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OPTIMIZATION OF ANTIFREEZE ADMIXTURE FORMULATIONS

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By

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Abstract

The objective of the study conducted was to optimize the proportions of three admixture formulations to be used for cold weather concreting and then to down select two for further research. For the purpose of the study an experimental plan was developed to conduct the optimization process based on the statistical method, Design of Experiments. The statistical data analysis program MINITAB was the tool used for generating the various runs of the experiments. The formulations were tested for the physical properties of mortars, Vicat Setting Time and Compressive Strengths. The experiments were carried out at varying temperatures in addition to varying proportions of the admixtures.

The results of the experimental runs were analyzed using the MINITAB program and a characteristic behavior pattern was developed over the range of admixture proportions for each formulation. The behavior pattern thus obtained was then used to predict the optimized proportions of the admixtures in each formulation using predefined criteria. The admixture formulations were down selected to two (one primary and one back up) based on the synergistic effects of the admixtures with each other.

Finally a detailed study of the effect of IPANEX, one of the admixtures used in the study, on the yields and its interaction with other admixtures was conducted using MINITAB. Thus the optimization of the admixture proportions was obtained through the statistical analysis.

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Chapter 1
Introduction

1.1 Background

Concreting during cold weather poses various challenges in terms of placement of concrete, strength gain, curing and so on. Behavior of concrete hydration changes with regards to temperature, requiring different heat inducing techniques for satisfactory placement at low temperatures. Employment of such techniques proves to be uneconomical. The cold weather experienced in certain areas for a considerable amount of time in a year reduces the construction season drastically [1]. The need to tackle the problem of reduced construction season has led to efforts of developing a concrete that is efficient at low temperatures.

Solutions such as heating enclosures and insulation have been applied to the problem of cold weather concreting since the 1930s. However these methods have not undergone any considerable amount of change until recently. Heating enclosures and insulation techniques are extremely costly, consume a huge amount of energy and also require skilled labor. The recent work focuses on the development of an antifreeze admixture to depress the freezing point of water thereby making it possible for the concrete to achieve maximum possible strength at low ambient temperatures. This reduces the cost of construction and energy required considerably.

The research undertaken by Small Business Innovation Research follows up on the earlier work conducted in the field of cold weather concreting. It aims at developing an antifreeze admixture formulation which is a combination of various admixtures. The proportions of the admixtures are optimized using statistical tools and experimentation.

1.2 Problem Statement

As mentioned in Section 1.1 the recent work in the area of cold weather concreting is focused on the development of an antifreeze admixture formulation for the placement of concrete at low temperatures in accordance with the specifications of ACI 306-02. Admixtures such as Pozzutec 20+ have been tested individually as possible solutions to the problem of cold weather concreting. Admixtures have also been tested in combination with each other. However the proportions of these admixtures were either as prescribed by the manufacturer or as mentioned in the ASTM 494 C.

The current cold weather concreting code ACI 306-02 does not provide any specific details about the methods by which the standard atmospheric conditions for concreting must be maintained at low ambient temperatures. Similarly ASTM 494 C, the standard for specifications of chemical admixtures for concrete, provides limiting values for the proportions of individual chemical admixtures to be used in concrete. However, it does not provide any clear idea about the recommended proportions for the admixtures when used in combination with each other.

A previous phase of the current research down selected three combinations of admixtures from 18 admixture combinations. The focus of the current research was to optimize the proportions of the admixtures in the formulations down selected in the previous phase of research.

1.3 Objectives

1) Optimization of admixture formulations identified in the Phase 1 of the project.
2) Down select to two admixture formulations.
3) Determining the effect of IPANEX on the yield and its interaction with the other factors.

1.4 Scope of Research

The research focuses on the optimization of the proportions of admixtures in the formulations based on the mortar properties. Three admixture formulations were down-selected from a pool of 18 in the Phase 1 of the current research. A hypothesis that higher concentrations of admixtures will depress the freezing point of the water in concrete was assumed for the optimization process [2]. The proportions of the admixtures were increased and a matrix of runs with the combinations of the admixtures at various levels of proportions was developed with the help of a statistical tool MINITAB, for the optimization process. The Vicat setting time test and the compressive strength test were conducted on each of the run combinations. Analysis of the test results was performed using the statistical tool MINITAB. Based on the results of the analysis and the initially determined criteria for optimization, the optimized proportions of the admixtures were finalized. The Vicat setting time and Compressive Strength test were then conducted on the optimized proportions of admixtures for the verification of the analysis. Analysis was conducted to determine the effect of IPANEX on the setting time and compressive strength and its synergistic effect on the other admixtures.

Chapter 2

Literature Review

2.1. Behavior of Concrete at Low Temperatures

"Cold weather as defined by ACI 306 - 2002 is a period when for more than 3 consecutive days the following conditions exist viz. the average daily temperature is less than 40°F and the air temperature is not greater than 50°F for more than one half of any 24 hour period" [3].

2.1.1. Mechanism of Hydration

Hydration of concrete is a chemical reaction between cement and water. The rate of a chemical reaction is influenced largely by the temperature at which the reaction takes place. This has been illustrated in the Arrhenius equation. The Arrhenius equation provides the relation for the dependence of the rate constant of a reaction on the temperature at which the reaction takes place. The equation [4] is as follows:

$$k = Ae^{-E_a/RT} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where,

k - Rate Constant

A - Prefactor

E_a - Activation Energy

R – Gas Constant

T – Temperature

The Equation (1) shows that the rate of a chemical reaction increases with an increase in temperature and decreases with a decrease in temperature. The collision theory states that for a chemical reaction to take place the reacting molecules must attain a certain minimum energy known as activation energy. The amounts of molecules that attain this energy collide with each other to form the product molecule. The activation energy required to initiate a reaction increases with the decrease in temperature. At low temperatures considerably more activation energy is required to sustain a chemical reaction at a particular rate [4].

At low temperatures the activation energy required for the hydration of concrete increases causing the rate of reaction to reduce. As the ambient temperature reduces further, at a certain temperature, in absence of external effort, the reaction of hydration ceases completely. Due to this there is no further gain in strength of the concrete. The partial gain in strength of concrete can prove to be hazardous to the concrete [4].

The low temperature also causes the water in the pores (interlinking voids between the cement and aggregate particles of concrete) of concrete to freeze. The pore water in the concrete starts freezing at -1^{o}C. As some water freezes the ion concentration in the unfrozen water rises, thus depressing the freezing point further. The pore water freezes completely at about -3^{o}C to -4^{o}C. At this point depending on the strength gained by the concrete due to partial hydration the frozen water may cause damage to the concrete due to its volume expansion [5].

2.1.2. Effect of Low Temperatures on the Concrete Properties

The concrete properties such as workability, setting time, compressive strength and freezing point are very important to the performance of concrete in cold weather conditions from the point of view of construction schedules and quality. Thus the effect of cold weather conditions on these properties is also of importance. The effect of low temperatures on the respective properties is presented here.

2.1.2.1. Compressive Strength:

The advantage shown by concrete placed at warm temperatures is short term and exhibits greater early strength. In long term these advantages are diminished and the concretes cured at low temperatures but above freezing point of water provide better results [6]. This can be illustrated by the results obtained by Korhonen in 1999 as shown in Table 1. Table 1 indicates the strength values as percentages of the strength of a control concrete cured at 20^{o}C. The values of strengths at 20^{o}C, -5^{o}C, -10^{o}C and -20^{o}C were as obtained in the Cold Regions Research and Engineering Laboratory (CRREL) testing of 0.39 W/c ratio concrete. The rest of the values of the strengths were as expected according to ACI 318 -2002.

Table 1: Strength Data for 0.39 W/c ratio concrete at varying temperatures and over a period of 56 days [6]

Temp(^{o}C)	Strength					
	1 day	3 days	7 days	14 days	28 days	56 days
50	17.7	33.6	58.1	69.3	75	77
40	16	38.1	69.7	79.2	85	85
30	13.1	36.6	72.1	87.3	94	93
20	10	30	70	90	100	105
10	3.7	15	49	75.6	97	110
5	2	14	30.1	56.7	83	100
-5	1	6.7	14	16.6	20.3	46.8
-10	0	0.2	1.7	2.7	4.2	51.1
-20	0	0	0	0.2	0.8	37.3

At temperatures lower than or close to freezing points the behavior of concrete changes drastically and the strength gain is reduced considerably. The rate of hydration reduces as the temperature decreases and the strength gain slows down. At a certain temperature the hydration process completely stops which results in partial strength gain of concrete. Also expansion of the water content in concrete at such low temperatures can cause damage to the concrete and consequently result in reduction of its strength. Ultimate strength reduction of about 50% can be observed in concrete if it freezes within the first 24 hours of casting or before reaching strength of 500 psi. There is a need to address the early strength gain problem of concrete at temperatures lower than freezing point of water [6].

***2.1.2.2. Workability*:**

Due to the reduction in the ambient temperature, the water in the mixture starts to freeze. The workability of concrete reduces in turn as the temperature reduces. There is an approximately 0.8-inch decrease in slump for every 20^{o}F decrease in concrete temperature [7]. Due to the reduction in the workability of concrete it becomes increasingly difficult to place concrete uniformly. It might also cause transport problems as well as get hardened inside the concrete mixer due to setting or freezing. Thus the concrete needs to be kept at a workable temperature externally or internally so that it can be placed with satisfactory results.

2.1.2.3. Setting time:

As the ambient temperature reduces the rate of hydration of concrete decreases, which in turn causes the time required for the complete hydration to increase as well. Thus the setting time goes on increasing with a decrease in temperature. In cold weather, an approximate 30% to 35% increase in set time can be expected for each 10^{o}F drop in ambient temperature [7]. As the setting time increases, the strength gain gets delayed. This in turn causes a delay in the finishing of concrete, the deforming of the molds. The schedule of construction is disrupted due to these problems and can translate into increased cost, labor and time. There is a need to address the problem of delay in the setting of concrete.

2.2.1.4. Freezing Point of Concrete

CRREL further performed a study of the effect of various chemical admixtures on the freezing point of the concrete mix. In this study, it was demonstrated that the freezing points of the concrete mixes can be depressed significantly by addition of simple chemical admixtures such as calcium chloride, calcium nitrite in various forms. The study showed that the freezing points of some of the mixes were depressed to about - 6^{o}C as shown in Table 2. The study however concluded that more work needed to be done to determine the right dosage of the various admixtures. Also there was a need to determine the effect of the chemical admixtures on the physical properties of concrete [6]. The study conducted in the previous phase of this

Table 2 Comparison of the Freezing Points of concrete with varying admixtures and their proportions [6]

Chemical	Percent by Weight of Cement	Freezing Point (^{o}C)
Calcium Chloride	1	-2.5
	2	-3.6
	3	-6.2
Calcium Bromide	4	-2.9
	6	-4
	8	na
Fertilizer (Calcium Nitrate)	3	-5.3
	6	-6.7
	9	-8.3
Calcium Nitrite	2	-3.5
	6	-5.7
	9	na
Calcium Chloride (deicer)	3	-1.5
	6	-2.25
	9	-4
Calcium acetate	2	-3
	4	-4.7
	6	-6.5
Calcium Formate	2	-3.3
	4	-4.5
	6	-5.5

2.1.3. Problems of concreting at low temperatures

At temperatures close to or below freezing point of water the concrete sets slowly and gains strength slowly. This can affect the construction schedule adversely and prove costly in terms of the manpower and time required.

The slow hydration results in delays in finishing of concrete. The season for concreting may be reduced or lost completely during cold weather due to these reasons. The labor, energy and time result in increased costs of construction.

2.2 Phase 1 of the SBIR study

2.2.1. Mortar Testing

The principal objective of the Phase I SBIR study was to develop an anti-freeze admixture formulation that would exhibit enhanced durability in the set concrete. 18 mix combinations were tested for the setting time and the compressive strength at 1 day, 3 day and 4 day for mortars. The combinations of admixtures and dosages are as given in the Table 3 and 4 were tested in Phase 1. As mentioned in Chapter 1 the admixture proportions to be used in concrete have limiting values for their individual use, however no limiting values have been specified when they are used in combinations. Thus the proportions were decided based on the specifications for the individual use of the admixtures.

Table 3: Admixture combinations tested in Phase 1 [2]

Admixture Combinations						
	Na-Si	Pozzotech 20+	K_2CO_3	Ca Formate	Ethylene Glycol	Pozzolith 122 HE
Naphthalene Sulfuric Acid Condensate	1	2	3	4	5	6
Polycarboxylic Acid	7	8	9	10	11	12
Polysaccharide	13	14	15	16	17	18

Table 4 Admixture Dosages [2]

High Range Water Reducer	Dosage
Rheobuild 1000 (Naphthalene Sulfuric Acid Condensate)	1125ml/100kg cement
PS 1466 (Polycarboxylic acid)	250ml/100kg cement
Rheomac VMA 450 (Polysaccharide)	146ml/100kg cement
Accelerator	Dosage (by weight of cement)
Pozzutec 20+ (Calcium Nitrite)	4%
Pozzolith 122 HE (Calcium Chloride)	4%
Sodium Silicate	1%
Potassium Carbonate	3%
Calcium Formate	4%
Ethylene Glycol	4%

All the 18 formulations were tested for setting times and compressive strengths. Out of the above admixture combinations the formulations 1, 8, 12 were down selected for further study and optimization in phase 2 of the project. The down selection was based on the performance of strength development of concrete. The formulations are as follows:

Mix 1

Cement: Sand = 1: 2.75

IPANEX = 0.008811mL/gr cement

W/C = 0.37

Na- Si = 1% by weight of cement

Rheobuild 1000 = 1125mL/100kg cement

Mix 8

Cement: Sand = 1: 2.75

IPANEX = 0.008811mL/gr cement

W/C = 0.42

Pozzutec 20+ = 4%

PS 1466 = 250 mL/ 100 kg cement

Mix 12

Cement: Sand = 1: 2.75

IPANEX = 0.008811mL/gr cement

W/C = 0.40

Pozzolith 122HE = 4%

PS 1466 = 250 mL/ 100 kg cement

2.2.2. Concrete Testing

The down selected combinations were also subjected to durability testing that included resistivity, surface air flow and half cell potential studies conducted on concrete. The concrete mix proportions were developed by Centre Concrete from the existing mortar formulations. The concrete mix proportions used for the durability studies were as provided in Table 5. The performance of the concrete which used IPANEX with the other admixtures was significantly better than the control concrete.

Table 5: Concrete formulations for durability testing [2]

Component	Control	Mix #1	Mix #8	Mix #12
Essroc Type I	7.08	7.08	7.08	7.08
Grancem 120 slag	3.81	3.81	3.81	3.81
Water	4.16	4.16	4.16	4.16
#57 limestone	34.48	34.48	34.48	34.48
sand	21.41	21.41	21.41	21.41
MBVR	5.71gr	5.71gr	5.71gr	5.71gr
Glenium 3030	9mL	9mL	9mL	9mL
IPANEX	---	43.55mL	43.55mL	43.55mL
Na-silicate	---	49.44grs	---	---
Rheobuild 1000	---	55.69mL	---	---
Pozzutec 20+	---	---	197.8grs	---
PS1466	---	---	12.38mL	12.38mL
Pozzolith 122HE	---	---	---	197.8grs

The admixture combinations 1, 8, 12 were supposed to be carried forward into the Phase 2 of the project for optimization of the admixture proportions.

2.3. Various Approaches of Cold Weather Concreting

For a long time it has been a goal of the construction industry to be able to place concrete in cold weather. There are two distinct approaches to tackle this problem. The first method used, to place concrete at low temperatures, was to provide heat externally to maintain the temperature of the concrete at or above the standard recommended values till the desired strengths were reached. However this method has certain disadvantages highlighted in section 2.3.1.3 which given way to another approach which consists of creating or providing heat to the concrete internally to sustain the rate of hydration of concrete even at low ambient temperatures. This was done by using various chemical admixtures to increase the rate of the hydration reaction and thus maintain the temperature of concrete.

2.3.1. Existing Methodology and its disadvantages

2.3.1.1. Existing Code Requirements

- **1905.11.1 IBC Regular (type I-II concrete) [8]:**

  Concrete (other than high early strength concrete) shall be maintained above 50^{o}F and in a moist condition for at least the first 7 days after placement except when using approved accelerated curing in accordance with ACI 318 section 5.11.3

- **1905.11.2 IBC High early strength (type III) [8]:**

  High early strength concrete shall be maintained above 50^{o}F and in a moist condition for at least the first 3 days except when using approved accelerated curing methods in accordance with ACI 318, section 5.11.3.

- **1905.12 IBC [8]**

Cold weather requirements: Concrete that is to be placed during freezing or near freezing weather shall comply with the following:

1. Adequate equipment shall be provided for heating concrete materials and protecting concrete during freezing or near freezing weather

2. Concrete materials and reinforcement, forms, fillers, and ground with which concrete is to come in contact shall be free from frost

3. Frozen materials or materials containing ice shall not be used.

The 2006 International Building Code requires concrete to be kept above 50^{o}F. The methods of required protection are no longer outlined. It is the contractor's responsibility to ensure the concrete is maintained at the proper temperatures.

- **ACI 306 (2002 Reaffirmed) [3]**

Concrete must be protected from freezing until it has reached a minimum strength of 3.5 MPa (500 psi), which typically happens within the first 24 hours. The current industry guidelines allow placing of concrete in cold weather only if the internal temperature can be maintained at 40^{o}C.

2.3.1.2. Current Industry Practices

The above requirements do not specify the method of achieving the required temperatures. The current practices for the cold weather concreting have largely remained unchanged since the 1930s. The temperatures are usually maintained by using tenting, heating enclosures and insulation. The raw materials used for the preparation of concrete need to be heated to account for the presence of ice and have to be maintained well above freezing temperatures. The substrate over which the concrete is to be placed also needs to be heated to above freezing temperatures [1,9].

2.3.1.3. Disadvantages of the current practices

The existing practices are very costly in terms of labor, material, power, fuel and budget. Also with the use of heating enclosures there is a significant risk of fire hazards. The construction industry in United States spends almost $800 million every year to tackle the problem of protection of concrete in freezing temperatures [1]. There is a need to eliminate such costly means of thermal insulation and substitute it with a method which allows the placement of concrete at an internal temperature below 40^{o}C.

2.3.2. Chemical Admixtures for Cold Weather Concreting

2.3.2.1. Types of Admixtures and Capabilities as Antifreeze Admixtures

2.3.2.1.1. Superplasticizers

Superplasticizers are used in cold weather concretes in order to increase the workability of the concrete. The water to cement ratio for cold weather concrete is reduced in order to limit the amount water available for freezing. To counter the loss of workability caused by reduction in the water content, superplasticizers are used [10].

2.3.2.1.2. Set Accelerator

The water contained in the concrete when exposed to low ambient temperatures freezes. It is necessary for the concrete to develop enough strength as required by the design before the water reaches temperatures where the hydration reaction ceases. Additionally the concrete needs to reach certain strength to resist the expansion of the freezing water which might lead to cracking and thus a loss of strength.

Antifreeze condition can also be obtained in the concrete by depressing the freezing point of water. This can be done by increasing the ions in the water. The set accelerator also helps in increasing the amount of ions present in water [10].

2.3.2.1.3. Water Tightening Agent

A water tightening agent is used to reduce the porosity of concrete enabling it to resist seepage of water through its microstructure. Water tightening agents such as IPANEX help in reducing the interlinking of the porous structure within the concrete by addition of CSH particles. This results in a more homogenous microstructure of concrete. A water tightening agent helps in increasing the durability of the concrete [10].

2.3.2.2. Early use of Chemical Admixtures

Since the early 1970s research has been performed to develop an admixture which would be capable of protecting concrete during placement without showing any undesirable effects to the final product. Experience in Europe mainly in the Soviet Union and Scandinavia [11] has proven that chemical admixtures can be used to depress the freezing point and allow hydration to take place below freezing point.

Soviet Union, Scandinavia, Canada, United States and Alaska usually experience a construction season of 8 to 9 months due to severe winter conditions in these countries. The use of chemical admixtures as a method to place concrete at low temperatures, although comparatively new to United States, has been a major source of extending the construction season in other regions around the world.

2.3.2.2.1. Calcium Chloride

Since early 1930s the use of calcium chloride has been a popular source for the placement of concrete at low temperatures. As early as 1951 it was identified that an addition of 1% of calcium chloride to the concrete mix can enhance its performance in cold weather [12]. However this could only be achieved if the concrete was kept at above 50^{o}C for the first three days. Although no special enclosures were required for this, the temperatures could only be maintained through insulation techniques. This still was a costly procedure.

Calcium chloride and sodium chloride were further tested over the years individually and in combination with each other as well as other compounds. A project involving the Army Foreign Science and Technology Centre in Charlottesville, VA investigated the addition of calcium chloride, sodium chloride and potassium carbonate to concretes and mortars for bridge construction. The strengths as a function of water cement ratio were presented [13].

Another study conducted in Vicksburg, MS also tested calcium chloride as an additive to concrete for antifreeze purposes. The study concludes that the amount of additive needed to create a mix that will perform satisfactorily increases with the decrease in the temperature [14].

2.3.2.1.2. Disadvantages

There were concerns however with the use of calcium chloride or other chloride based admixtures. Problems such as enhanced corrosion in the rebars or undesirable chemical reactions with the aggregate were cited as reasons to discourage the use of such admixtures. Due to these observations there developed a need to find a chloride free admixture.

2.3.2.3. Chloride Free Admixtures

Due to the disadvantages of the chloride based admixtures, mainly its corrosive nature research in development of new chloride free admixtures began. The research in antifreeze admixture development was revived in the USA in the late 1980s when a non-chloride based low temperature admixture was introduced in the market by Master Builders called Pozzutec 20 [9]. Since then CRREL has been performing research in cold weather concreting admixtures. Grace Construction Products also started investigation for antifreeze admixtures [9].

Various admixtures began to be used for this purpose. Accelerating agents, superplasticizers, air entraining agents etc. were tested for their effects on the mechanical properties of concrete at low temperatures. The specifications for the use of concrete admixtures provided basic guidelines to their usage regarding various variables such as the proportions of individual admixtures. Various combinations of admixtures were also tested.

2.3.2.3.1. Nitrate based Admixtures

The major chemical admixtures investigated which were chloride free were calcium nitrate and sodium nitrate. The results of these investigation indicated that the concretes using these additives showed strengths at below freezing temperatures which were comparable to concretes without the additives at much higher temperatures [15].

Research was conducted in Norway to develop calcium nitrate as a multifunctional admixture. It was tested as a set accelerator, long term strength enhancer and a corrosion inhibitor. The secondary result of this investigation was the successful application of calcium nitrate as an antifreeze agent [16].

Concurrently research was also conducted in Finland to test concrete under arctic conditions wherein the changes or variability of concrete strengths was tested at arctic temperature going up to -65^{o}C to -70^{o}C [17]. Also various researches were conducted in places like China, Malaysia and Singapore to develop a antifreeze admixture. Several types of admixtures were experimented with such as set accelerators superplasticizers and so on.

Similarly wide scale research has been undertaken at the CRREL since 1990 by Charles Korhonen, Peter Semen, Sherri Orchino [1, 6, 9, 10, 18, 19, 20, 21, 22, 23 and 24]. The crux of this research has been the development of an antifreeze admixture which will help in reducing the cost of construction in cold weather, extending the season of concrete construction and repair. To a lesser extent, the research also deals with defense against freeze thaw cycles and salt scaling on the concrete.

2.3.2.4. CRREL Research

Charles Korhonen, since 1990 has done considerable amount of work in the field of developing an antifreeze admixture. There has been a continuous and progressive study by CRREL in the development of a commercially viable antifreeze admixture. In this section the various steps in the previously mentioned study have been described. The studies conducted by CRREL were considered to have considerable relevance to the current research.

2.3.2.4.1. Pozzutec 20

CRREL carried out a comprehensive study of Pozzutec 20 as a cold weather admixture. This study consisted of three distinct phases, the first dealing with the evaluation of Pozzutec 20, the second with the development of another admixture which performed better than Pozzutec 20 and the third was evaluation of the admixture developed in phase 2. Korhonen concluded that Pozzutec 20 provided distinct advantage in terms of accelerating the hydration process and the generation of heat evident from the temperature rise in the concrete mix [9]. The significant part of these experiments was that the materials were mixed and cast at room temperatures. Also the new prototype admixture EY-11 tested in phase 2 of the research did not perform as well as Pozzutec 20+ in terms of reducing set times [9]. Korhonen recommended that the prototype admixture would require more development in order to fit in the existing ASTM guidelines.

2.3.2.4.2. Field evaluation and comparison of EY 11 (L and H) and DP

In 1997 the above prototype admixture EY-11 (L and H) and another similar admixture named DP both produced by different manufacturers, Master Builders and Grace and Co. respectively were tested in the field [1]. The field evaluation was done at Soo Locks in Michigan. The concrete in this case was mixed using heated water, unheated aggregates and at ambient temperatures. The field evaluation concluded that the new prototype admixtures perform satisfactorily in the field. The performance was evaluated on the basis of the data obtained for the compressive strengths as compared against a control mix that did not contain any admixtures. This is shown in Table 6.

Table 6 Comparison of EY11 and DP against Control mix [1]

Mix	Compressive Strength (Mpa)
Control	46.7
DP	46.0
EY11L	50.6
EY11H	55.5

2.3.2.4.4. Development of Antifreeze Admixture Formulation

In 2001 CRREL carried out an initial laboratory investigation on a set of admixtures whose identity was not revealed. They were instead identified by their function viz. water reducing agent, accelerator and so on. The important part of this study was that it was carried out with mortars instead of concrete. The reasons cited were that the only difference between mortar and concrete is the presence of coarse aggregate and the mortar acts exactly like concrete with respect to the admixtures. This was because the admixtures have the same amount of cement and water to react with, similar voids to fill in and similar aggregate to paste transition zones to deal with. Also, mortar allows for simpler mixing process, lesser material handling and smaller testing equipment. The study concluded that it was possible to develop freezing points as low as 15.4^{o}F by a combination of admixtures. Also, the study suggested that future work should be done to explore various combinations of the commercially available admixtures and also include cement types other than Portland cement [20].

