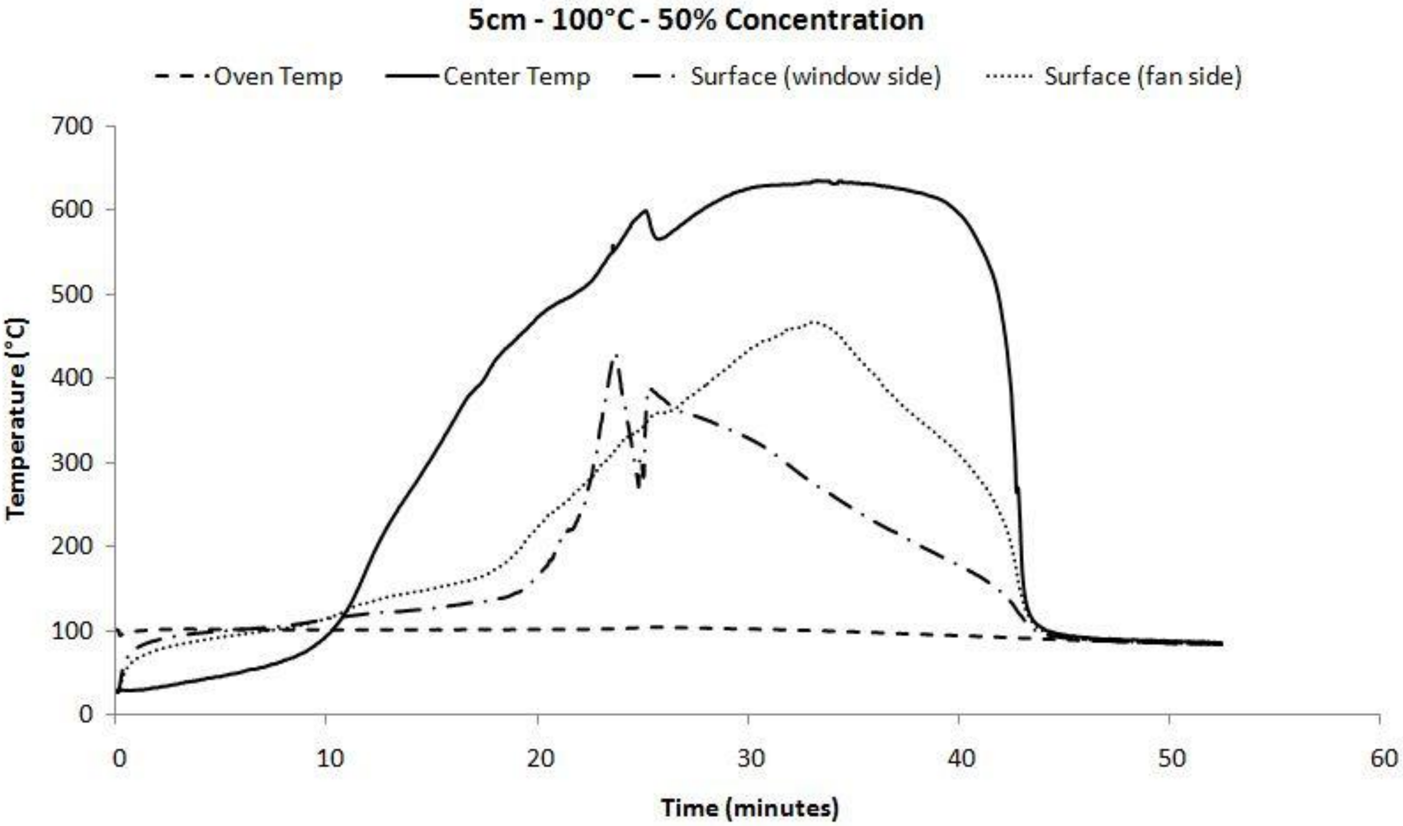


Figure 6.44: 5cm - 100°C – 50% Concentration

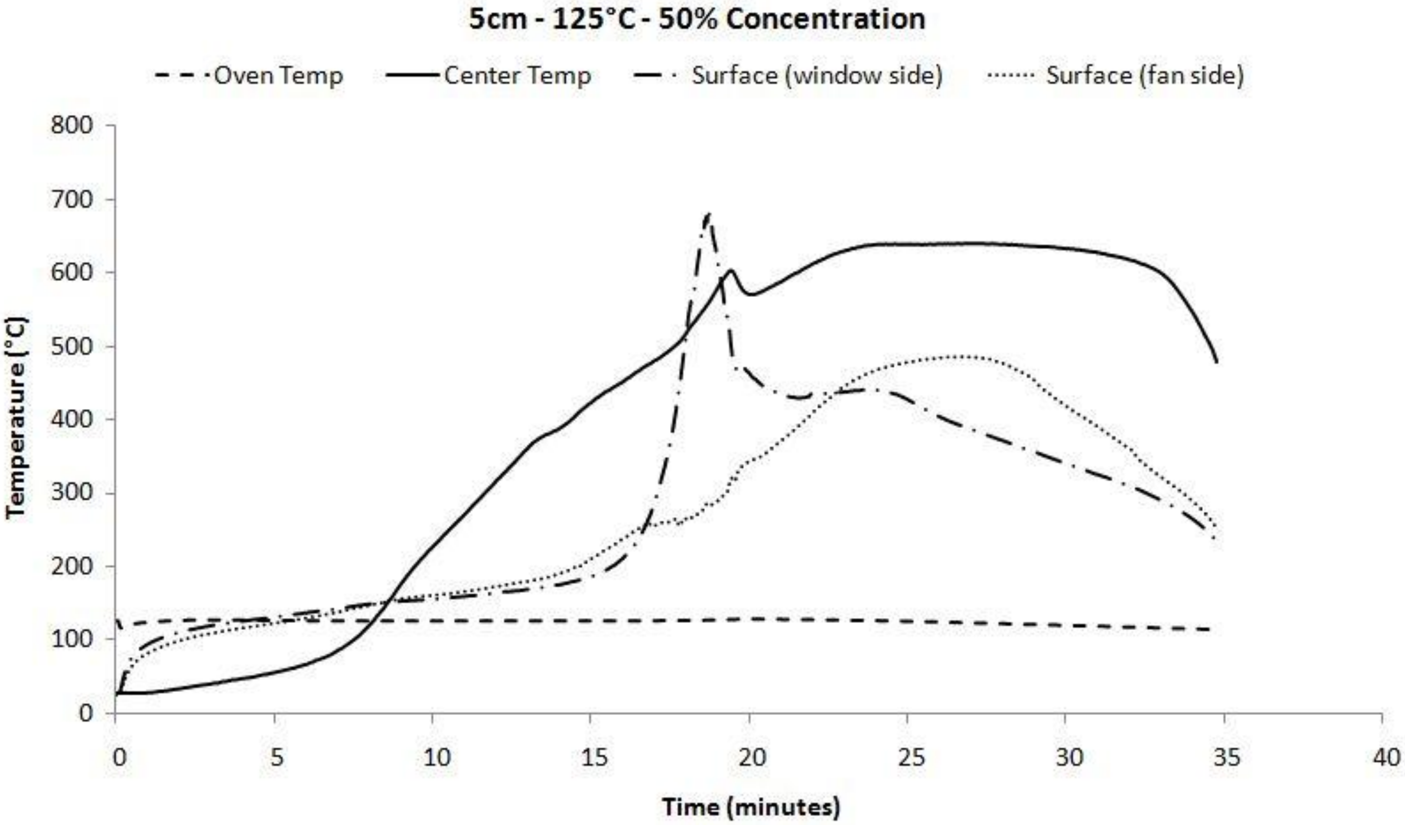


Figure 6.45: 5cm - 125°C – 50% Concentration

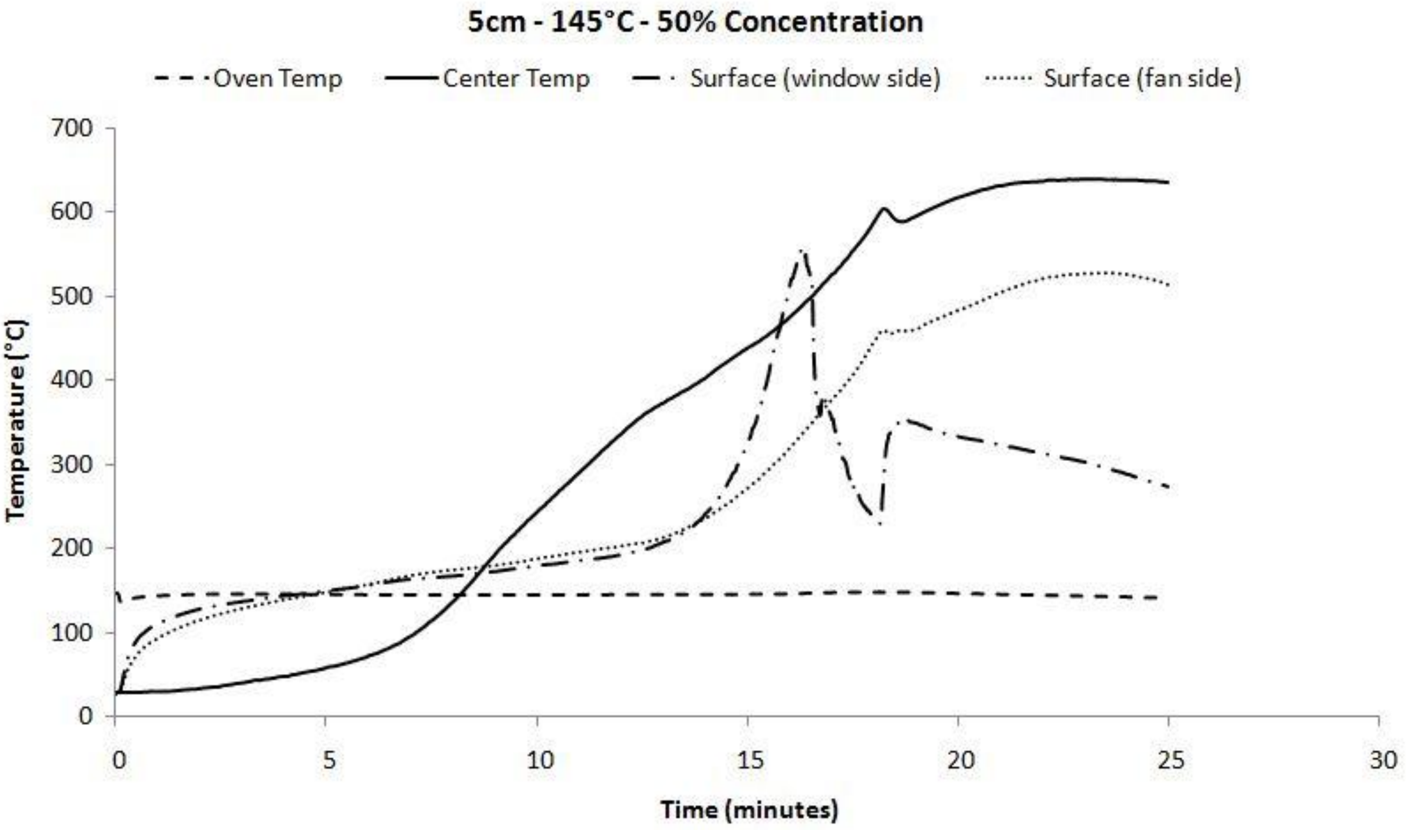


Figure 6.46: 5cm - 145°C – 50% Concentration

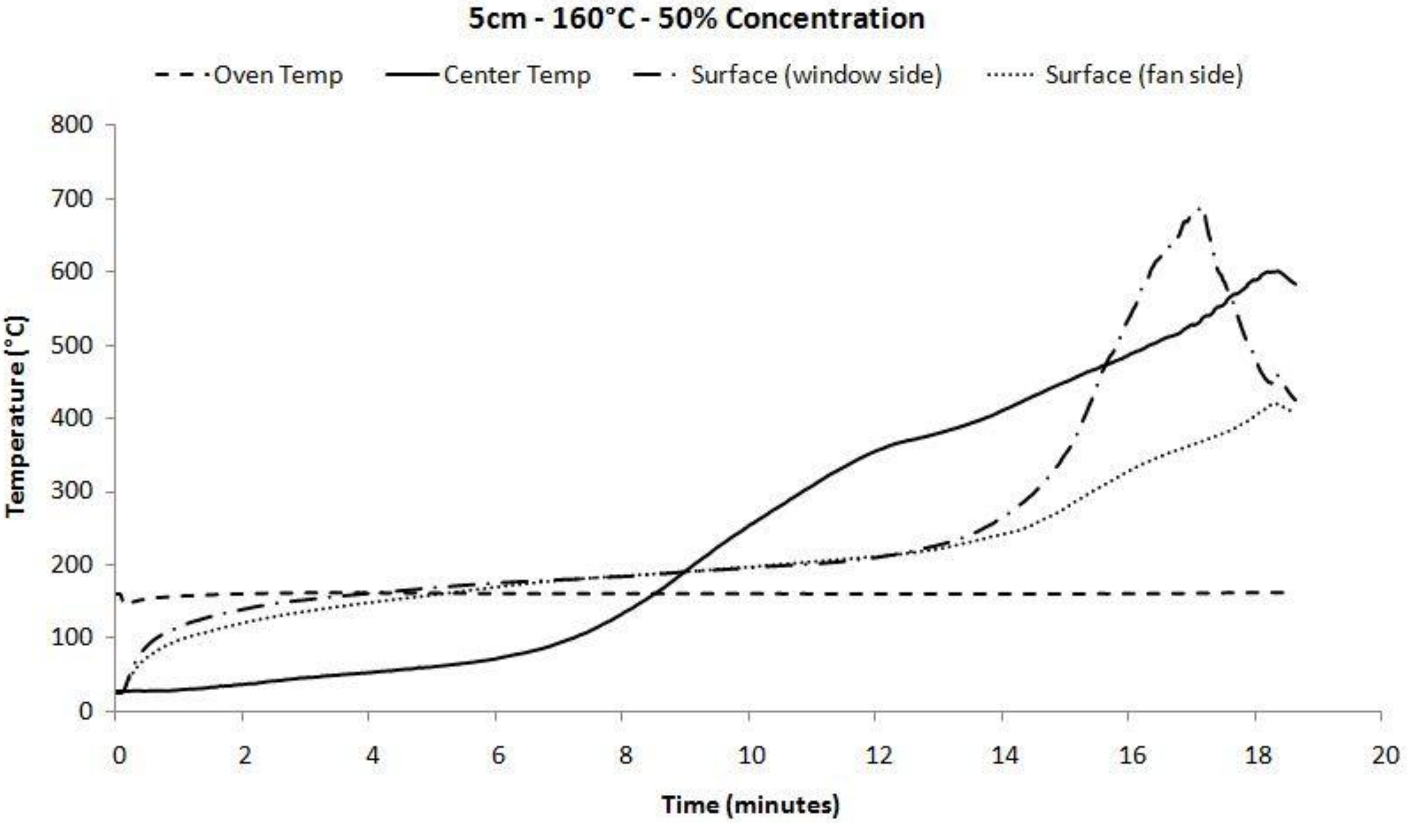


Figure 6.47: 5cm - 160°C – 50% Concentration

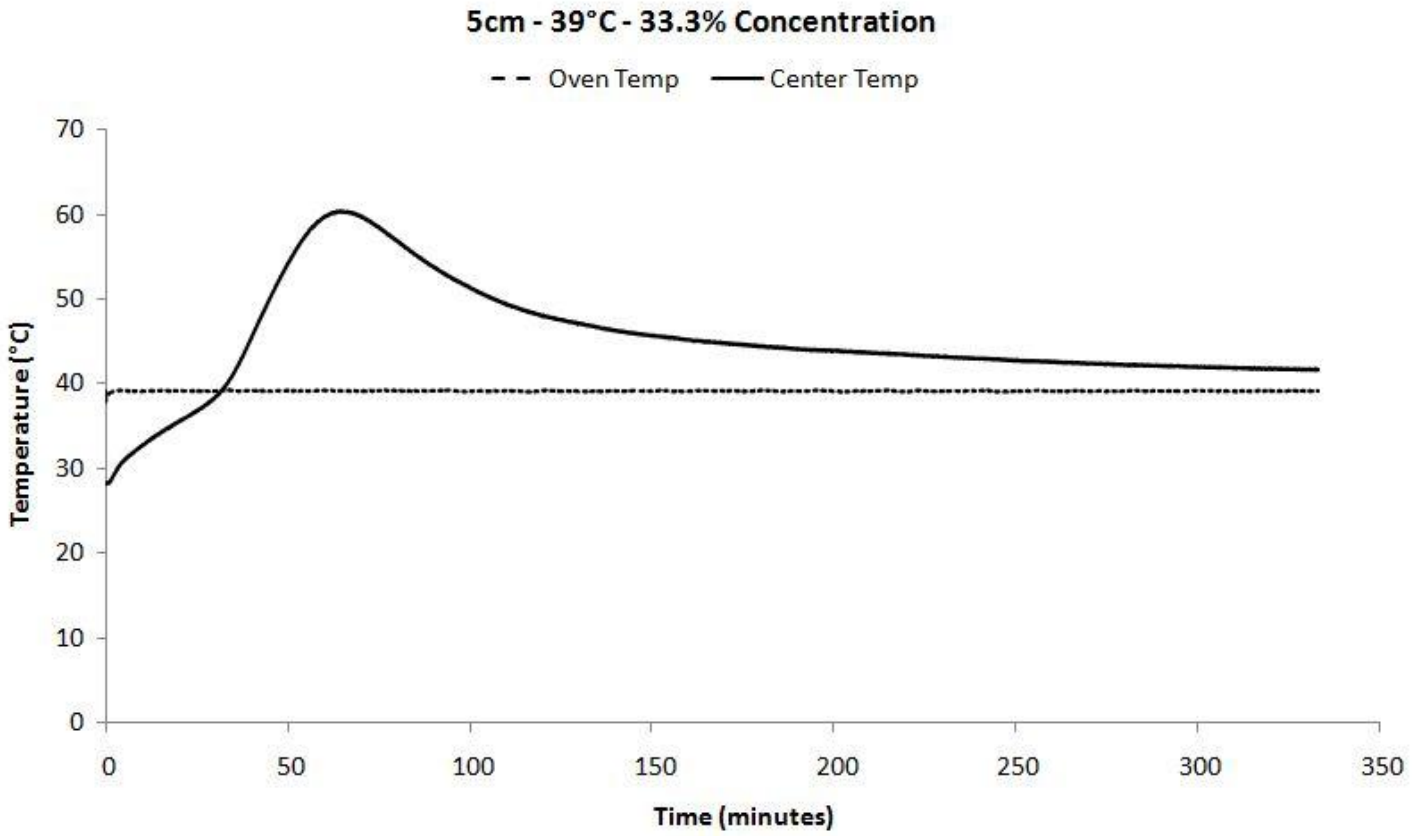


Figure 6.48: 5cm - 39°C – 33.3% Concentration

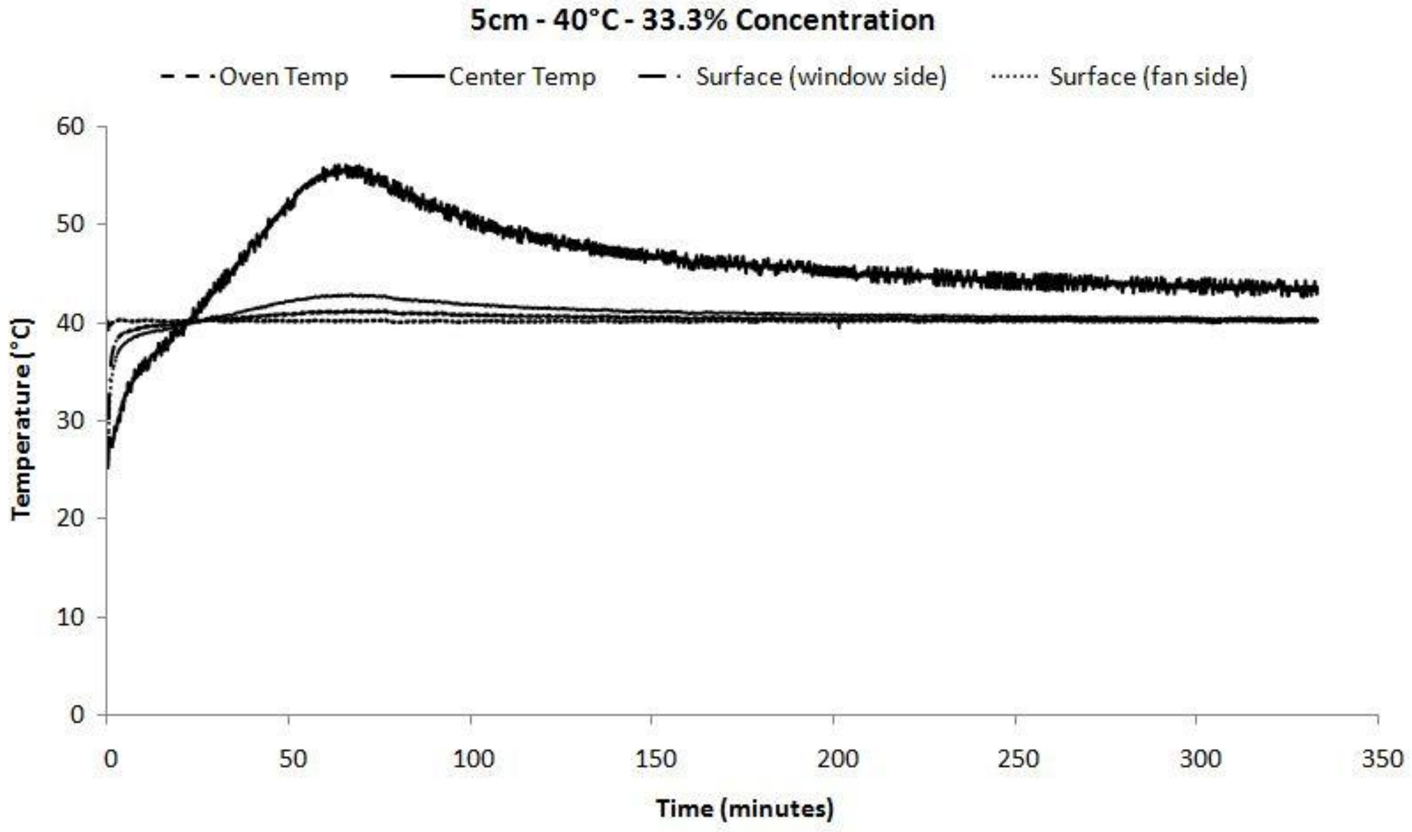


Figure 6.49: 5cm - 40°C – 33.3% Concentration

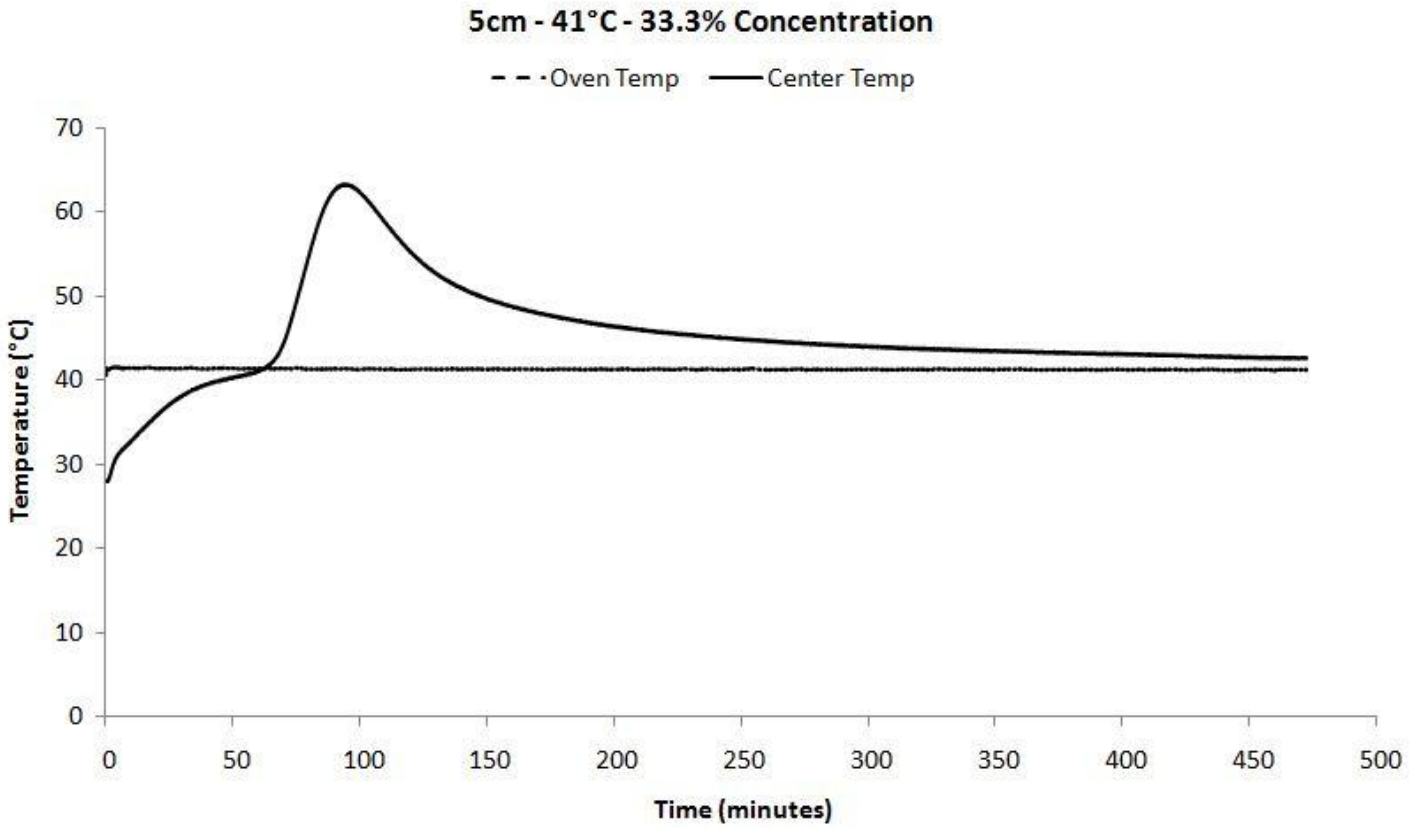


Figure 6.50: 5cm - 41°C – 33.3% Concentration

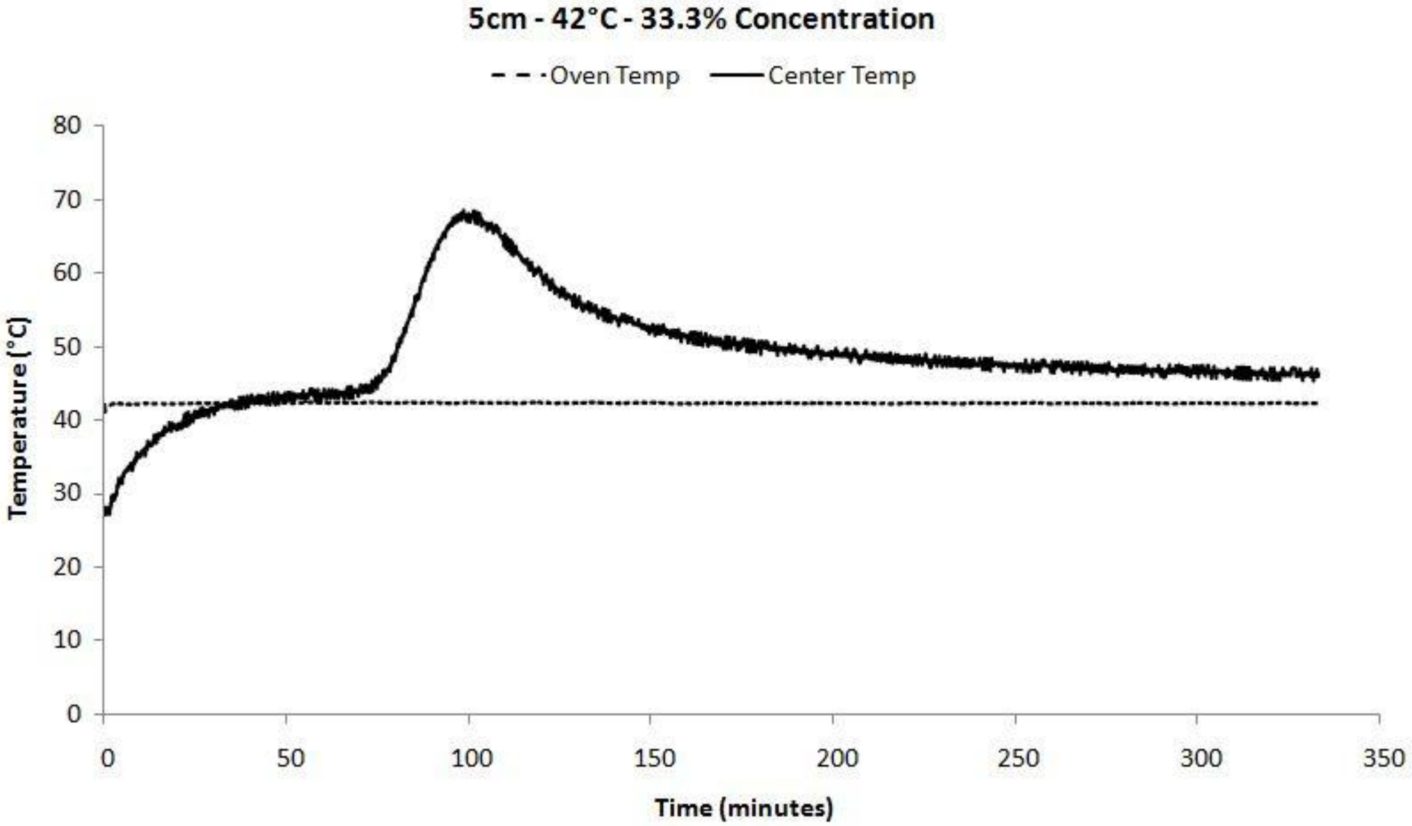


Figure 6.51: 5cm - 42°C – 33.3% Concentration

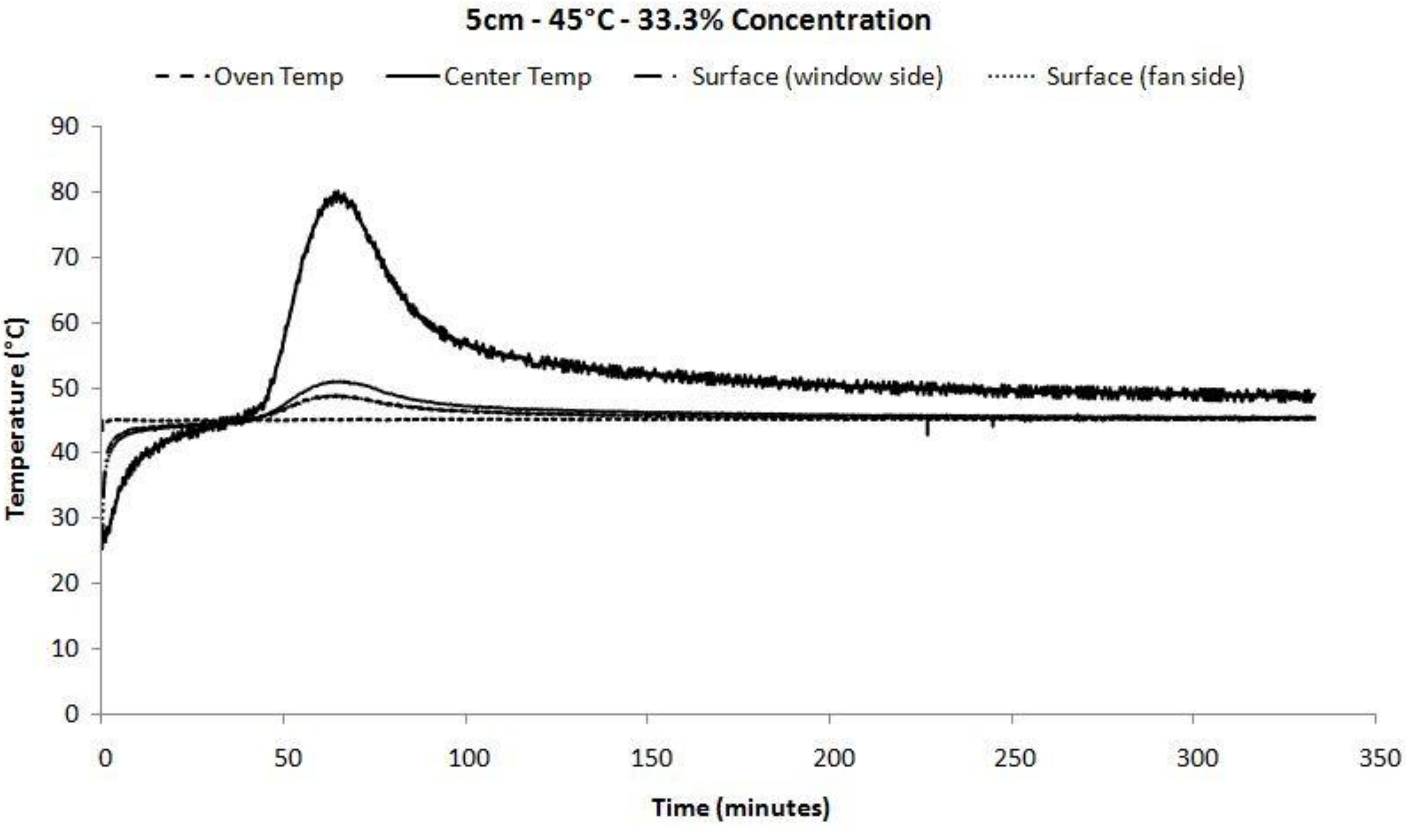


Figure 6.52: 5cm - 45°C – 33.3% Concentration

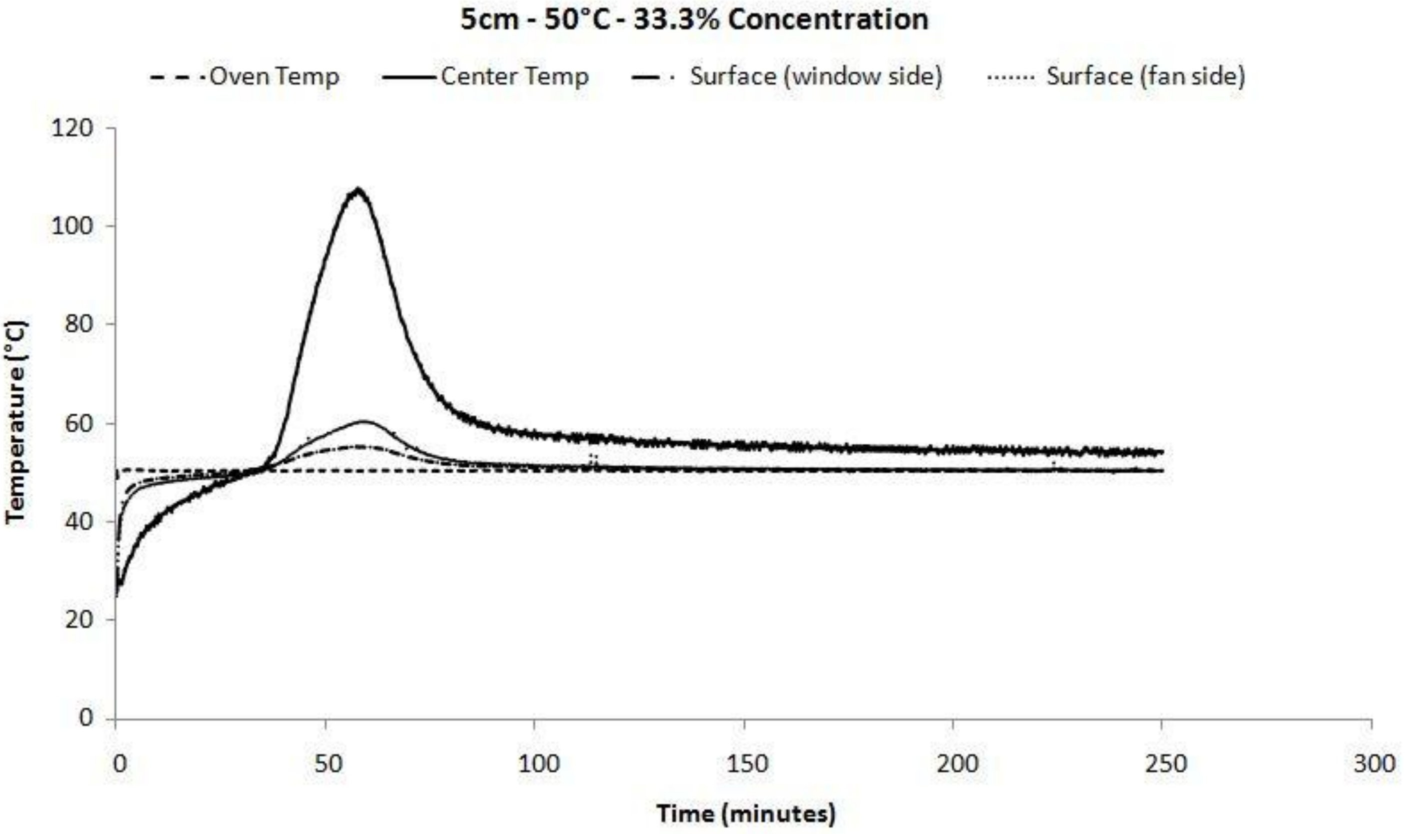