The effect of freeze thaw cycles on the durability of concrete had also been of concern for a long time. In 2002, CRREL studied the causes of the damage caused by the freeze thaw cycles on the durability of concrete. The study identified the various causes of frost damage to concrete such as the porous nature of concrete which led to the development of a capillary structure and the mount of freezable water in concrete. The study concluded that it was necessary to find a solution to the problem of frost damage and that it could be done in two ways, by reducing the water content of concrete or to depress its freezing point. The code specifications, advice against the use of high volumes of admixtures to prevent damage to concrete. However, this study cites the use of high volumes of admixtures in Soviet Union as a reference point [21].

CRREL further continued their research to develop an antifreeze admixture formulation. Phase 1 of this research dealt with the identification of various combinations of admixtures and evaluation of their low temperature performances. Various admixtures such as water reducers, accelerators, air entrainers were considered. A detailed study was conducted to evaluate the effect of each of the admixtures on the physical nature of concrete. Based on the results obtained from these experiments in terms of properties such as workability, entrained air, initial freezing points, various suites of combinations were selected. The admixtures used were commercially available products from W R Grace and Master Builders. These down selected combinations were then tested for low temperature performance with respect to the compressive strengths, setting times and freeze thaw durability [10].

Phase 2 of the CRREL research reported in February 2004 furthered the process of the freeze thaw testing in the Phase 1. The results in Phase 1 suggested that some of the admixtures improved the freeze thaw resistance of the concrete. The study however concluded that more work needs to be done in the area of freeze thaw testing in terms of extending the freeze thaw testing to a 1000 cycles.

2.4. IPANEX

IPANEX is a water tightening agent produced by IPA systems. The primary function of IPANEX is to increase the durability of the concrete. IPANEX is a state of the art admixture that is commercially sold as a water tightening and corrosion inhibitor agent. IPANEX is composed of nanometer sized seeds of C-S-H (calcium silicate hydrate) ions which template the growth of microstructure.

Tricalcium-silicate and dicalcium-silicate are two main constituents of cement. These constituents react with the water and form C-S-H compound structure. This structure serves as the glue in the concrete. As the C-S-H pores increase during hydration, they form a structure which allows easy passage of water and other fluids through it [2].

IPANEX is a state of the art admixture that is commercially sold as a water tightening and corrosion inhibitor agent. The result of the microstructure control is more homogenous growth of C-S-H and a uniformly distributed distribution of CH thus minimizing stress concentrators and distributing the otherwise deleterious effects of highly soluble CH. The principal function of the seeding is to shift the pore sized distribution to smaller sizes thus minimizing that portion of the distribution that can transmit fluid [2].

Below shown in Figure 1 is the comparison between a concrete using IPANEX and control concrete, using a Scanning Electron Microscope. As shown the concrete microstructure when IPANEX is used is significantly denser with lesser amount of voids and interconnecting pores than in control concrete. This helps in reducing, the amount of water trapped in the pores of concrete which causes freezing of concrete before setting, cracking of concrete and retardation of strength gain.

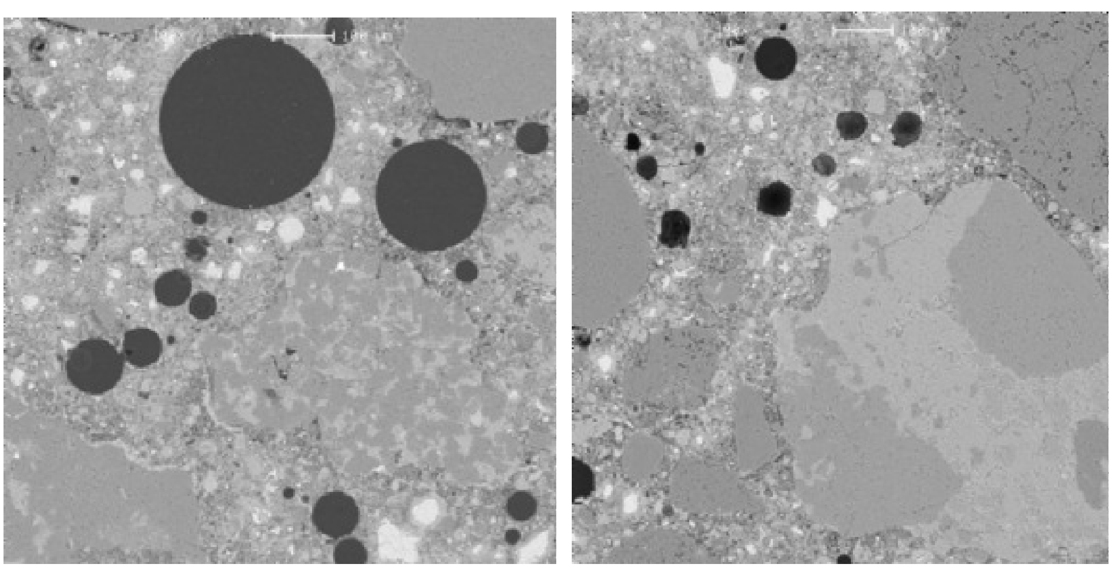

Figure 1 Comparison of SEM images of concrete microstructure with and without IPANEX [25]

2.5 Design of Experiments

Design of Experiments is a statistical method used to setup a matrix of experimental runs. The method is based on important characteristics such as comparison, randomization, replication, blocking, orthogonality and factorial experimentation.

Validity of an experiment is compromised if it is approached randomly. Numerous factors affect the behavior of the yield during an experiment. It is necessary to design the experiment in order to account for the variances involved. Design of Experiments as a method helps us determine the number of replicates needed for each run, how many blocks are needed in order to account for the batches of runs performed at the same time and so on.

The method establishes an interaction between the factors and the yield of a process. Any experiment has a number of variables involved and these variables need to be accounted for in the results. There are different kinds of variables such as controllable and nuisance variables. The method helps in determining the effect of controllable variables on the yield and also reduces the errors in the behavior patterns due to nuisance or uncontrollable variables.

The results of the analysis can be obtained in terms of plots of the various factors against the yield. Also the interaction between the individual factors can be determined b this method. The method is based on fundamental statistical concepts such as analysis of variance, p-value determination, and regression analysis.

The biggest advantage of designing an experiment is that a number of factors can be considered at the same time instead of one factor at a time.

2.5.1 Types of designs

There are various types of designs that can be used to setup experimental runs. These are the Factorial Design, the Response Surface Design, the Taguchi Design and the Mixture Design [26].

The Factorial Design being a very robust design technique can be applied to almost any kind of experimental setup. However it has several drawbacks. It provides a large number of experimental runs when the factors involved in the experiments are numerous or the levels of these factors being considered are greater than 3. This can prove to be time consuming and costly in most of the cases. Also it does not provide us with other advantageous analysis outputs that are required for the optimization problems such as the optimization graphs. Most of the times it is not necessary to perform all the runs as required by the full factorial design and a partial factorial design consisting of either half, a fourth or similar denominations of a full factorial design does suffice. Thus, when the experiment consists of several factors it proves beneficial not to opt for a factorial design.

In case of the Taguchi Design the higher order interactions are confounded and difficult to resolve. With the Mixture Design, various constraints on the number of factors that can be used and their levels make them a less beneficial design option for the optimization problem as compared to the Response Surface Design. Thus the only viable option that is available for the optimization problem with several factors is the Response Surface Design. Response Surface Design is explained in detail in section 2.5.1.1.

2.5.1.1. Response Surface Design

Response surface methodology or RSM is a collection of mathematical and statistical techniques that are useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response [26].

The eventual objective of the RSM is to determine the optimum operating conditions for the system or to determine a region of the factor space in which operating requirements are satisfied [26]. In the current study, the purpose was to consider the six admixtures of the antifreeze formulations, and temperature and establish the optimum proportions of the components that would meet or exceed the performance criteria established in Phase I as mentioned later in Section 4.4. Temperature is considered as a factor as the variation of the response with temperatures is required to apply the performance criteria to the model. MINITAB [27] was used as the software to create and analyze the design.

2.6 Summary

The literature review gives a background of the research conducted in the area of antifreeze admixtures for placing concretes at low temperatures. The behavior of concrete at low temperatures has been explained in this section along with the effect of low temperatures on the various properties of concrete. A need to address the effect of low temperatures on the properties of concrete has been established in the section 2.1.2.

Chapter 2 primarily attempts to provide a basis for the necessity of using antifreeze admixtures as a solution for the problem of cold weather concreting. For this purpose it presents the various techniques that can be used to place concrete at low temperature in accordance with the ACI and IBC specifications, and their comparison. It highlights the problems faced by the current methods of external heating, for the problem of concreting at low temperatures.

Most importantly Chapter 2 provides the background of the research performed with admixture formulations at low temperatures. Various conclusions relevant to the current study have been provided in each section of this chapter.

A brief description of the procedure and results of Phase 1 of the research has been provided in this chapter. It also mentions the reasoning behind the selection of the various admixtures including IPANEX and their proportions.

The process of design of experiments has been explained in detail in section 2.5.

Chapter 3
Experimental Plan

3.1 Design Aspects

Response surface design has been explained in detail in the section 2.5. The various design aspects mentioned in the section 2.5 are discussed in detail here. The concentrations of IPANEX, Rheobuild 1000, Na Silicate, Pozzutec 20+, Pozzolith 122 HE, PS 1466 (it can be seen from section 2.2 that these are the admixtures whose proportions are to be optimized) and temperature were considered as factors in the design process. These factors were all applied at five different levels, except for temperature which was modified within the program to represent only three levels. This had to be done as the temperature could be controlled only at three levels due to the constraints on the laboratory equipment.

For the design process two levels of each factor have to be input. The design then created a centre level and two extreme levels beyond each value provided at the beginning. Blocks can be specified if the experiments are not going to be conducted at one time.

In the data table for mix 1 [Table 7] the factor IPANEX has levels 6.2, 6.4, 6.6, 6.8, 7. Now according to the above procedure the MINITAB requires an input of only the cuboidal (i.e. 2^{nd} and 4^{th} level) levels. Thus, in this example we input only the factor levels 6.4 and 6.8. The other three levels are generated by the program itself. These levels are the levels of the proportions of the factors which are used in the experiment.

Table 7 : Run combinations for the Response Surface Design

Blocks	IPANEX	Na Si	Rheobuild 1000	Temperature
1	6.8	7.5	8.25	20
1	6.8	7.5	8.75	73
1	6.6	8	8.5	40
1	6.8	8.5	8.75	20
1	6.6	8	8.5	40
1	6.4	7.5	8.25	73
1	6.8	8.5	8.25	73
1	6.4	8.5	8.75	73
1	6.4	8.5	8.25	20
1	6.4	7.5	8.75	20
3	6.6	8	8.5	40
3	6.2	8	8.5	40
3	6.6	7	8.5	40
3	6.6	8	8.5	73
3	6.6	8	8.5	20
3	6.6	9	8.5	40
3	6.6	8	9	40
3	6.6	8	8	40
3	7	8	8.5	40
3	6.6	8	8.5	40
2	6.8	7.5	8.75	20
2	6.8	8.5	8.25	20
2	6.6	8	8.5	40
2	6.4	8.5	8.25	73
2	6.6	8	8.5	40
2	6.4	7.5	8.75	73
2	6.4	8.5	8.75	20
2	6.4	7.5	8.25	20
2	6.8	7.5	8.25	73
2	6.8	8.5	8.75	73

The MINITAB program when provided with the factor levels and the blocks then creates a design with appropriate number of runs. The minimum number of runs that were available in a Response Surface Design for 4 factors at 3 levels was 30. Table 7 provides the run combinations for Mix 1. The steps of the design process have been presented in the Figure 2.

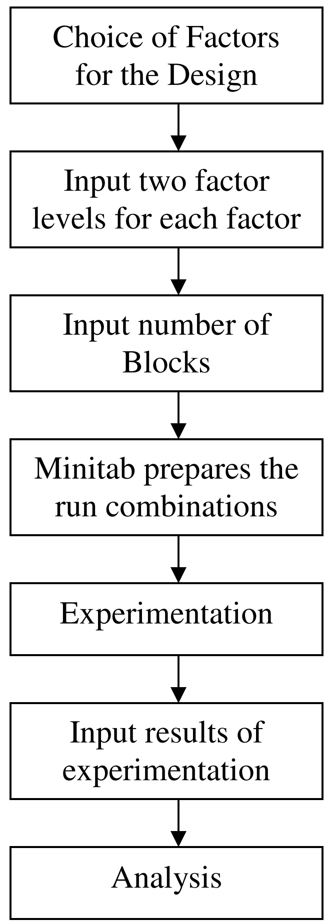

Figure 2: Flow Chart for procedure of the Design of Experiments

The run combinations obtained in Table 7 were the combinations of the proportions of the various admixtures used to prepare the mortar for testing. Similar combinations were obtained for the Mixes 8 and 12.

3.2 Experimental Plan

The run combinations obtained in the section 3.1 are then tested for both the setting time and the compressive strengths of the mortars. The Phase 1 study of the current research had conducted these tests on the mortar to down select the three admixture formulations. The Criteria of Optimization as explained in section 4.4 was based on these tests. Thus it was imperative to test the mortar for the setting time and compressive strengths at various temperatures.

3.2.1. Tests Conducted

3.2.1.1 Vicat Setting Time Test (ASTM C 191)

There are two stages of setting time, initial setting time and final setting time. The initial setting time is defined as the time elapsed between the molding of the specimen and the first penetration of the needle less than 25 mm. The final setting time is defined as the time elapsed between molding the specimen and the time when the needle does not penetrate through the specimen.

The setting time of mortar is determined by the Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle as described in ASTM C 191.

The standard specifications given in ASTM C 150, state a minimum initial set of 45 minutes and a maximum final set of 375 minutes.[28]

3.2.1.2. Compressive Strength Test, Mortar (ASTM C 109)

Compressive strength by definition is that value of uniaxial compressive stress reached at ultimate failure of the concrete. The compressive strength is determined by the Standard Test Method for Compressive Strength of Hydraulic Cement Concrete as described in ASTM C 109. The three day test should yield approximately 1740 psi of compressive strength [29].

3.2.1.3. Compressive Strength Test, Concrete

The Compressive Strength Test on concrete was carried out using 6” x 12” cylinders. The cylinders were tested under the compression testing machine. The pressure applied was between 36 psi/sec to 45 psi/sec. While placing the cylinders the testing surfaces were leveled using the saw dust so as to ensure equal distribution of the applied pressure

3.3. Experimentation

3.3.1. Raw Material

Mortar testing was the first step that was undertaken. Sand, Cement, and admixtures, Na Silicate, Rheobuild 1000, PS 1466, Pozzutec 20+ and Pozzolith 122HE [30] were the materials that were used for the experiments. The sand used was natural sand provided by Center Concrete. The cement used was the ESSROC Type 1 cement. Na Silicate was obtained from PQ Corporation. All the other admixtures were supplied by the Master Builders.

3.3.2. Storage

The sand to cement ratio and the water to cement ratio were both carried over from the Phase 1 of the project. The material was then batched for the tests and kept in respective temperatures for over 24 h.

The cement was batched and stored at the respective temperatures directly. The sand was first dried in an oven at 110 +- 5^0F for 24 h. It was then batched and placed at the respective temperatures in zip lock bags to provide an air tight atmosphere so as to protect against water absorption.

3.3.3. Curing of Concrete

Curing of concrete was done by moist curing at 73^oF in a room kept at 73^oF and 95% humidity. In the Cold Room (at 40^oF) and Cold Chamber (at 20^oF) the sealed curing was undertaken.

3.3.4. Determination of water absorption capacity

To compensate for the water absorption capacity of sand a correction had to be applied to the amount of water that was being used. This had to be done as dry sand was being used for all the mixes and it would mean that the sand would first absorb the moisture till it is saturated and then leave the rest for hydration. The water absorption capacity provided by the supplier was used in the experiments. The water absorption capacity for the sand used in this particular experimentation had a capacity of 1.17% by weight of sand. This meant that water equal to 1.17% of sand by weight was needed to be increased in the amount obtained by the water cement ratio as dry sand was being used.

3.4. Conversion of Mortar to Concrete

For the second part of the experimentation which involved testing of concrete the mortar mixes down selected from the optimization process needed to be converted to concrete mixes. The conversion from mortar mix to concrete mix was done based on the values provided by Centre Concrete. The concrete mix proportions were designed for 1 cubic yard of concrete for 6% of air. The values for Glenium 3030 and MB-VR were recommended values provided by the manufacturer. The sand to cement ratio and the water cement ratio for the mixes were slightly adjusted in the concrete mixes. This was done to conform to the PennDOT mix proportions of the AA concrete. The values obtained in this manner are provided in Chapter 4.

Chapter 4
Results and Analysis

This chapter provides a detailed explanation of the analysis process and the discussion of the results obtained. The experimentation was performed on all run combinations for the three admixture formulations and the results, thus obtained, were entered into MINITAB in a tabulated manner for analysis. The Table 8 presents the tabulated data for Mix 1 with the results in terms of the setting times in minutes, and compressive strengths at day 1, 3 and 4 in psi. Each test consisted of two specimens for each data value. An average of the test values for the two specimens was considered to be the final value. The Phase 1 of the SBIR study had used this method for the testing. The same method was adopted in the current research to maintain identical procedures for comparison of the results. Each part of Table 8, a, b and c represents the three different blocks of experiments conducted at different times. The tables for mix 8 and mix 12 have been provided in the Appendix A.

Table 8: The experimental data obtained for tests conducted on mortar for Mix 1 in Subtask A

(a)

Blocks	IPANEX	Na Si	Rheobuild 1000	Temperature	Vicat Setting Time	CST DAY 1	CST DAY 3	CST DAY 4
1	6.8	7.5	8.25	20	30	35.5	116.5	120.5
1	6.8	7.5	8.75	73	150	4419	5230	5207.5
1	6.6	8	8.5	40	285	940	1979	2611
1	6.8	8.5	8.75	20	60	26.5	158	112
1	6.6	8	8.5	40	300	995.5	1915	2687
1	6.4	7.5	8.25	73	210	3704.5	3848.5	4078
1	6.8	8.5	8.25	73	60	3771.5	3701.5	4050
1	6.4	8.5	8.75	73	120	4316	5270.5	5302.5
1	6.4	8.5	8.25	20	120	53	152.5	209
1	6.4	7.5	8.75	20	90	138	169	265

(b)

Blocks	IPANEX	Na Si	Rheobuild 1000	Temperature	Vicat Setting Time	CST DAY 1	CST DAY 3	CST DAY 4
2	6.8	7.5	8.75	20	120	23	100	358
2	6.8	8.5	8.25	20	90	39	126.5	420.5
2	6.6	8	8.5	40	255	714.5	1870	2550
2	6.4	8.5	8.25	73	120	3634.5	5278	4823
2	6.6	8	8.5	40	255	707.5	1992	2441.5
2	6.4	7.5	8.75	73	135	3952	4771	5290
2	6.4	8.5	8.75	20	150	92.5	117.5	279
2	6.4	7.5	8.25	20	150	44	86.5	289
2	6.8	7.5	8.25	73	120	3712.5	4823.5	5176
2	6.8	8.5	8.75	73	165	4464	4599	5636.5

(c)

Blocks	IPANEX	Na Si	Rheobuild 1000	Temperature	Vicat Setting Time	CST DAY 1	CST DAY 3	CST DAY 4
3	6.6	8	8.5	40	345	970	1890	2507
3	6.2	8	8.5	40	300	1810	2207.5	2586
3	6.6	7	8.5	40	315	567	617.5	626
3	6.6	8	8.5	73	135	4536	5004	5243
3	6.6	8	8.5	20	135	28	122	471
3	6.6	9	8.5	40	285	473	1117.5	1557.5
3	6.6	8	9	40	195	450	1715.5	2151
3	6.6	8	8	40	195	550.5	600	1005
3	7	8	8.5	40	285	512	1731.5	2314
3	6.6	8	8.5	40	240	916.5	1954	2671.5

4.1. P-value

The P-value is the measure of the significance of the factors in affecting the response variables in the experiment. This experiment was based on a 95% confidence interval (which is the default value in MINITAB) i.e. for the factor to have a significant effect on the response variables it must have a ‘p’ value of less than 0.05. This is illustrated in the following example (Table 9). Here after the analysis the program provides us with the values of the coefficients of each of the terms. It also gives us the P value. Thus, in the example given below, the terms (highlighted in red) that have a significant effect on the Vicat Setting time based on the P values are Rheobuild 1000*Rheobuild 1000 (P = 0.008) and temperature* Temperature(P = 0.000). We can also consider IPANEX * Rheobuild (P = 0.062) as it has a P value very close to 0.05. Similar analysis for each mix combination and for each response variable has been provided in the Appendix B (Tables B1- B12).

Table 9: Response Surface Regression Analysis for Mix 1: Vicat Setting Time

Term	Coeff	P
Constant	5065.83	0
Block 1	-9.25	0.376
Block 2	4.25	0.68
IPANEX	-4282.7	0.102
Na Si	-891.849	0.284
Rheobuild 1000	2994.68	0.609
Temperature	17.4034	0.106
IPANEX*IPANEX	62.5	0.728
Na Si*Na Si	17.5	0.545
Rheobuild 1000*Rheobuild 1000	-350	0.008
Temperature*Temperature	-0.22338	0
IPANEX*Na Si	18.75	0.841
IPANEX*Rheobuild 1000	375	0.062
IPANEX*Temperature	1.16042	0.51
Na Si*Rheobuild 1000	60	0.428
Na Si*Temperature	-0.83236	0.246
Rheobuild 1000*Temperature	0.34551	0.805

4.2. Residual Plots

The purpose of the residual plots (Fig. 3, 4, 5, 6) is to check the adequacy of the model. The design of experiments undertaken in earlier sections is based on two primary assumptions of normality and randomization of the errors or residuals. All of the subsequent mathematical manipulations depend upon the data being normally distributed in order to be valid.

The normal probability plot is used to check this assumption. An example of a normal probability plot is shown in Figure 3 (which has been selected randomly for the sake of illustration). If this assumption is correct, then the plot should have all the points along the line shown on the graph which is an almost 45 degree line. Any point away from the line is an unusual observation normally called as an outlier which are either retested or if justified, discarded.

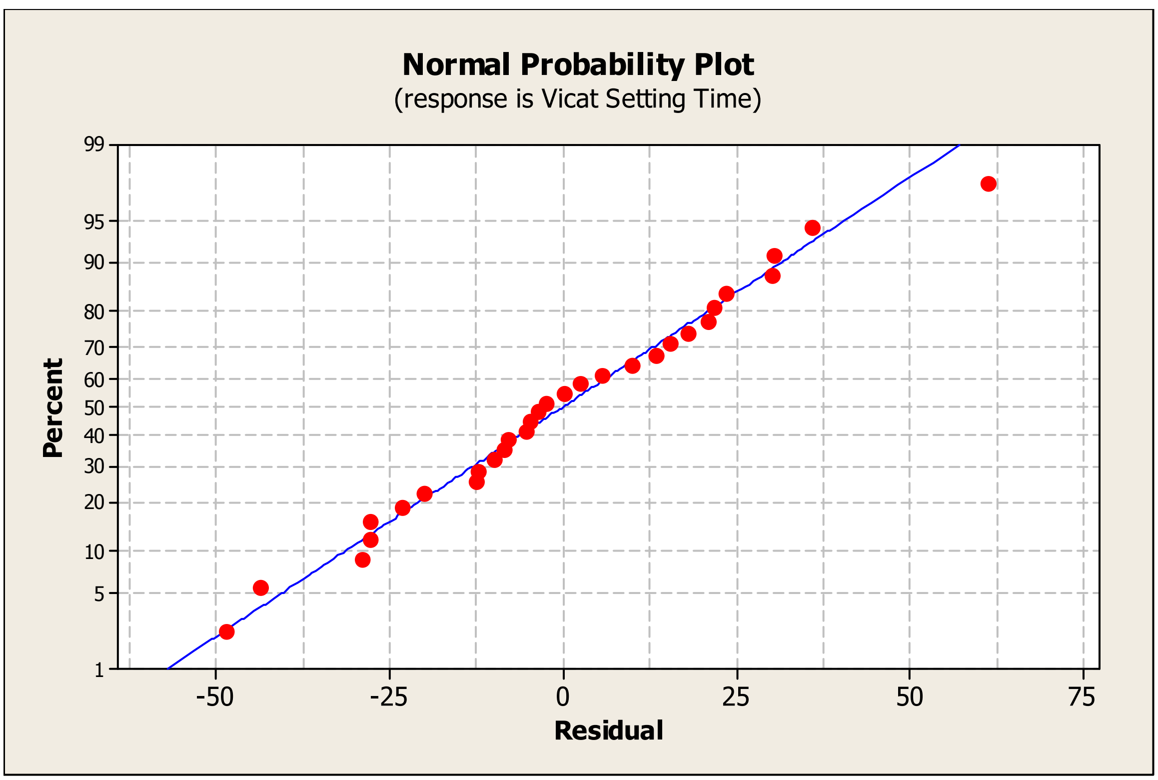

Figure 3: Example of a Normality Plot for the residuals (MINITAB).

The Histogram plot (Figure 4) shows the frequency distribution of the residuals which shows that the residuals are distributed normally. This means that maximum frequency of the residuals is close to 0 which shows that the error margin is less and the deviation of the residuals from mean is not large.

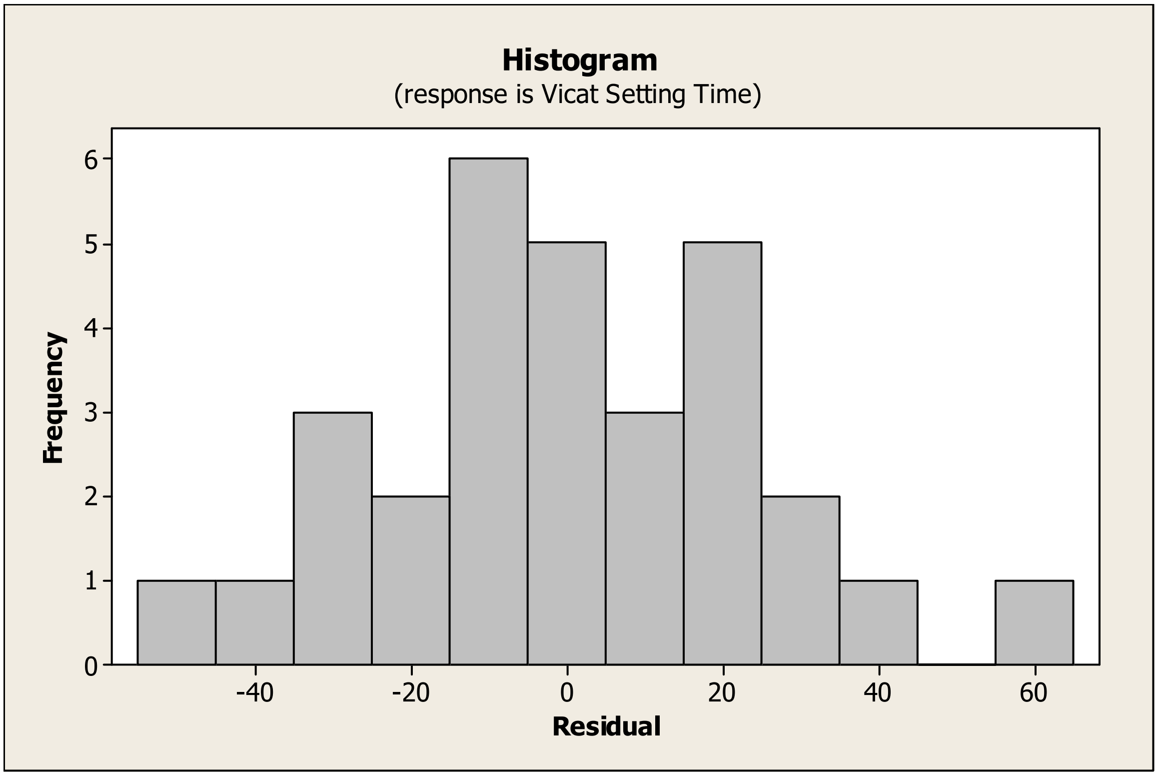

Figure 4: Example of Histogram (MINITAB)

The residuals versus fits plot (Figure 5) is the plot of the residuals against the fitted values. Now for the model to be correct and the assumption of randomization to be satisfied this graph must show no correlation between the residuals and the fitted values. In other words the graph should be without a pattern and the residuals should be unrelated to any other variable including the predicted response [26].

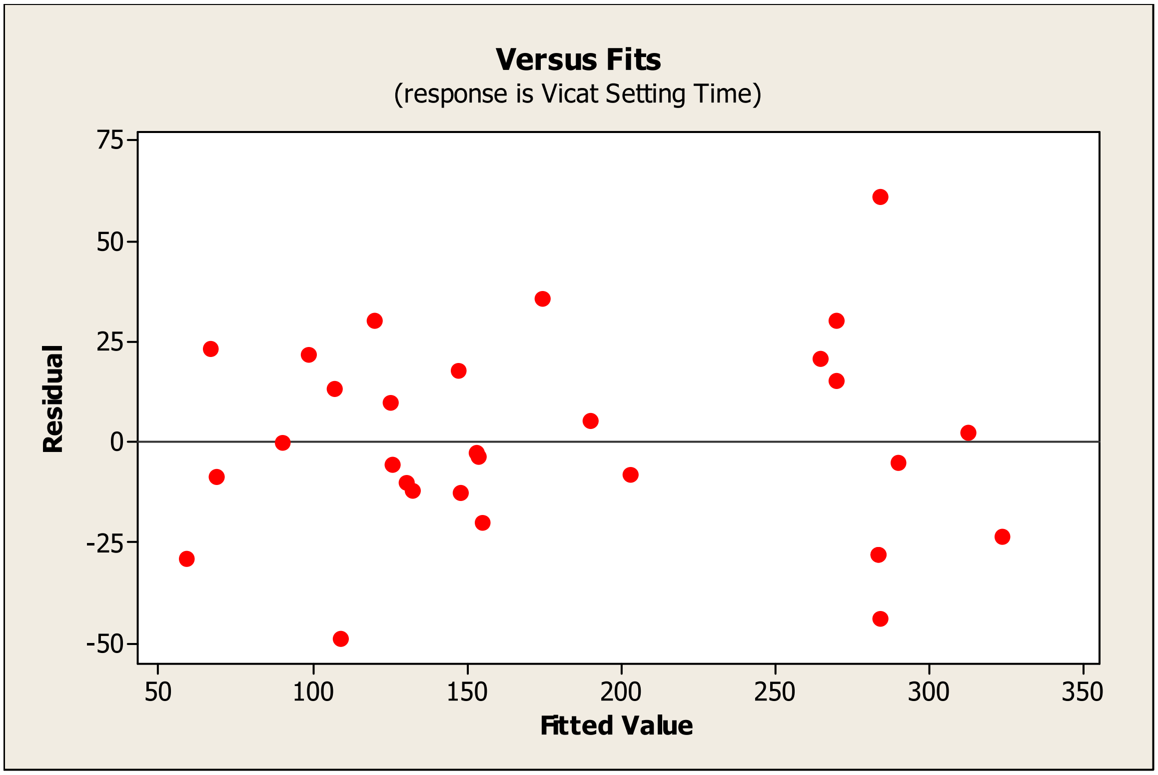

Figure 5: The Residuals vs. Fitted value plot (MINITAB)

The fourth graph (Figure 6) indicates the correlation between the residuals and their order. If the graph indicates a tendency to have consistent runs of positive or negative residuals then it would indicate positive correlation between the residuals which could prove to be a potential problem. Ideally this graph should show equal number of residuals on both the positive and negative side. The remaining residual plots have been provided in the Appendix B. Fig B1, B2, B3, B4, B8, B9, B10, B11, B15, B16, B17, B18.

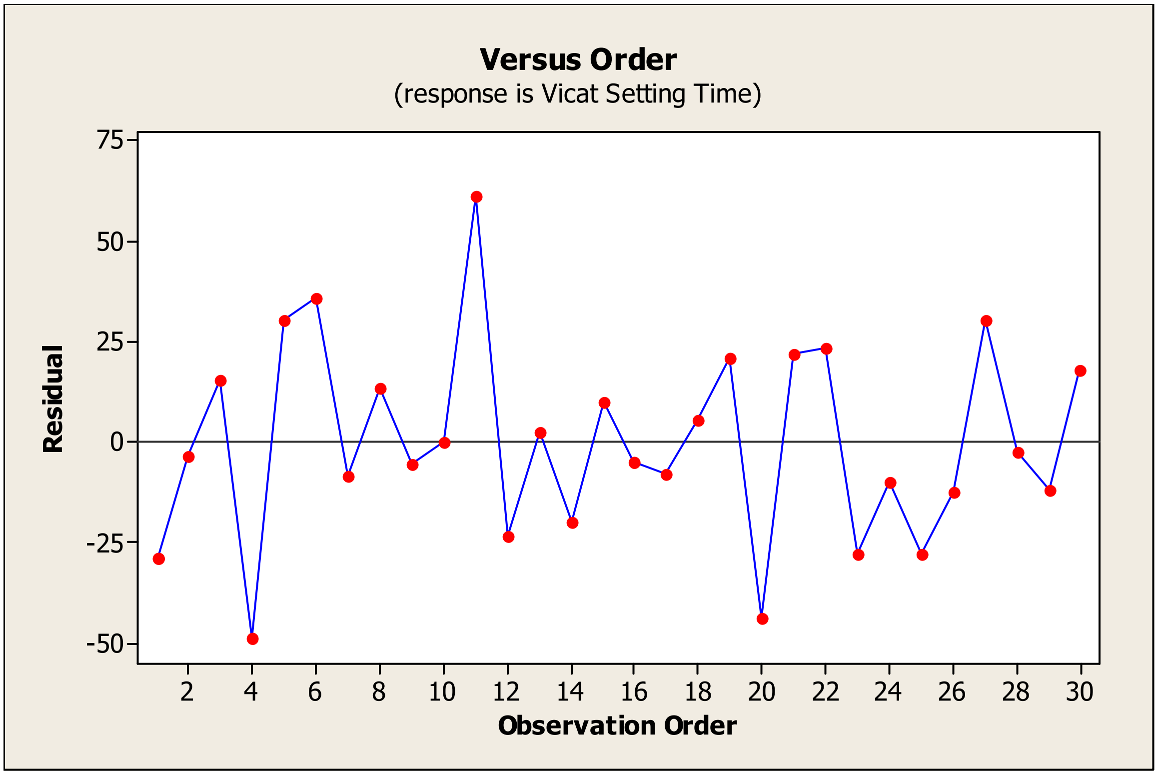

Figure 6: Plot of the Residuals vs. Observation Order (MINITAB)

4.3. Optimization Graphs

The optimization graphs [Fig. 7] is the unique feature of the response surface design. The optimization graph is obtained after the analysis where in the behaviors of all the various yields with respect to the factors are plotted on the same graph. It allows for manual adjusting of the values of the factors in one single graph to obtain the required values for all of the response variables (yields).

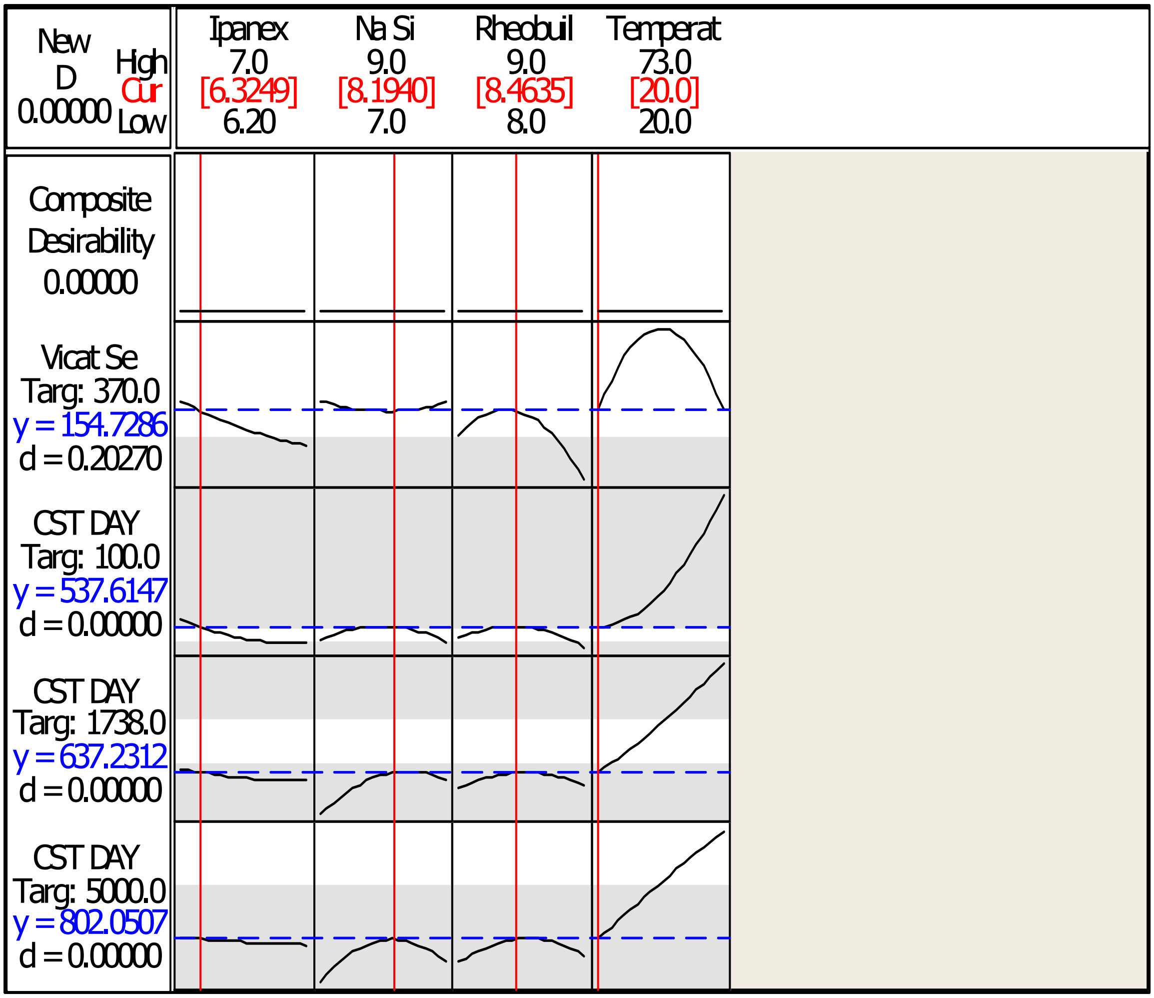

Figure 7: Example for the Optimization graph (MINITAB)

In Figure 7 the factors such as IPANEX, Rheobuild, NaSi and temperature are shown at the top. The yields such as the setting time and the compressive strengths are shown on the left. For the factors the value in the bracket represents the position of the red line. The values above and below this value represent the levels of the factors over which the optimization process was performed. For the yield the value of 'y' is the value corresponding to the factor levels. The red line can be moved between the levels of the factors to get the corresponding values of the yields.

Thus in this experiment, the amounts of admixtures could be adjusted to provide acceptable values (as mentioned in section 4.4) of the response variables viz. the setting time and the compressive strengths at day 1, day 3, day 4. Therefore the mixture formulation was easily optimized on one single graph. This is illustrated in Figure 7.

The remaining optimization graphs have been provided in Appendix B Fig. B5, B6, B7, B12, B13, B14, B19, B20, B21

4.4 Performance Criteria for Optimization

For the optimization of the proportions of the admixture formulations certain performance criteria were decided. These performance criteria were then utilized in the optimization graphs to adjust the values of the factors. The performance criteria were as follows:

1) The setting time was selected to be between 90 min and 150 min. This was done on the basis that the setting time of the mixture should be long enough to enable mixing of the concrete, transport and placement without the concrete getting set. However, it also needs to be short enough so that the concrete does not freeze up before being completely set.

2) The compressive strength for day 1 should be at the minimum greater than that obtained in the suite of each of the combination tested in the Phase 1 of the project. Also, the three day strength should be greater than 1740 psi as stated by ASTM 150 at room temperature. The day 4 strength was considered to be at least 4500 psi at room temperature.

4.5 Optimized Proportions of Admixture Formulations

On the basis of the performance criteria presented in Section 4.4 the proportions of the admixture formulations were optimized. The final proportions were as presented below:

Mix 1

Sand: cement =1: 2.75

W/c ratio = 0.37

IPANEX =0.009035 mL/ gr cement

Rheobuild = 1209.07 mL/ 100 kg cement

Na silicate = 1.17%

Mix 8

Sand: cement = 1:2.75

W/c ratio = 0.42

IPANEX = 0.009957 mL/gr cement

Pozzutec 20+ = 4.025% cement

PS1466 = 478.6 mL /100 kg cement

Mix 12

Sand: cement = 1:2.75

W/c ratio = 0.40

IPANEX = 0.0096401 mL/ gr cement

Pozzolith 122HE = 4.906% cement

PS1466 = 447.42 mL/ kg cement

A comparison between the starting values of the admixture formulations as concluded in the Phase 1 of the project and those arrived at, at the end of the optimization analysis in the Phase 2 is given below in Table 10.

Table.10 Comparison between the Original formulations from Phase I and the optimized values determined in this study

Component	Original Value	Final Value
Mix 1		
IPANEX	0.008811 mL/ gr cement	0.009035 mL/ gr cement
Rheobuild	1125mL / 100 kg cement	129.07mL/ 100 kg cement
Na Silicate	1% of cement	1.17% of cement
Mix 8		
IPANEX	0.008811mL/ gr cement	0.009957 mL/ gr cement
Pozzutec 20+	4% of cement	4.025% of cement
PS 1466	250 mL / 100 kg cement	478.6 mL/ 100 kg cement
Mix 12		
IPANEX	0.008811mL/gr cement	0.009640 mL/ gr cement
Pozzolith 122HE	4% of cement	4.906 % of cement
PS 1466	250 mL/ 100 kg cement	447.42 mL/ 100kg cement

All the admixtures except for Rheobuild showed a significant increase in the proportions with respect to the values prior to the optimization process. In mix 1, Rheobuild, showed a significant decrease in its proportion. This meant that the value of Rheobuild after optimization lay outside the range of values considered for the optimization process. This could have been due to various reasons such as inadequacy of the optimization model, bad results due to reaction between the admixtures in Mix 1. The Mix 8 and Mix 12 both showed increase in the admixture proportions which signified that the optimization model was accurate and that the optimized values lay within the range of values considered.

4.6. Down selecting two admixtures formulations

The objective of this subtask was to down select from the three admixture formulations in the previous section to two admixture formulations based on the evidence obtained during the analysis of the mortar testing.

4.6.1. Down Selection

All three mixes showed that they met the required results in terms of setting times and compressive strengths at 20^{o}F. However the mix 1 showed very poor consistency while mixing and placing. It also showed a tendency of freezing at 20^{o}F very rapidly. Na Silicate and Rheobuild 1000 reacted with each other when mixed in water which might prove to have an extreme effect on the strength of the mixture.

Based on this approach, the Mix 8 and Mix 12 were down selected to be tested further and Mix 1 was eliminated. These mix proportions were as follows:

Mix 8

Sand: cement = 1:2.75

W/c ratio = 0.42

IPANEX = 0.009957 mL/gr cement

Pozzutec 20+ = 4.025% cement

PS1466 = 478.6 mL /100 kg cement

Mix 12

Sand: cement = 1:2.75

W/c ratio = 0.40

IPANEX = 0.0096401 mL/gr cement

Pozzolith 122HE = 4.906% cement

PS1466 = 447.42 mL/ 100 kg cement

4.7. Verification of Analytical Results for Mix 8 and Mix 12

The optimized mix proportions for mix 8 and mix 12 were obtained from the analytical study. These mix proportions as mentioned in the section 4.3 had corresponding values for the setting time and compressive strengths. These values however were theoretical values and needed to be verified by actual experimentation. The optimized proportions were thus tested for setting times and compressive strengths and their results were compared to the analytically obtained values. In this case however the number of specimens per data value was 3. The comparison of these results is provided below in Table 11

Table 11 Comparison of Analytical and Experimental Data from Optimized Admixture Proportions

(a) Day 1

Temperatures (^oF)	Compressive Strength (psi)			
	Mix 8		Mix 12	
	Analytical	Experimental	Analytical	Experimental
20	251	288	180	225
73	2481	2420	4443	2883

(b) Day 3

Temperatures (^oF)	Compressive Strength (psi)			
	Mix 8		Mix 12	
	Analytical	Experimental	Analytical	Experimental
20	455	515	600	555
73	4678	3551	4704	4794

(c) Day 4

Temperatures (^oF)	Compressive Strength (psi)			
	Mix 8		Mix 12	
	Analytical	Experimental	Analytical	Experimental
20	694	671	848	756
73	4709	4305	5401	5496

(d) 20^oF

Vicat Setting Time (min)			
Mix 8		Mix 12	
Analytical	Experimental	Analytical	Experimental
105	105	134	120

4.8. Conversion of Mortar Mixes to Concrete Mixes

The mortar mixes as discussed above were converted to concrete mixes after the down selection to two admixture formulations. The conversion was done based on the values provided by Centre Concrete as per their standard AA PennDOT mixes. The values of the air entraining agent and the Glenium 3030 were decided as per values recommended by the manufacturer and industry standards. The converted mixes were as follows:

Mix 8

Sand/ Cement = 2.78

W/C ratio = 0.418

Table 12: Concrete Mix Proportions for PennDOT AA Mix for Mix 8

(a)

Material	Amount (ft^3)
Cement	3.154
Coarse Aggregate	11.322
Fine Aggregate	7.217
Water	3.688
Air	1.620

The admixture amounts extrapolated from the mortar values were as follows

(b)

Material	Amount (oz/cwt)
Glenium 3030	1.96
MB-VR	0.5
IPANEX	15.3
Pozzutec 20+	45.9
PS 1466	7.3

Mix 12

Sand/ Cement = 2.85

W/C ratio = 0.401

Table 13: Concrete Mix Proportions for PennDOT AA Mix for Mix 12

(a)

Material	Amount (ft^3)
Cement	3.154
Coarse Aggregate	11.322
Fine Aggregate	7.217
Water	3.688
Air	1.620

The admixture amounts extrapolated from the mortar values were as follows

(b)

Material	Amount (oz/cwt)
Glenium 3030	1.96
MB-VR	0.5
IPANEX	14.8
Pozzutec 20+	56.3
PS 1466	6.9

4.9 Compressive Strength and Temperature Gradient Results for Down Selected Mixes

4.9.1. Compressive Strength Test

The concrete formulations obtained as explained in section 4.8 were then tested for compressive strengths. Three specimens were tested for each day. The testing was conducted at 20^{o}F. The results for these tests are provided in Tables 14 and 15 respectively. The graphical representation of the results in Tables 14 and 15 has been provided in Figures 8 and 9.

Mix 8

The data obtained for the mix 8 is as given below. The strength values are denoted in terms of psi.

Table 14 Compressive Strength results for Mix 8 Concrete

Days	Mix 8 (psi)	Standard Deviation (psi)
0	0	0
1	297	+/- 0
3	915	+/- 4
7	1296	+/- 67
14	1610	+/- 37
28	1760	+/- 107
90	1850	+/- 80

Mix 12

The data obtained for the mix 12 is as given below. The strength values are denoted in terms of psi.

Table 15 Compressive Strength results for Mix 12 Concrete

Days	Mix 12 (psi)	Standard Deviation (psi)
0	0	0
1	277	+/- 5
3	851	+/- 9
7	998	+/- 65
14	1443	+/- 15
28	1570	+/- 59
90	1700	+/- 60