Figure 6.53: 5cm - 50°C – 33.3% Concentration

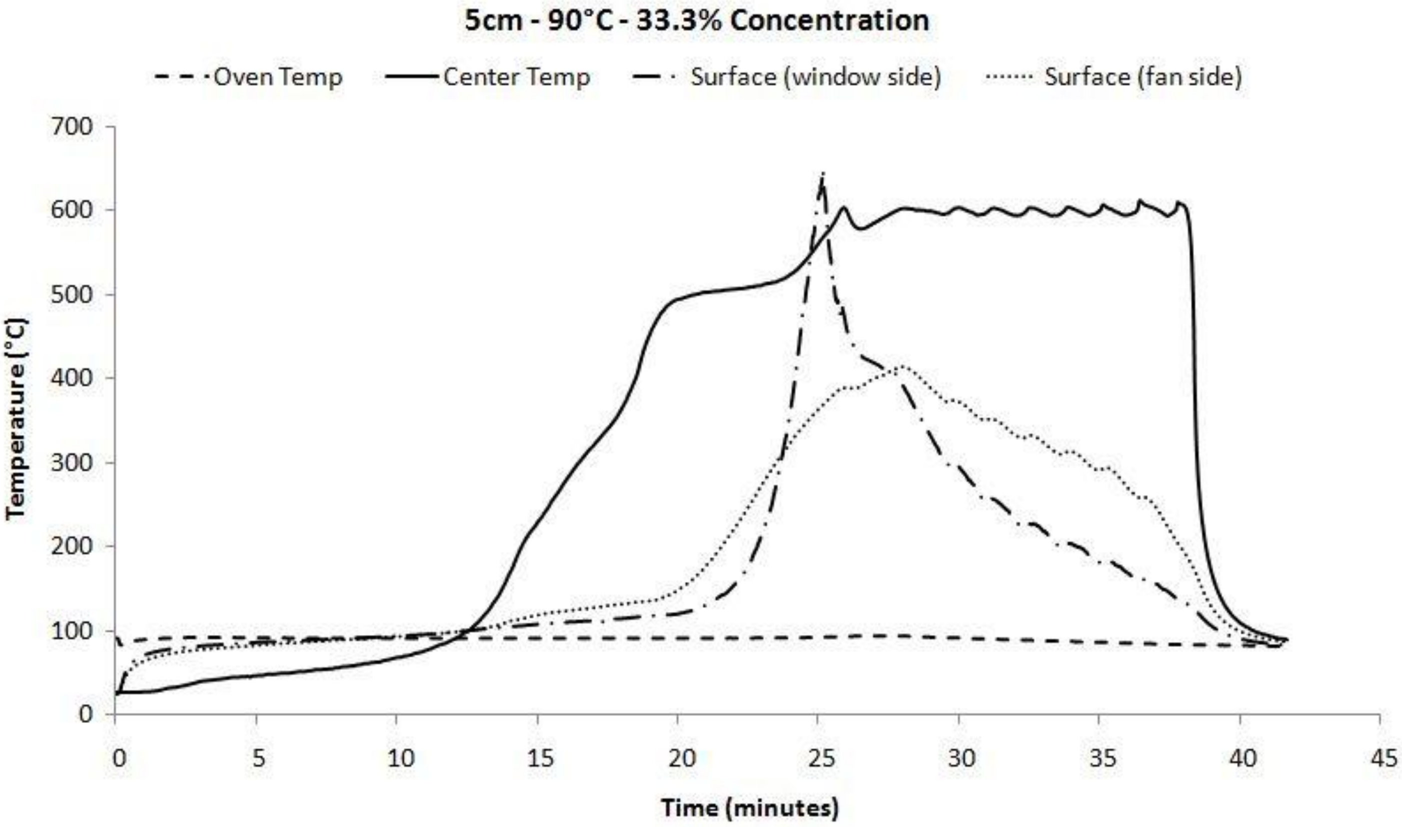


Figure 6.54: 5cm - 90°C – 33.3% Concentration

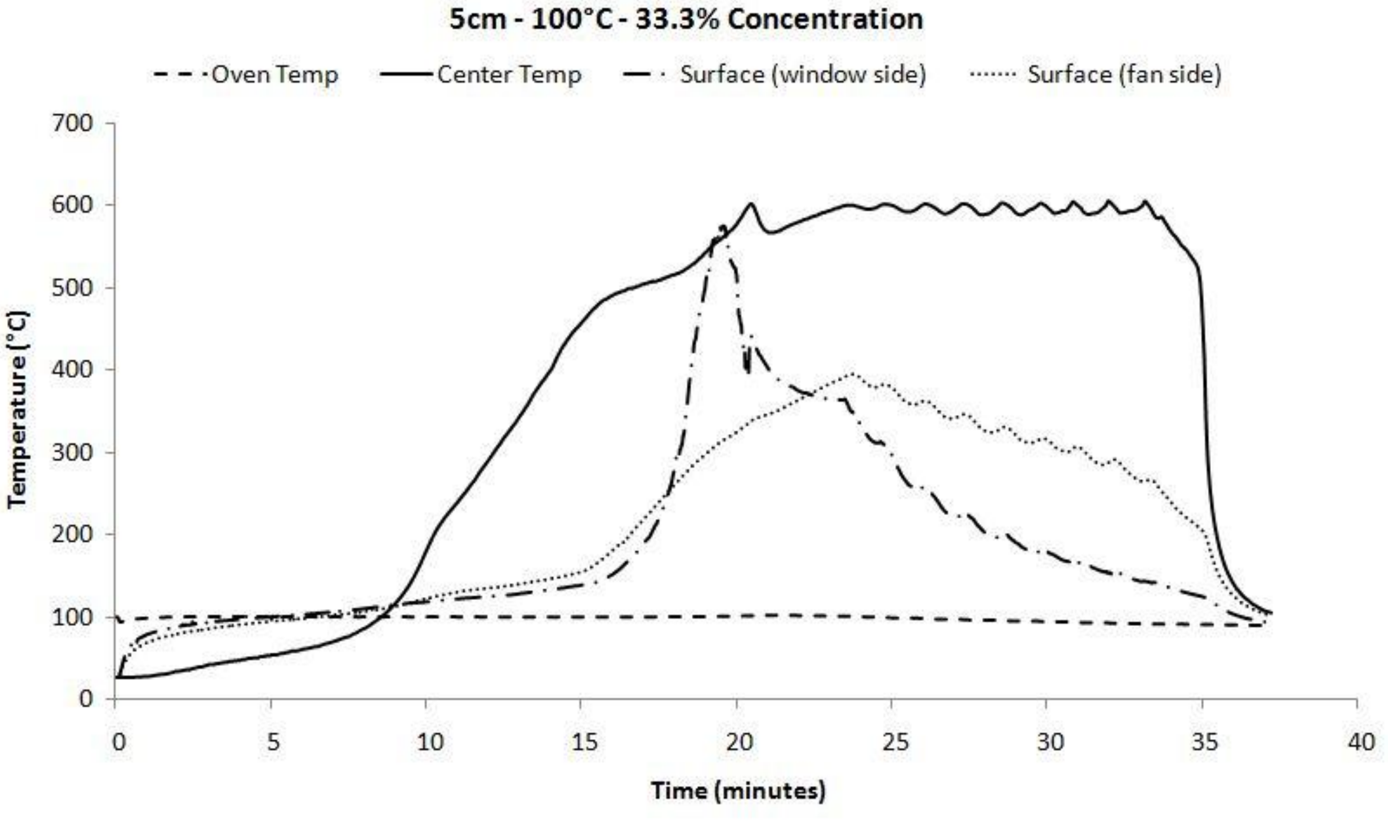


Figure 6.55: 5cm - 100°C – 33.3% Concentration

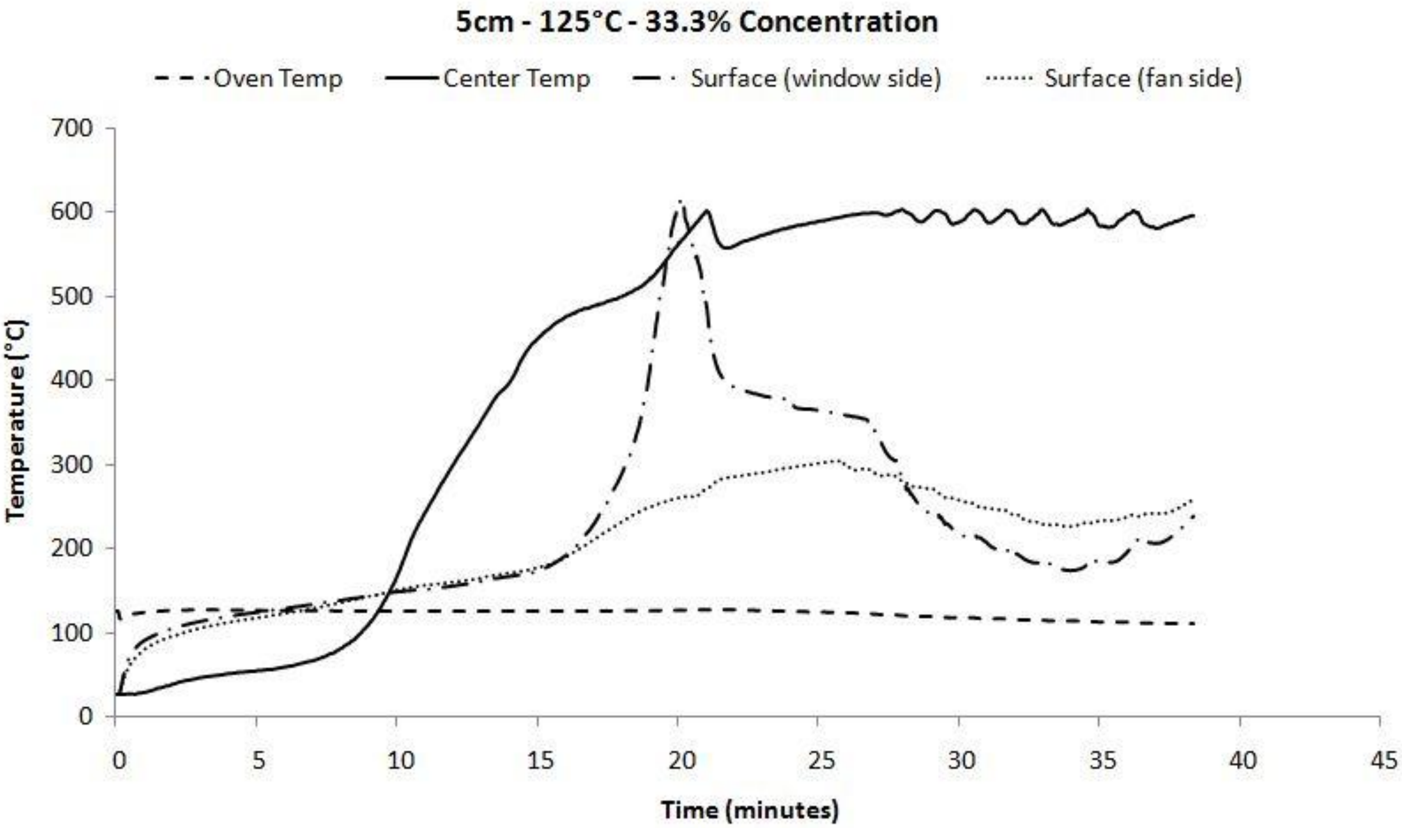


Figure 6.56: 5cm - 125°C – 33.3% Concentration

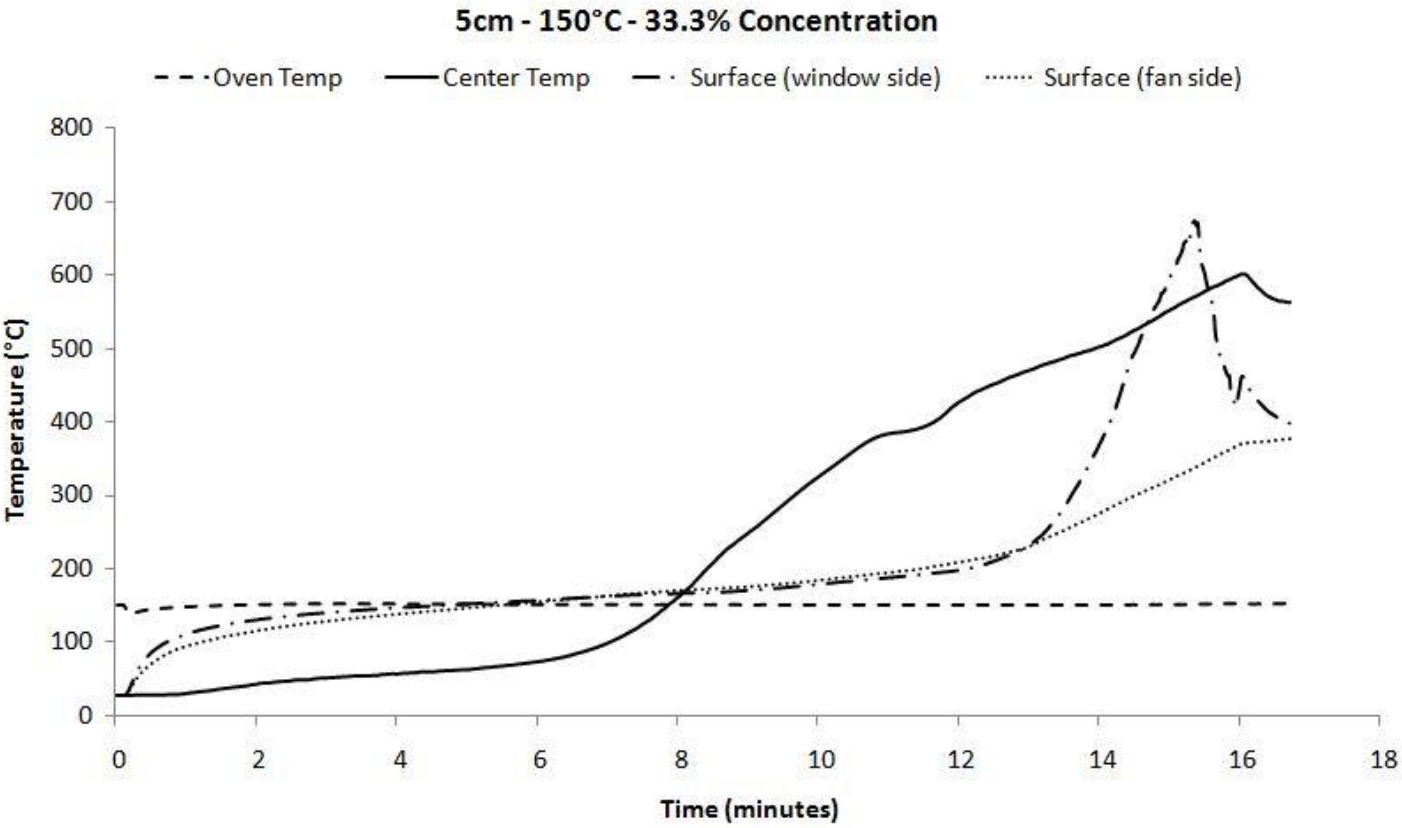


Figure 6.57: 5cm - 150°C – 33.3% Concentration

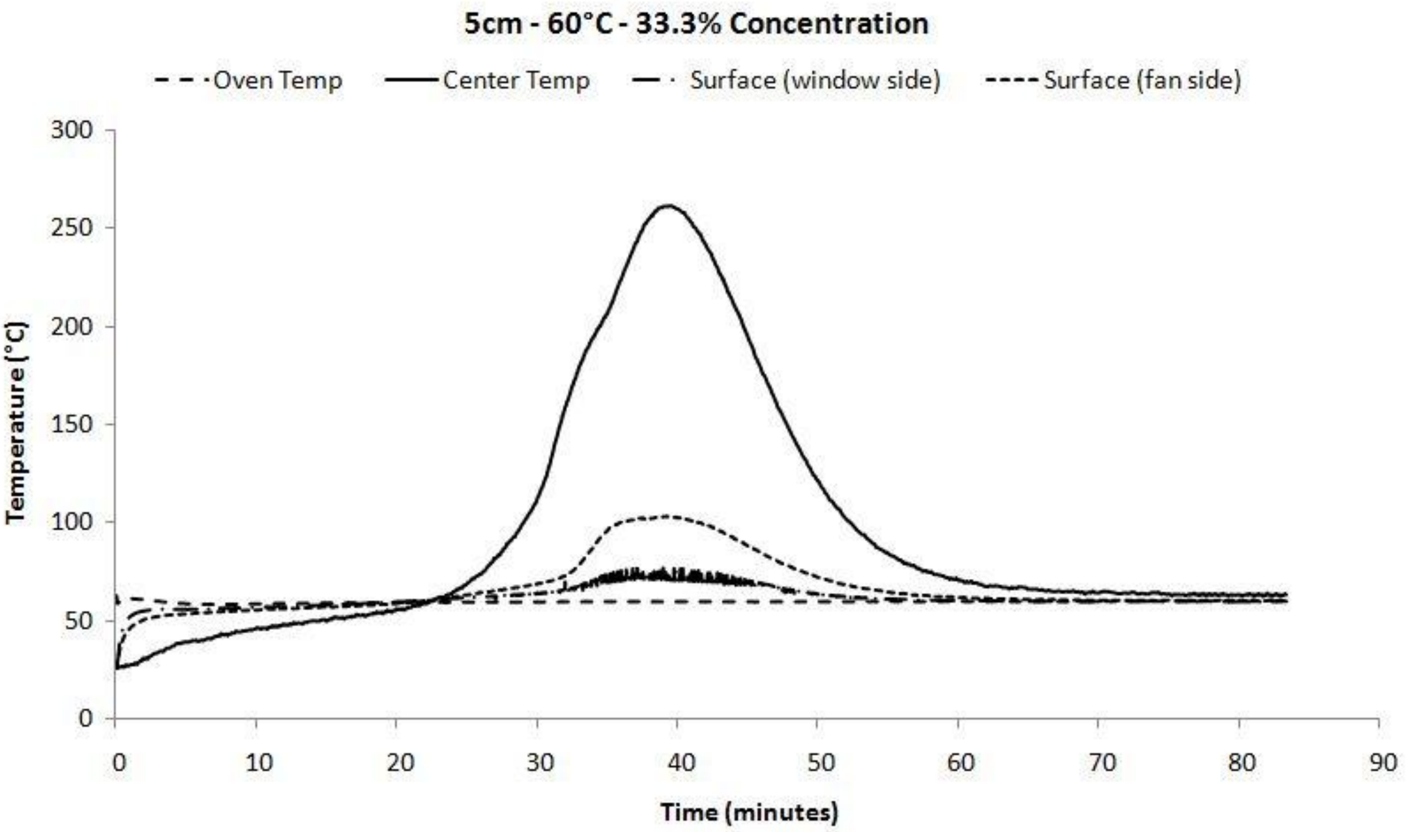


Figure 6.58: 5cm - 60°C – 33.3% Concentration

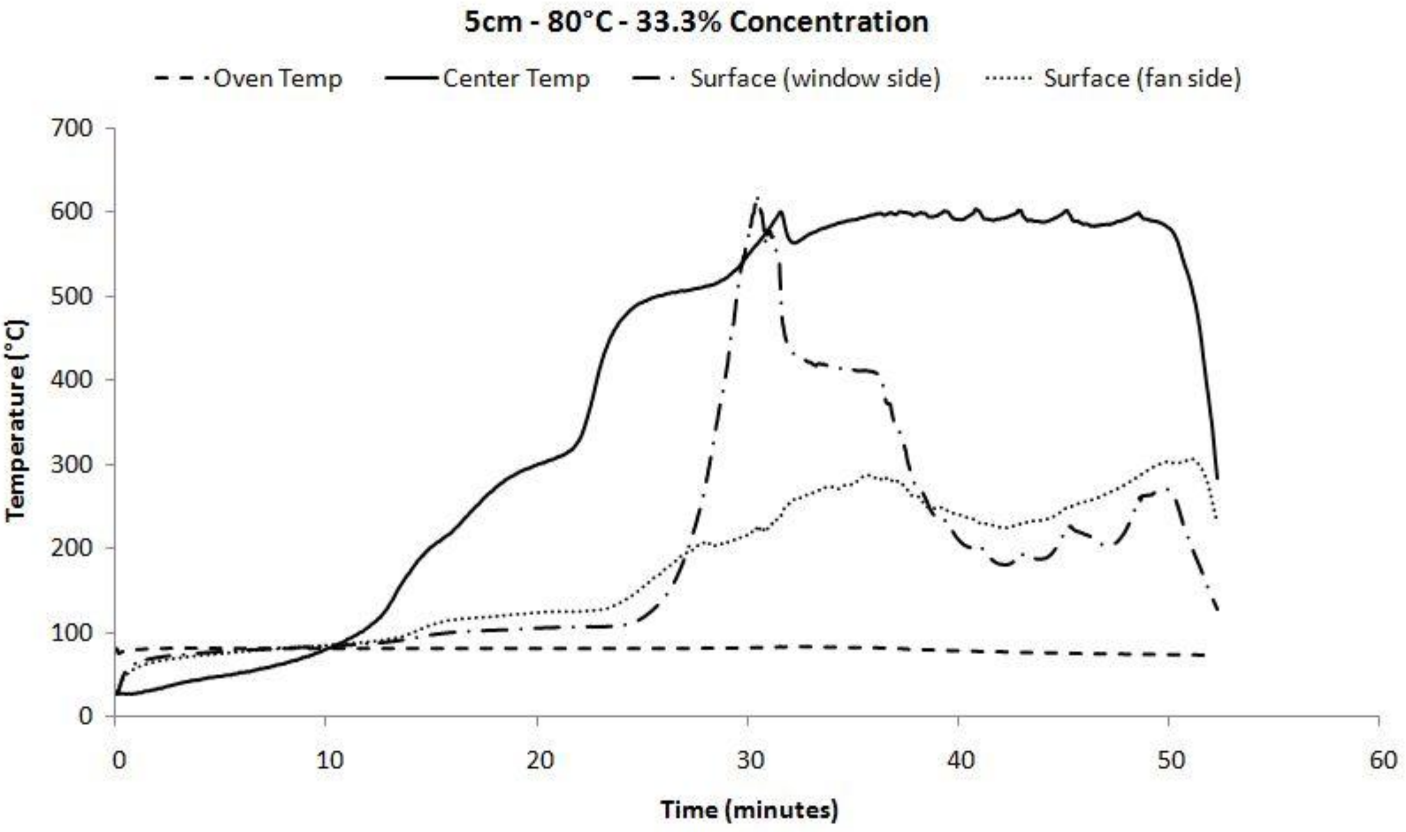


Figure 6.59: 5cm - 80°C – 33.3% Concentration

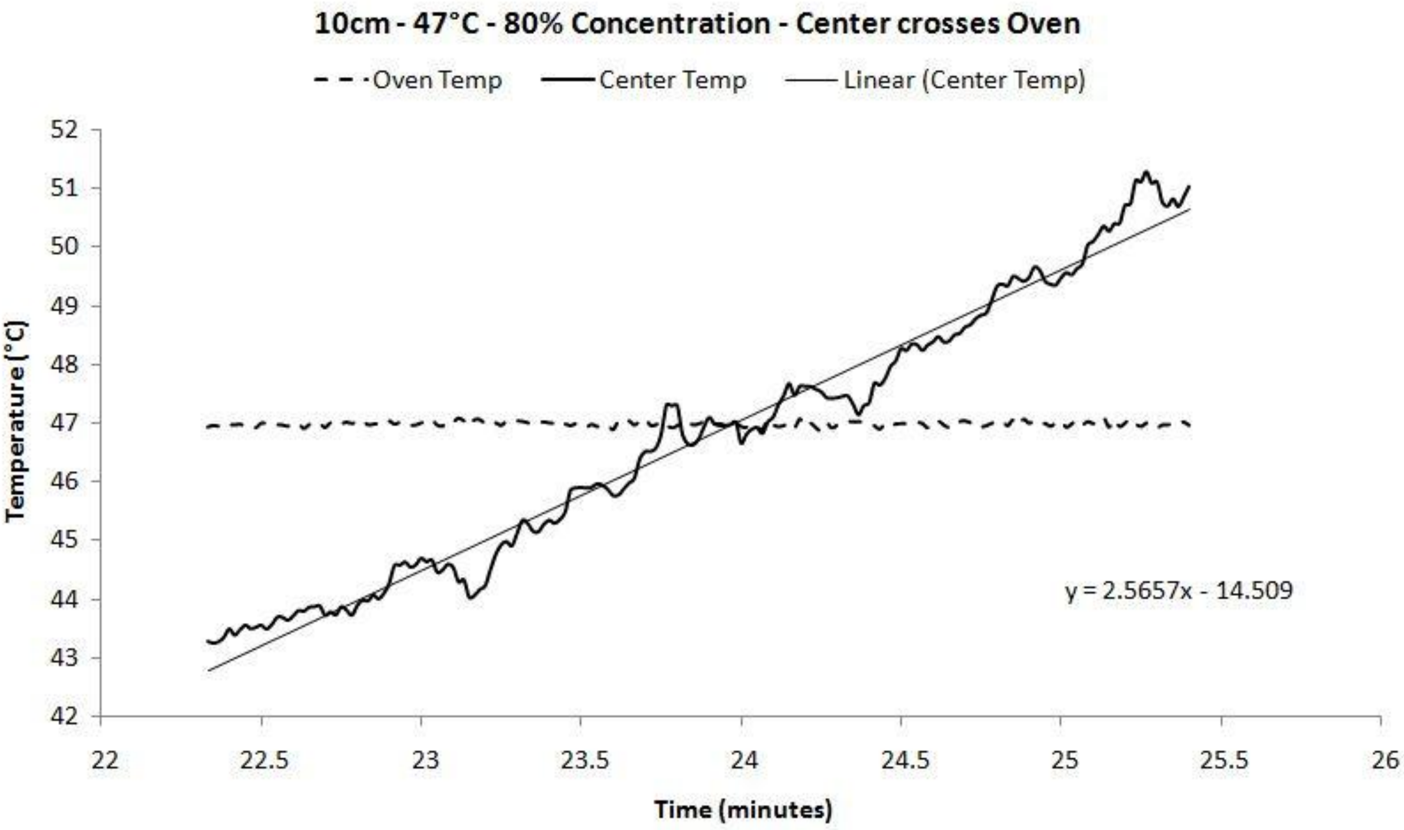


Figure 6.60: 10cm - 47°C – 80% Concentration – Center crosses Oven

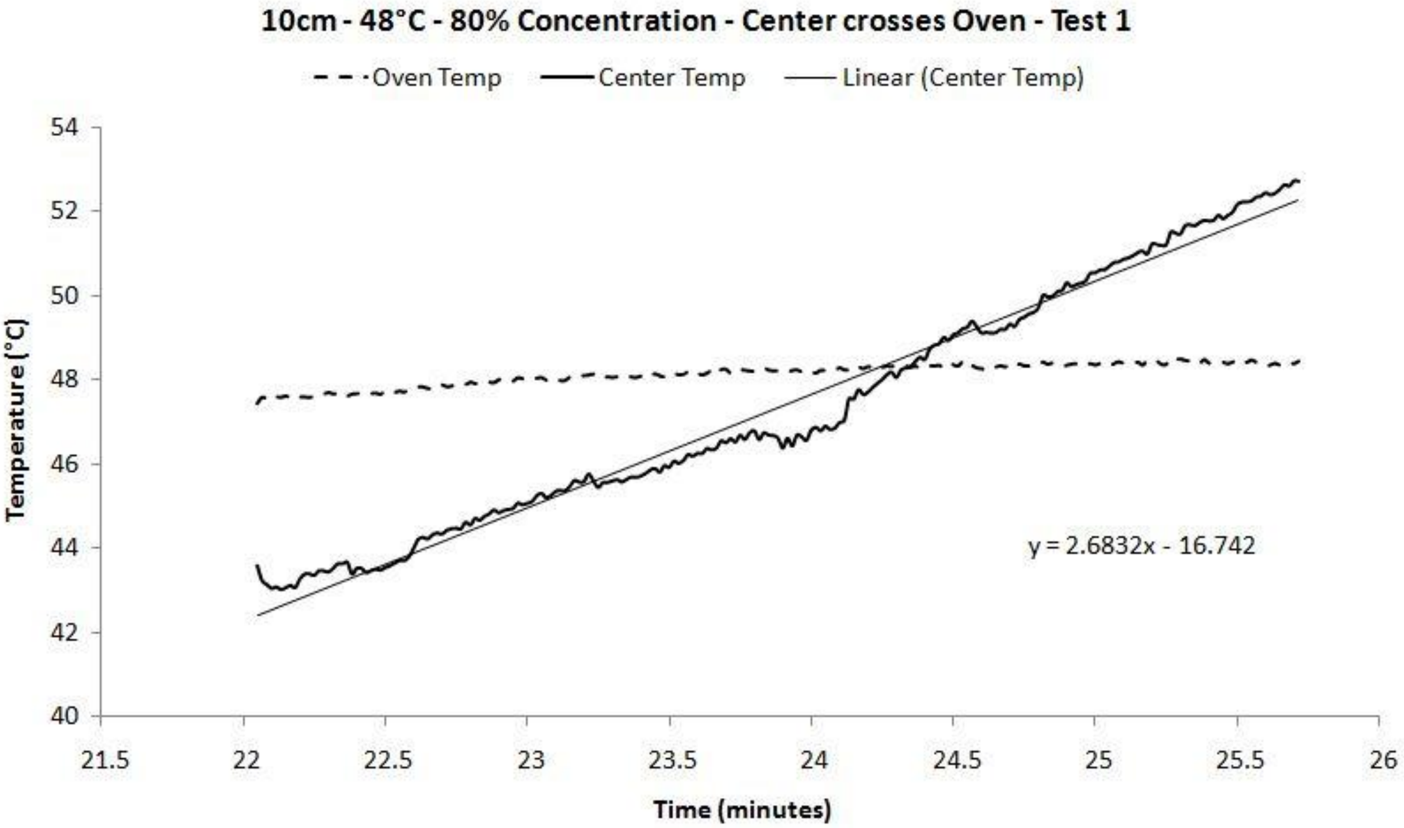


Figure 6.61: 10cm - 48°C – 80% Concentration – Center crosses Oven – Test 1

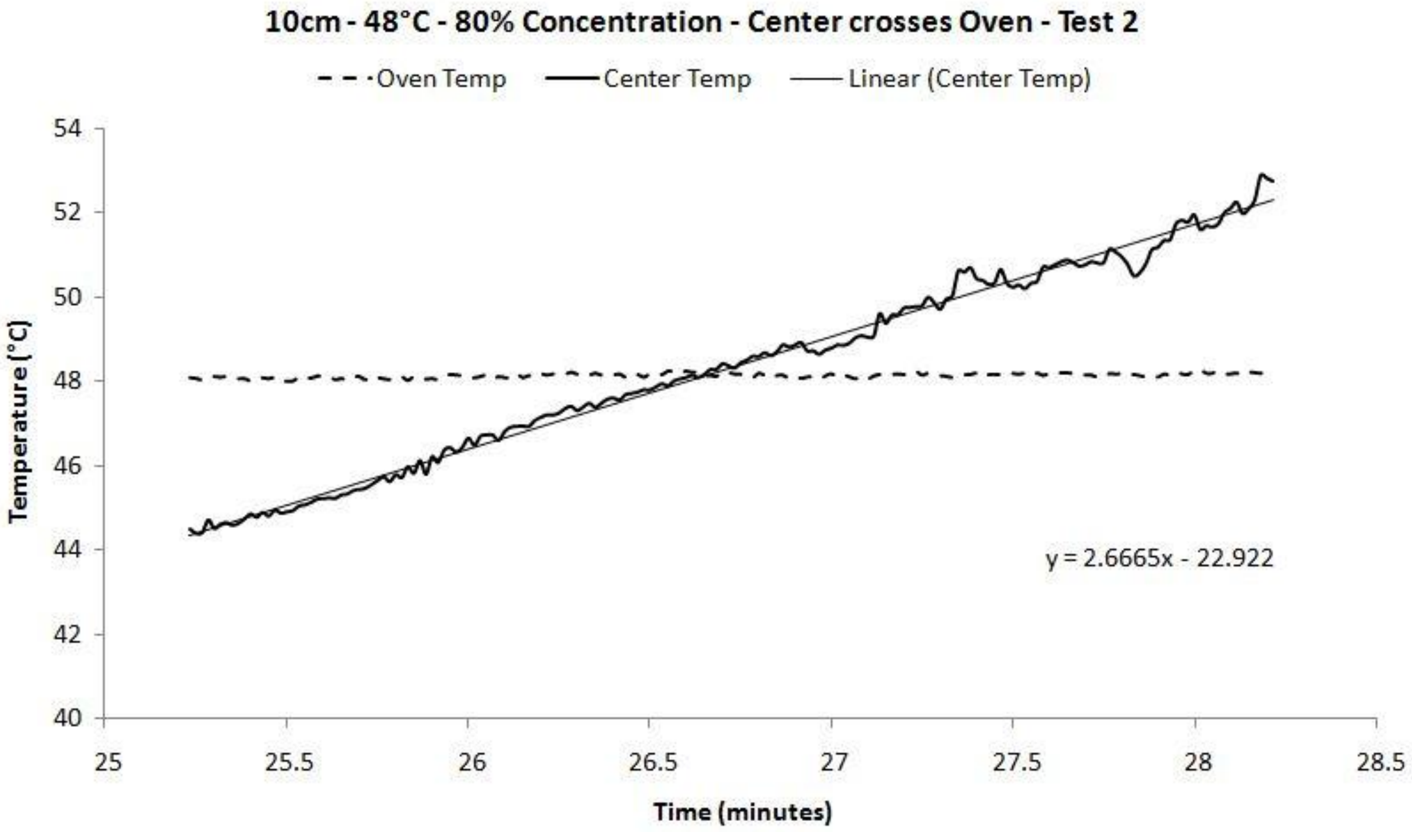


Figure 6.62: 10cm - 48°C – 80% Concentration – Center crosses Oven – Test 2

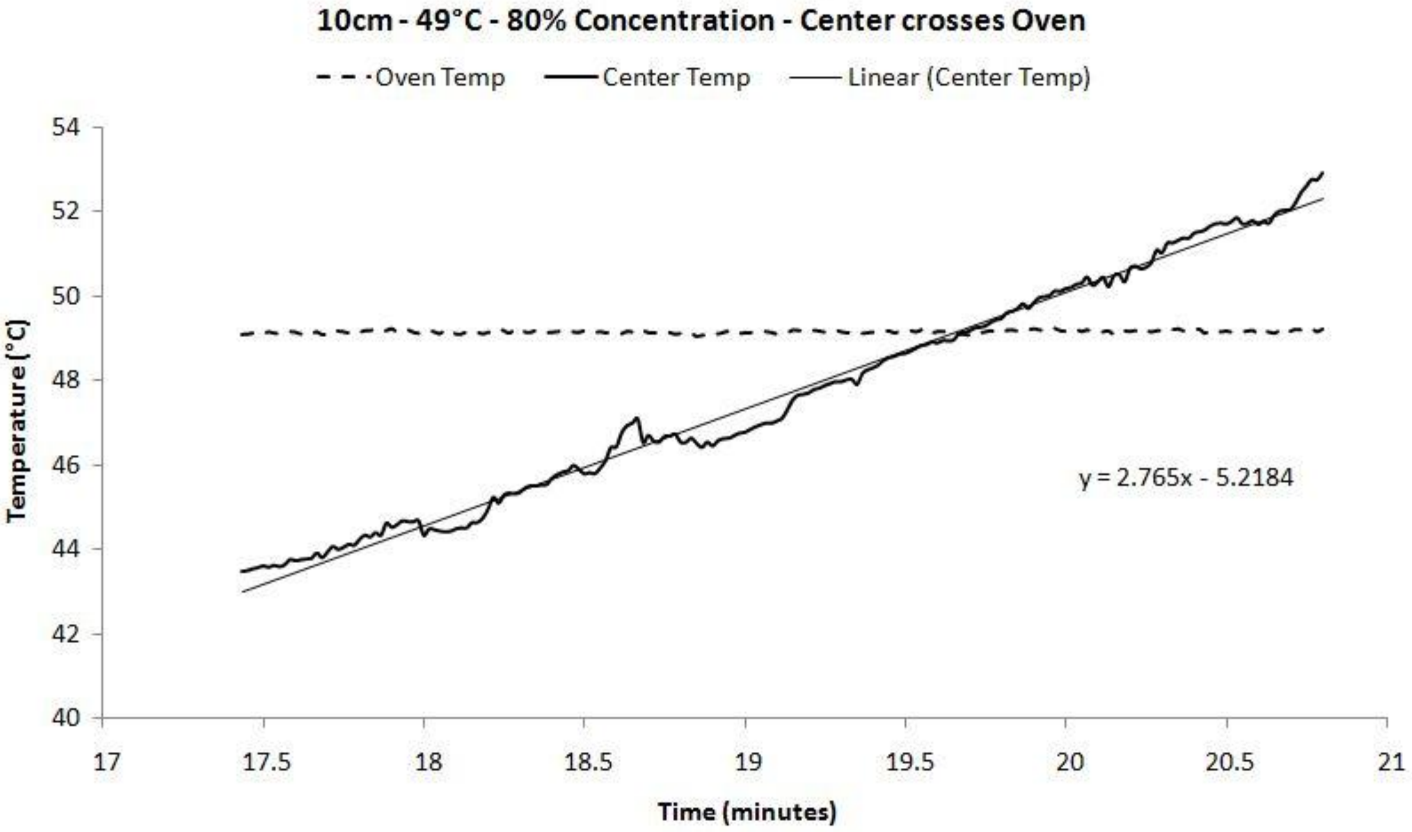


Figure 6.63: 10cm - 49°C – 80% Concentration – Center crosses Oven

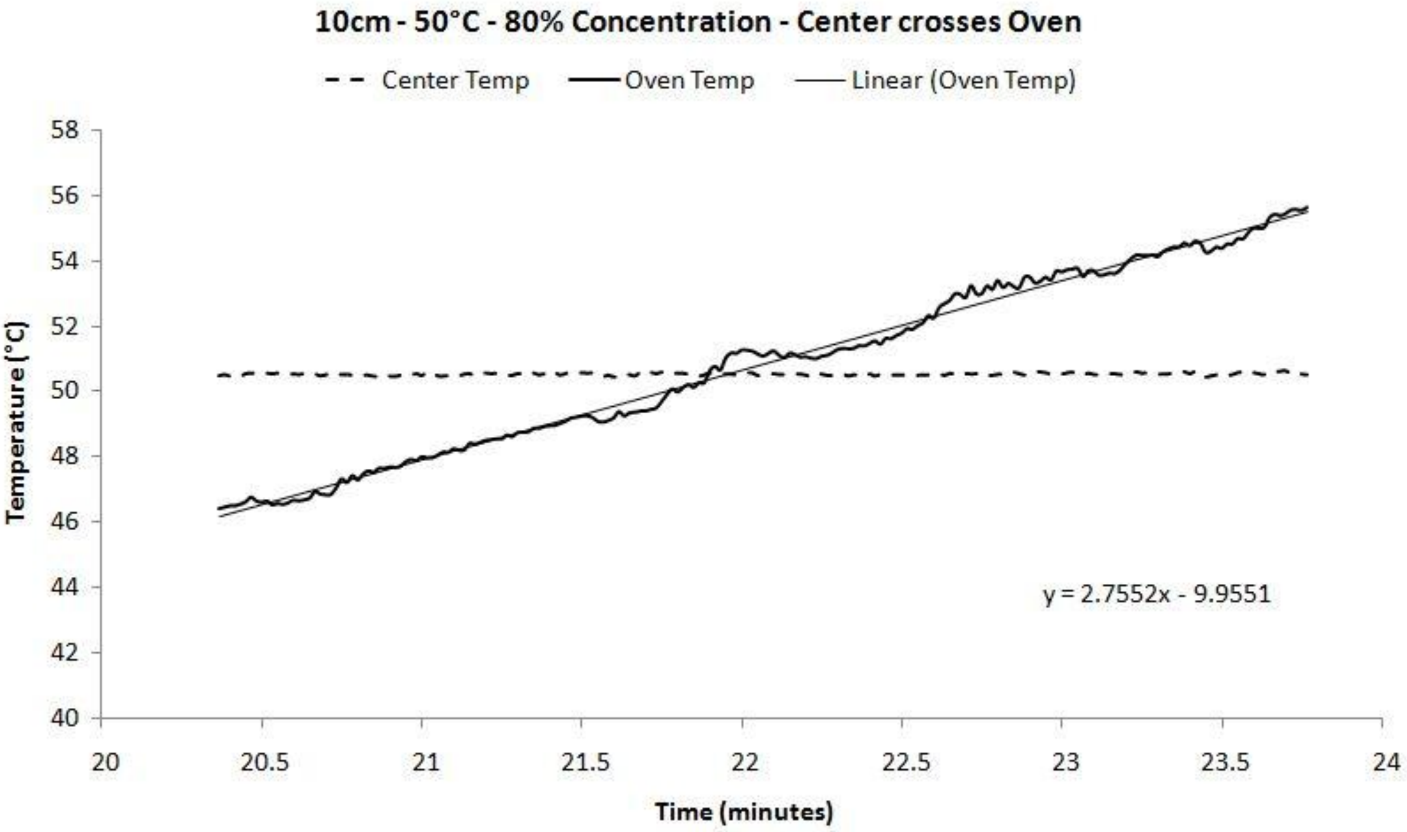


Figure 6.64: 10cm - 50°C – 80% Concentration – Center crosses Oven

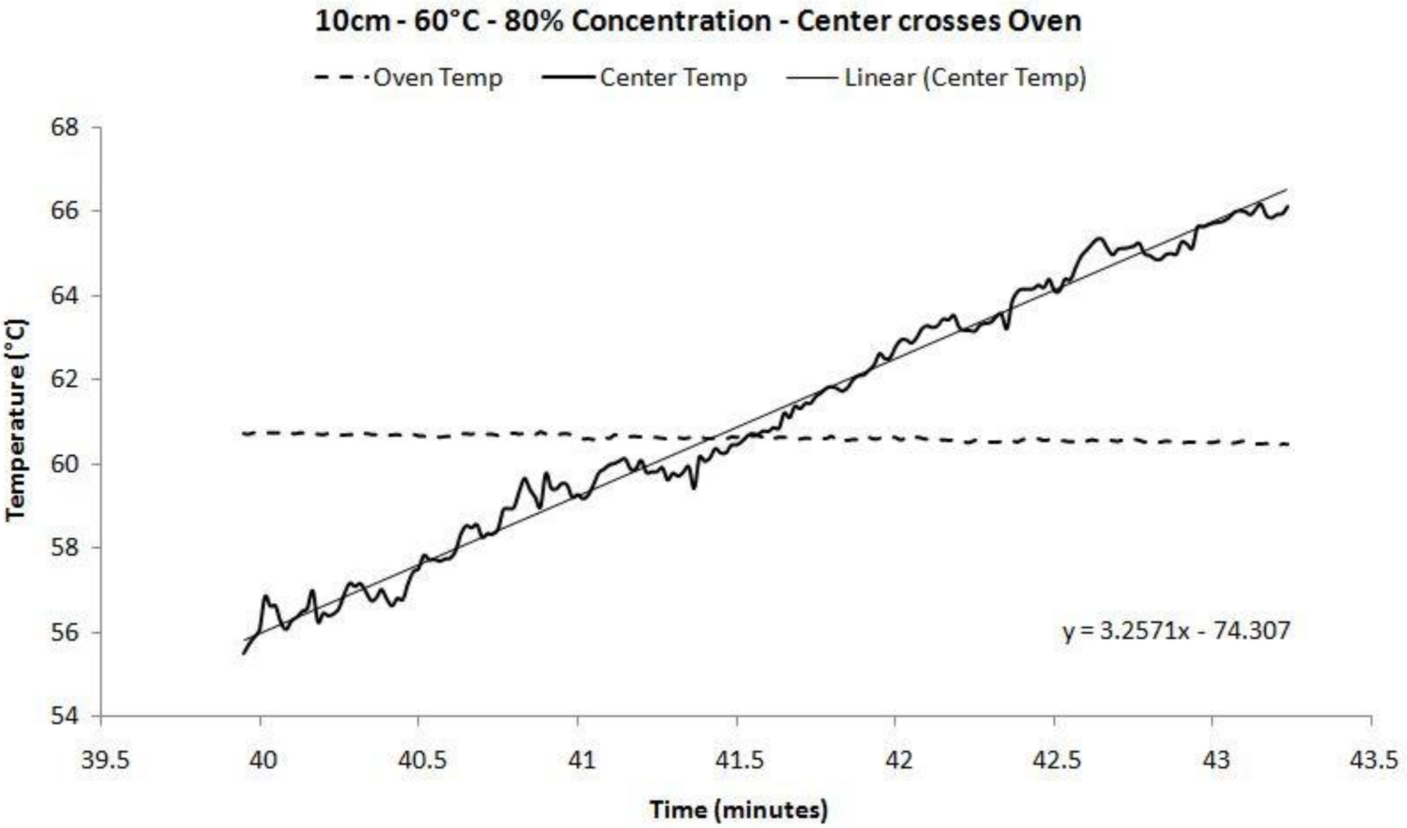


Figure 6.65: 10cm - 60°C – 80% Concentration – Center crosses Oven

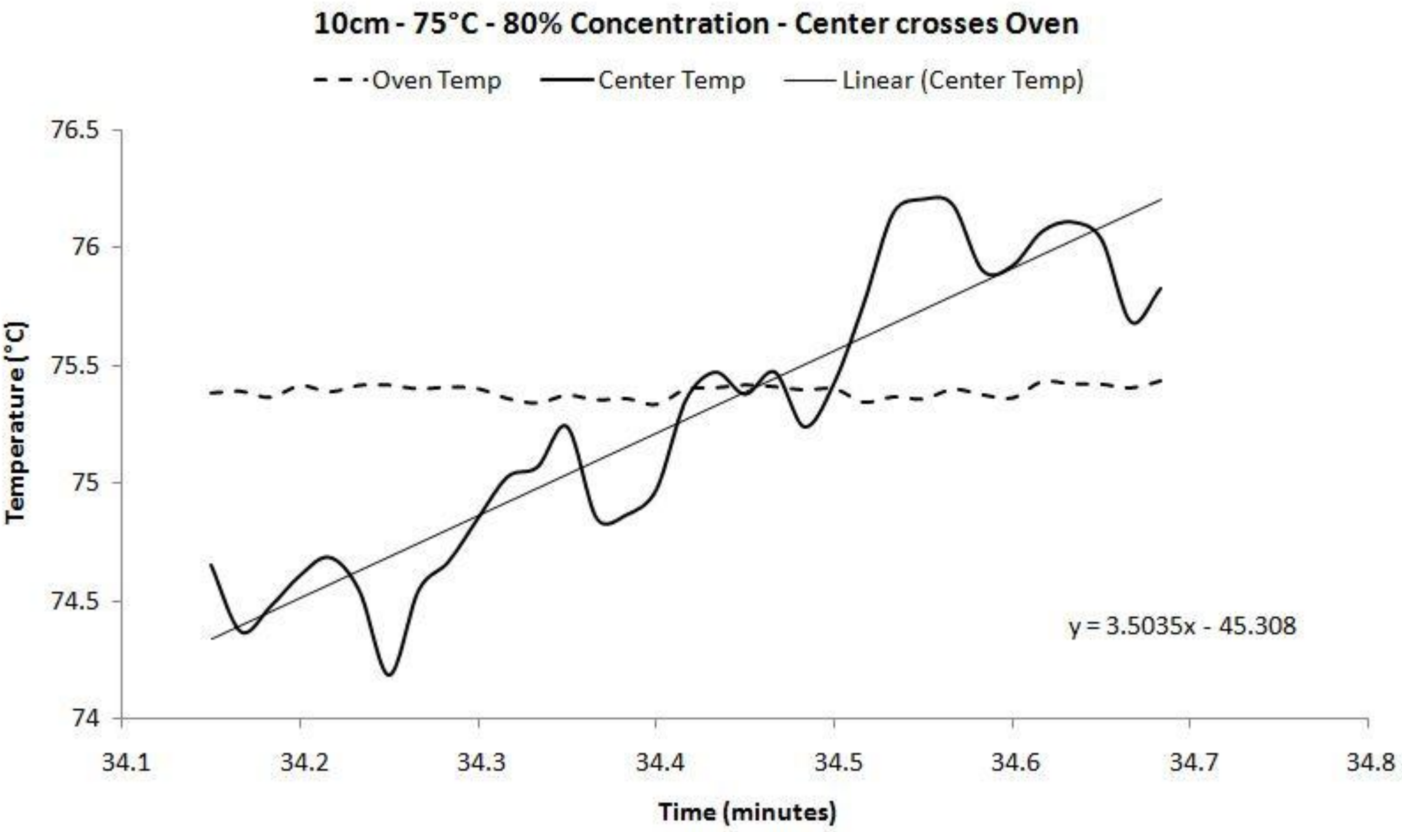


Figure 6.66: 10cm - 75°C – 80% Concentration – Center crosses Oven

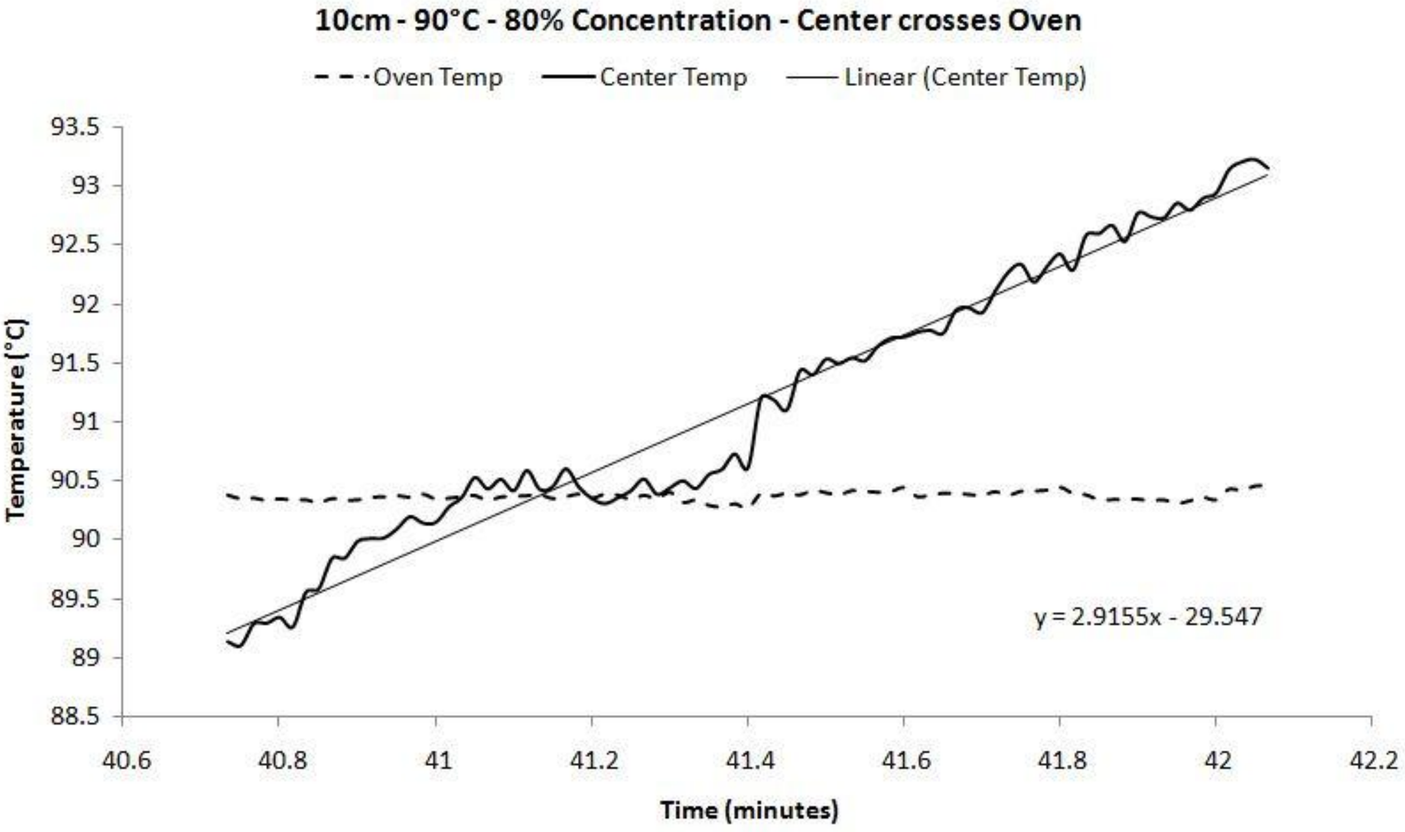


Figure 6.67: 10cm - 90°C – 80% Concentration – Center crosses Oven

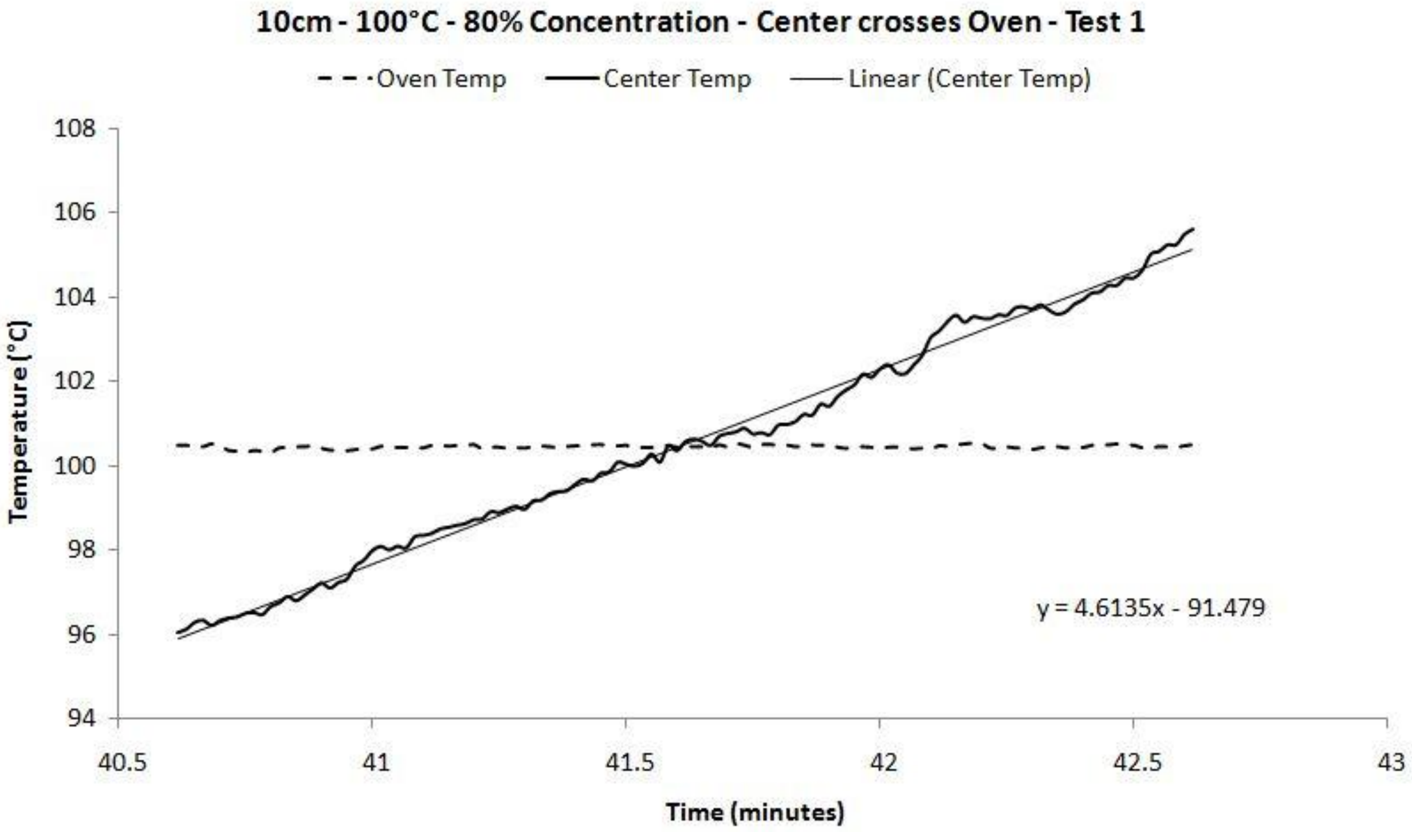


Figure 6.68: 10cm - 100°C – 80% Concentration – Center crosses Oven – Test 1

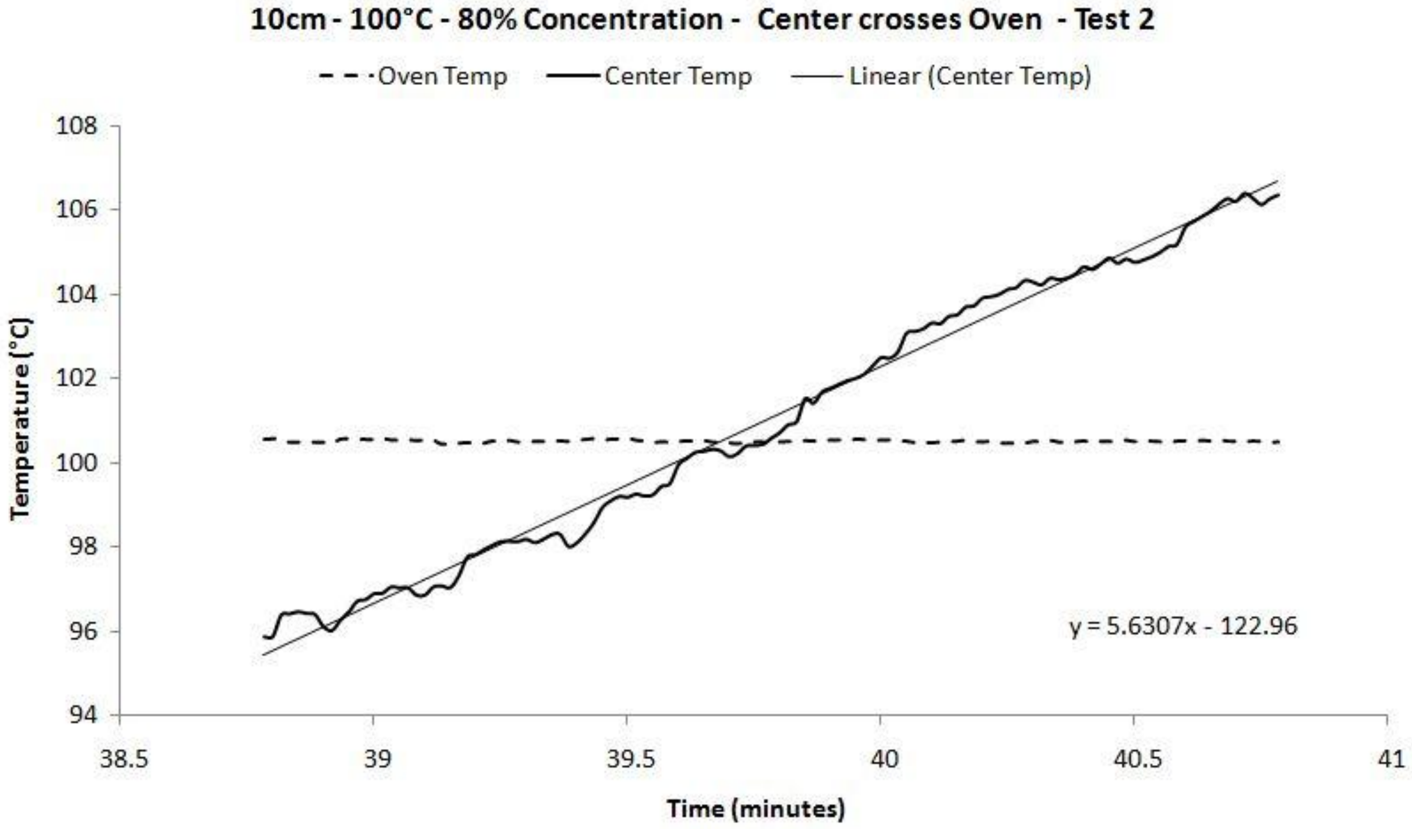