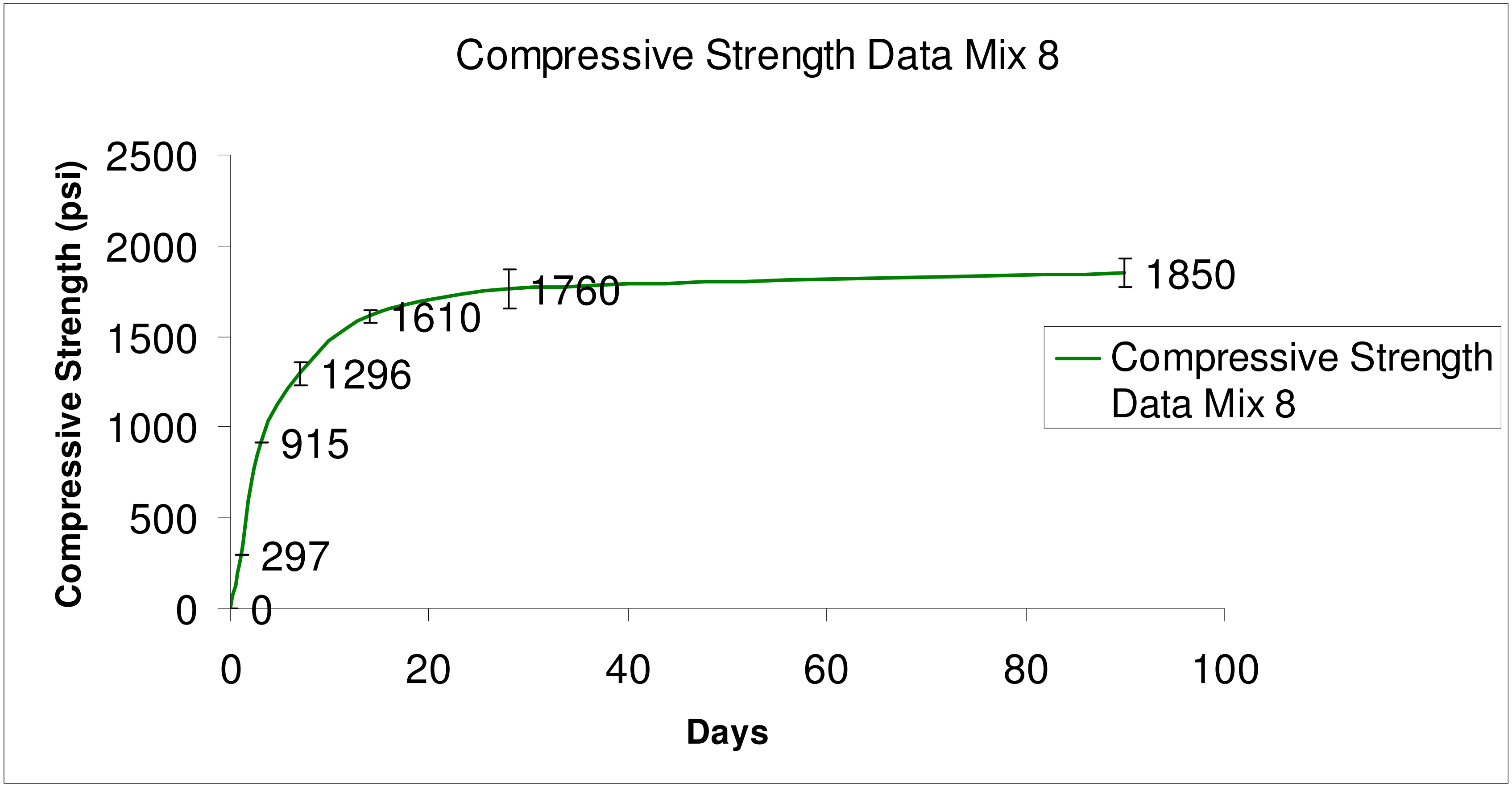

Figure 8 Graphical representation of compressive strength results of Table 14

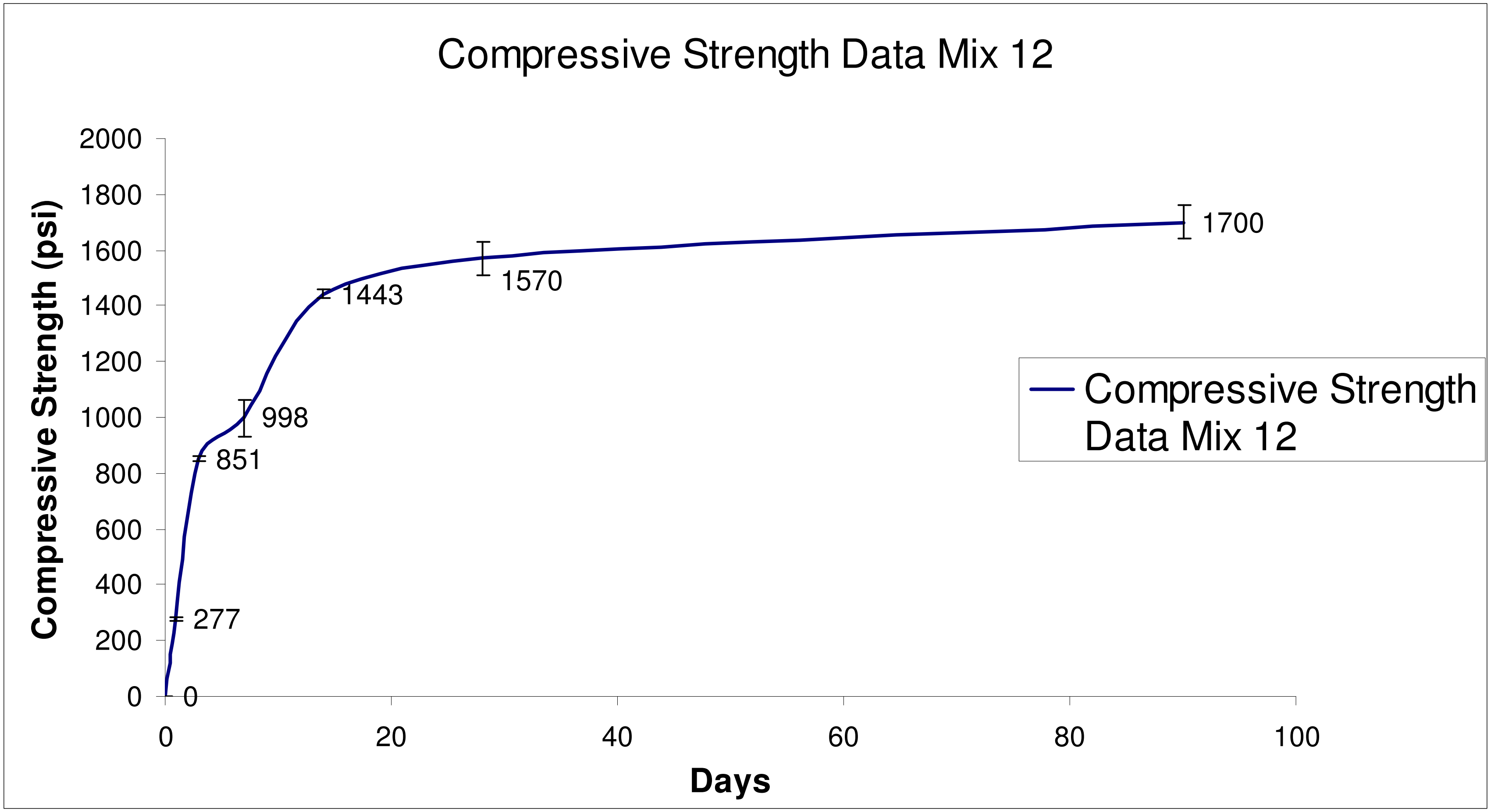

Figure 9 Graphical representation of compressive strength results of Table 15

From the Figure 8 we can see that at 20^{o}F the concrete formulation of Mix 8 gains strength smoothly and at about 28 days shows a distinct plateau and reduction in strength gain. Thus it can be seen that Mix 8 gains most of its strength in the first 28 days.

From Figure 9 it can be seen that Mix 12 also shows behavior of strength gain similar to Mix 8. It also shows a slight plateau in the strength gain from 3 days to 7 days.

4.9.2. Temperature Gradients

As mentioned in Chapter 2, the principle of antifreeze concreting was to maintain the internal temperature of concrete above the freezing point of water thereby allowing the concrete to set before freezing. Thus it was necessary to monitor the internal temperature gradient of the concrete mixes. For each of the mixes temperature gradients were obtained for internal temperature for the first day till the internal temperature reached the ambient temperature. The gradients were obtained by placing thermocouples inside the specimens and then reading the temperatures using a maturity meter. The Figures 10 and 11 provide the Temperature gradients for Mix 8 and 12 respectively.

The temperature gradients for both Mix 8 and Mix 12 show that the internal temperature of the concrete does not reach the ambient temperature of 20^{o}F (approximately -5^{o}C) for at least 15 hours. It can be concluded from this that the concrete mix has 15 hours to form set before freezing.

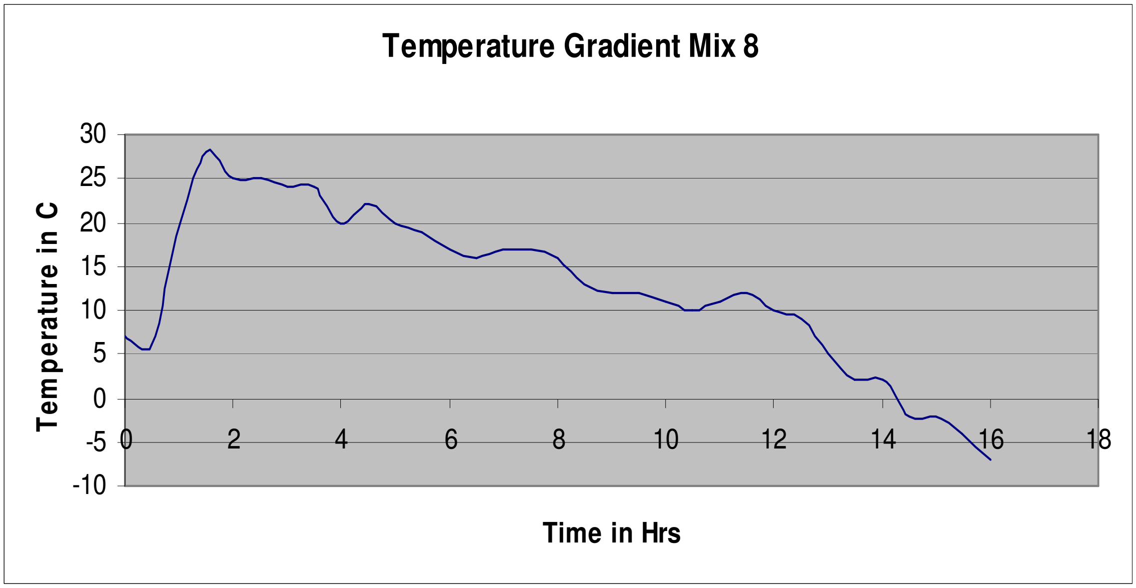

Figure 10: Internal temperature gradient for Mix 8

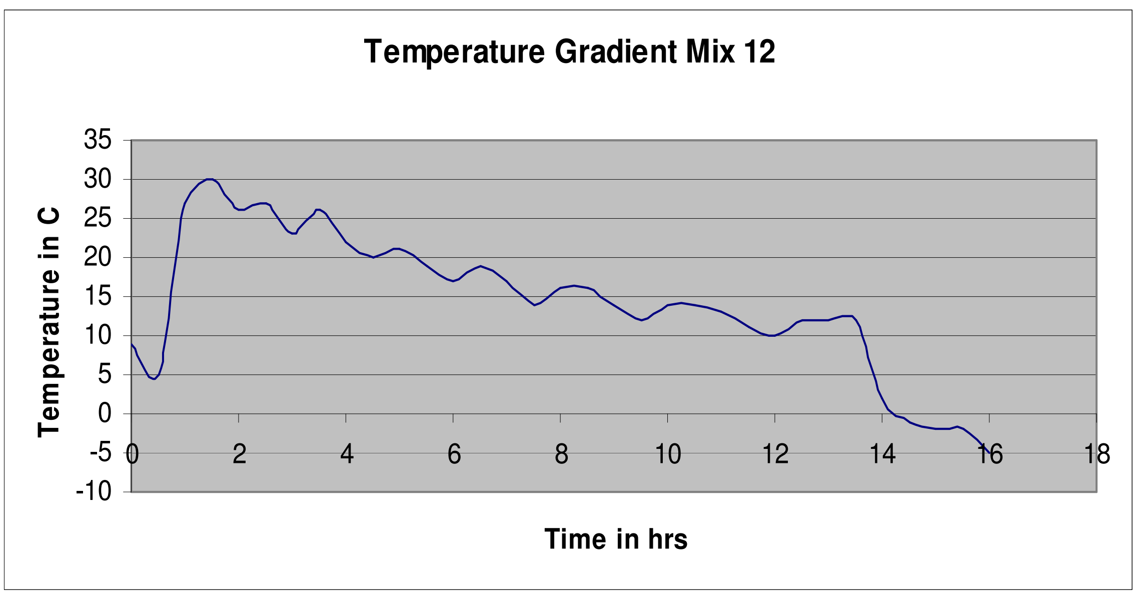

Figure 11: Internal temperature gradient for Mix 12

4.10. Effect of IPANEX

The effect of IPANEX on the properties of the mortar mixes is shown below in Figure 16 with the help of contour plots. Figure 12 is a contour plot which shows the variation of the Vicat setting time with respect to the values of temperature and IPANEX. The contour plot shown in Figure 12 is held at low settings, which signifies the fact that the remaining two factors NaSi, and Rheobuild are held at their lowest individual values. Similarly, in the middle settings, they are held at their centre levels while high settings they are held at their highest levels. Only the graphs for the Vicat setting time in Mix 1 are shown here as an example. The rest of the graphs are provided in the Appendix C.

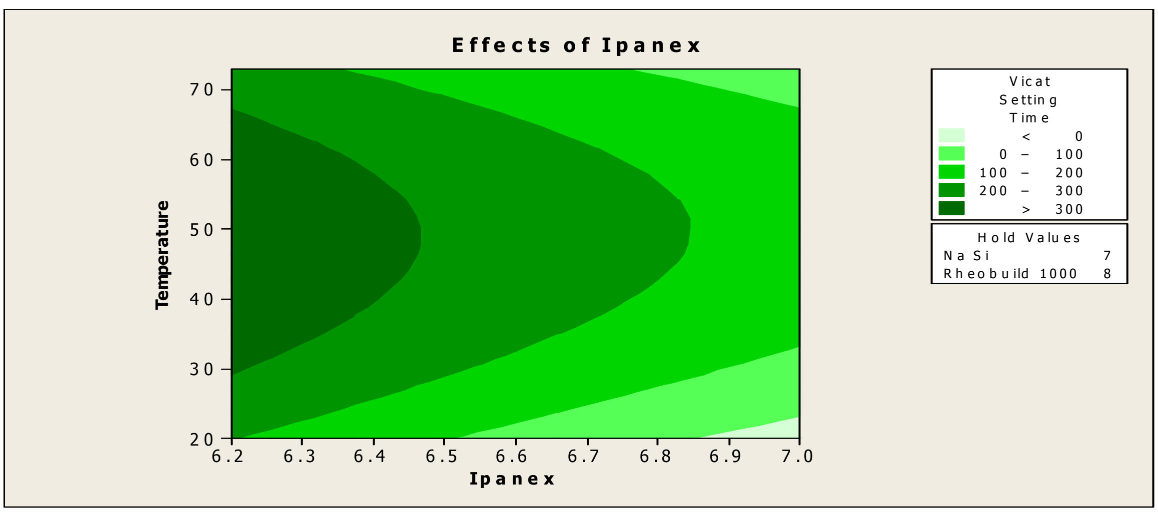

Figure 12: Contour Plot of Vicat Setting Time for Temperature vs IPANEX for the Mix 1 keeping all the other Factors at their low settings.

As seen in the Figure 12, the Temperature vs IPANEX plot has horizontal trending contours at all the settings. Also the value of the setting time decreases as the amount of IPANEX increases. These data suggest that setting varies inversely with the concentration of IPANEX with slightly longer working times at the mid-range temperatures of the study. The observations for the remaining experiments has been given below and all the graphical data have been provided in the Appendix C

The observations for the effects of IPANEX on the yield values and its interaction with other admixtures were as follows:

Mix 1

1) Vicat Setting Time: From the graphs the effect of IPANEX is found to be generally independent of Na silicate and temperature but is slightly dependent on Rheobuild 1000.
2) CST Day 1: It is seen that the effect of IPANEX is independent of the Temperature, Na silicate and Rheobuild 1000 as the contour lines in all three are practically horizontal. The higher values of IPANEX seem to possess higher Compressive strength at Day 1.
3) CST Day 3: The effect of IPANEX on the Day 3 strength is found to be greatly dependent on Na silicate and slightly dependent on Temperature. However it is seen to be independent of Rheobuild 1000.
4) CST Day 4: From the contour lines which are practically horizontal for each of the plot the effect of IPANEX on the compressive strength at day 4 is found to be independent of all the other three factors.

Mix 8

1) Vicat Setting Time.: The effect of IPANEX seems to be independent of temperature however it is seen to be slightly dependent on the PS 1466 and largely dependent on pozzutec 20+

2) CST Day 1: The effect of IPANEX seems very slightly dependent on the Pozzutec 20+ and PS1466 but is independent of temperature.

3) CST Day 3: The effect of IPANEX on the Day 3 compressive strength is independent of temperature but is greatly dependent on the PS 1466 and Pozzutec 20+

4) CST Day 4: IPANEX seems to have an effect on the day 4 compressive strength which is independent of temperature but is highly dependent on the PS1466 and pozzutec 20+.

Mix 12

1) Vicat Setting Time: The effect of IPANEX on the setting time for mix 12 is highly dependent on pozzutec 20+ but is independent of temperature and PS 1466.

2) CST Day 1: The effect of IPANEX on the Day 3 strength is dependent on pozzutec 20 + but is either lightly or not dependent on temperature and PS 1466.

3) CST Day 3: It is found that IPANEX affects the compressive strength t day 3 independent of temperature and PS 1466 but is dependent slightly on pozzutec 20+

4) CST Day 4: The effect of IPANEX seems to be practically independent of all the other three factors.

Chapter 5
Conclusions

1. The three mixes (1, 8, and 12) were analyzed and it was found that the optimization models for Mix 8 and Mix 12 were adequate. The optimization model for the Mix 1 was considered to be inadequate. This was concluded due to the fact that the optimized value of Rheobuild lay outside the range considered for the optimization process. Hence the hypothesis that increased admixture proportions would result in a better performance failed for Mix 1. However for Mix 8 and Mix 12, it was found that the optimized values of the proportions were in the range of values considered for the optimization process. The assumptions made at the start of the analysis (such as the normality assumption) were valid. The number of outliers was small.
2. Two out of three admixture formulations were down selected as primary and back up formulations. Mix 1 as mentioned above proved to have an inadequate optimization model. Also Rheobuild reacted with other admixtures in the formulations during mixing. While placing and finishing the mortar mixes at 20^{o}F Mix 1 showed very poor consistency. All these factors were considered while during the down selection process. Mix 8 and Mix 12 did not show any of the above mentioned drawbacks and were thus down selected as the primary and back up formulations.
3. It was also concluded that IPANEX shows a distinct effect on the various yields such as the Vicat setting time and compressive strength as well as other

admixtures in the formulations. The detailed effects have been presented in section 4.10. This is explained using the contour plots provided in the Appendix C.

4. The comparison of the Analytical and Experimental values (Table 11) of the compressive strengths and Vicat setting times of the optimized proportions of Mix 8 and Mix 12 shows that there is disparity (around 400 psi to 2000 psi) in three values and more testing should be conducted for verification of the optimized proportions. This disparity was concluded to be due to the use of two specimens for the optimization process which was the basis for the analytical values while the use of three specimens for each of the experimental values.

5.1 Future Scope

Extensive concrete testing of the optimized admixture formulations should be undertaken before the formulations can be introduced commercially. It is necessary to perform concrete durability testing as well as other fundamental testing for the concrete mixes 8 and 12 as mortar testing may not be sufficient to justify use of the optimized proportions in concrete.

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Appendix A: Data Tables

Table A1: Phase 2 Vicat setting time and Compressive Strength Data for Mix 8

Blocks	IPANEX	Pozzutec 20+	PS 1466	Temperature	Vicat Setting Time	CST DAY 1	CST DAY 3	CST DAY 4
2	6.4	34	3.5	20	90	261	651	486
2	6.4	30	2.5	20	90	100	329.5	233.5
2	6.4	34	2.5	73	150	2396.5	4618.5	5333
2	6.6	32	3	40	435	142.5	2171.5	2492.5
2	6.8	34	3.5	73	150	2015.5	4035.5	5003
2	6.6	32	3	40	405	140.5	2198	2489
2	6.4	30	3.5	73	150	2065.5	4200.5	4888
2	6.8	30	2.5	73	120	2327.5	5020.5	4735
2	6.8	30	3.5	20	90	75.5	231.5	288
2	6.8	34	2.5	20	90	126	352.5	373.5
3	6.6	28	3	40	420	94	2355.5	2597.5
3	6.6	32	3	20	90	139.5	442	500
3	6.2	32	3	40	390	45.5	2204	2406.5
3	6.6	32	3	40	420	170	2118	2432.5
3	6.6	32	3	73	150	1920	3575	4542
3	7	32	3	40	405	111	1972.5	2556
3	6.6	36	3	40	435	53	2079	2499.5
3	6.6	32	4	40	420	89	2310	2594.5
3	6.6	32	3	40	435	143	2146	2426.5
3	6.6	32	2	40	390	215.5	2506.5	2810
1	6.8	30	3	73	225	2545.5	4617	4712.5
1	6.8	34	2.5	73	165	2167	4377	5025.5
1	6.6	32	3	40	450	134	2178.5	2525.5
1	6.8	34	3.5	20	90	125	338	448
1	6.4	34	3.5	73	240	2050	4282	5203
1	6.4	34	2.5	20	90	76	315	300.5
1	6.6	32	3	40	420	147.5	2203.5	2532.5
1	6.4	30	3.5	20	90	99.5	286	376.5
1	6.4	30	2.5	73	180	2127	4519.5	4175.5
1	6.8	30	2.5	20	90	86.5	309	410.5

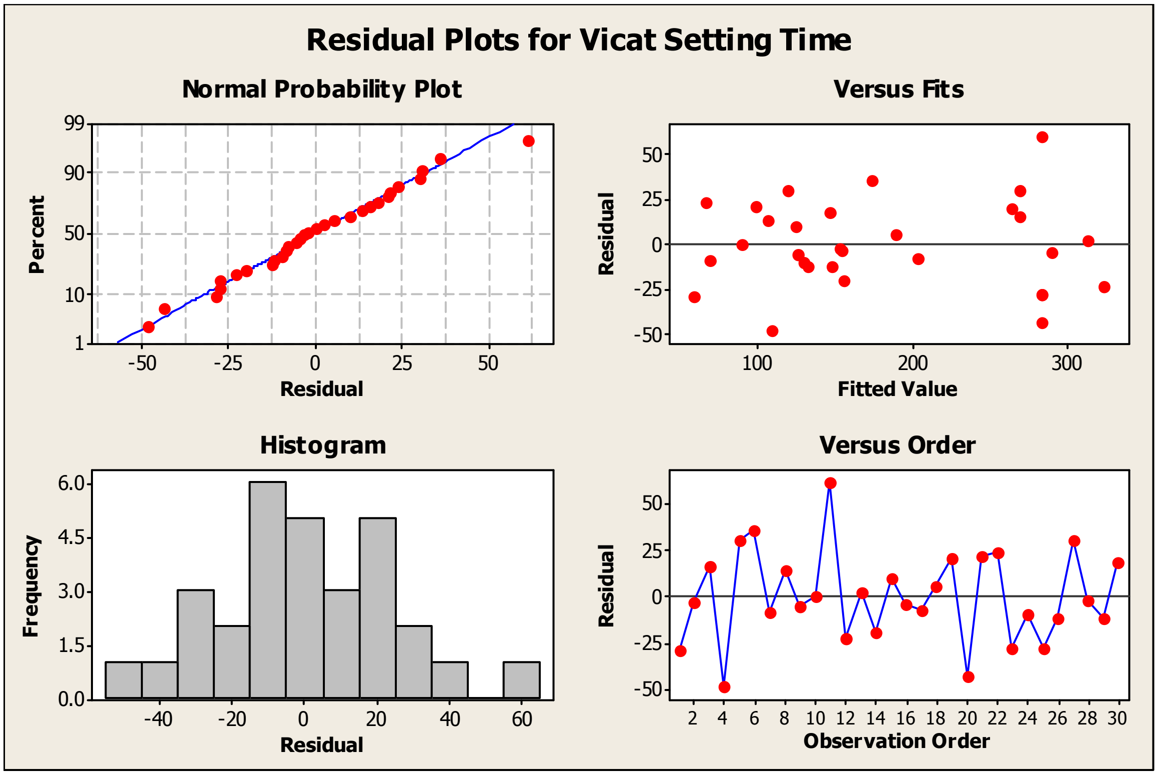
Table A2: Phase 2 Vicat setting time and Compressive Strength Data for Mix 12

IPANEX	Pozzolith 122HE	PS 1466	Temperature	Vicat Setting Time	CST DAY1	CST DAY3	CST DAY4	Blocks
6.8	30	2.5	20	120	133	895	734.5	1
6.4	34	2.5	20	90	98.5	556.5	906.5	1
6.4	30	3.5	20	120	91	529.5	770.5	1
6.8	34	2.5	73	180	5024.5	4904.5	5459.5	1
6.6	32	3	40	435	665	3092	3968	1
6.4	30	2.5	73	210	4745	4810	5439	1
6.6	32	3	40	420	664	3071	4089	1
6.8	30	3.5	73	210	3828.5	4563	5723	1
6.4	34	3.5	73	195	4254.5	4380.5	5247	1
6.8	34	3.5	20	120	50.5	594.5	989	1
6.6	28	3	40	465	578	3145	4119	3
6.2	32	3	40	405	676.5	2809	3118.5	3
6.6	32	3	73	255	4204	4889	5821.5	3
6.6	32	3	40	390	649	3155	3943.5	3
6.6	32	3.5	40	420	804.5	3572	4590.5	3
6.6	32	3	40	465	683	3126.5	3944.5	3
7	32	3	40	480	886	3208	3413.5	3
6.6	36	3	40	375	815	3489	3671.5	3
6.6	32	2	40	300	902.5	3300.5	3409.5	3
6.6	32	3	20	90	53.5	548.5	925.5	3
6.6	32	3	40	450	630.5	3160	4072	2
6.4	34	2.5	73	180	3553	4464	5539.5	2
6.4	30	3.5	73	270	3633.5	4916.5	5517	2
6.8	34	3.5	73	225	4244	4457.5	4920	2
6.8	34	2.5	20	120	29.5	245	436.5	2
6.8	30	2.5	73	180	3487	4457.5	5304.5	2
6.6	32	3	40	420	660	3155.5	4149	2
6.4	30	2.5	20	150	44	328	524.5	2
6.8	30	3.5	20	180	28.5	170.5	444	2
6.4	34	3.5	20	90	31.5	128	479	2

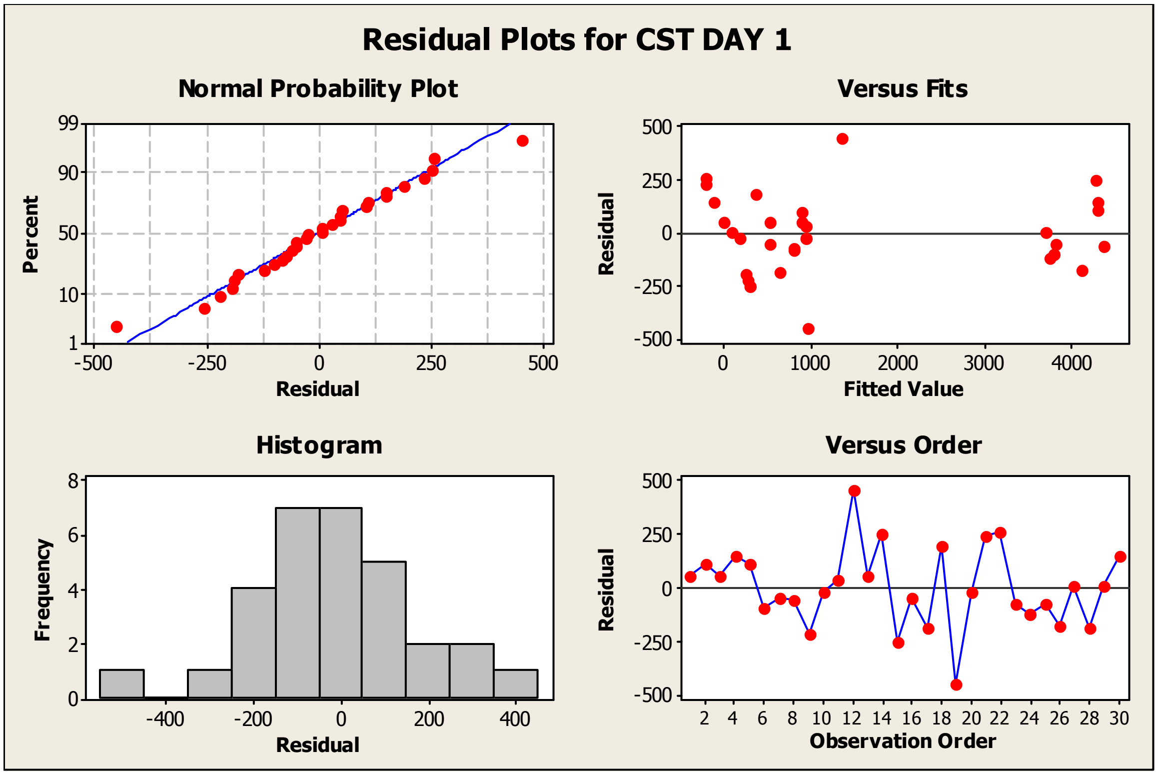
Appendix B

Analysis of the Mix 1 Data in table 3 is presented below.