Figure 6.69: 10cm - 100°C – 80% Concentration – Center crosses Oven – Test 2

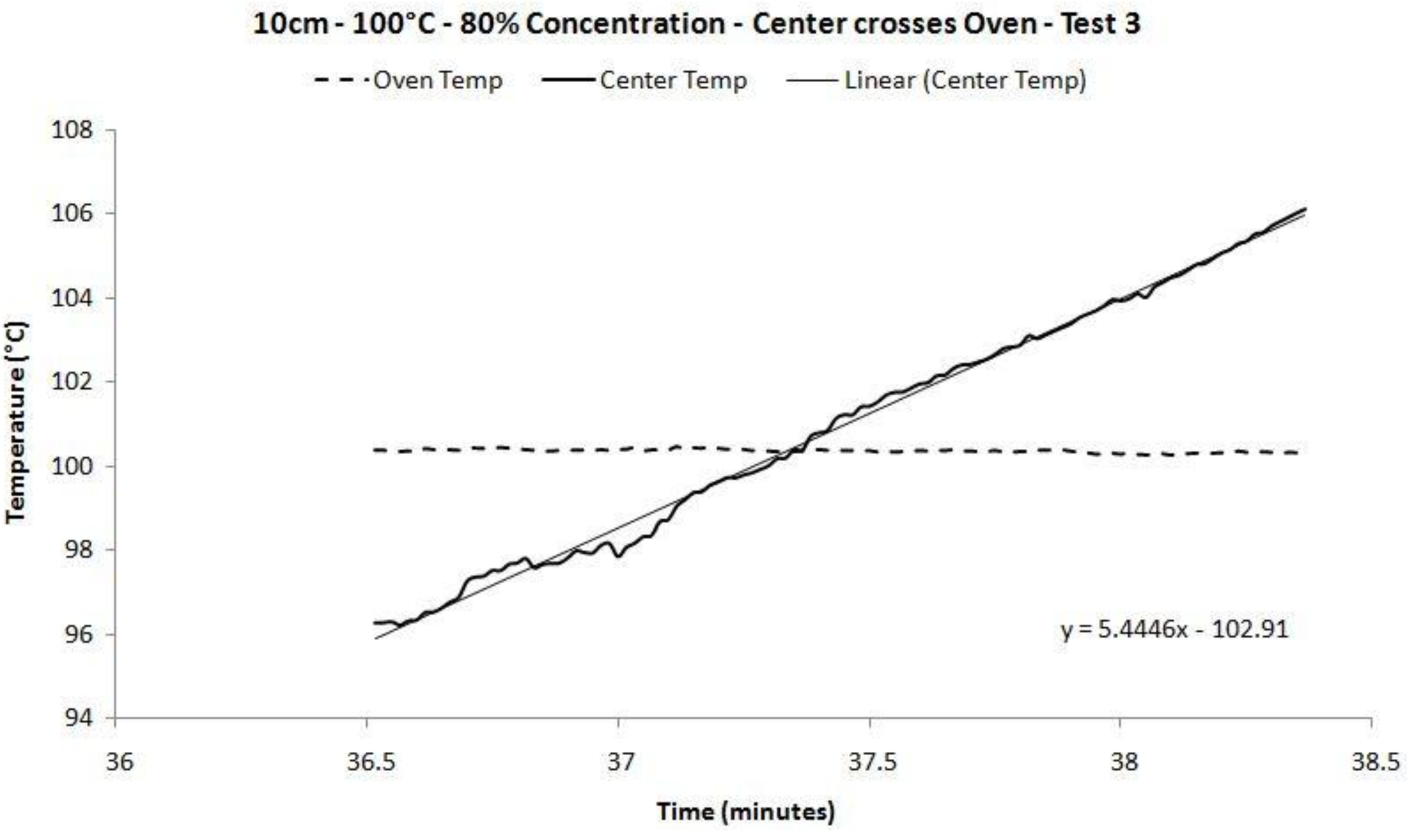


Figure 6.70: 10cm - 100°C – 80% Concentration – Center crosses Oven

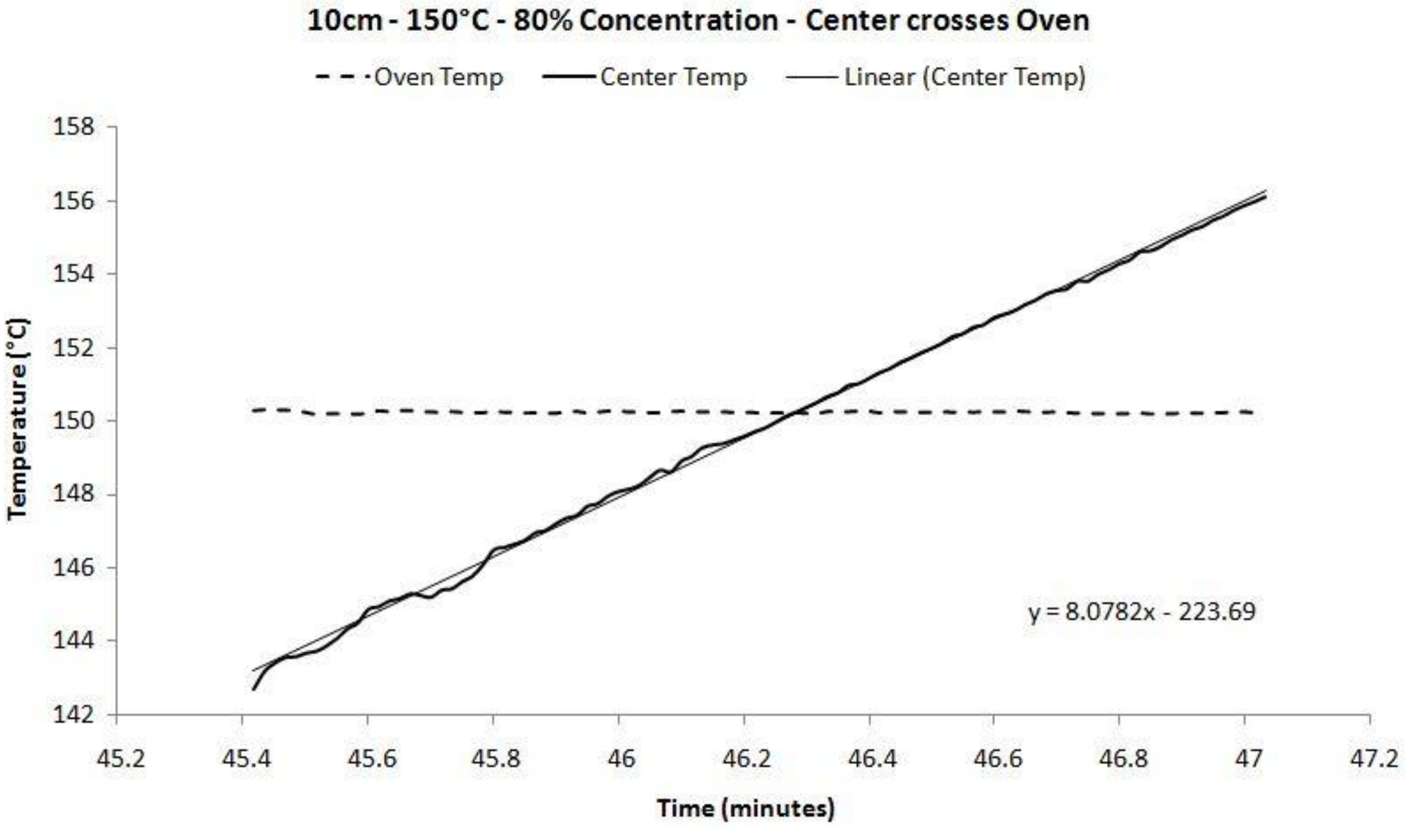


Figure 6.71: 10cm - 150°C – 80% Concentration – Center crosses Oven

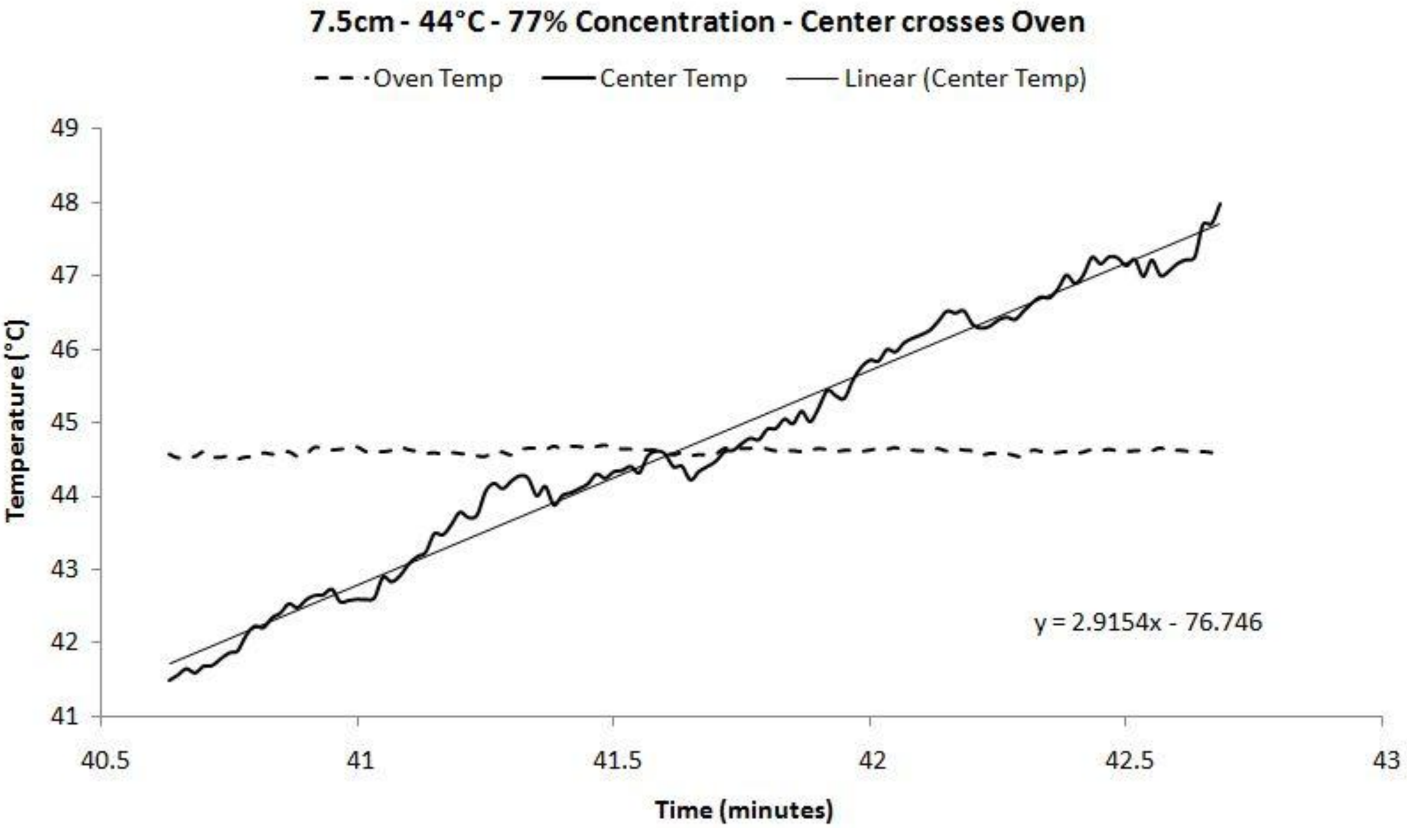


Figure 6.72: 7.5cm - 44°C – 77% Concentration – Center crosses Oven

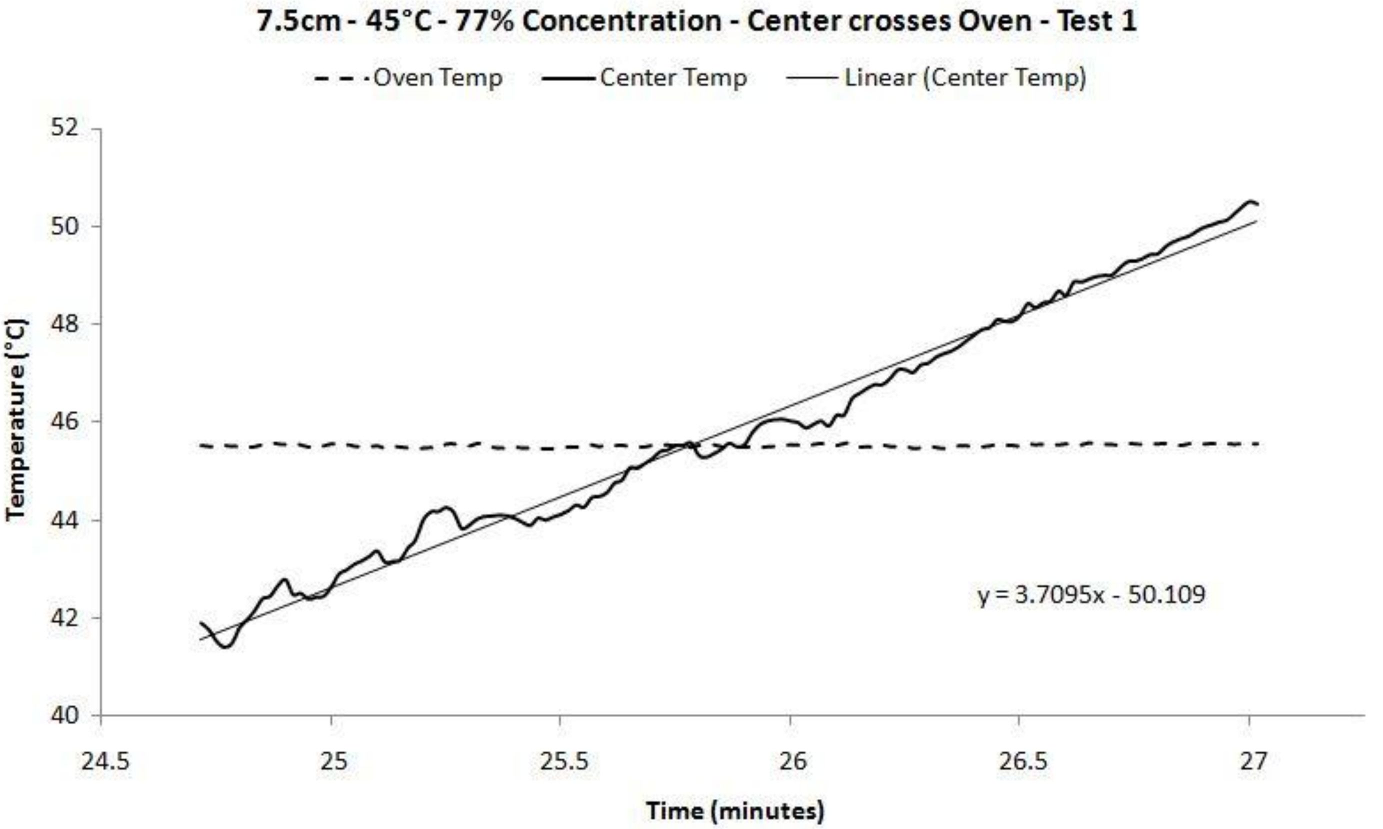


Figure 6.73: 7.5cm - 45°C – 77% Concentration – Center crosses Oven – Test 1

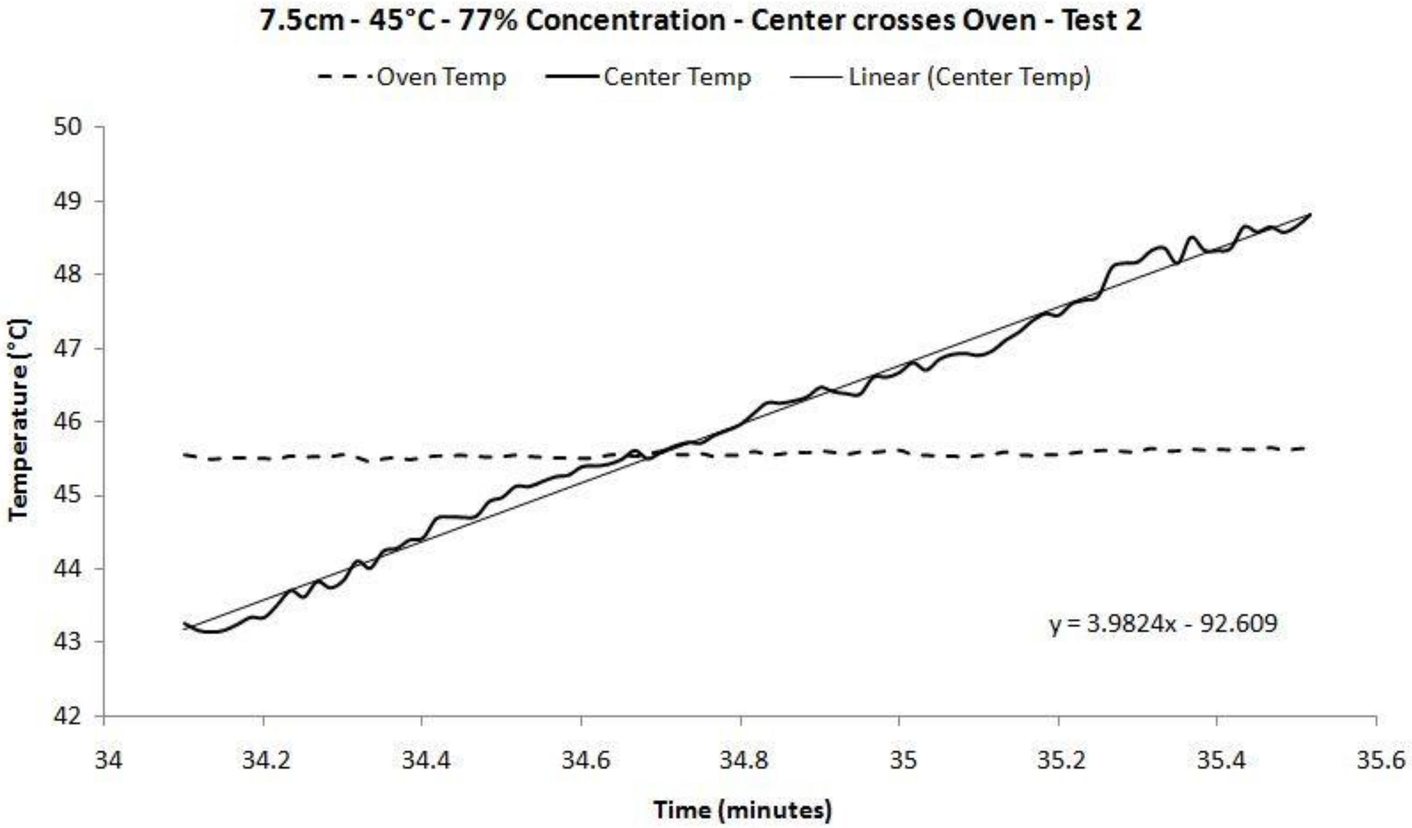


Figure 6.74: 7.5cm - 45°C – 77% Concentration – Center crosses Oven – Test 2

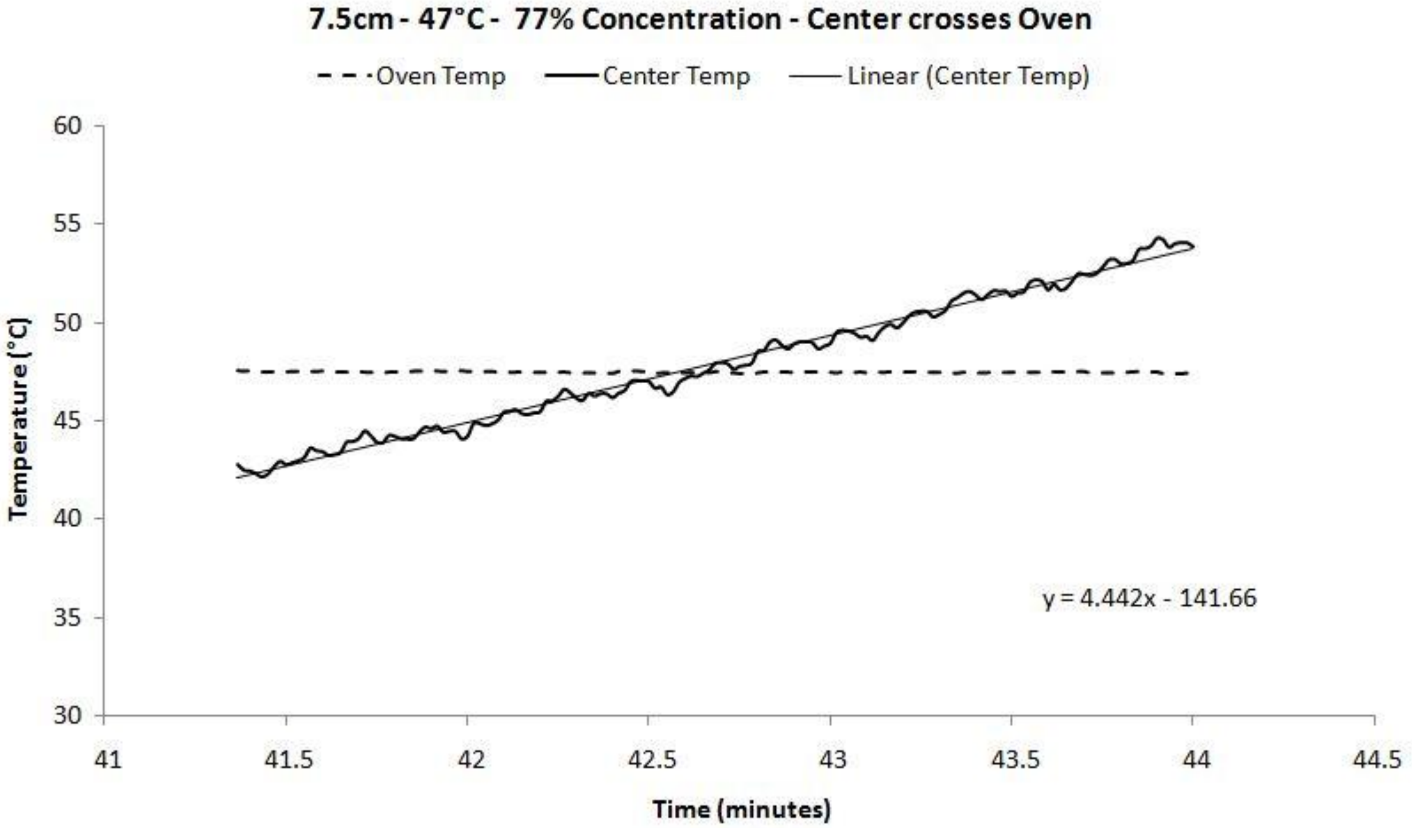


Figure 6.75: 7.5cm - 47°C – 77% Concentration – Center crosses Oven

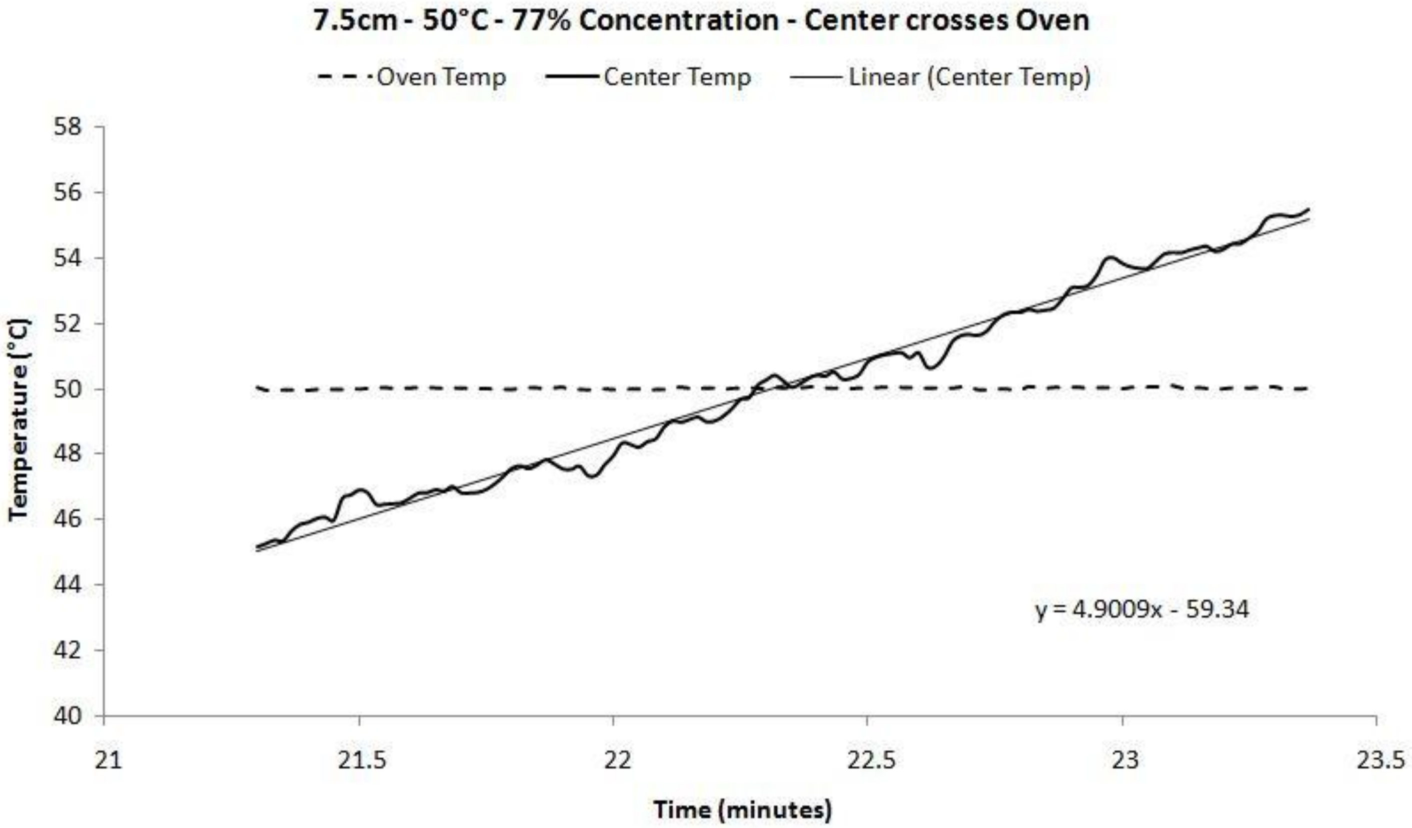


Figure 6.76: 7.5cm - 50°C – 77% Concentration – Center crosses Oven

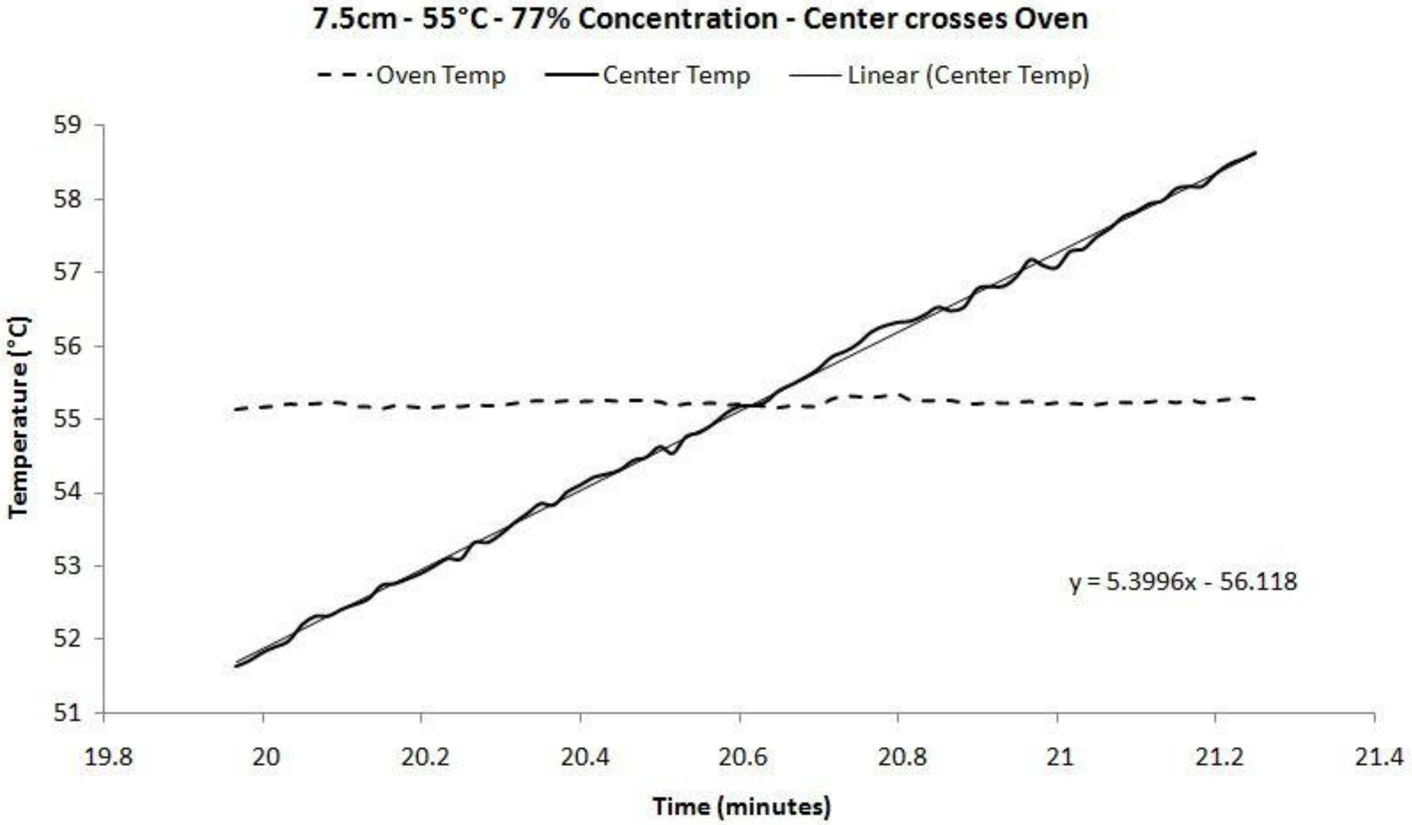


Figure 6.77: 7.5cm - 55°C – 77% Concentration – Center crosses Oven

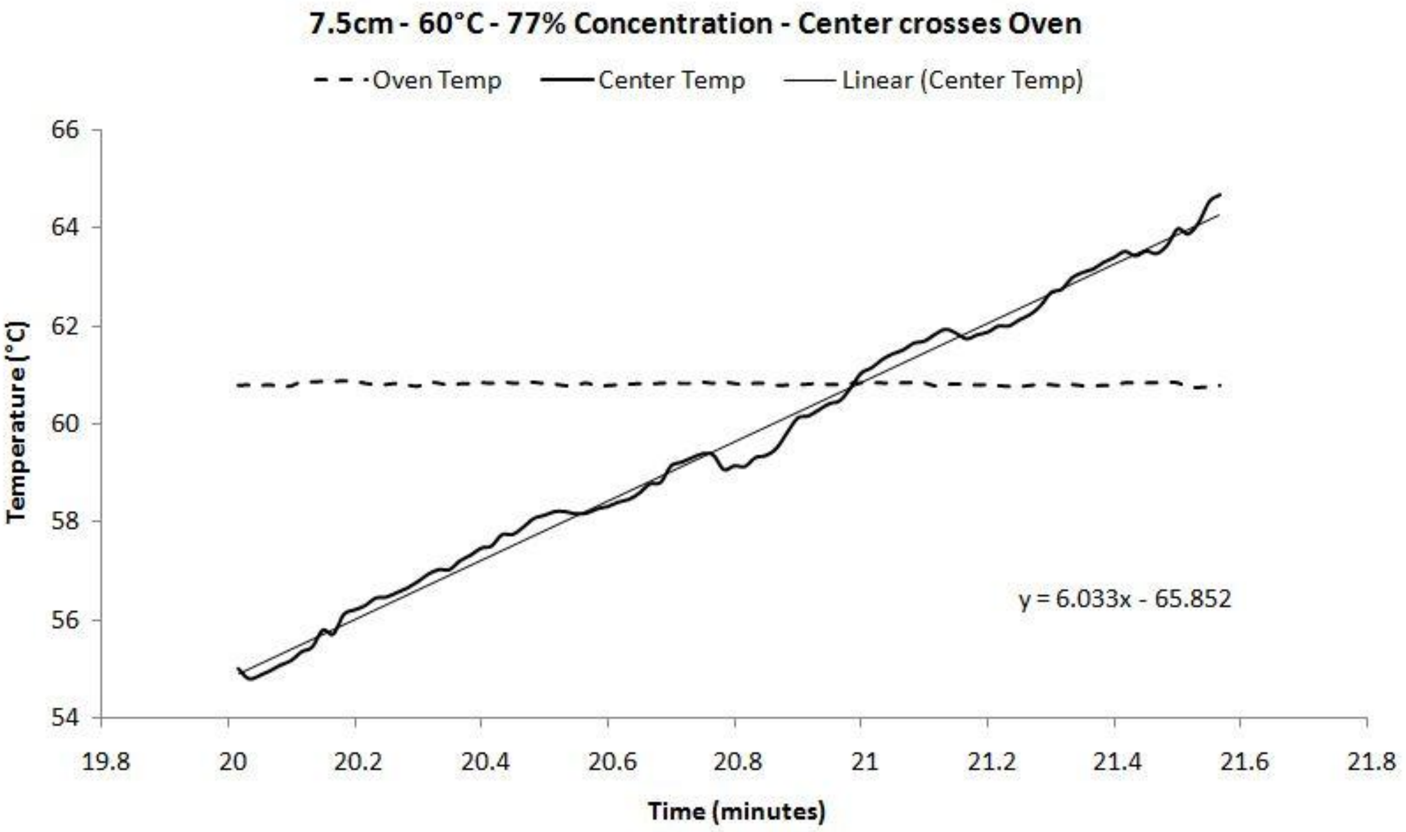


Figure 6.78: 7.5cm - 60°C – 77% Concentration – Center crosses Oven

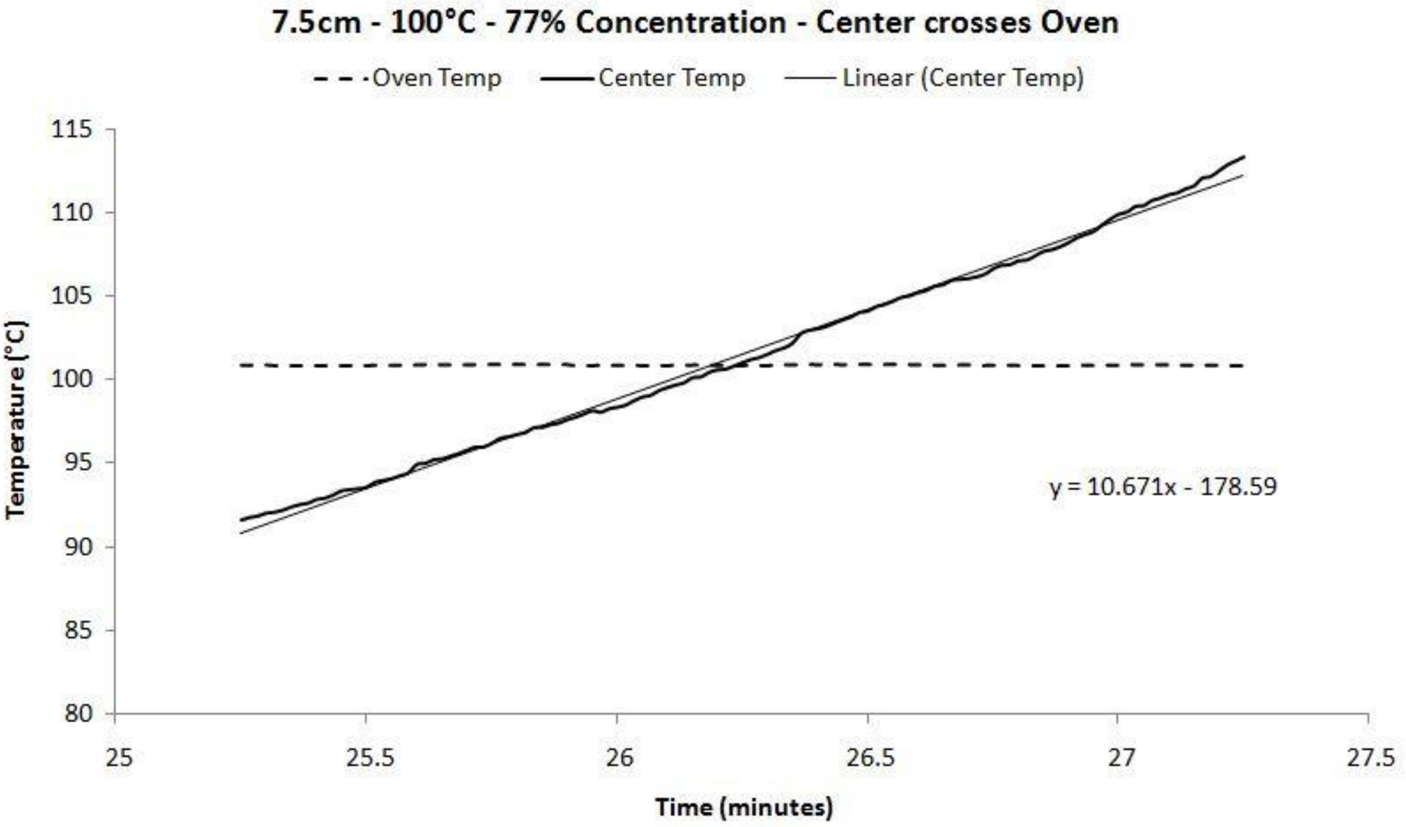


Figure 6.79: 7.5cm - 100°C – 77% Concentration – Center crosses Oven

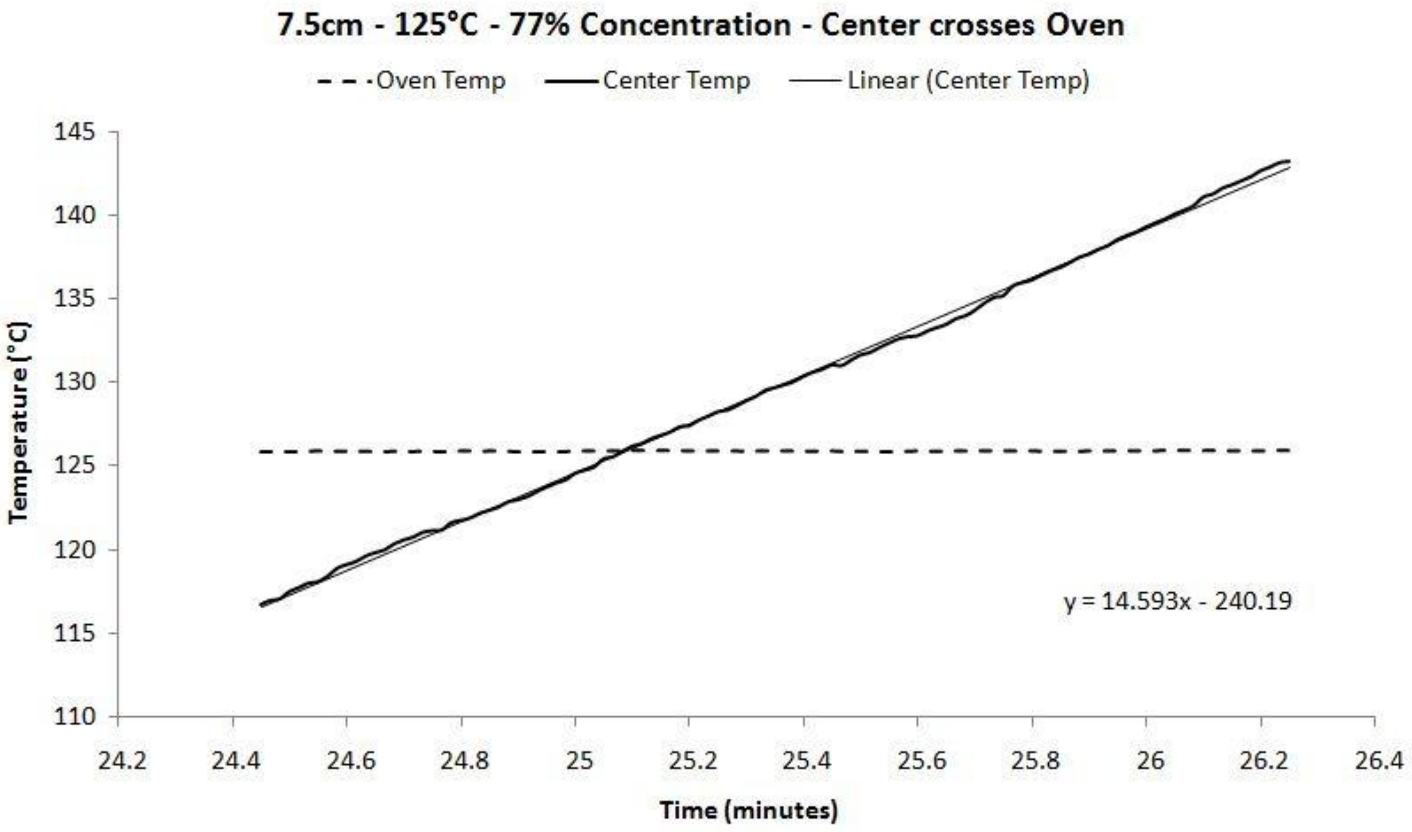


Figure 6.80: 7.5cm - 125°C – 77% Concentration – Center crosses Oven

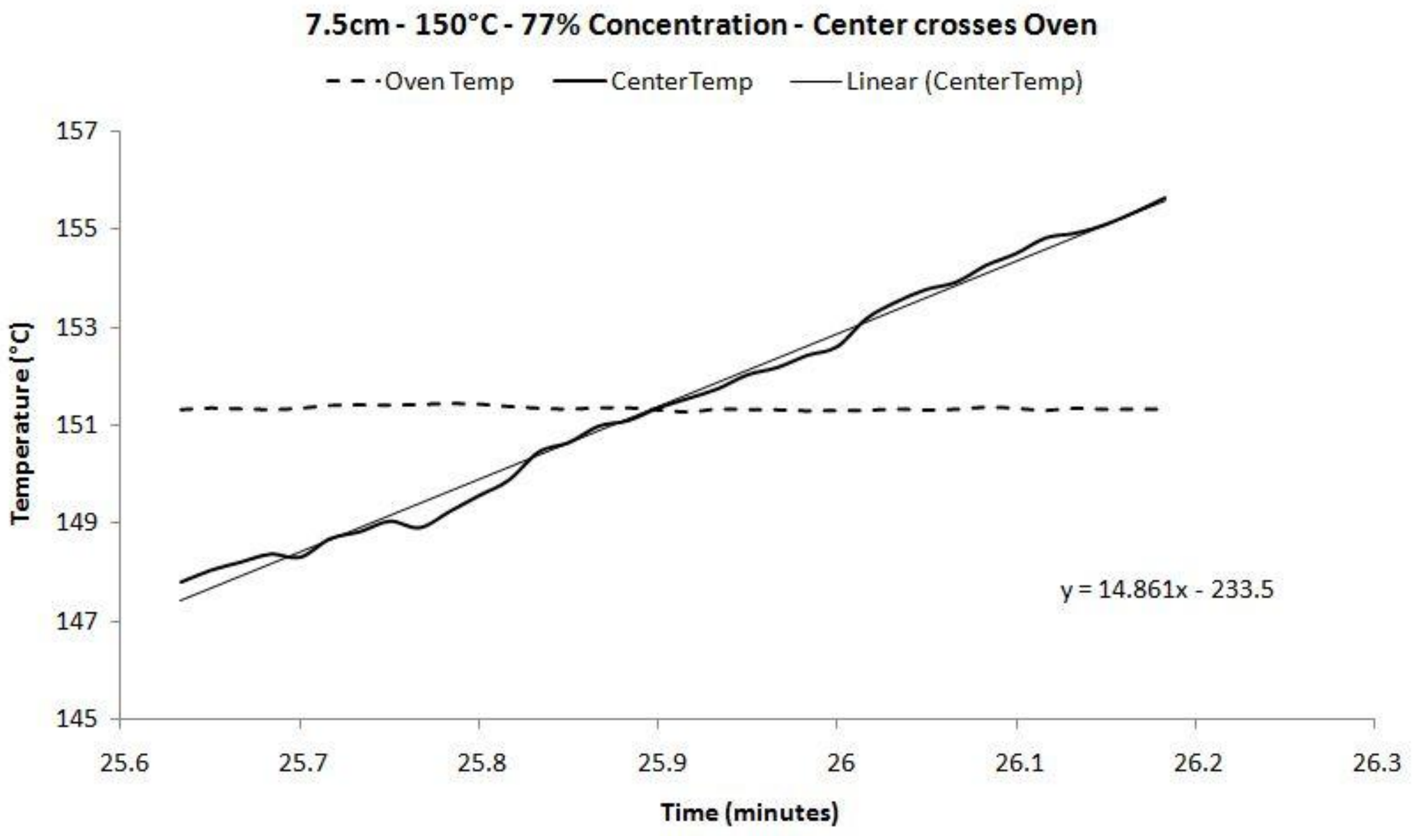


Figure 6.81: 7.5cm - 150°C – 77% Concentration – Center crosses Oven

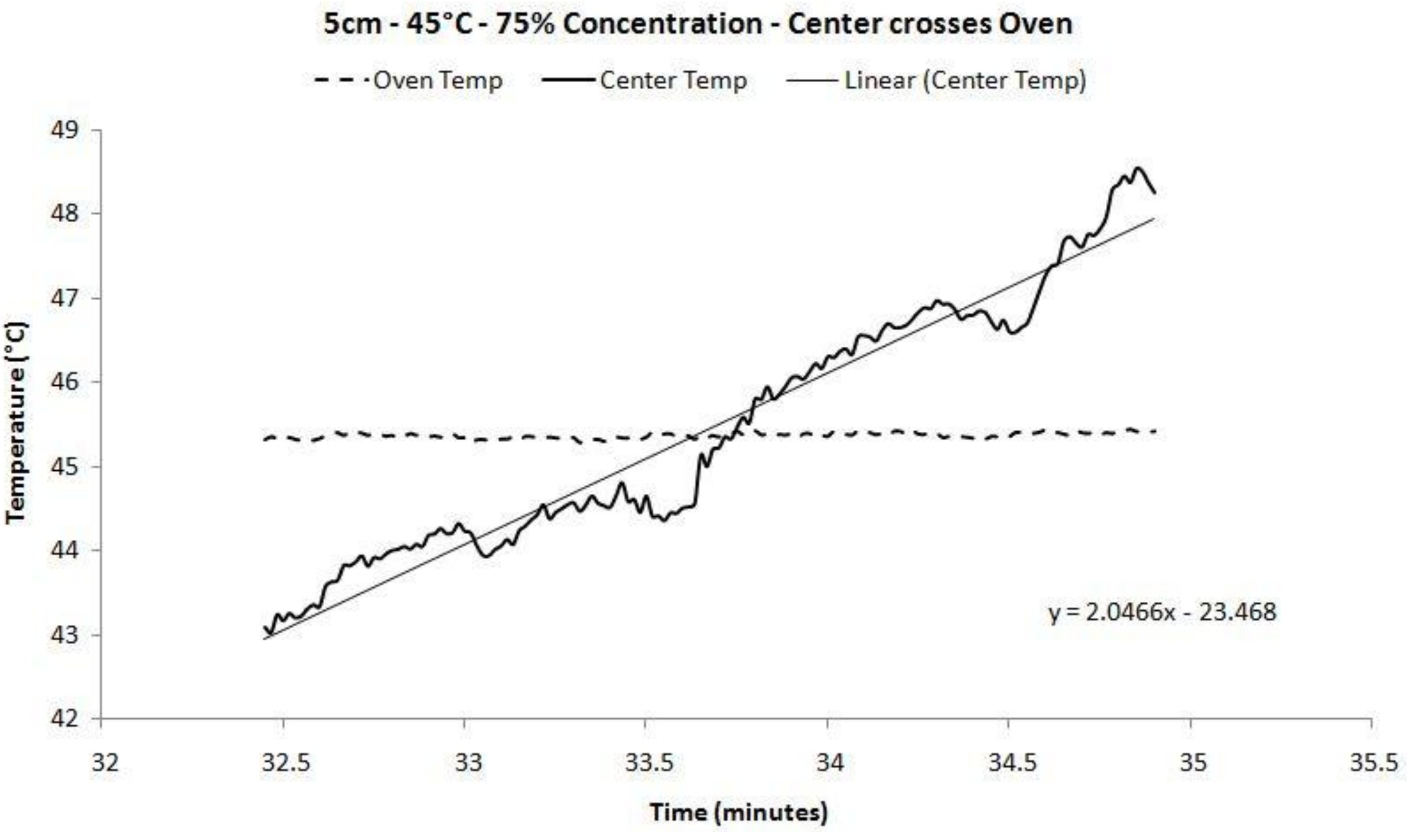


Figure 6.82: 5cm - 45°C – 75% Concentration – Center crosses Oven

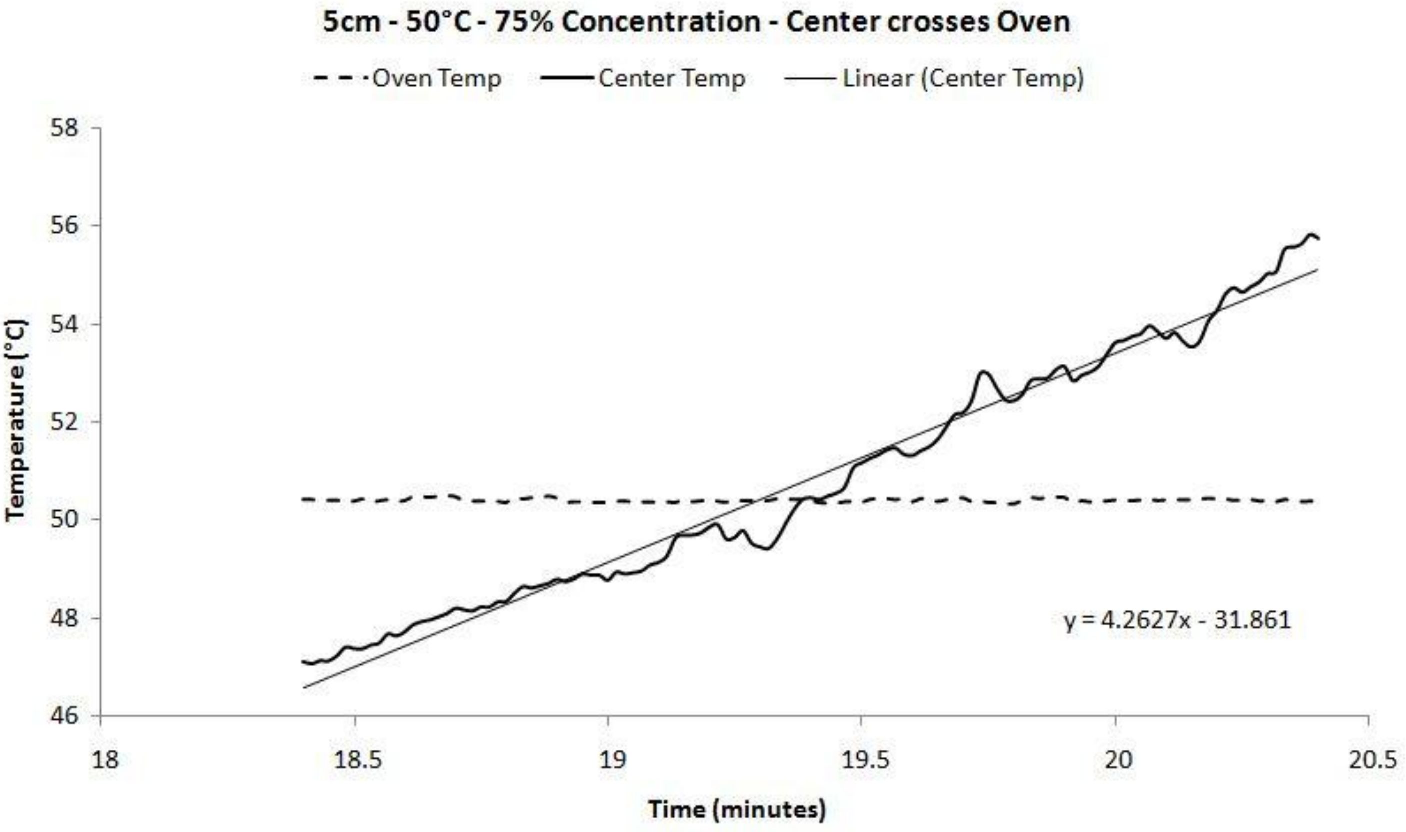


Figure 6.835cm - 50°C – 75% Concentration – Center crosses Oven

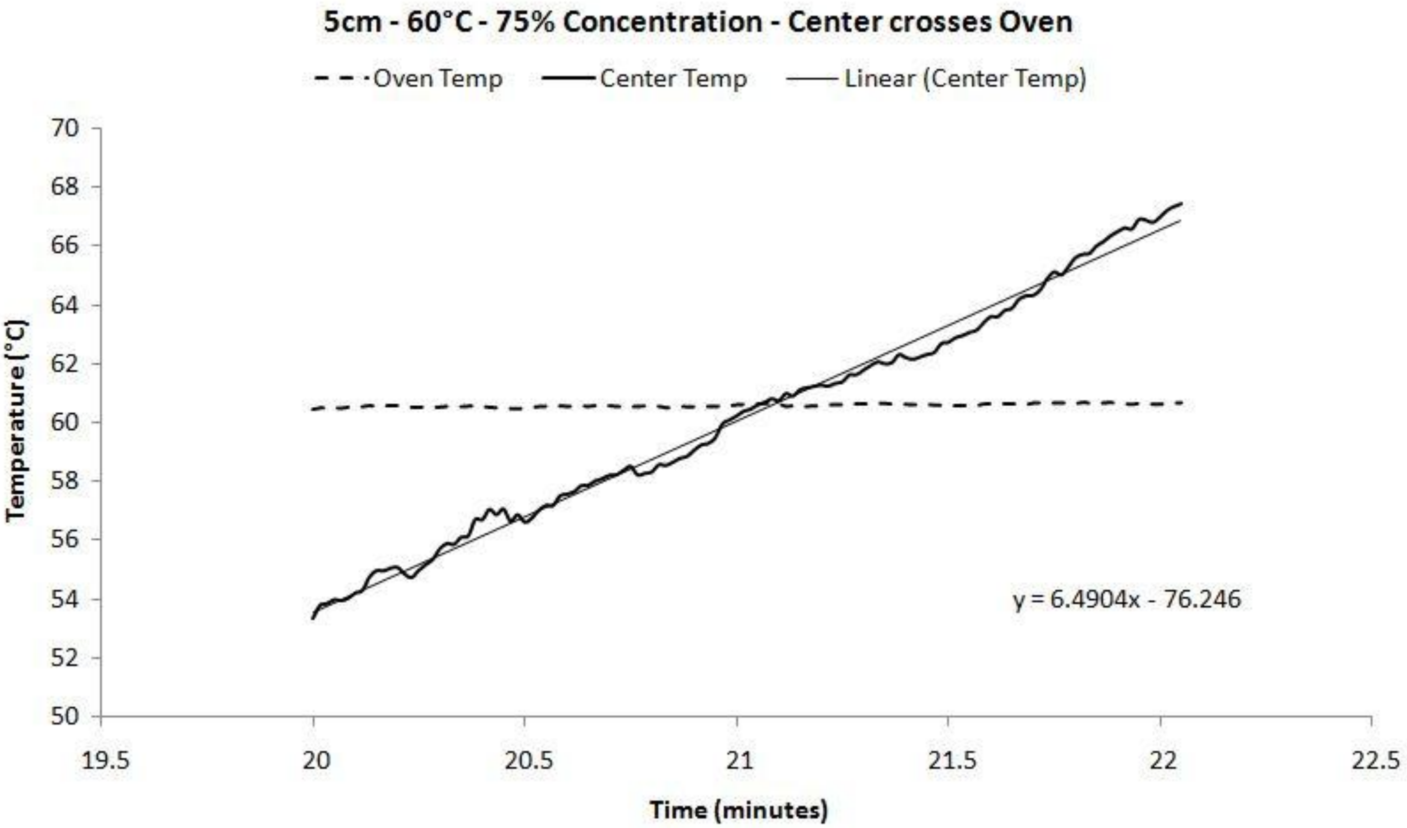