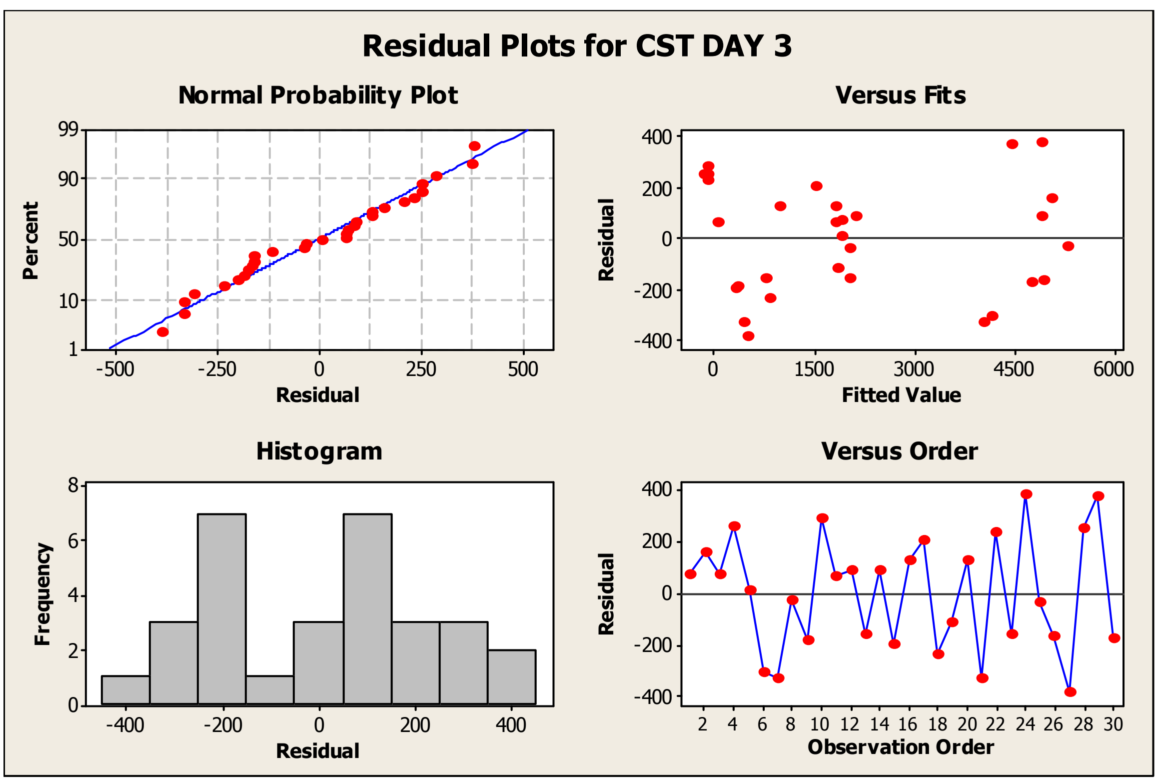
Response Surface Regression: Vicat Setting Time



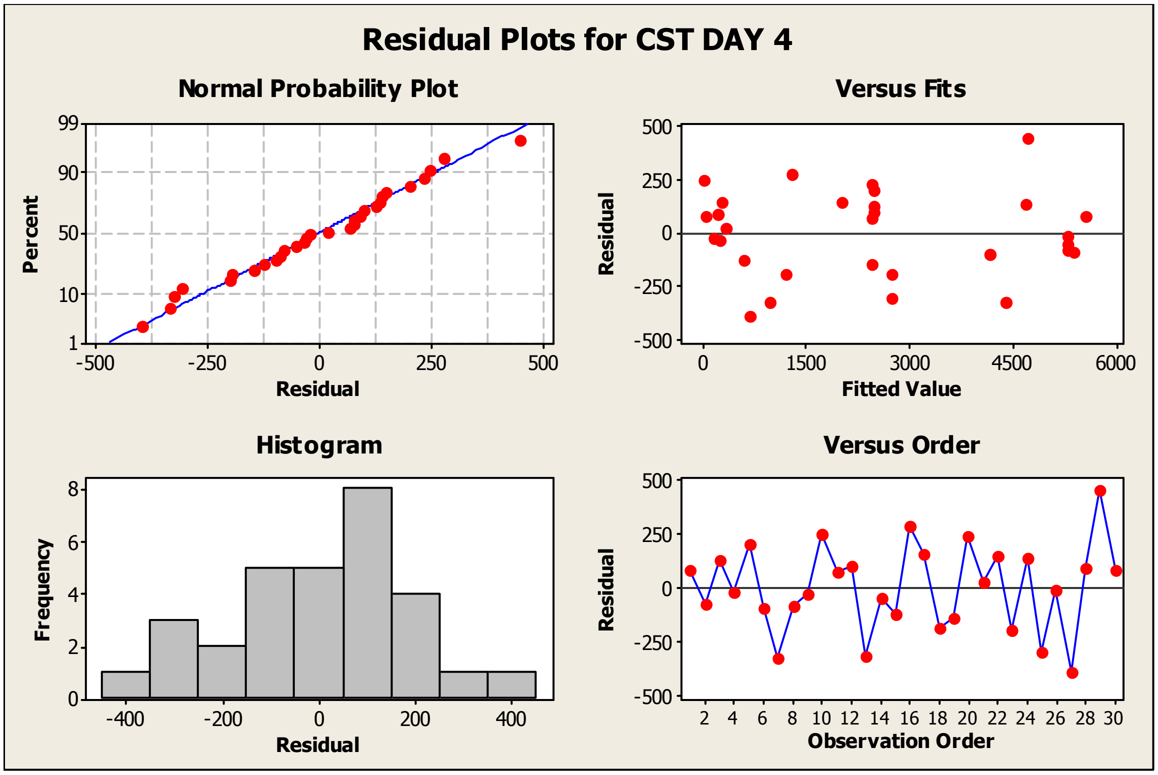
Appendix B1: Residual plots for Mix 1 for the Vicat Setting Time Data



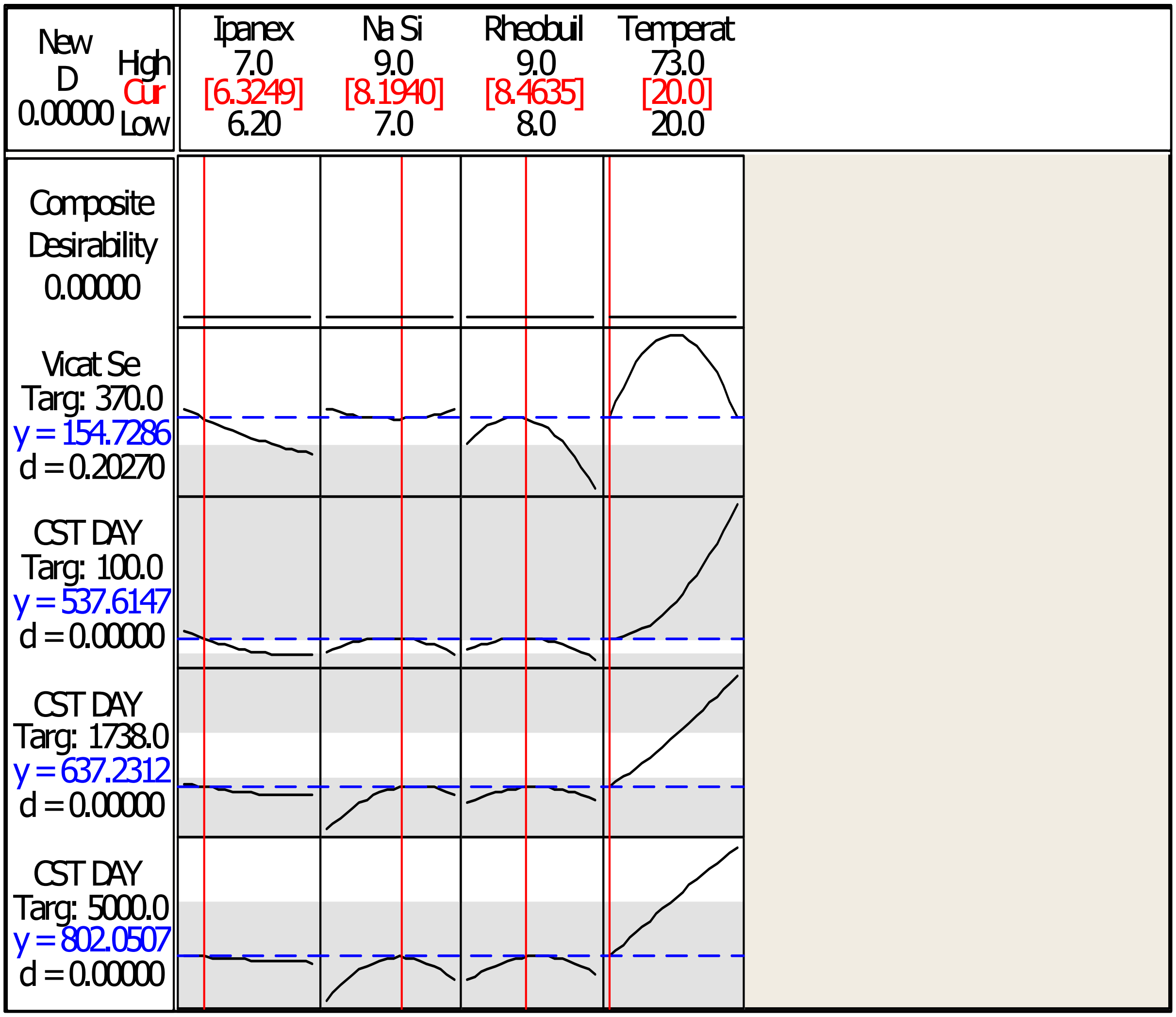
Appendix B2: Residual plots for Mix 1 for the CST Day 1 Data



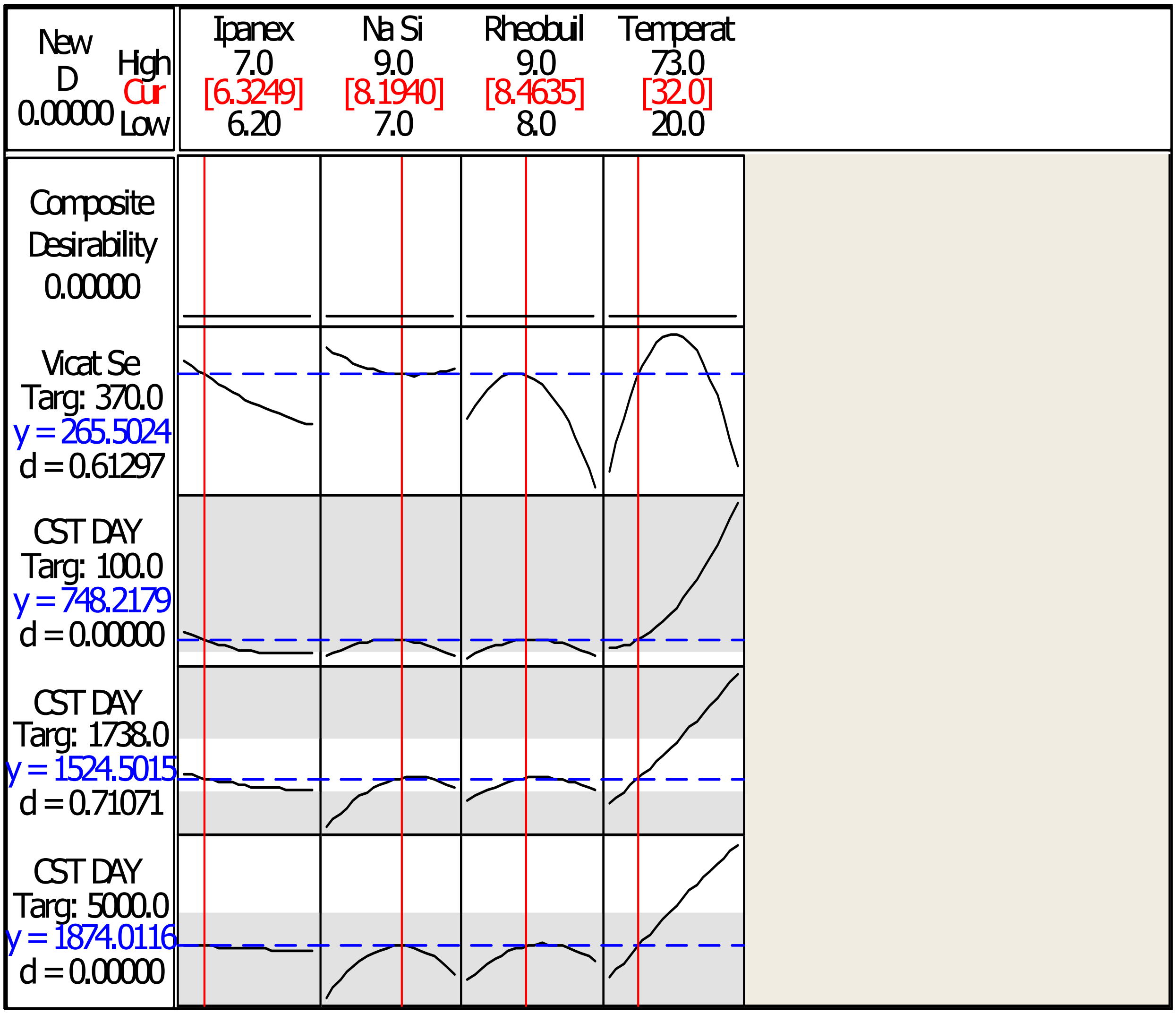
Appendix B3: Residual plots for Mix 1 for the CST Day 3 Data



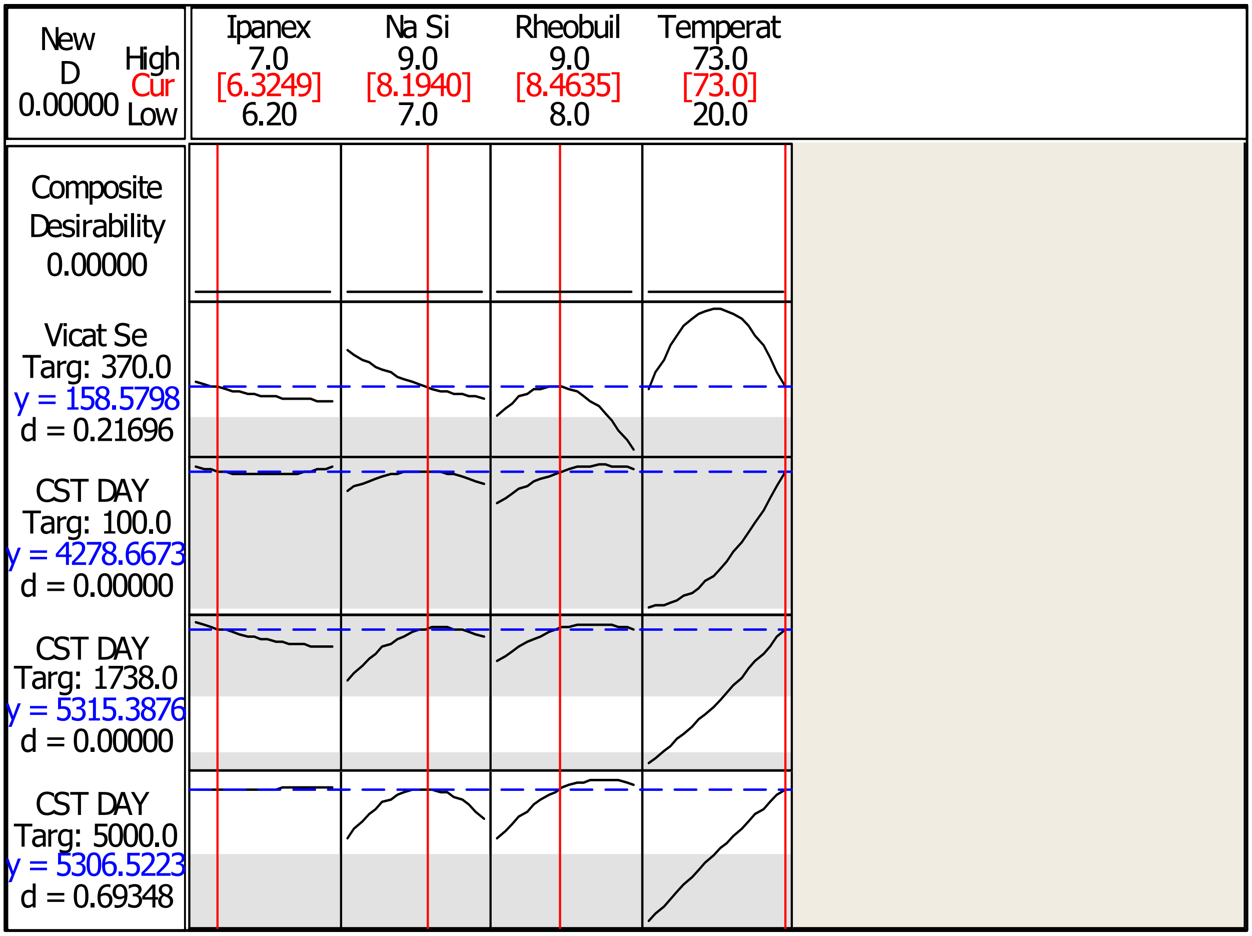
Appendix B4: Residual plots for Mix 1 for the CST Day 4 Data



Appendix B5: Optimization Graph for the Mix 1 at 20F



Appendix B6: Optimization Graph for the Mix 1 at 32F



Appendix B7: Optimization Graph for the Mix 1 at 73F

Table B1: Response Surface Regression Analysis (Minitab Output) for Mix 1: Vicat Setting Time

The analysis was done using coded units.

Estimated Regression Coefficients for Vicat Setting Time

Term	Coef	SE Coef	T	P
Constant	291.867	15.437	18.907	0.000
Block 1	-9.250	10.084	-0.917	0.376
Block 2	4.250	10.084	0.421	0.680
IPANEX	-26.494	15.042	-1.761	0.102
Na Si	-16.803	15.042	-1.117	0.284
Rheobuild 1000	7.874	15.042	0.524	0.609
Temperature	15.000	8.642	1.736	0.106
IPANEX*IPANEX	10.000	28.156	0.355	0.728
Na Si*Na Si	17.500	28.156	0.622	0.545
Rheobuild 1000*Rheobuild 1000	-87.500	28.156	-3.108	0.008
Temperature*Temperature	-156.867	18.616	-8.427	0.000
IPANEX*Na Si	7.500	36.664	0.205	0.841
IPANEX*Rheobuild 1000	75.000	36.664	2.046	0.062
IPANEX*Temperature	12.300	18.151	0.678	0.510
Na Si*Rheobuild 1000	30.000	36.664	0.818	0.428
Na Si*Temperature	-22.058	18.151	-1.215	0.246
Rheobuild 1000*Temperature	4.578	18.151	0.252	0.805

S = 36.6645   PRESS = 88438.1
R-Sq = 91.98% R-Sq(pred) = 59.44% R-Sq(adj) = 82.12%

Estimated Regression Coefficients for Vicat Setting Time using data in uncoded units

Term	Coef
Constant	5065.83
Block 1	-9.25000
Block 2	4.25000
IPANEX	-4282.70
Na Si	-891.849
Rheobuild 1000	2994.68
Temperature	17.4034
IPANEX*IPANEX	62.5000
Na Si*Na Si	17.5000
Rheobuild 1000*Rheobuild 1000	-350.000
Temperature*Temperature	-0.223378
IPANEX*Na Si	18.7500
IPANEX*Rheobuild 1000	375.000
IPANEX*Temperature	1.16042
Na Si*Rheobuild 1000	60.0000
Na Si*Temperature	-0.832364
Rheobuild 1000*Temperature	0.345510

Table B2: Response Surface Regression Analysis (Minitab Output) for Mix 1: CST Day 1

Response Surface Regression: CST DAY 1

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY 1

Term	Coef	SE Coef	T	P
Constant	1309.79	114.69	11.420	0.000
Block 1	17.80	74.92	0.238	0.816
Block 2	-83.80	74.92	-1.119	0.284
IPANEX	-155.60	111.75	-1.392	0.187
Na Si	19.93	111.75	0.178	0.861
Rheobuild 1000	210.85	111.75	1.887	0.082
Temperature	2001.69	64.20	31.177	0.000
IPANEX*IPANEX	222.10	209.19	1.062	0.308
Na Si*Na Si	-418.90	209.19	-2.003	0.067
Rheobuild 1000*Rheobuild 1000	-438.65	209.19	-2.097	0.056
Temperature*Temperature	908.40	138.30	6.568	0.000
IPANEX*Na Si	-36.63	272.40	-0.134	0.895
IPANEX*Rheobuild 1000	77.88	272.40	0.286	0.779
IPANEX*Temperature	175.67	134.85	1.303	0.215
Na Si*Rheobuild 1000	91.37	272.40	0.335	0.743
Na Si*Temperature	59.85	134.85	0.444	0.664
Rheobuild 1000*Temperature	300.42	134.85	2.228	0.044

S = 272.398   PRESS = 7580646
R-Sq = 98.88% R-Sq(pred) = 91.20% R-Sq(adj) = 97.50%

Estimated Regression Coefficients for CST DAY 1 using data in uncoded units

Term	Coef
Constant	-49373.2
Block 1	17.8048
Block 2	-83.7952
IPANEX	-22059.7
Na Si	5668.32
Rheobuild 1000	25164.0
Temperature	-364.932
IPANEX*IPANEX	1388.10
Na Si*Na Si	-418.904
Rheobuild 1000*Rheobuild 1000	-1754.62
Temperature*Temperature	1.29355
IPANEX*Na Si	-91.5625
IPANEX*Rheobuild 1000	389.375
IPANEX*Temperature	16.5722
Na Si*Rheobuild 1000	182.750
Na Si*Temperature	2.25835
Rheobuild 1000*Temperature	22.6734

Table B3: Response Surface Regression Analysis (Minitab Output) for Mix 1:CST Day 3

Response Surface Regression: CST DAY 3 versus Block, IPANEX, Na Si, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY 3

Term	Coef	SE Coef	T	P
Constant	2468.46	138.48	17.825	0.000
Block 1	-13.65	90.46	-0.151	0.882
Block 2	108.70	90.46	1.202	0.251
IPANEX	-156.25	134.93	-1.158	0.268
Na Si	104.39	134.93	0.774	0.453
Rheobuild 1000	395.86	134.93	2.934	0.012
Temperature	2298.75	77.52	29.652	0.000
IPANEX*IPANEX	159.13	252.58	0.630	0.540
Na Si*Na Si	-942.87	252.58	-3.733	0.003
Rheobuild 1000*Rheobuild 1000	-652.62	252.58	-2.584	0.023
Temperature*Temperature	245.41	166.99	1.470	0.165
IPANEX*Na Si	-907.13	328.91	-2.758	0.016
IPANEX*Rheobuild 1000	89.13	328.91	0.271	0.791
IPANEX*Temperature	-86.07	162.83	-0.529	0.606
Na Si*Rheobuild 1000	-127.13	328.91	-0.387	0.705
Na Si*Temperature	-5.99	162.83	-0.037	0.971
Rheobuild 1000*Temperature	242.42	162.83	1.489	0.160

S = 328.907   PRESS = 12112386
R-Sq = 98.67% R-Sq(pred) = 88.54% R-Sq(adj) = 97.03%

Estimated Regression Coefficients for CST DAY 3 using data in uncoded units

Term	Coef
Constant	-318800
Block 1	-13.6478
Block 2	108.702
IPANEX	1213.58
Na Si	32329.5
Rheobuild 1000	43412.1
Temperature	-45.8663
IPANEX*IPANEX	994.551
Na Si*Na Si	-942.872
Rheobuild 1000*Rheobuild 1000	-2610.49
Temperature*Temperature	0.349461
IPANEX*Na Si	-2267.81
IPANEX*Rheobuild 1000	445.625
IPANEX*Temperature	-8.11998
Na Si*Rheobuild 1000	-254.250
Na Si*Temperature	-0.226064
Rheobuild 1000*Temperature	18.2955

Table B4: Response Surface Regression Analysis (Minitab Output) for Mix 1: CST Day 4

Response Surface Regression: CST DAY 4 versus Block, IPANEX, Na Si, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY 4