Figure 6.84: 5cm - 60°C – 75% Concentration – Center crosses Oven

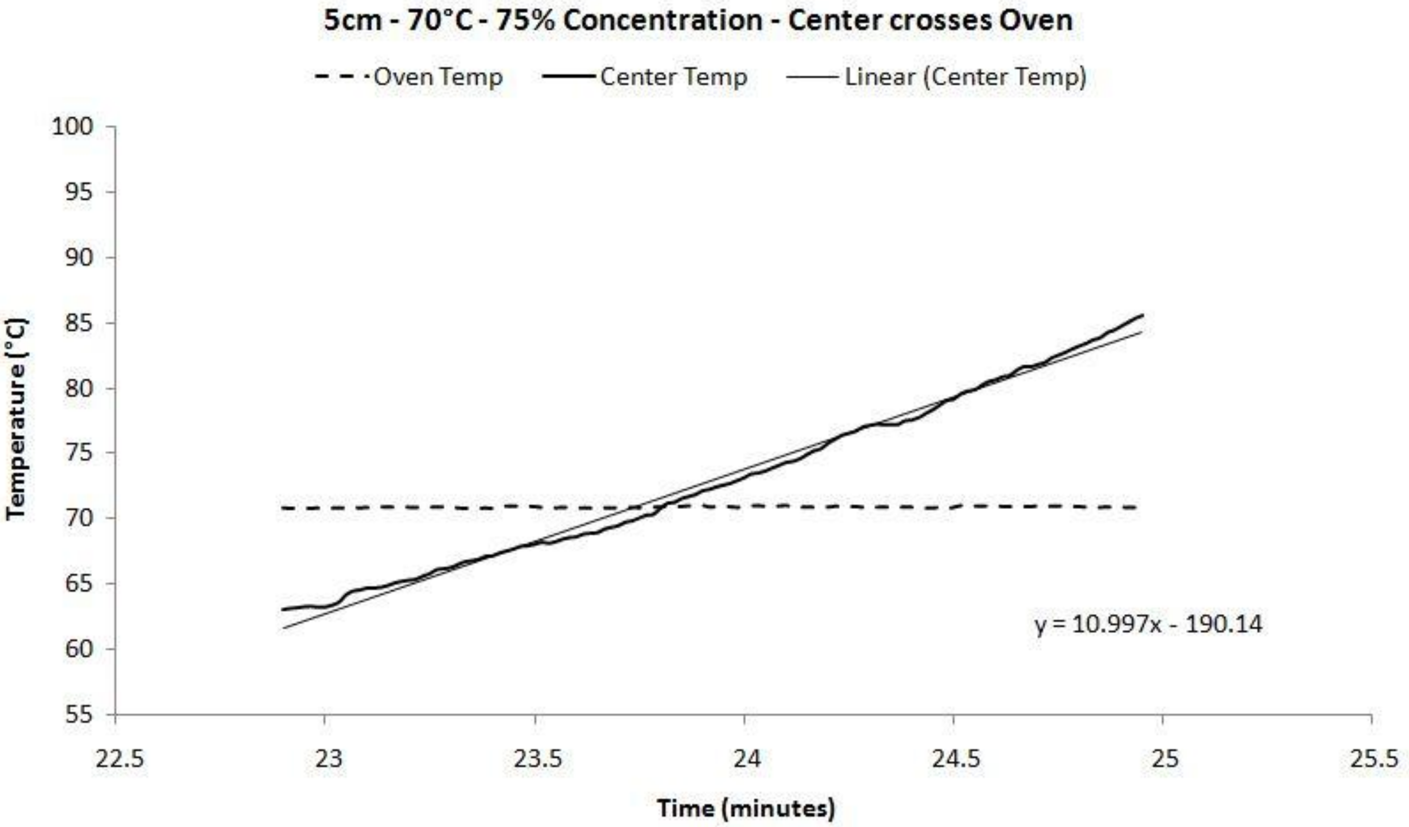


Figure 6.85: 5cm - 70°C – 75% Concentration – Center crosses Oven

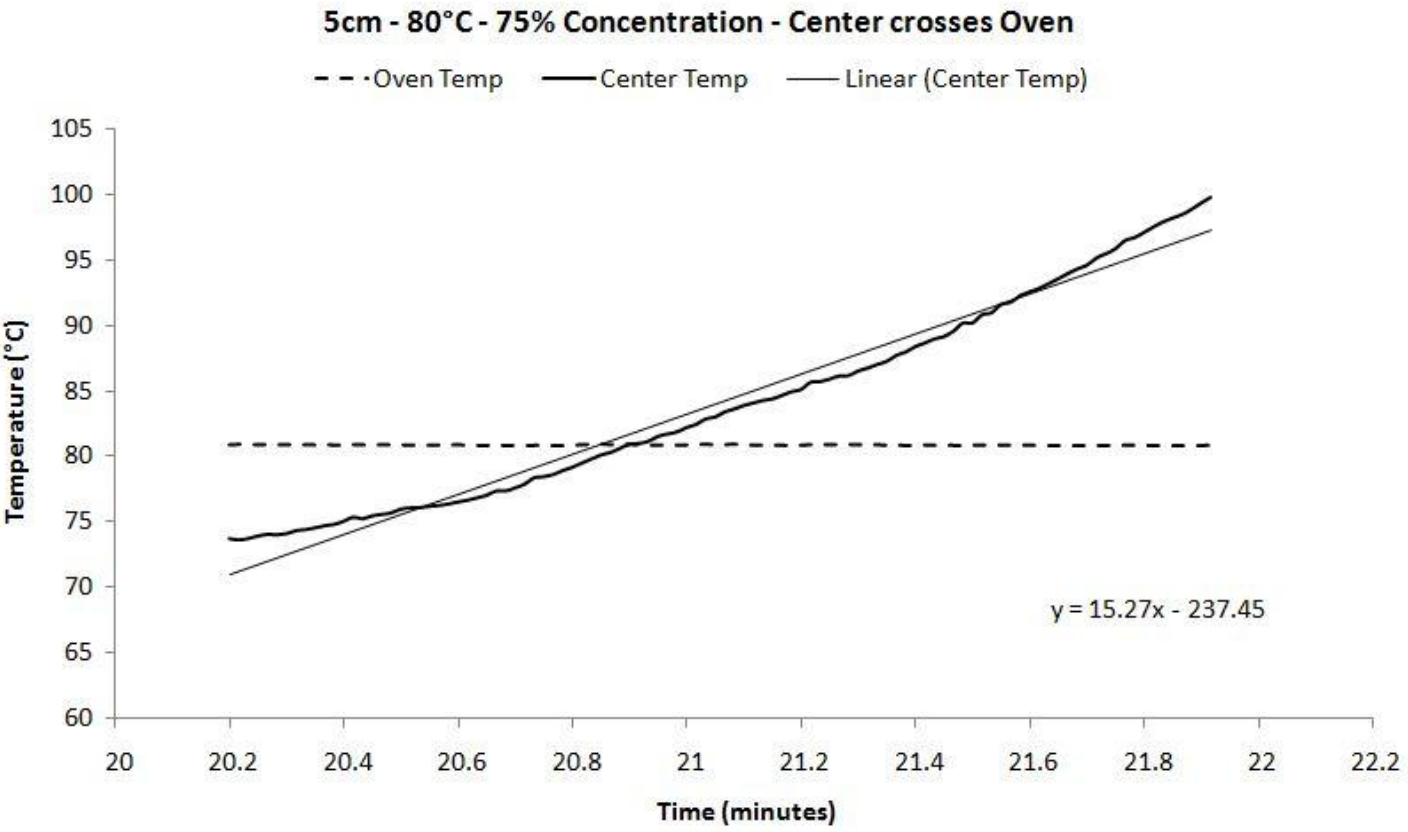


Figure 6.86: 5cm - 80°C – 75% Concentration – Center crosses Oven

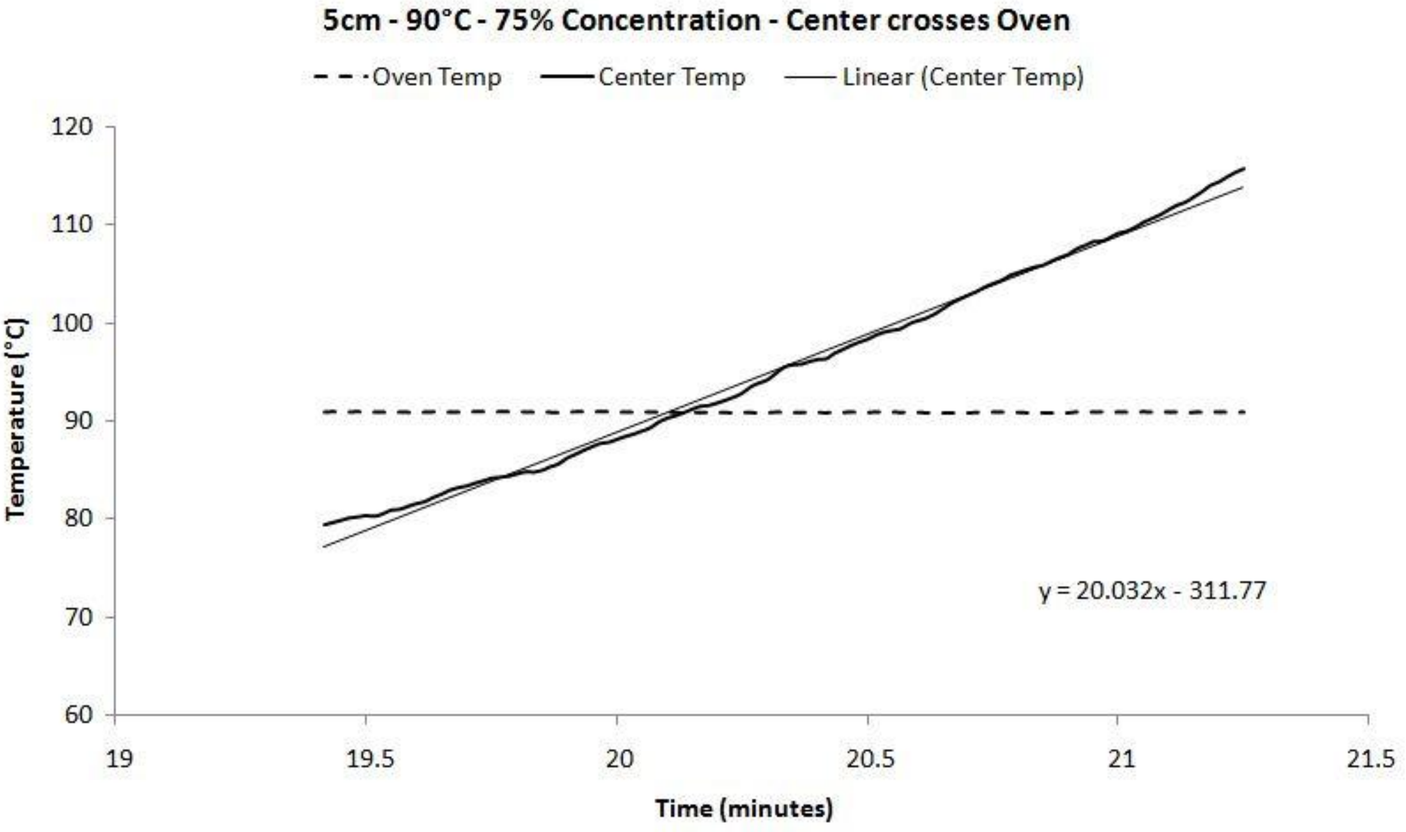


Figure 6.87: 5cm - 90°C – 75% Concentration – Center crosses Oven

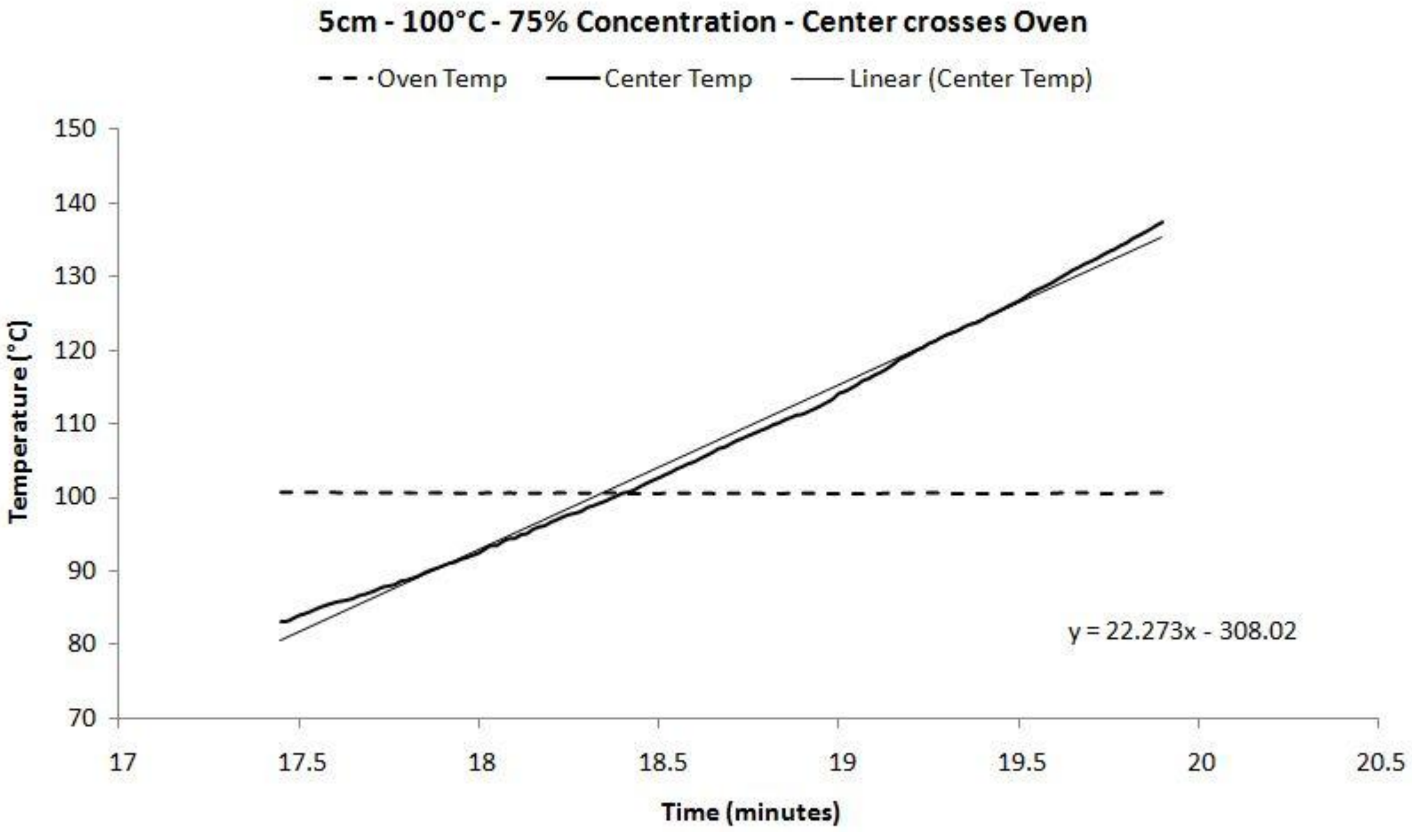


Figure 6.88: 5cm - 100°C – 75% Concentration – Center crosses Oven

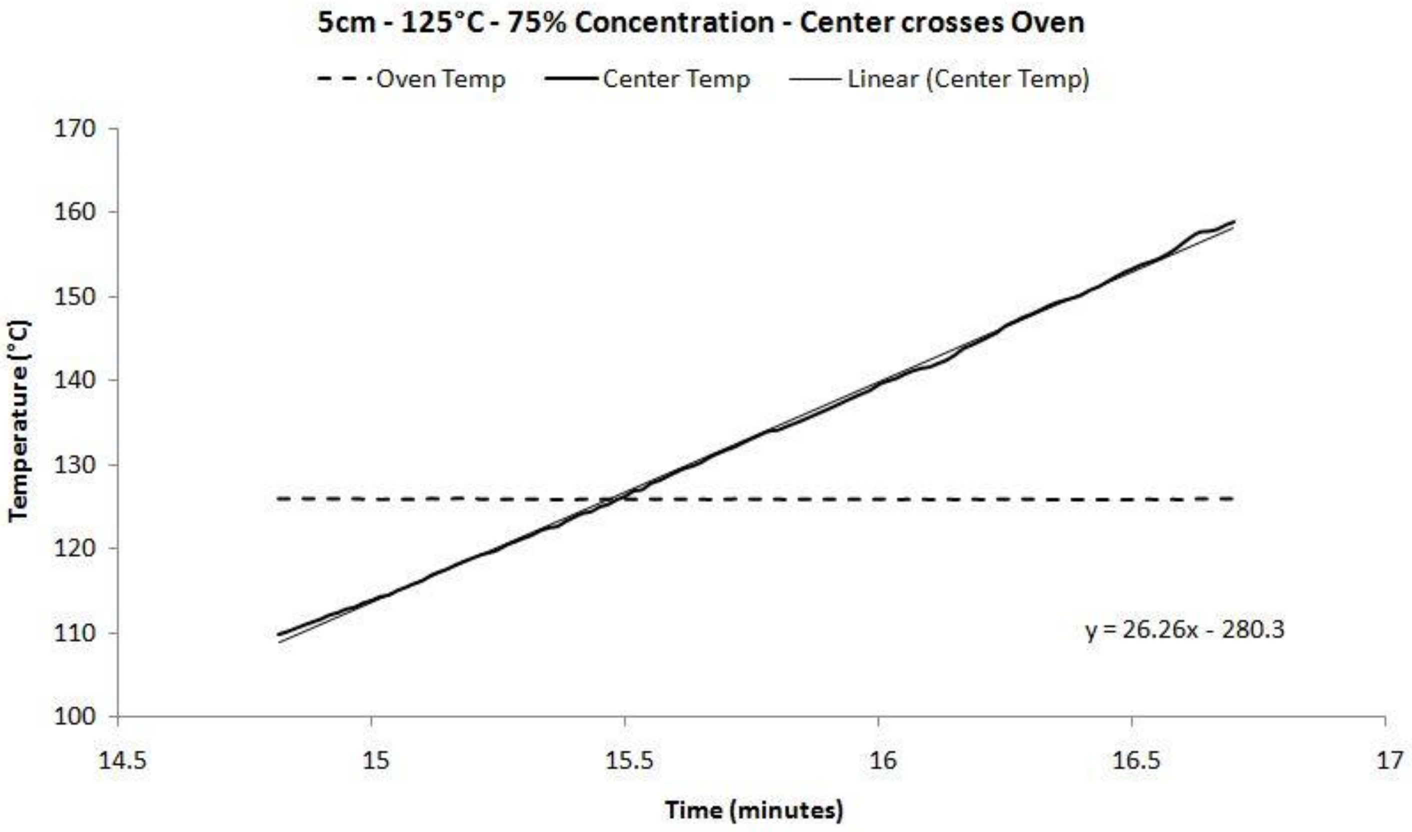


Figure 6.89: 5cm - 125°C – 75% Concentration – Center crosses Oven

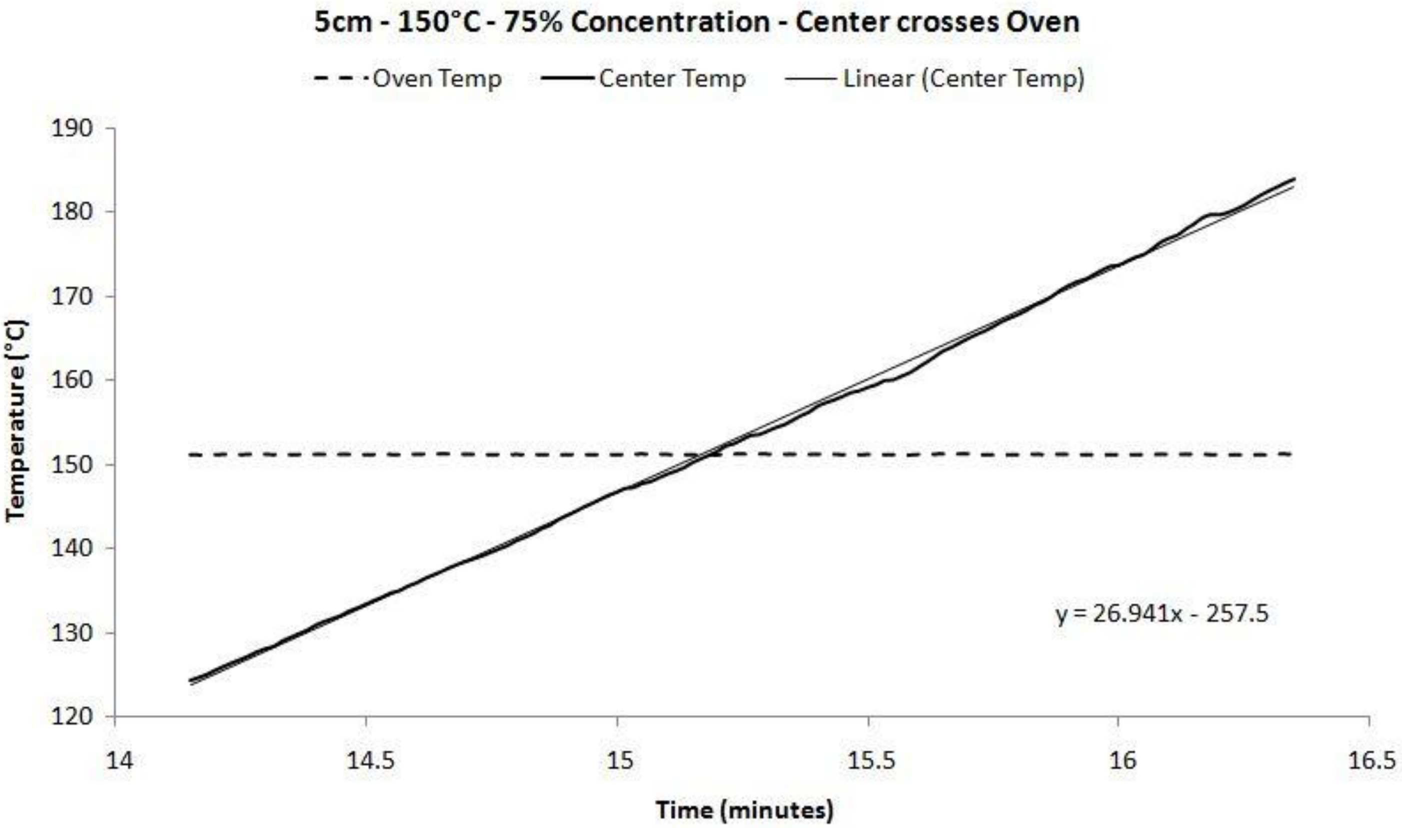


Figure 6.90: 5cm - 150°C – 75% Concentration – Center crosses Oven

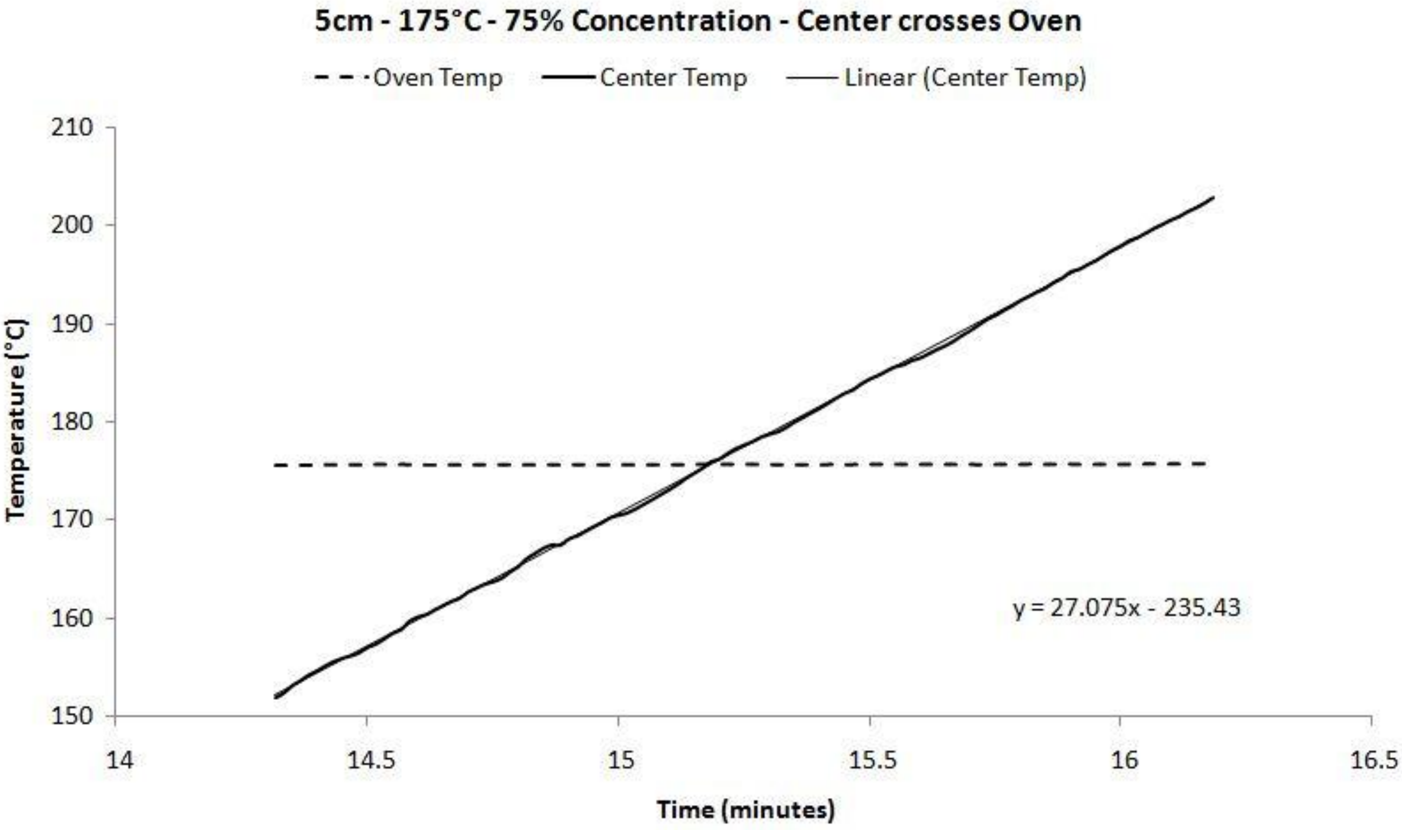


Figure 6.91: 5cm - 175°C – 75% Concentration – Center crosses Oven