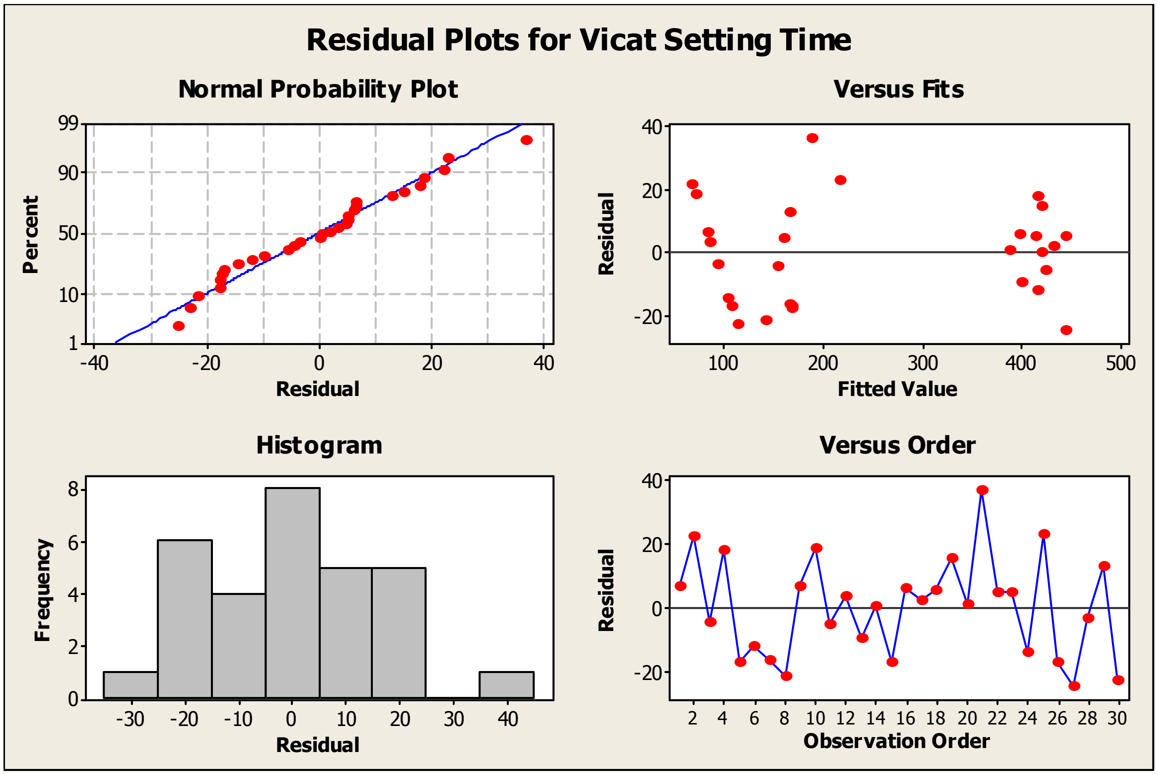
Term	Coef	SE Coef	T	P
Constant	3136.96	125.56	24.984	0.000
Block 1	-71.61	82.02	-0.873	0.398
Block 2	190.49	82.02	2.323	0.037
IPANEX	7.55	122.34	0.062	0.952
Na Si	157.01	122.34	1.283	0.222
Rheobuild 1000	497.05	122.34	4.063	0.001
Temperature	2349.03	70.29	33.420	0.000
IPANEX*IPANEX	34.25	229.01	0.150	0.883
Na Si*Na Si	-1324.00	229.01	-5.781	0.000
Rheobuild 1000*Rheobuild 1000	-837.75	229.01	-3.658	0.003
Temperature*Temperature	-74.34	151.41	-0.491	0.632
IPANEX*Na Si	-333.63	298.21	-1.119	0.283
IPANEX*Rheobuild 1000	-47.62	298.21	-0.160	0.876
IPANEX*Temperature	90.81	147.63	0.615	0.549
Na Si*Rheobuild 1000	92.62	298.21	0.311	0.761
Na Si*Temperature	-27.96	147.63	-0.189	0.853
Rheobuild 1000*Temperature	395.60	147.63	2.680	0.019

S = 298.211   PRESS = 8729422
R-Sq = 98.94% R-Sq(pred) = 91.99% R-Sq(adj) = 97.63%

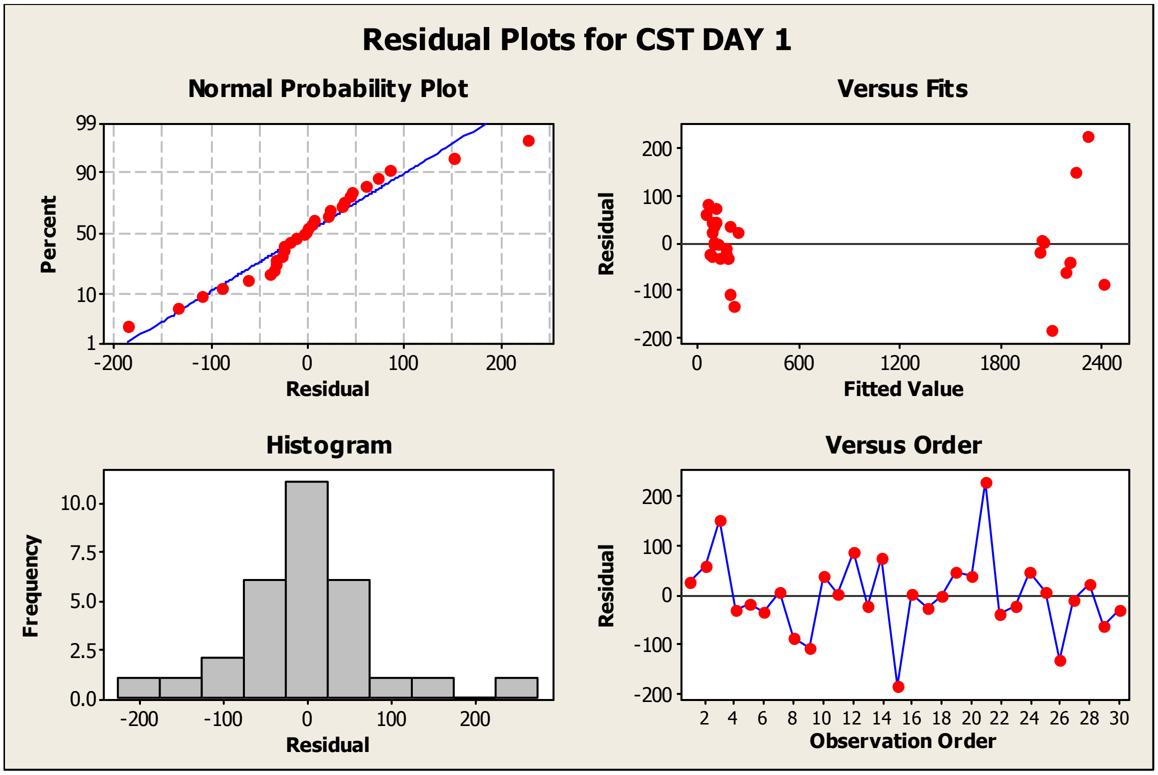
Estimated Regression Coefficients for CST DAY 4 using data in uncoded units

Term	Coef
Constant	-359328
Block 1	-71.6125
Block 2	190.487
IPANEX	5491.44
Na Si	25320.3
Rheobuild 1000	56662.4
Temperature	-203.394
IPANEX*IPANEX	214.063
Na Si*Na Si	-1324.00
Rheobuild 1000*Rheobuild 1000	-3351.00
Temperature*Temperature	-0.105855
IPANEX*Na Si	-834.063
IPANEX*Rheobuild 1000	-238.125
IPANEX*Temperature	8.56707
Na Si*Rheobuild 1000	185.250
Na Si*Temperature	-1.05514
Rheobuild 1000*Temperature	29.8564

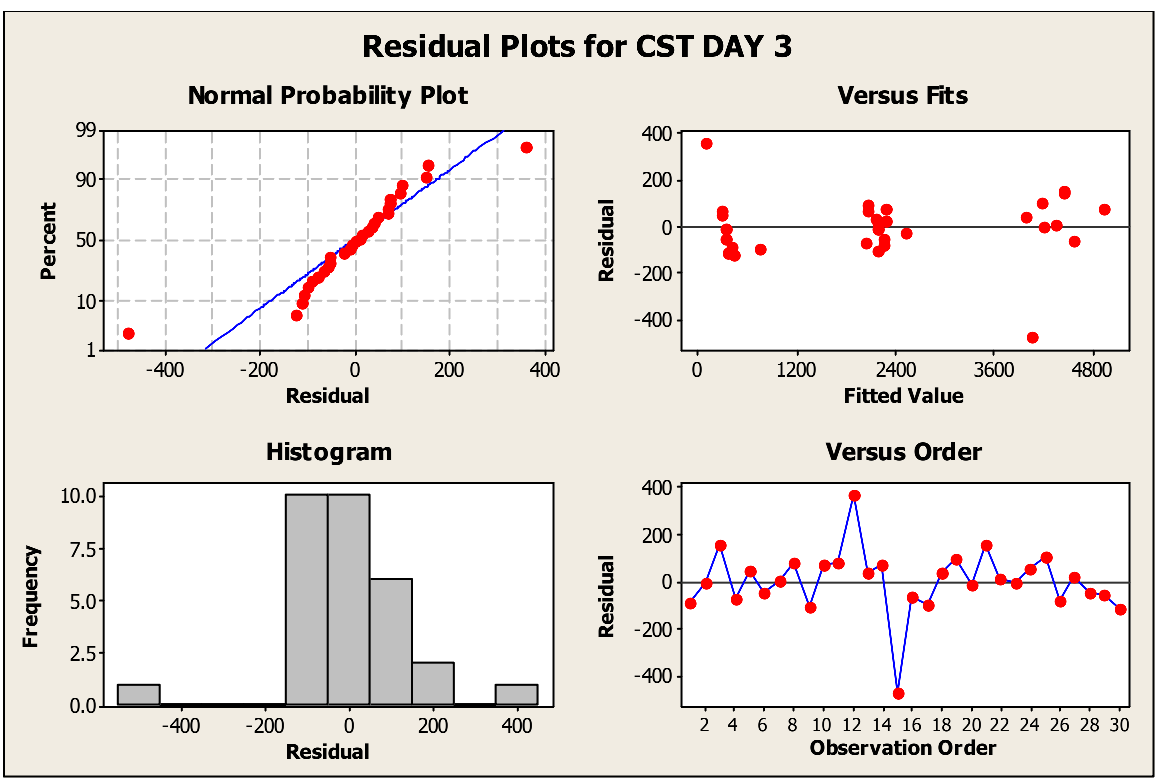
Analysis of the Mix 8 Data in table 4 is presented below.
Response Surface Regression: Vicat Setting Time



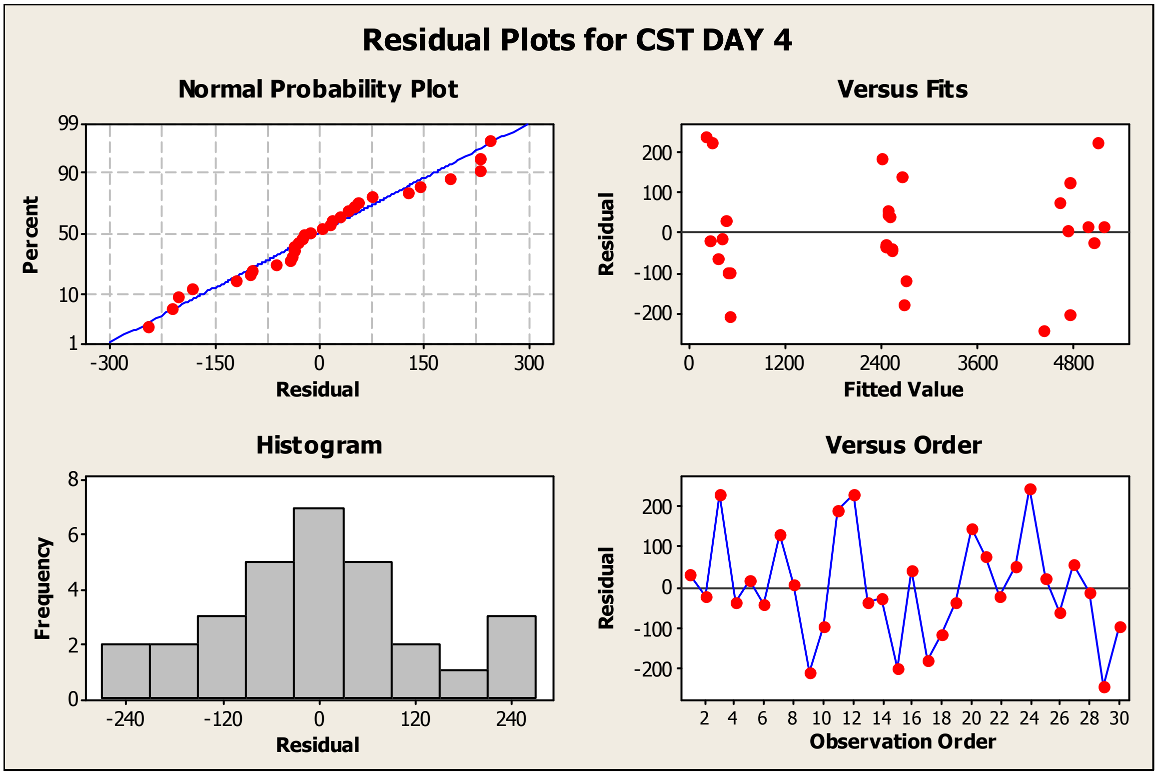
Appendix B8: Residual plots for Mix 8 for the Vicat Setting Time Data



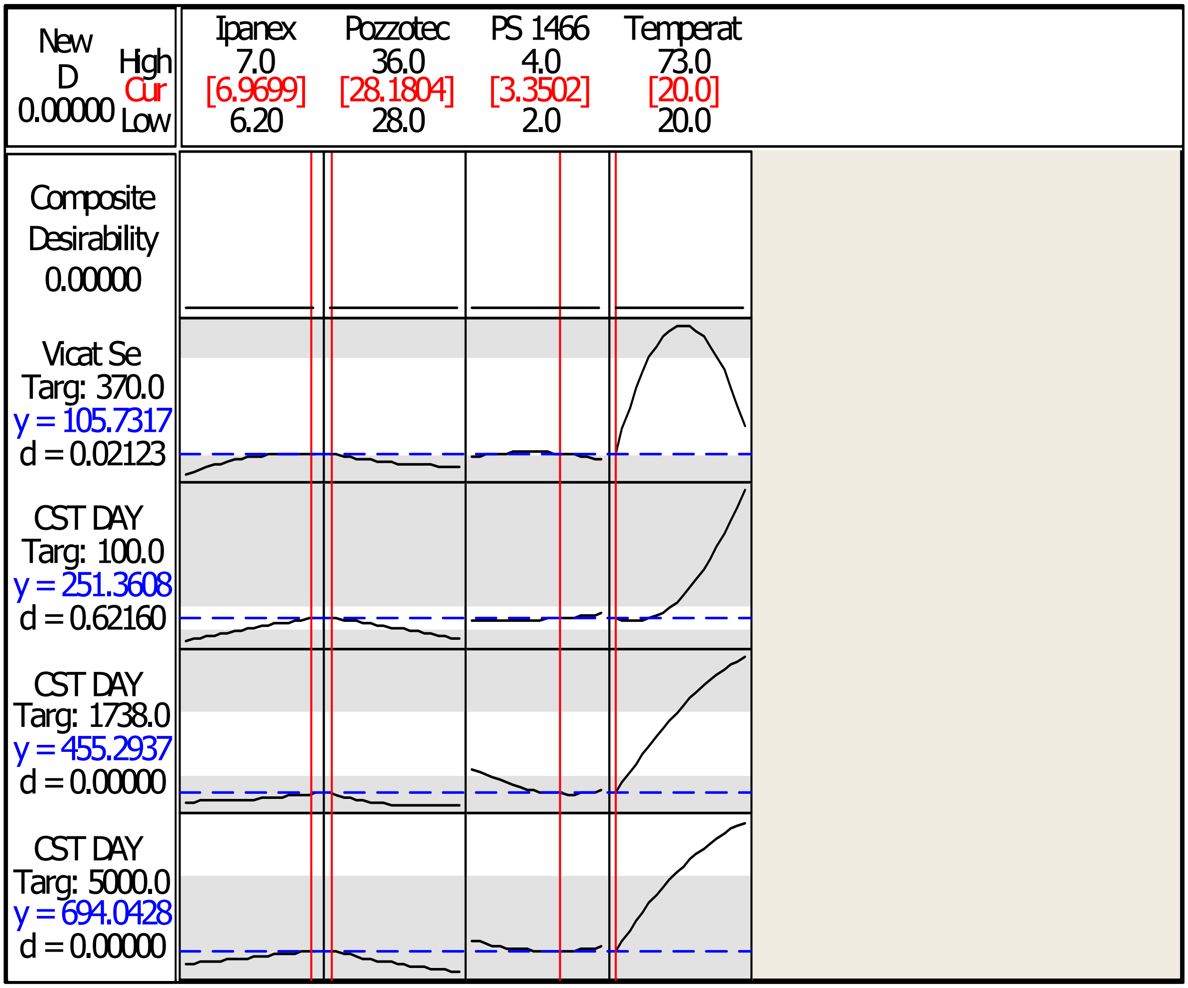
Appendix B9: Residual plots for Mix 8 for the CST Day 1 Data



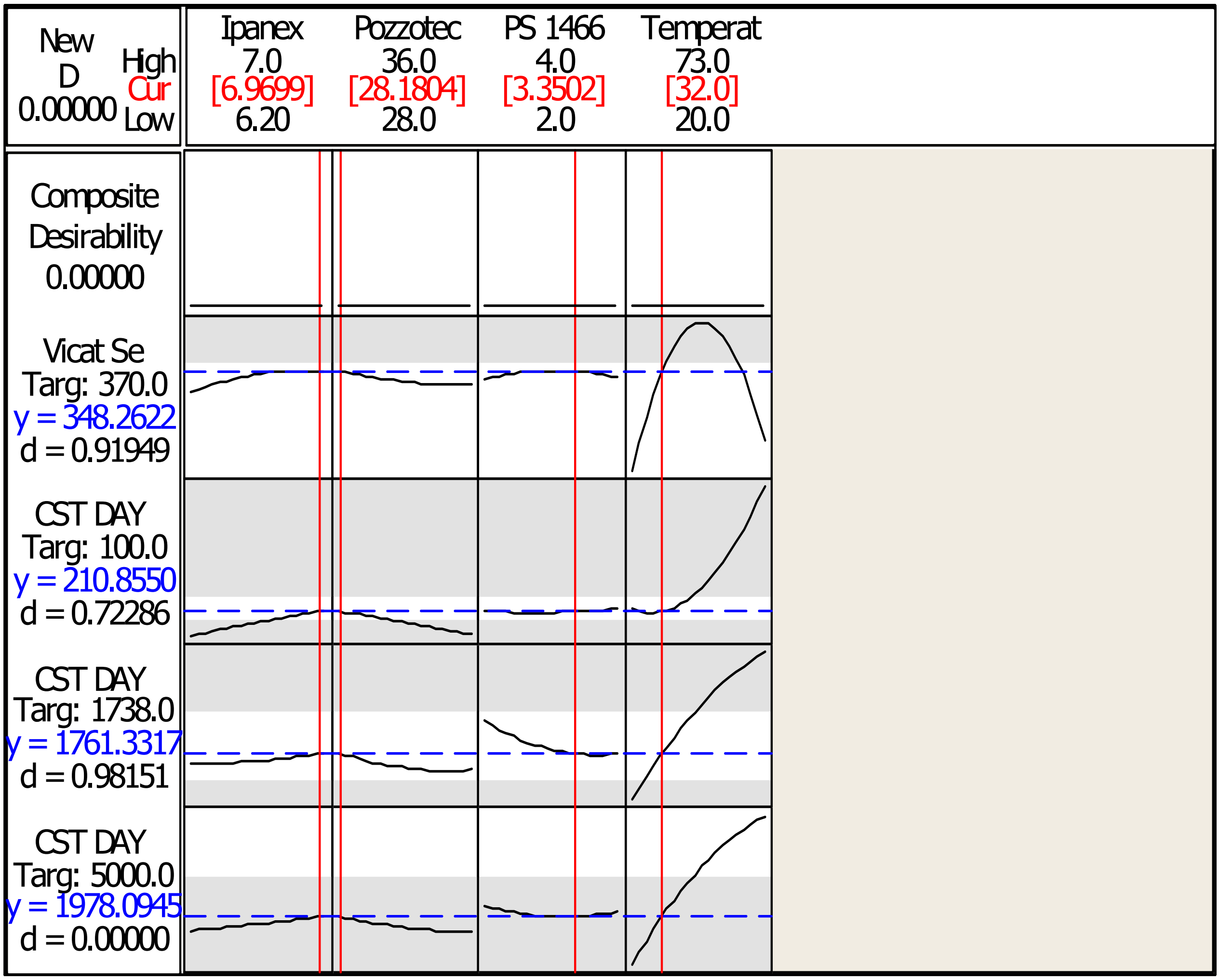
Appendix B10: Residual plots for Mix 8 for the CST Day 3 Data



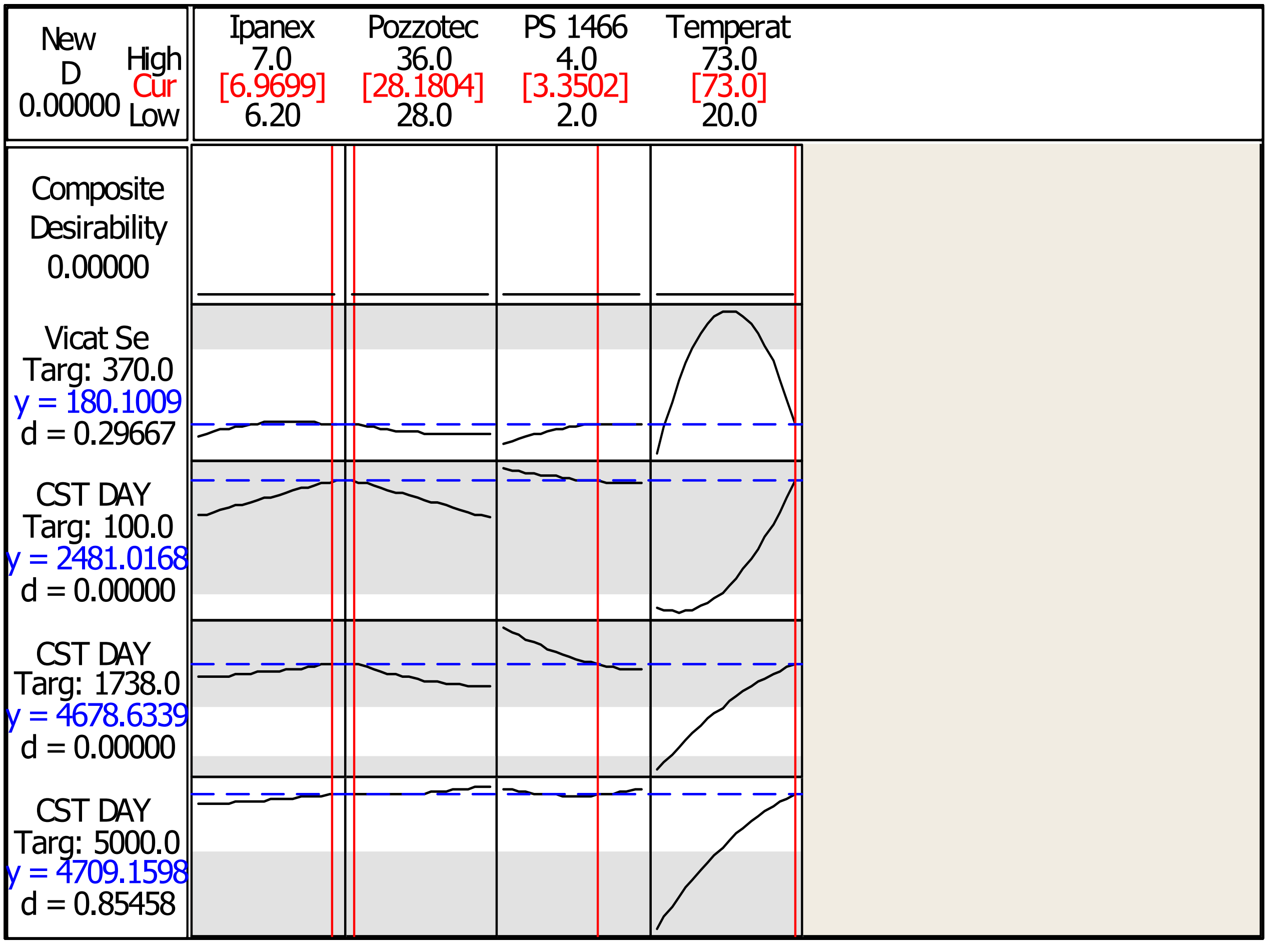
Appendix B11: Residual plots for Mix 1 for the CST Day 4 Data



Appendix B12: Optimization Graph for the Mix 8 at 20F



Appendix B13: Optimization Graph for the Mix 8 at 32F



Appendix B14: Optimization Graph for the Mix 8 at 73F

Table B5: Response Surface Regression Analysis (Minitab Output) for Mix 8: Vicat Setting Time

Response Surface Regression: Vicat Settin versus Block, IPANEX, Pozzotech 20, .

The analysis was done using coded units.