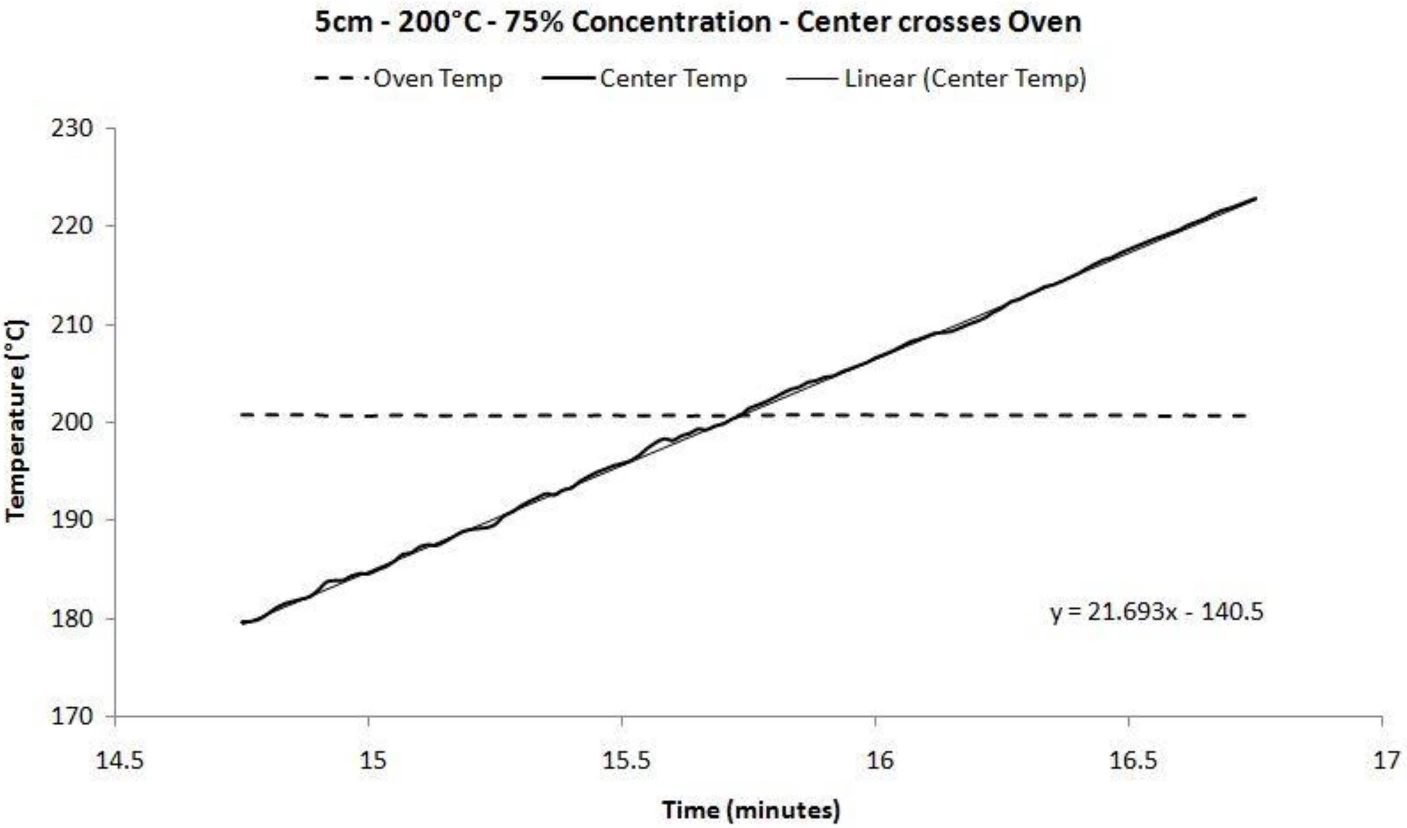


Figure 6.92: 5cm - 200°C – 75% Concentration – Center crosses Oven

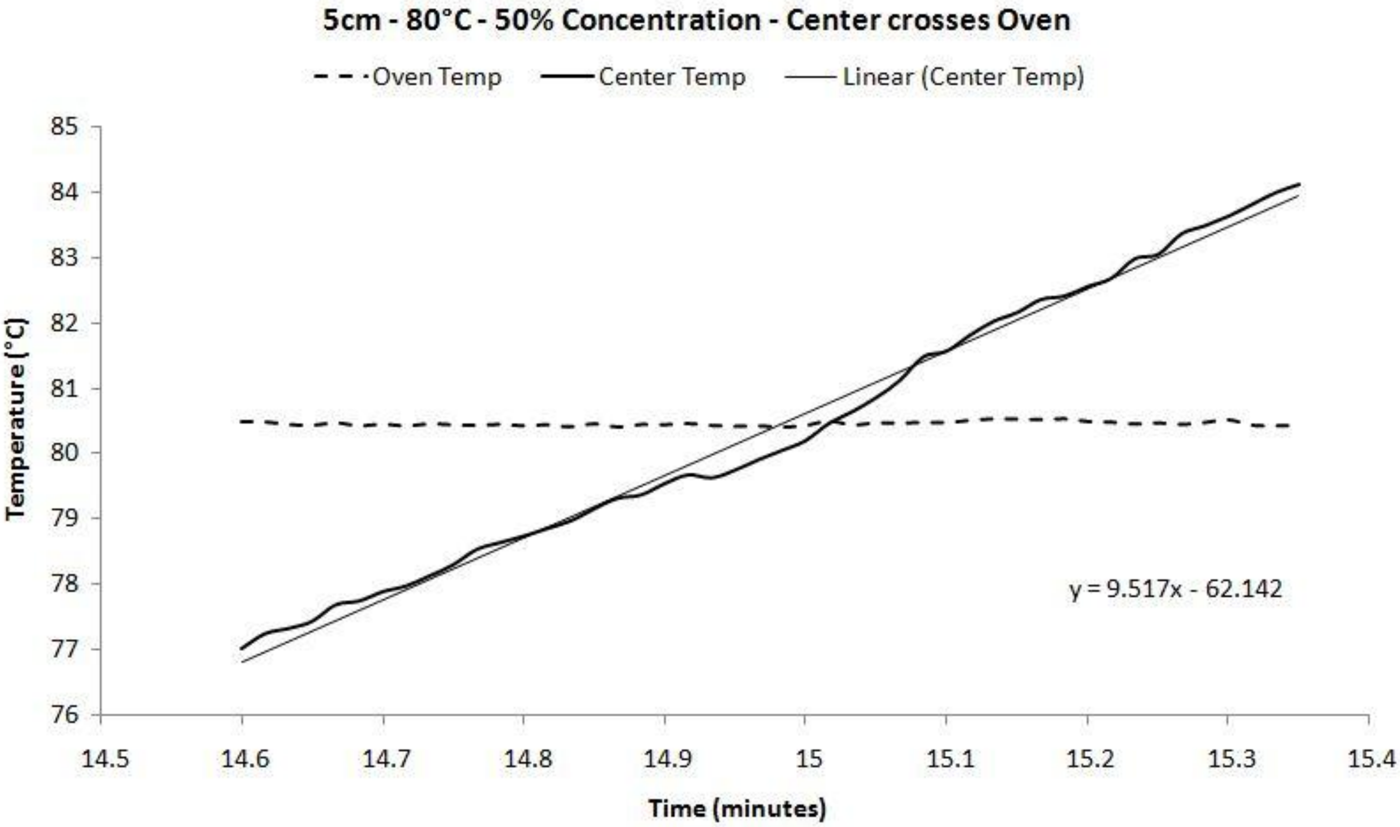


Figure 6.93: 5cm - 80°C – 50% Concentration – Center crosses Oven

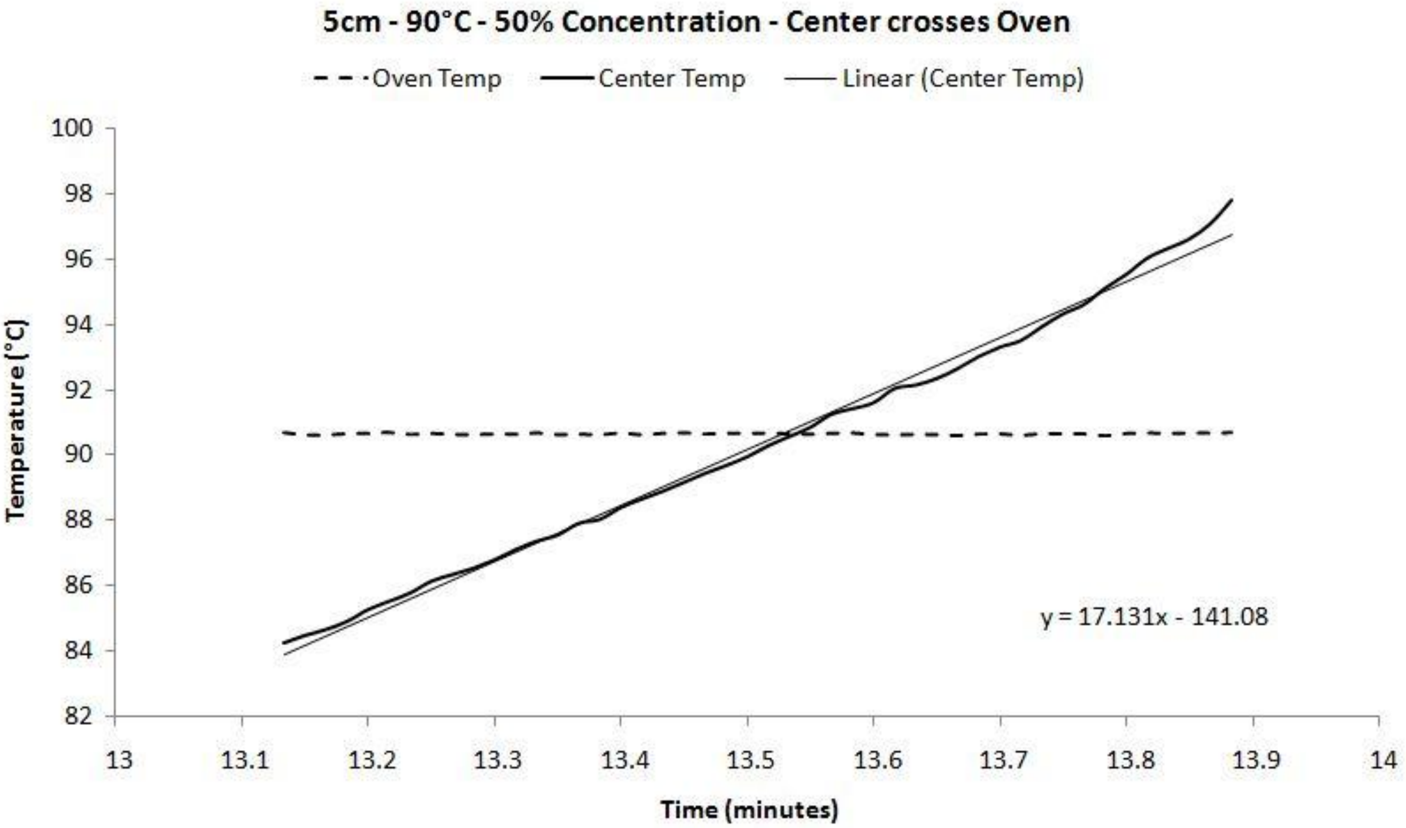


Figure 6.94: 5cm - 90°C – 50% Concentration – Center crosses Oven

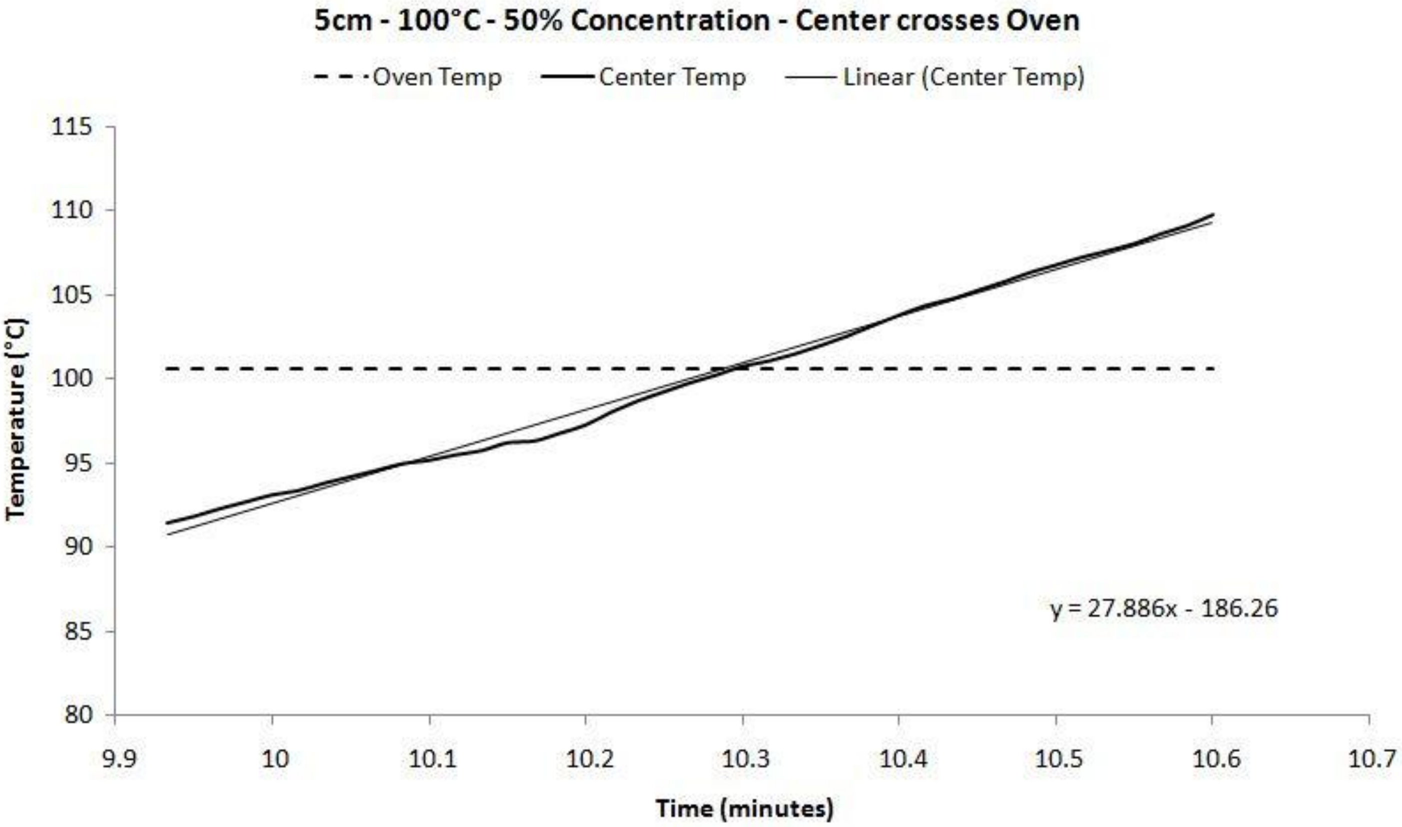


Figure 6.95: 5cm - 100°C – 50% Concentration – Center crosses Oven

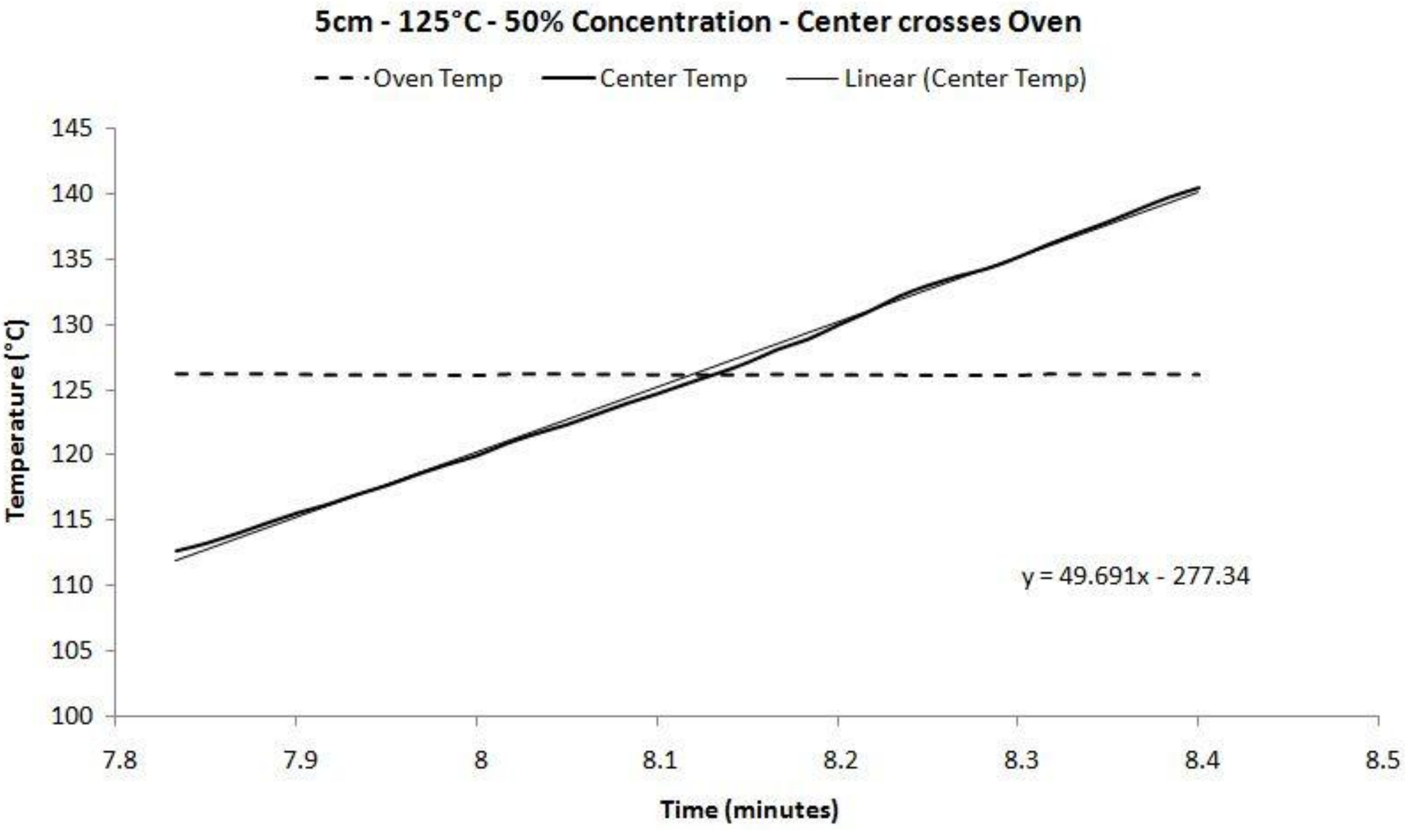


Figure 6.96: 5cm - 125°C – 50% Concentration – Center crosses Oven

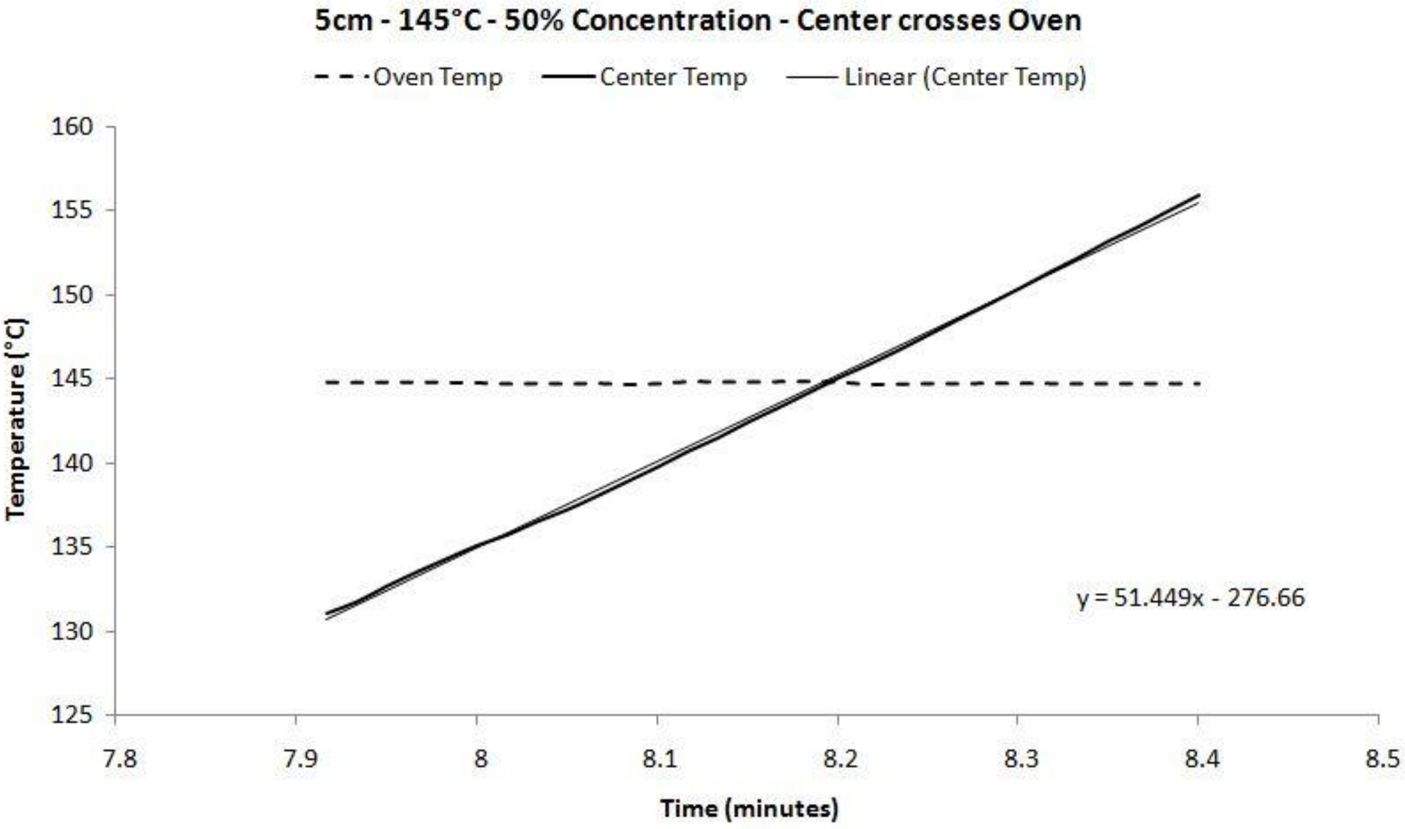


Figure 6.97: 5cm - 145°C – 50% Concentration – Center crosses Oven

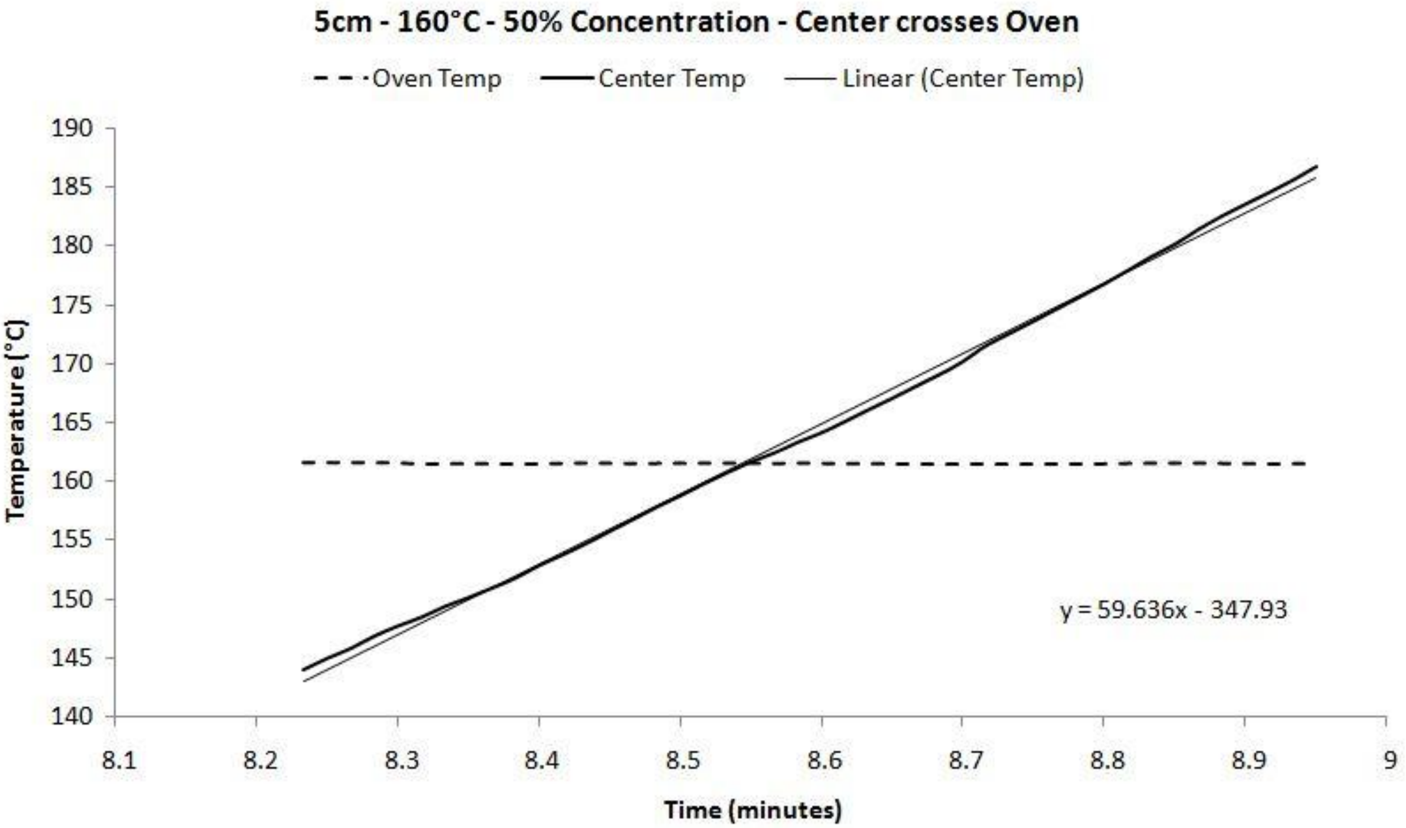


Figure 6.98: 5cm - 90°C – 50% Concentration – Center crosses Oven

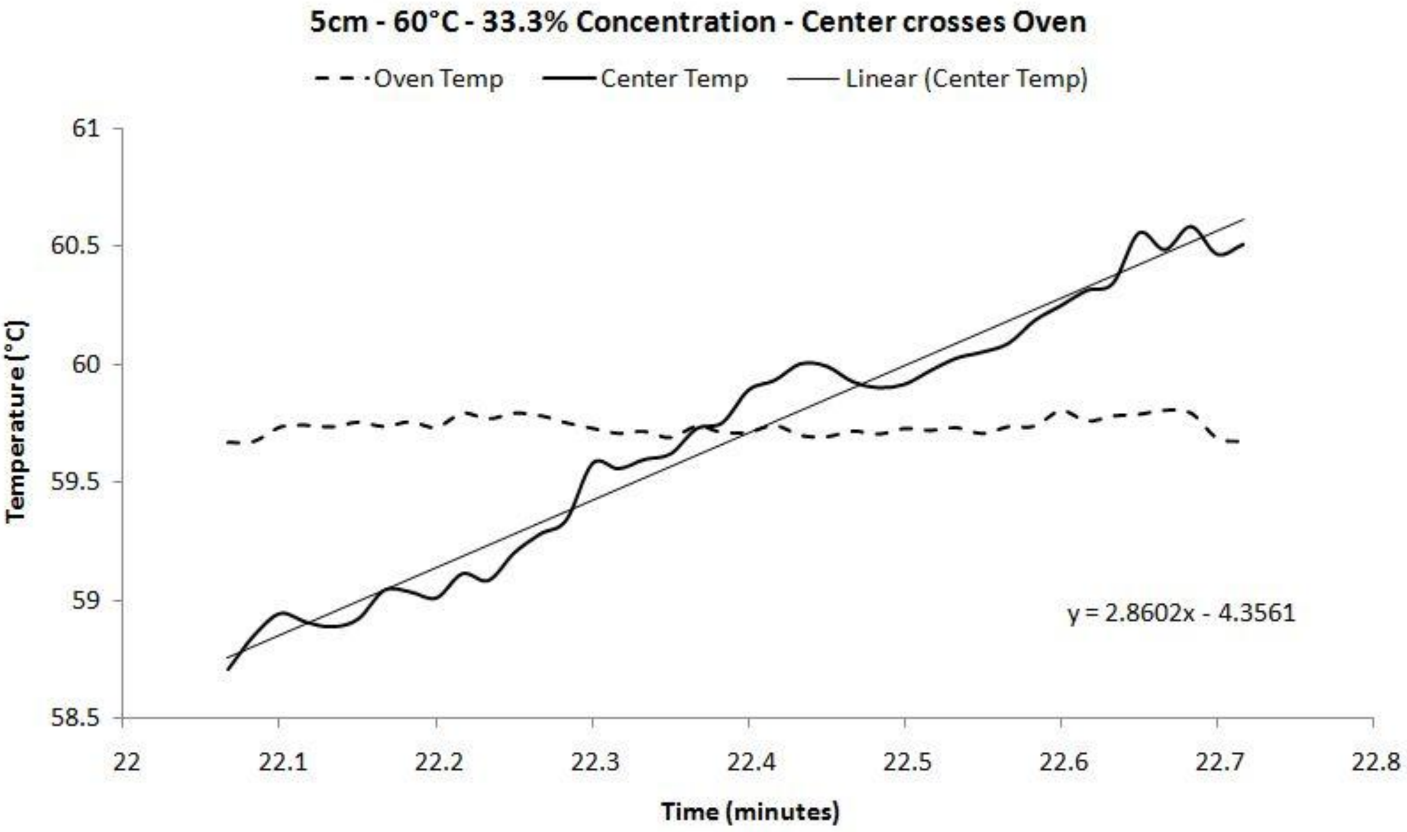