Estimated Regression Coefficients for Vicat Setting Time

Term	Coef	SE Coef	T	P
Constant	456.606	9.725	46.950	0.000
Block 1	17.723	6.450	2.748	0.017
Block 2	-10.268	6.385	-1.608	0.132
IPANEX	-2.266	9.605	-0.236	0.817
Pozzotech 20+	4.342	9.605	0.452	0.659
PS 1466	16.500	10.050	1.642	0.125
Temperature	40.551	5.522	7.344	0.000
IPANEX*IPANEX	-20.570	17.805	-1.155	0.269
Pozzotech 20+*Pozzotech 20+	9.430	17.805	0.530	0.605
PS 1466*PS 1466	-17.665	17.767	-0.994	0.338
Temperature*Temperature	-322.139	11.739	-27.441	0.000
IPANEX*Pozzotech 20+	-24.979	23.460	-1.065	0.306
IPANEX*PS 1466	0.791	24.740	0.032	0.975
IPANEX*Temperature	-7.240	11.636	-0.622	0.545
Pozzotech 20+*PS 1466	6.709	24.740	0.271	0.791
Pozzotech 20+*Temperature	2.061	11.636	0.177	0.862
PS 1466*Temperature	15.124	12.359	1.224	0.243

S = 23.1580   PRESS = 48128.5
R-Sq = 98.92% R-Sq(pred) = 92.54% R-Sq(adj) = 97.59%

Estimated Regression Coefficients for Vicat Setting Time using data in uncoded units

Term	Coef
Constant	-9007.19
Block 1	17.7234
Block 2	-10.2683
IPANEX	2216.80
Pozzotech 20+	60.4696
PS 1466	29.2275
Temperature	46.3652
IPANEX*IPANEX	-128.565
Pozzotech 20+*Pozzotech 20+	0.589354
PS 1466*PS 1466	-17.6645
Temperature*Temperature	-0.458724
IPANEX*Pozzotech 20+	-15.6119
IPANEX*PS 1466	1.97675
IPANEX*Temperature	-0.683048
Pozzotech 20+*PS 1466	1.67732
Pozzotech 20+*Temperature	0.0194448
PS 1466*Temperature	0.570731

Table B6: Response Surface Regression Analysis (Minitab Output) for Mix 8: CST Day 1

Response Surface Regression: CST DAY 1 versus Block, IPANEX, Pozzotech 20, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY 1

Term	Coef	SE Coef	T	P
Constant	347.94	49.81	6.986	0.000
Block 1	15.30	33.03	0.463	0.651
Block 2	30.92	32.70	0.945	0.362
IPANEX	34.77	49.19	0.707	0.492
Pozzotech 20+	-25.02	49.19	-0.509	0.620
PS 1466	-67.59	51.47	-1.313	0.212
Temperature	1025.66	28.28	36.268	0.000
IPANEX*IPANEX	-6.15	91.19	-0.067	0.947
Pozzotech 20+*Pozzotech 20+	-10.90	91.19	-0.119	0.907
PS 1466*PS 1466	39.25	90.99	0.431	0.673
Temperature*Temperature	778.37	60.12	12.946	0.000
IPANEX*Pozzotech 20+	-232.33	120.15	-1.934	0.075
IPANEX*PS 1466	-3.64	126.71	-0.029	0.978
IPANEX*Temperature	58.04	59.60	0.974	0.348
Pozzotech 20+*PS 1466	-41.61	126.71	-0.328	0.748
Pozzotech 20+*Temperature	-73.26	59.60	-1.229	0.241
PS 1466*Temperature	-98.39	63.30	-1.554	0.144

S = 118.605 PRESS = 1270932
R-Sq = 99.32% R-Sq(pred) = 95.30% R-Sq(adj) = 98.49%

Estimated Regression Coefficients for CST DAY 1 using data in uncoded units

Term	Coef
Constant	-32955.4
Block 1	15.2969
Block 2	30.9152
IPANEX	5013.19
Pozzotech 20+	1059.04
PS 1466	262.482
Temperature	-67.2602
IPANEX*IPANEX	-38.4112
Pozzotech 20+*Pozzotech 20+	-0.680987
PS 1466*PS 1466	39.2467
Temperature*Temperature	1.10840
IPANEX*Pozzotech 20+	-145.206
IPANEX*PS 1466	-9.08973
IPANEX*Temperature	5.47561
Pozzotech 20+*PS 1466	-10.4035
Pozzotech 20+*Temperature	-0.691174
PS 1466*Temperature	-3.71285

Table B7: Response Surface Regression Analysis (Minitab Output) for Mix 8: CST Day 3

Response Surface Regression: CST DAY 3 versus Block, IPANEX, Pozzotech 20, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY 3

Term	Coef	SE Coef	T	P
Constant	2678.17	84.74	31.605	0.000
Block 1	24.95	56.20	0.444	0.664
Block 2	87.17	55.64	1.567	0.141
IPANEX	-45.84	83.69	-0.548	0.593
Pozzotech 20+	-83.96	83.69	-1.003	0.334
PS 1466	-185.74	87.57	-2.121	0.054
Temperature	1986.24	48.11	41.284	0.000
IPANEX*IPANEX	53.99	155.14	0.348	0.733
Pozzotech 20+*Pozzotech 20+	182.99	155.14	1.180	0.259
PS 1466*PS 1466	355.23	154.80	2.295	0.039
Temperature*Temperature	-499.30	102.29	-4.881	0.000
IPANEX*Pozzotech 20+	-341.83	204.41	-1.672	0.118
IPANEX*PS 1466	-215.70	215.56	-1.001	0.335
IPANEX*Temperature	74.01	101.39	0.730	0.478
Pozzotech 20+*PS 1466	218.95	215.56	1.016	0.328
Pozzotech 20+*Temperature	-152.06	101.39	-1.500	0.158
PS 1466*Temperature	-251.88	107.68	-2.339	0.036

S = 201.779   PRESS = 2529026
R-Sq = 99.28% R-Sq(pred) = 96.57% R-Sq(adj) = 98.40%

Estimated Regression Coefficients for CST DAY 3 using data in uncoded units

Term	Coef
Constant	-22613.0
Block 1	24.9522
Block 2	87.1688
IPANEX	3560.72
Pozzotech 20+	559.596
PS 1466	-67.6947
Temperature	169.410
IPANEX*IPANEX	337.456
Pozzotech 20+*Pozzotech 20+	11.4371
PS 1466*PS 1466	355.227
Temperature*Temperature	-0.710996
IPANEX*Pozzotech 20+	-213.646
IPANEX*PS 1466	-539.246
IPANEX*Temperature	6.98250
Pozzotech 20+*PS 1466	54.7371
Pozzotech 20+*Temperature	-1.43452
PS 1466*Temperature	-9.50481

Table B8: Response Surface Regression Analysis (Minitab Output) for Mix 8: CST Day 4

Response Surface Regression: CST DAY 4 versus Block, IPANEX, Pozzotech 20, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY 4

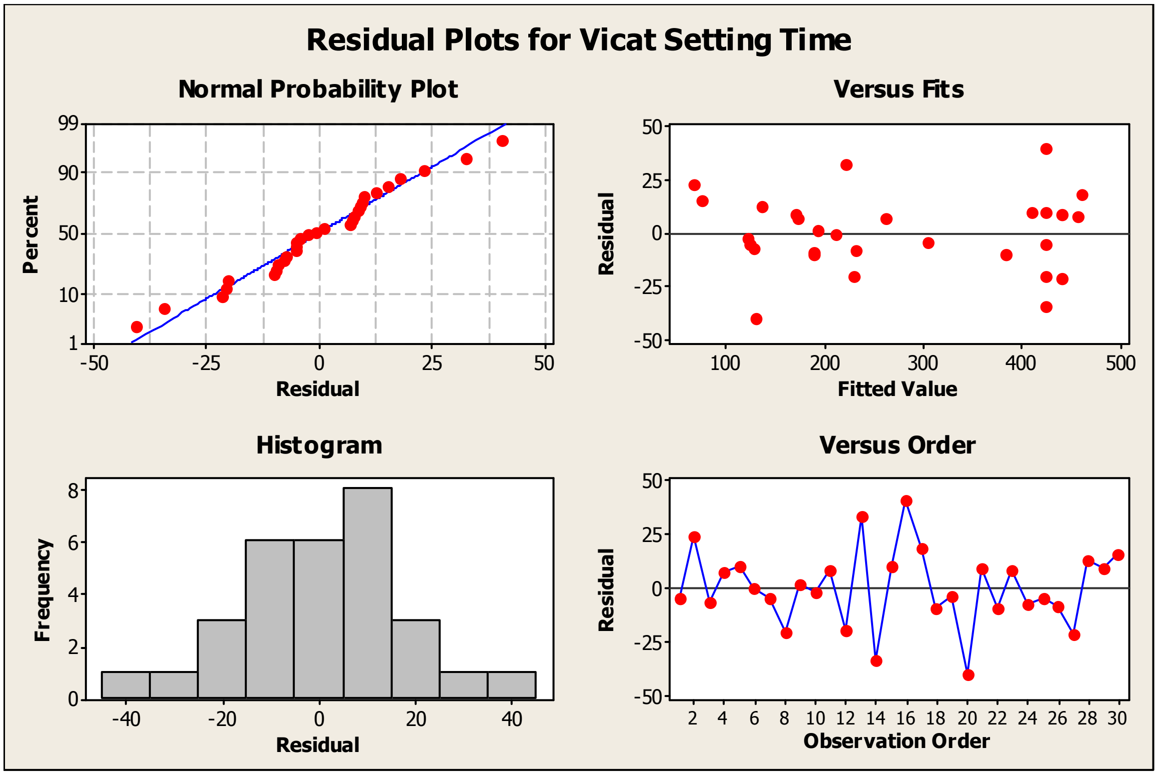
Term	Coef	SE Coef	T	P
Constant	3070.55	80.47	38.160	0.000
Block 1	-13.15	53.37	-0.246	0.809
Block 2	40.66	52.83	0.770	0.455
IPANEX	28.87	79.47	0.363	0.722
Pozzotech 20+	192.65	79.47	2.424	0.031
PS 1466	35.97	83.16	0.433	0.672
Temperature	2237.47	45.69	48.975	0.000
IPANEX*IPANEX	15.93	147.31	0.108	0.916
Pozzotech 20+*Pozzotech 20+	83.18	147.31	0.565	0.582
PS 1466*PS 1466	227.44	147.00	1.547	0.146
Temperature*Temperature	-535.21	97.13	-5.510	0.000
IPANEX*Pozzotech 20+	-254.62	194.11	-1.312	0.212
IPANEX*PS 1466	-251.60	204.69	-1.229	0.241
IPANEX*Temperature	-26.52	96.28	-0.275	0.787
Pozzotech 20+*PS 1466	-150.15	204.69	-0.734	0.476
Pozzotech 20+*Temperature	232.70	96.28	2.417	0.031
PS 1466*Temperature	48.01	102.26	0.469	0.647

S = 191.606   PRESS = 3803153
R-Sq = 99.48% R-Sq(pred) = 95.82% R-Sq(adj) = 98.83%

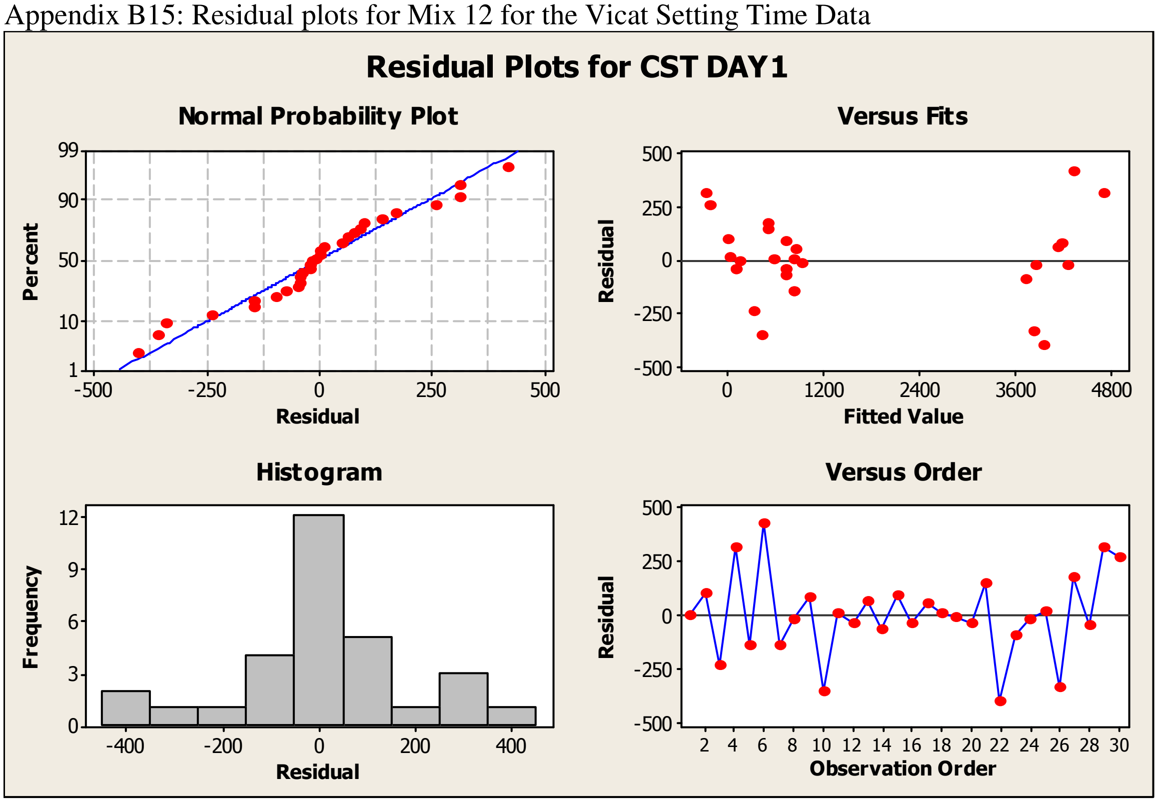
Estimated Regression Coefficients for CST DAY 4 using data in uncoded units

Term	Coef
Constant	-39837.5
Block 1	-13.1455
Block 2	40.6556
IPANEX	5853.61
Pozzotech 20+	776.284
PS 1466	3939.68
Temperature	96.1382
IPANEX*IPANEX	99.5723
Pozzotech 20+*Pozzotech 20+	5.19885
PS 1466*PS 1466	227.445
Temperature*Temperature	-0.762131
IPANEX*Pozzotech 20+	-159.139
IPANEX*PS 1466	-629.004
IPANEX*Temperature	-2.50167
Pozzotech 20+*PS 1466	-37.5371
Pozzotech 20+*Temperature	2.19529
PS 1466*Temperature	1.81157

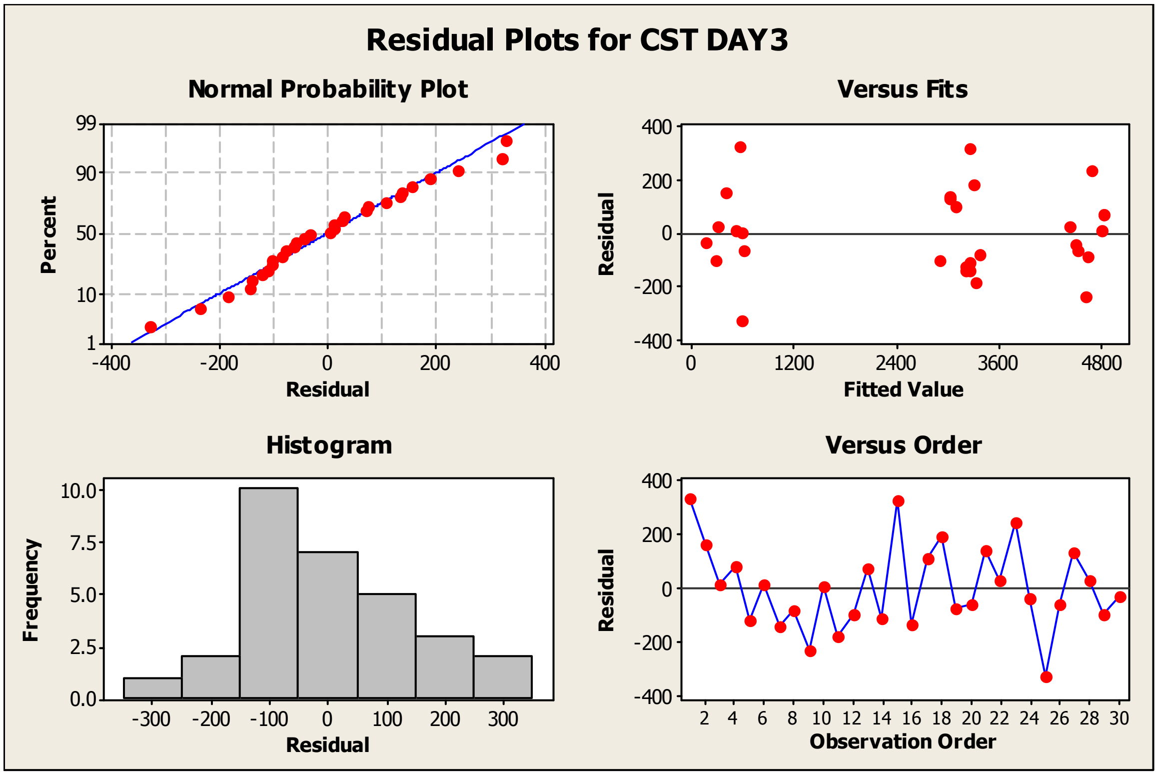
Analysis of the Mix 12 Data in table 5 is presented below.
Response Surface Regression: Vicat Setting Time



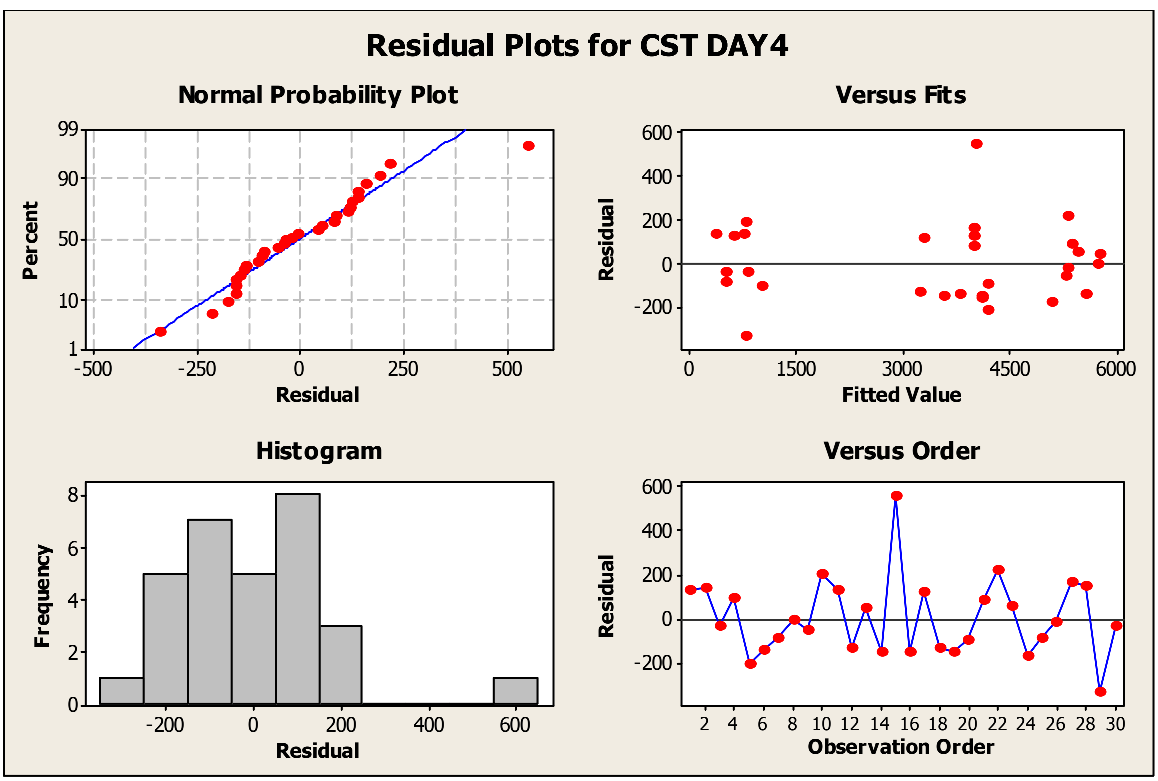
Appendix B15: Residual plots for Mix 12 for the Vicat Setting Time Data



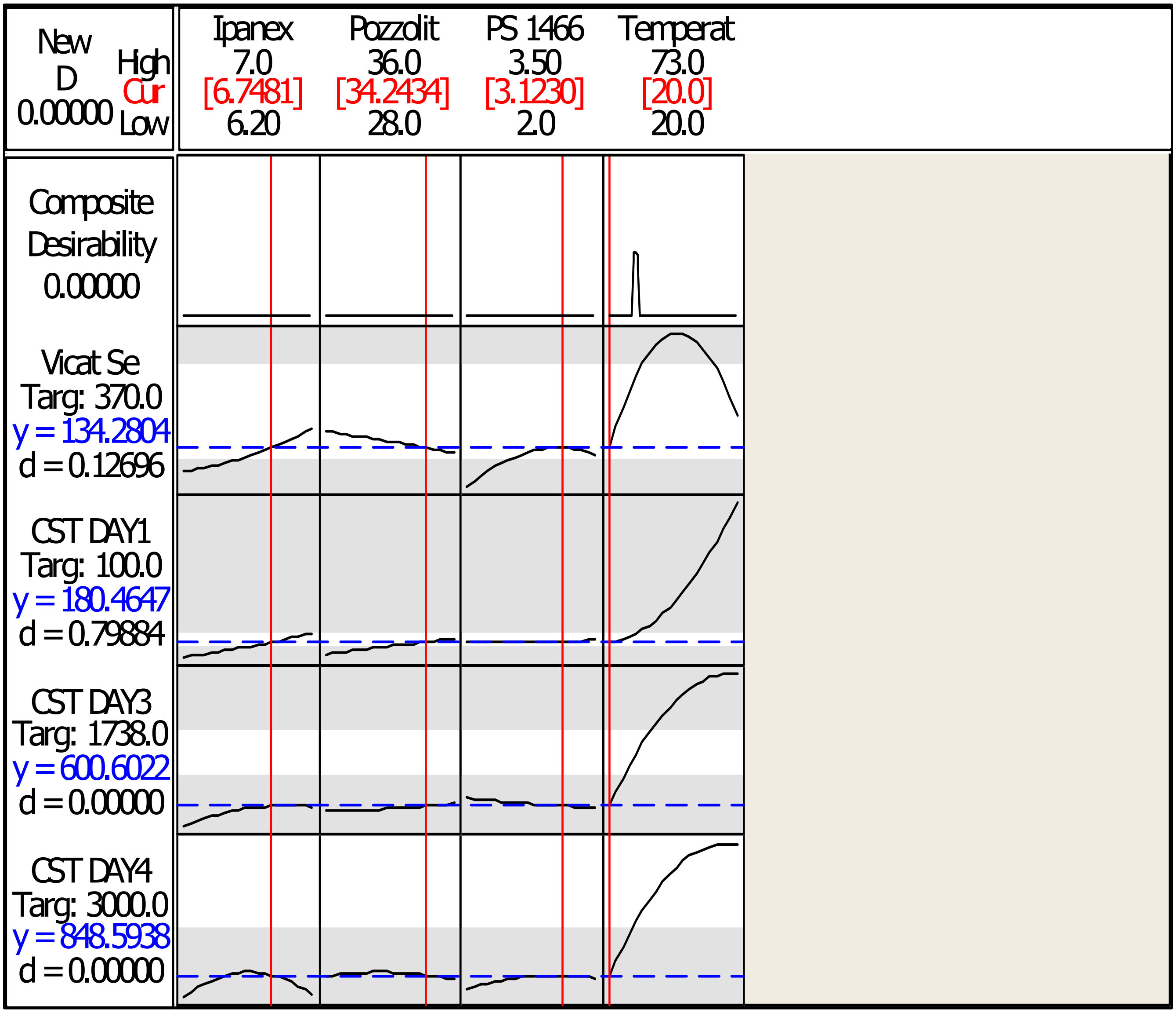
Appendix B16: Residual plots for Mix 12 for the CST Day 1 Data



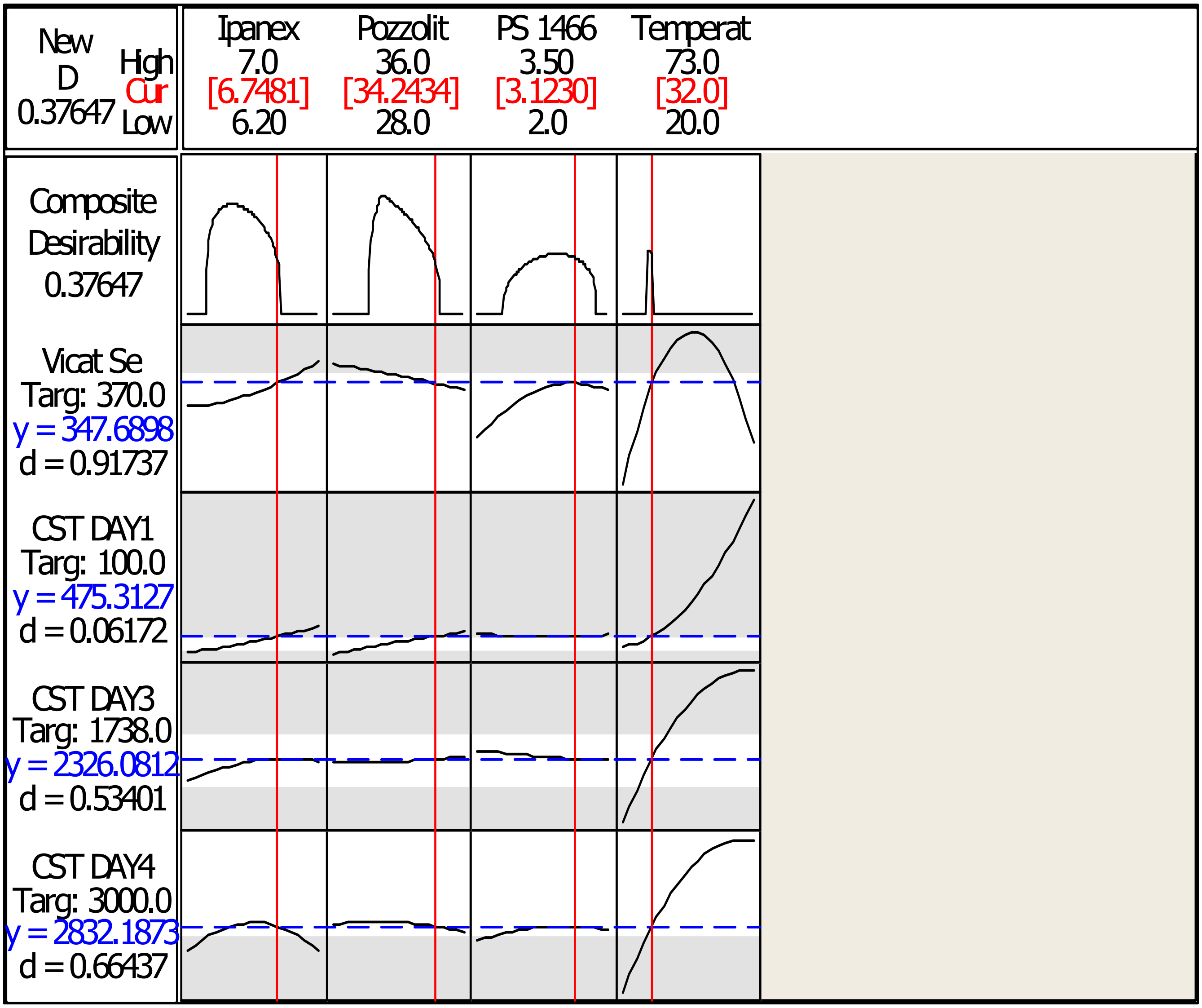
Appendix B17: Residual plots for Mix 12 for the CST Day 3 Data