Figure 6.99: 5cm - 60°C – 33.3% Concentration – Center crosses Oven

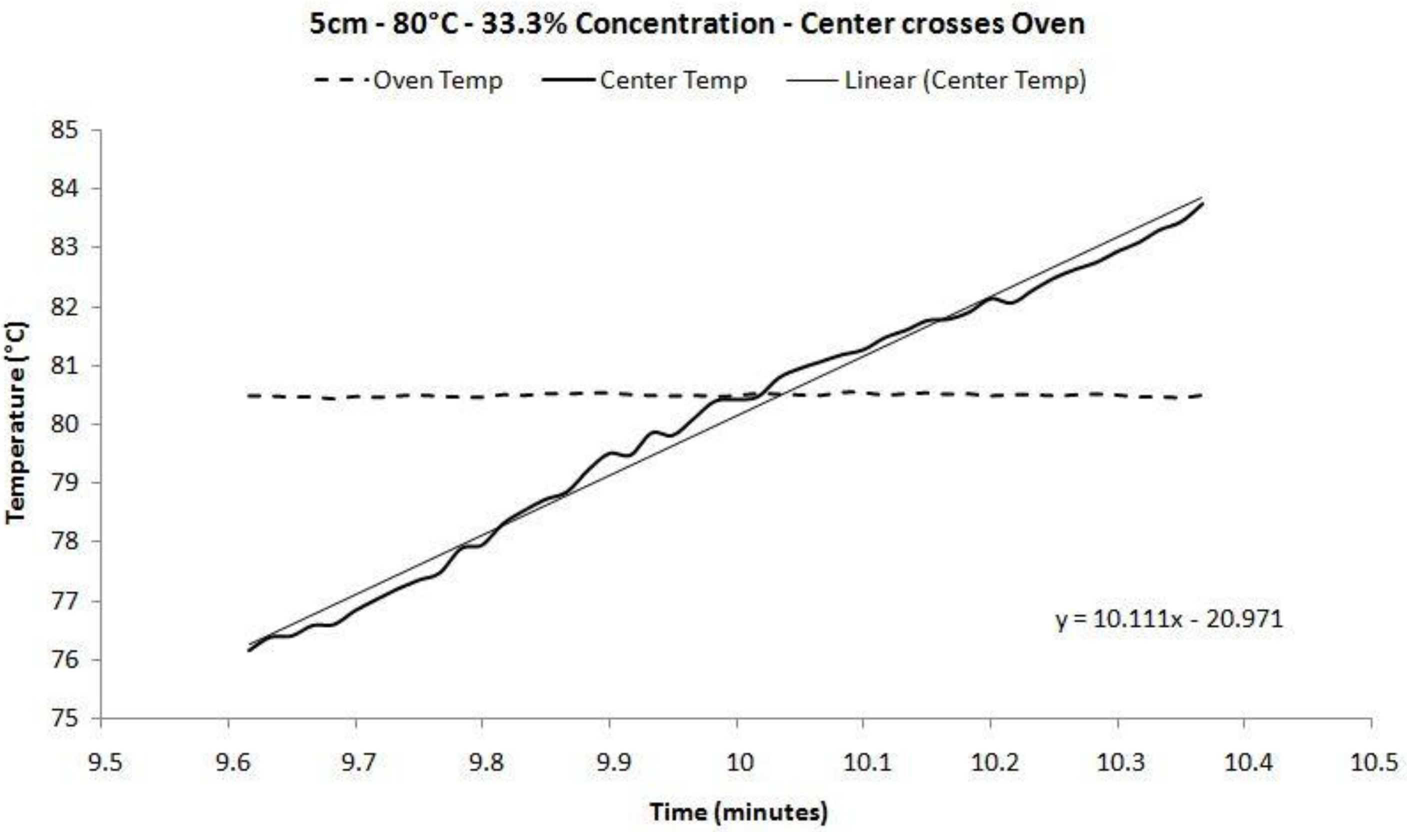


Figure 6.100: 5cm - 80°C – 33.3% Concentration – Center crosses Oven

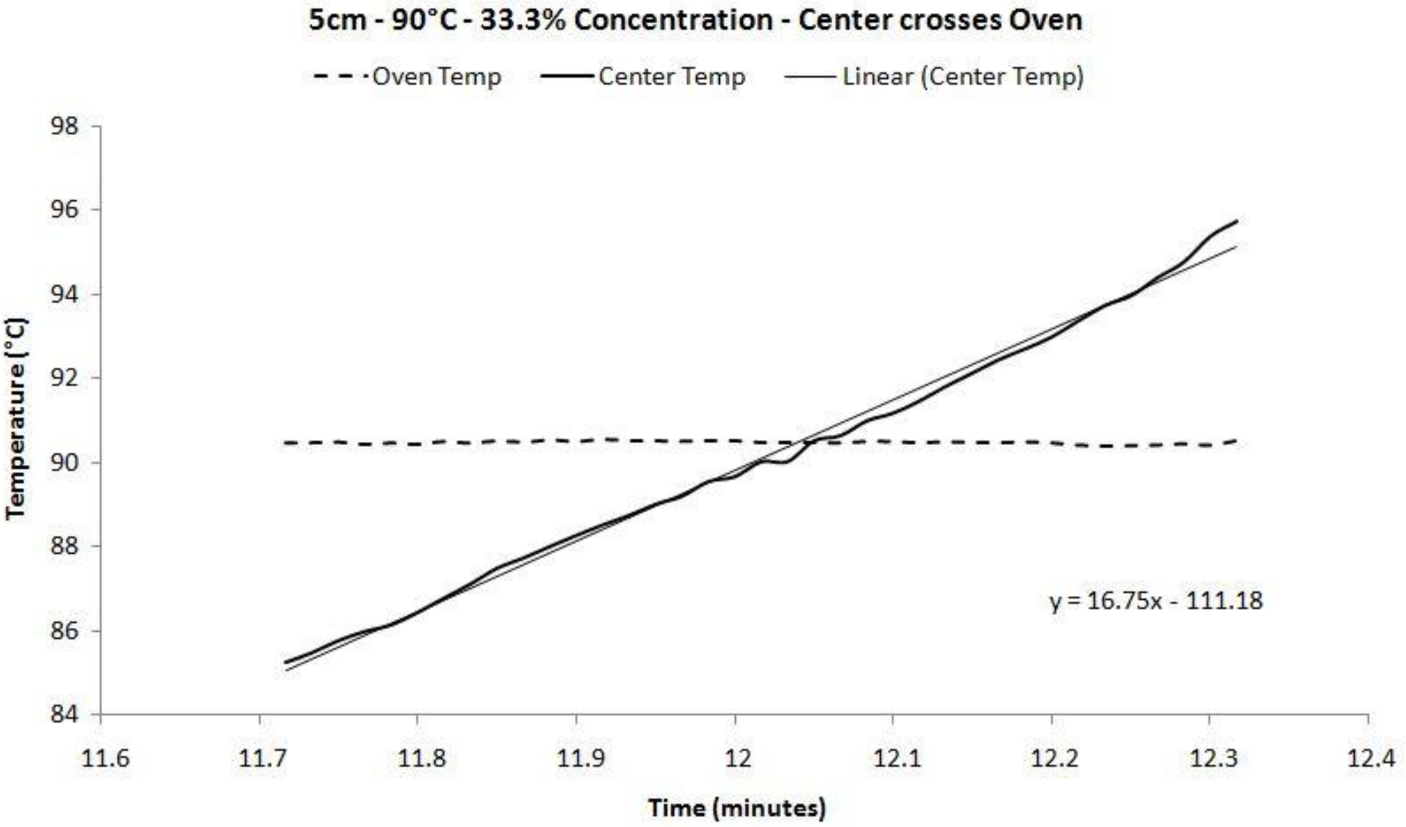


Figure 6.101: 5cm - 90°C – 33.3% Concentration – Center crosses Oven

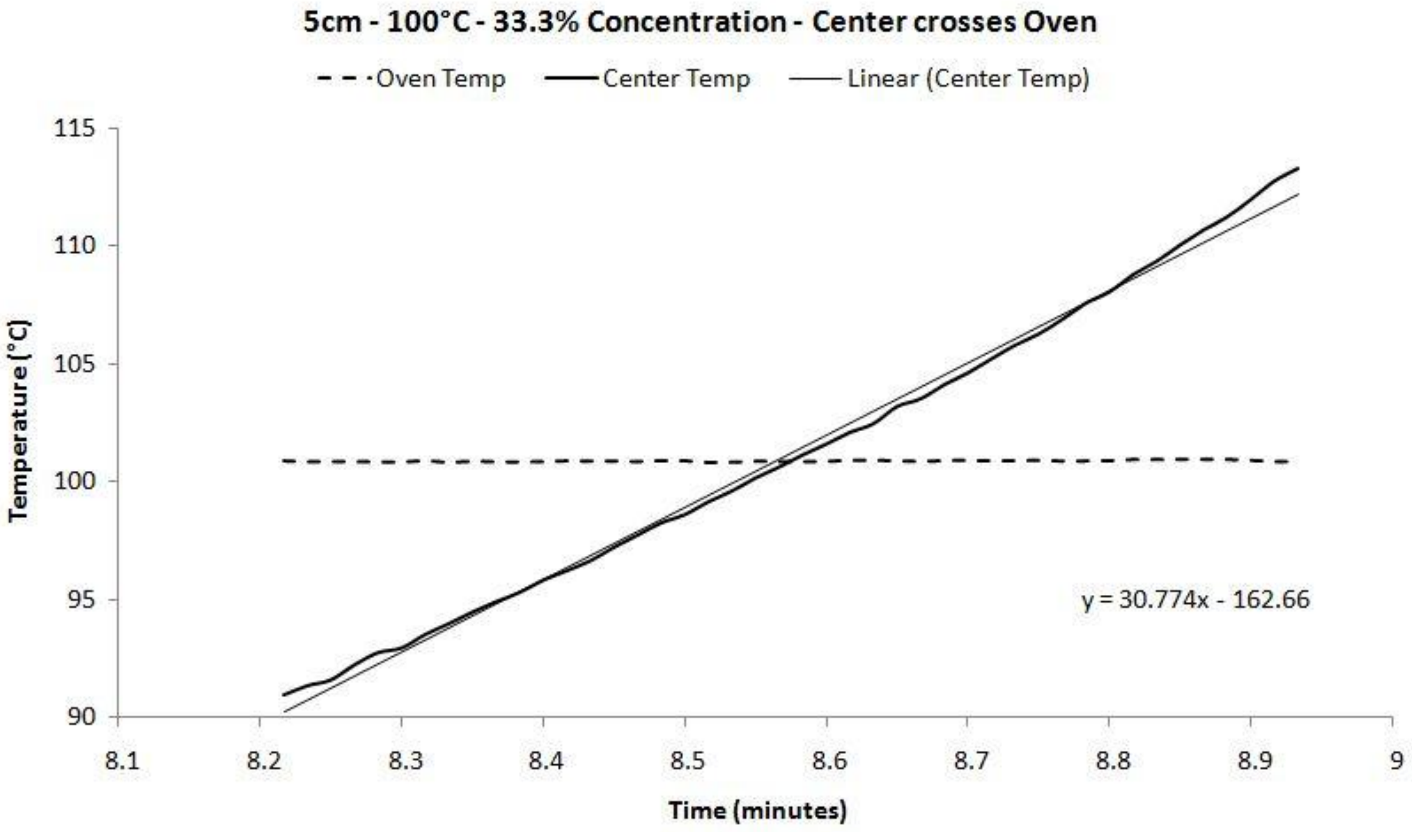


Figure 6.102: 5cm - 100°C – 33.3% Concentration – Center crosses Oven

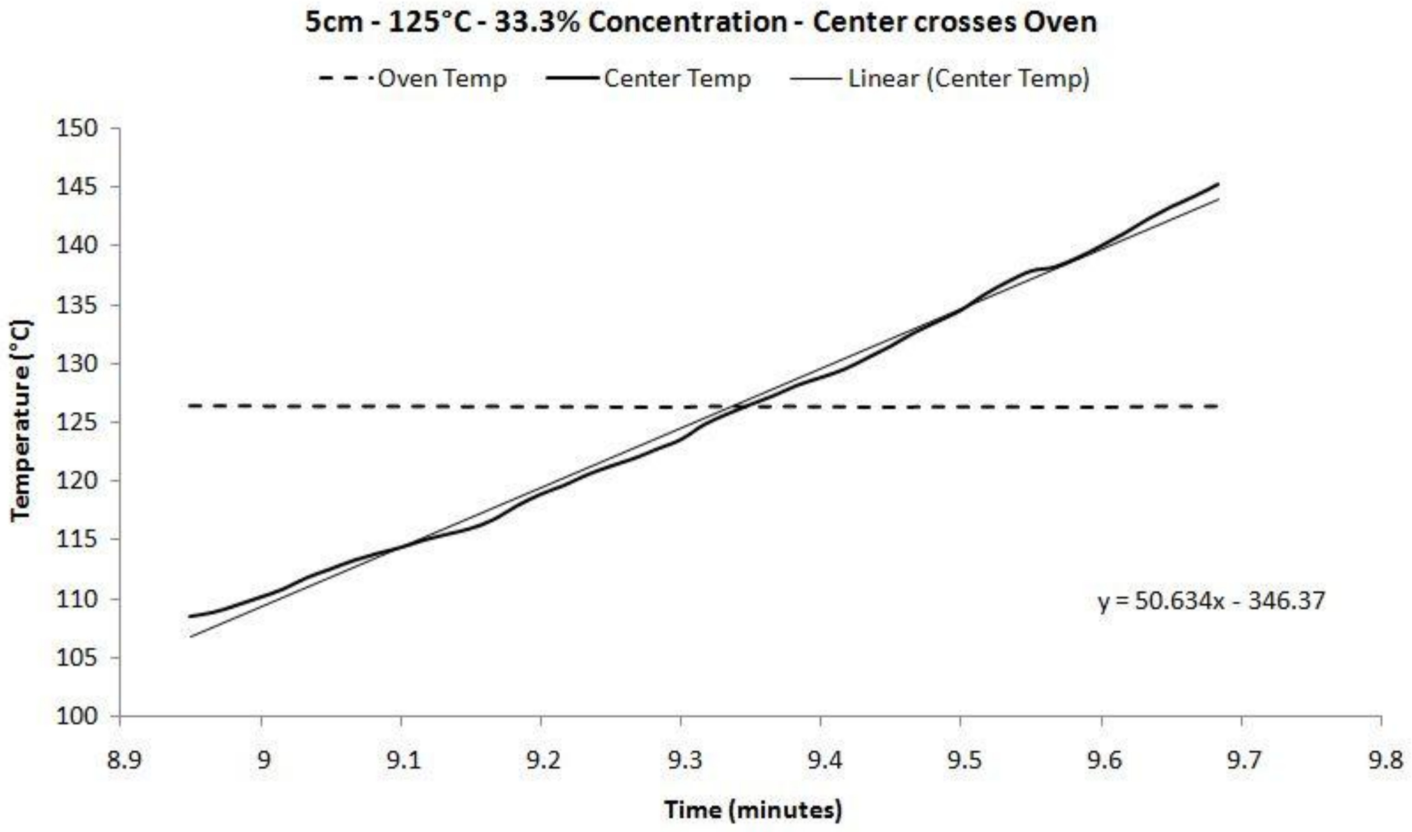


Figure 6.103: 5cm - 125°C – 33.3% Concentration – Center crosses Oven

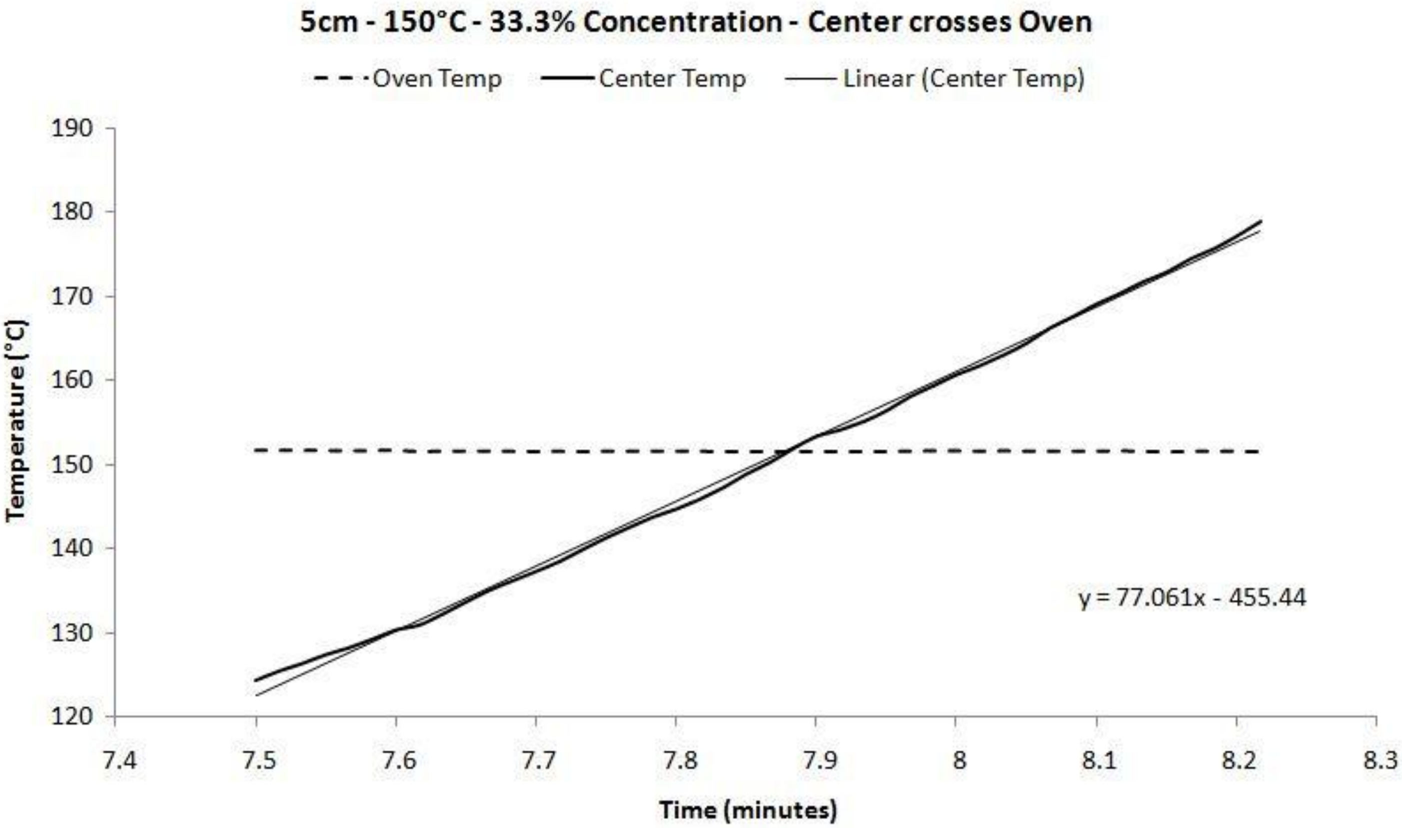


Figure 6.104: 5cm - 150°C – 33.3% Concentration – Center crosses Oven

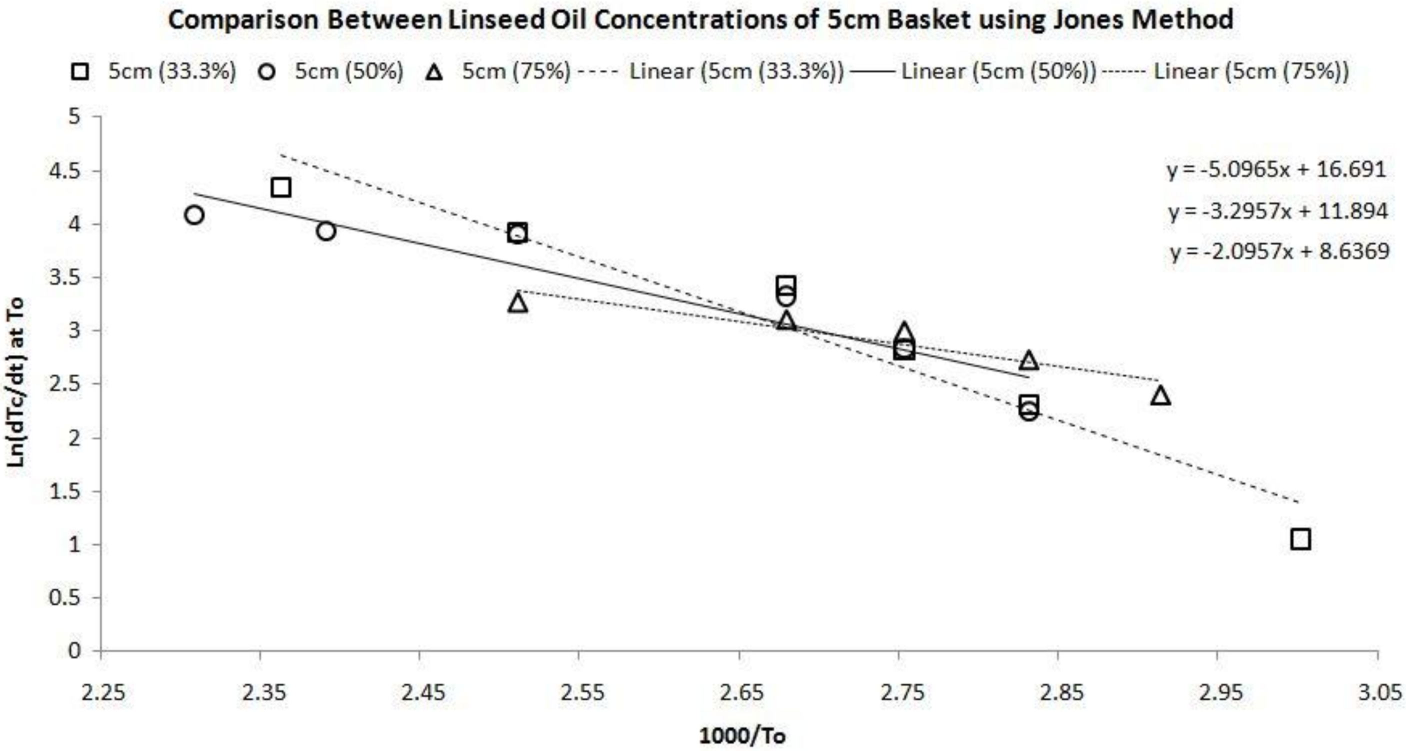


Figure 6.105: Comparison between Linseed Oil Concentrations of the 5cm Basket Size using Jones Method

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