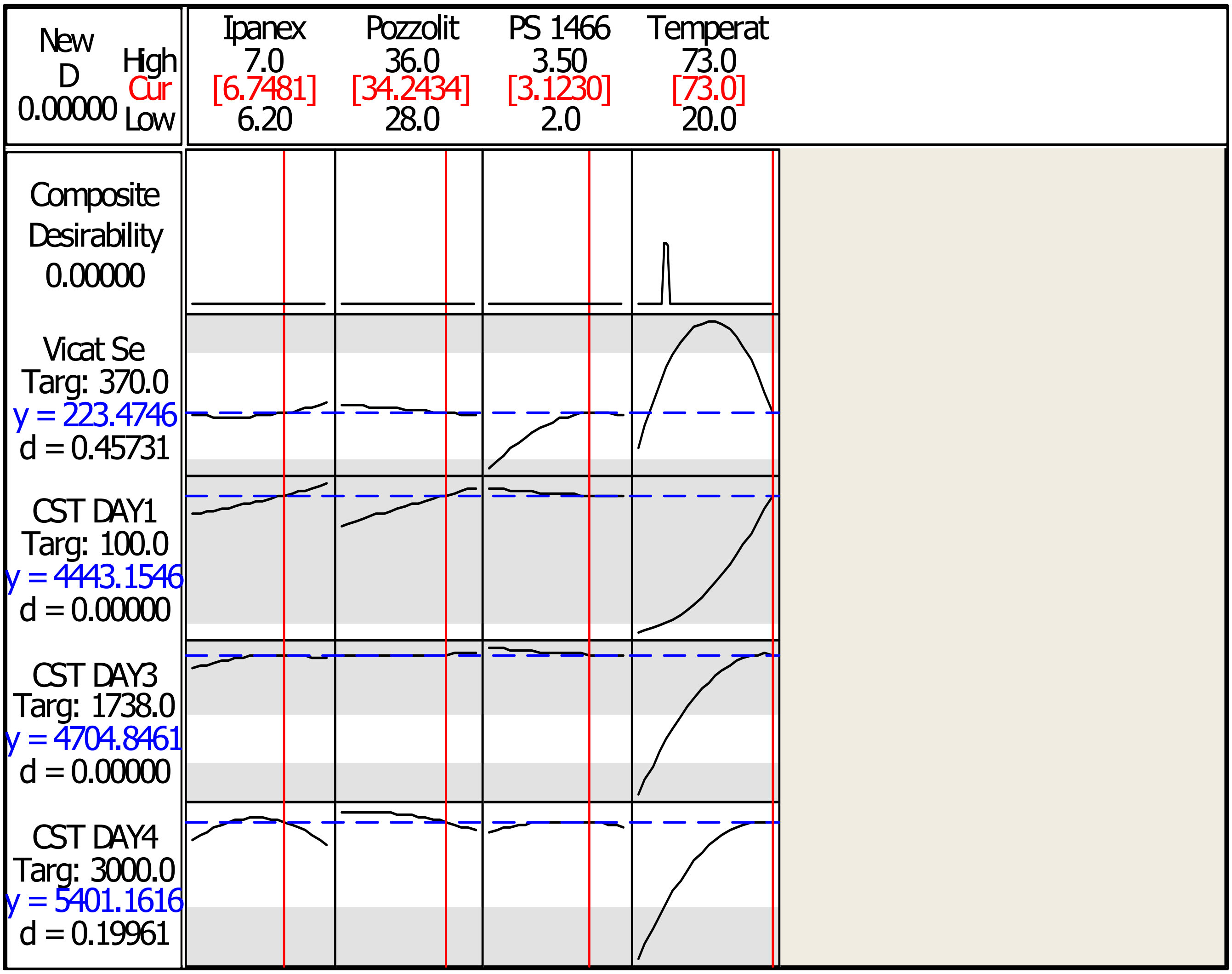
Appendix B18: Residual plots for Mix 12 for the CST Day 4 Data



Appendix B19: Optimization Graph for the Mix 12 at 20F



Appendix B20: Optimization Graph for the Mix 12 at 32F



Appendix B21: Optimization Graph for the Mix 12 at 73F

Table B9: Response Surface Regression Analysis (Minitab Output) for Mix 12: Vicat Setting Time

Response Surface Regression: Vicat Settin versus Block, IPANEX, Pozzolith 12, .

The analysis was done using coded units.

Estimated Regression Coefficients for Vicat Setting Time

Term	Coef	SE Coef	T	P
Constant	445.705	10.992	40.547	0.000
Block 1	-5.297	7.272	-0.728	0.479
Block 2	11.203	7.272	1.541	0.147
IPANEX	7.651	12.722	0.601	0.558
Pozzolith 122HE	-30.551	12.722	-2.401	0.032
PS 1466	55.617	11.341	4.904	0.000
Temperature	42.225	7.059	5.982	0.000
IPANEX*IPANEX	19.136	20.165	0.949	0.360
Pozzolith 122HE*Pozzolith 122HE	-3.364	20.165	-0.167	0.870
PS 1466*PS 1466	-55.519	15.711	-3.534	0.004
Temperature*Temperature	-275.782	13.764	-20.036	0.000
IPANEX*Pozzolith 122HE	37.500	26.481	1.416	0.180
IPANEX*PS 1466	16.875	19.861	0.850	0.411
IPANEX*Temperature	-21.087	13.110	-1.608	0.132
Pozzolith 122HE*PS 1466	-11.250	19.861	-0.566	0.581
Pozzolith 122HE*Temperature	8.555	13.110	0.653	0.525
PS 1466*Temperature	10.826	9.893	1.094	0.294

S = 26.4807   PRESS = 42642.8
R-Sq = 98.31% R-Sq(pred) = 92.11% R-Sq(adj) = 96.24%

Estimated Regression Coefficients for Vicat Setting Time using data in uncoded units

Term	Coef
Constant	8904.95
Block 1	-5.29732
Block 2	11.2027
IPANEX	-2371.78
Pozzolith 122HE	-142.310
PS 1466	340.427
Temperature	47.1645
IPANEX*IPANEX	119.600
Pozzolith 122HE*Pozzolith 122HE	-0.210248
PS 1466*PS 1466	-98.7000
Temperature*Temperature	-0.392712
IPANEX*Pozzolith 122HE	23.4375
IPANEX*PS 1466	56.2500
IPANEX*Temperature	-1.98930
Pozzolith 122HE*PS 1466	-3.75000
Pozzolith 122HE*Temperature	0.0807061
PS 1466*Temperature	0.544694

Table B10: Response Surface Regression Analysis (Minitab Output) for Mix 12: CST Day 1

Response Surface Regression: CST DAY1 versus Block, IPANEX, Pozzolith 12, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY1

Term	Coef	SE Coef	T	P
Constant	1145.02	117.98	9.705	0.000
Block 1	136.46	78.05	1.748	0.104
Block 2	-184.84	78.05	-2.368	0.034
IPANEX	75.75	136.54	0.555	0.588
Pozzolith 122HE	118.55	136.54	0.868	0.401
PS 1466	-121.95	121.72	-1.002	0.335
Temperature	2047.27	75.77	27.021	0.000
IPANEX*IPANEX	56.75	216.43	0.262	0.797
Pozzolith 122HE*Pozzolith 122HE	-28.00	216.43	-0.129	0.899
PS 1466*PS 1466	66.91	168.63	0.397	0.698
Temperature*Temperature	956.73	147.73	6.476	0.000
IPANEX*Pozzolith 122HE	611.88	284.22	2.153	0.051
IPANEX*PS 1466	-17.34	213.16	-0.081	0.936
IPANEX*Temperature	47.01	140.70	0.334	0.744
Pozzolith 122HE*PS 1466	131.72	213.16	0.618	0.547
Pozzolith 122HE*Temperature	183.44	140.70	1.304	0.215
PS 1466*Temperature	-72.82	106.18	-0.686	0.505

S = 284.217   PRESS = 9654152
R-Sq = 98.83% R-Sq(pred) = 89.22% R-Sq(adj) = 97.38%

Estimated Regression Coefficients for CST DAY1 using data in uncoded units

Term	Coef
Constant	100355
Block 1	136.456
Block 2	-184.844
IPANEX	-16777.4
Pozzolith 122HE	-2583.57
PS 1466	-1669.90
Temperature	-124.015
IPANEX*IPANEX	354.700
Pozzolith 122HE*Pozzolith 122HE	-1.74987
PS 1466*PS 1466	118.952
Temperature*Temperature	1.36238
IPANEX*Pozzolith 122HE	382.422
IPANEX*PS 1466	-57.8125
IPANEX*Temperature	4.43469
Pozzolith 122HE*PS 1466	43.9062
Pozzolith 122HE*Temperature	1.73052
PS 1466*Temperature	-3.66401

Table B11: Response Surface Regression Analysis (Minitab Output) for Mix 12: CST Day 3

Response Surface Regression: CST DAY3 versus Block, IPANEX, Pozzolith 12, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY3

Term	Coef	SE Coef	T	P
Constant	3768.88	96.52	39.047	0.000
Block 1	45.79	63.85	0.717	0.486
Block 2	-145.61	63.85	-2.280	0.040
IPANEX	106.38	111.71	0.952	0.358
Pozzolith 122HE	-5.62	111.71	-0.050	0.961
PS 1466	-60.63	99.58	-0.609	0.553
Temperature	2097.42	61.99	33.838	0.000
IPANEX*IPANEX	-259.96	177.07	-1.468	0.166
Pozzolith 122HE*Pozzolith 122HE	48.54	177.07	0.274	0.788
PS 1466*PS 1466	28.87	137.96	0.209	0.838
Temperature*Temperature	-1136.47	120.86	-9.403	0.000
IPANEX*Pozzolith 122HE	292.63	232.52	1.258	0.230
IPANEX*PS 1466	-96.09	174.39	-0.551	0.591
IPANEX*Temperature	-81.82	115.11	-0.711	0.490
Pozzolith 122HE*PS 1466	-55.97	174.39	-0.321	0.753
Pozzolith 122HE*Temperature	-40.54	115.11	-0.352	0.730
PS 1466*Temperature	15.56	86.87	0.179	0.861

S = 232.521   PRESS = 5542037
R-Sq = 99.16% R-Sq(pred) = 93.39% R-Sq(adj) = 98.13%

Estimated Regression Coefficients for CST DAY3 using data in uncoded units

Term	Coef
Constant	-43845.6
Block 1	45.7936
Block 2	-145.606
IPANEX	17100.1
Pozzolith 122HE	-1333.55
PS 1466	2311.57
Temperature	290.685
IPANEX*IPANEX	-1624.76
Pozzolith 122HE*Pozzolith 122HE	3.03367
PS 1466*PS 1466	51.3179
Temperature*Temperature	-1.61833
IPANEX*Pozzolith 122HE	182.891
IPANEX*PS 1466	-320.312
IPANEX*Temperature	-7.71929
Pozzolith 122HE*PS 1466	-18.6562
Pozzolith 122HE*Temperature	-0.382445
PS 1466*Temperature	0.782995

Table B12: Response Surface Regression Analysis (Minitab Output) for Mix 12: CST Day 4

Response Surface Regression: CST DAY4 versus Block, IPANEX, Pozzolith 12, ...

The analysis was done using coded units.

Estimated Regression Coefficients for CST DAY4

Term	Coef	SE Coef	T	P
Constant	4707.42	107.70	43.711	0.000
Block 1	92.15	71.24	1.293	0.218
Block 2	-101.85	71.24	-1.430	0.176
IPANEX	-22.63	124.64	-0.182	0.859
Pozzolith 122HE	-52.64	124.64	-0.422	0.680
PS 1466	222.21	111.11	2.000	0.067
Temperature	2395.21	69.16	34.633	0.000
IPANEX*IPANEX	-825.57	197.57	-4.179	0.001
Pozzolith 122HE*Pozzolith 122HE	-196.32	197.57	-0.994	0.339
PS 1466*PS 1466	-248.05	153.93	-1.611	0.131
Temperature*Temperature	-1360.00	134.85	-10.085	0.000
IPANEX*Pozzolith 122HE	-80.50	259.44	-0.310	0.761
IPANEX*PS 1466	100.69	194.58	0.517	0.614
IPANEX*Temperature	-47.69	128.44	-0.371	0.716
Pozzolith 122HE*PS 1466	-217.31	194.58	-1.117	0.284
Pozzolith 122HE*Temperature	-128.41	128.44	-1.000	0.336
PS 1466*Temperature	-58.81	96.93	-0.607	0.554

S = 259.438   PRESS = 7281413
R-Sq = 99.20% R-Sq(pred) = 93.32% R-Sq(adj) = 98.21%

Estimated Regression Coefficients for CST DAY4 using data in uncoded units

Term	Coef
Constant	-258834
Block 1	92.1537
Block 2	-101.846
IPANEX	68949.0
Pozzolith 122HE	1359.71
PS 1466	2962.14
Temperature	347.088
IPANEX*IPANEX	-5159.80
Pozzolith 122HE*Pozzolith 122HE	-12.2699
PS 1466*PS 1466	-440.982
Temperature*Temperature	-1.93663
IPANEX*Pozzolith 122HE	-50.3125
IPANEX*PS 1466	335.625
IPANEX*Temperature	-4.49897
Pozzolith 122HE*PS 1466	-72.4375
Pozzolith 122HE*Temperature	-1.21143
PS 1466*Temperature	-2.95881

Appendix C

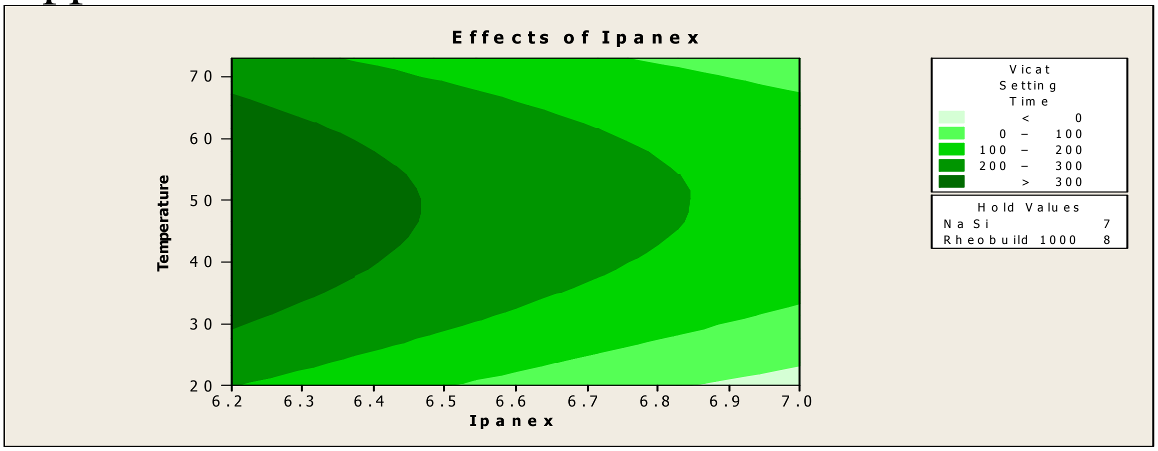


Figure C1: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

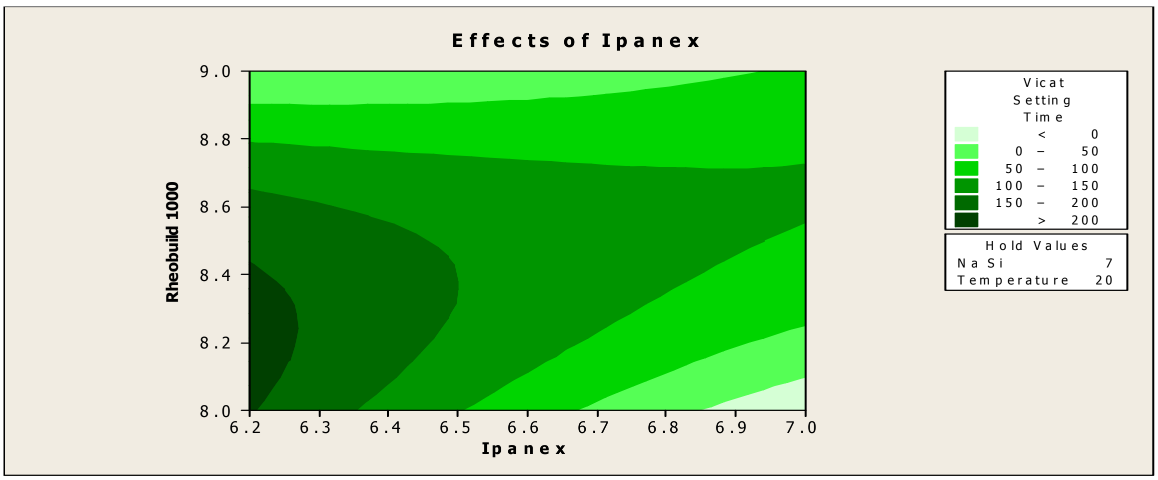


Figure C2: Contour Plot of Vicat Setting Time for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

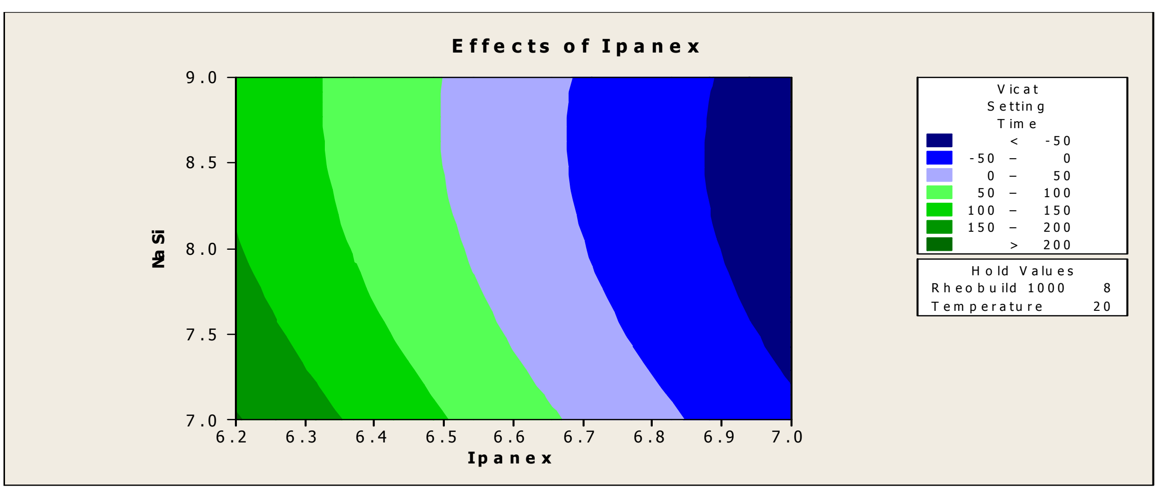


Figure C3: Contour Plot of Vicat Setting Time for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

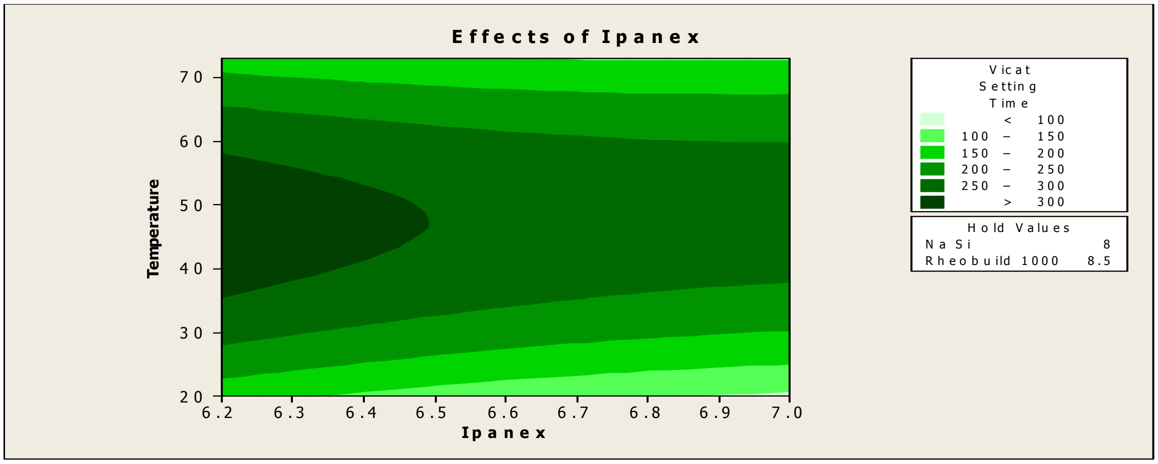


Figure C4: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

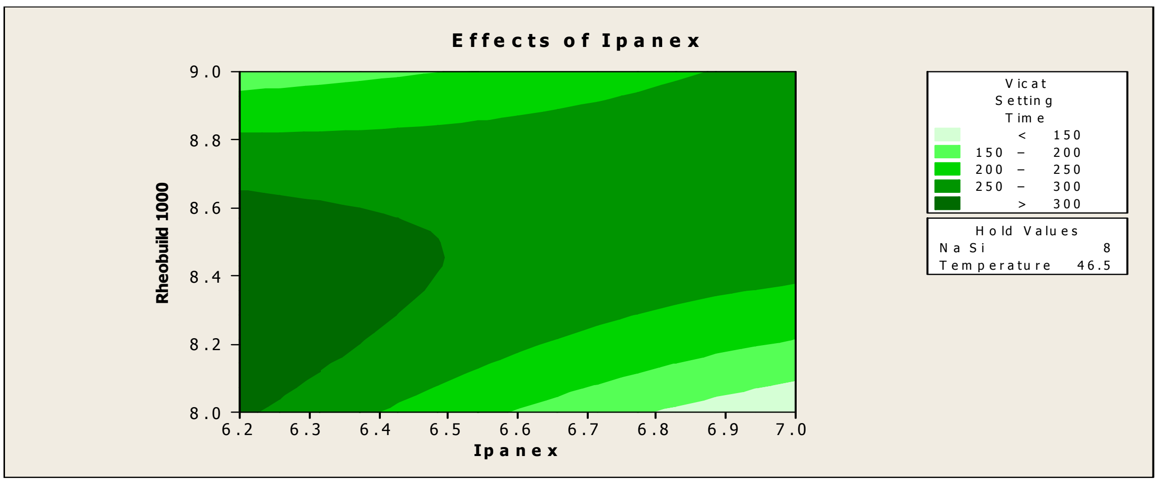


Figure C5: Contour Plot of Vicat Setting Time for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

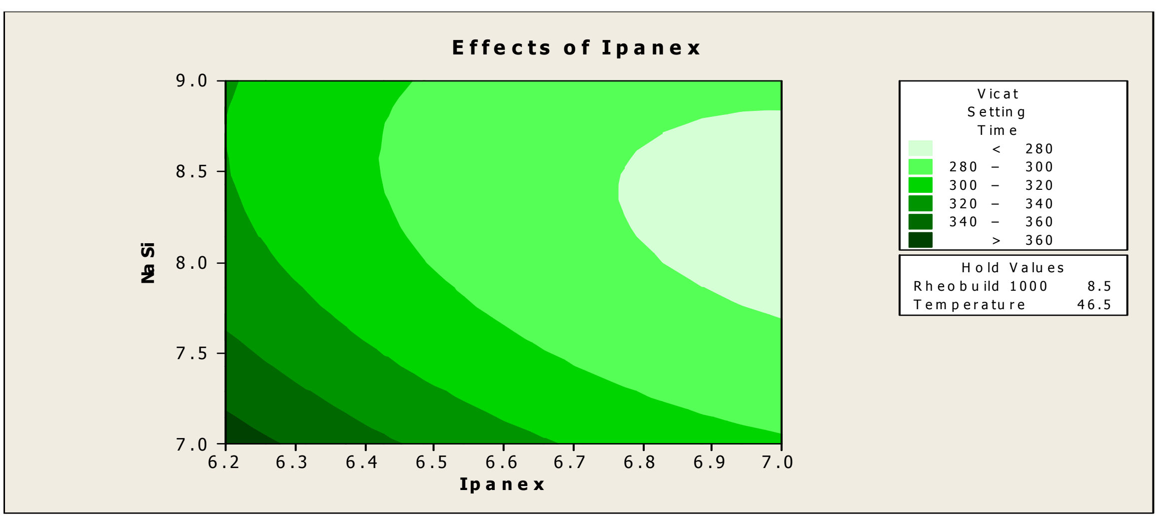


Figure C6: Contour Plot of Vicat Setting Time for Na Silicate vs Ipanex for the Mix 1 keeping all the other Factors at their medium settings.

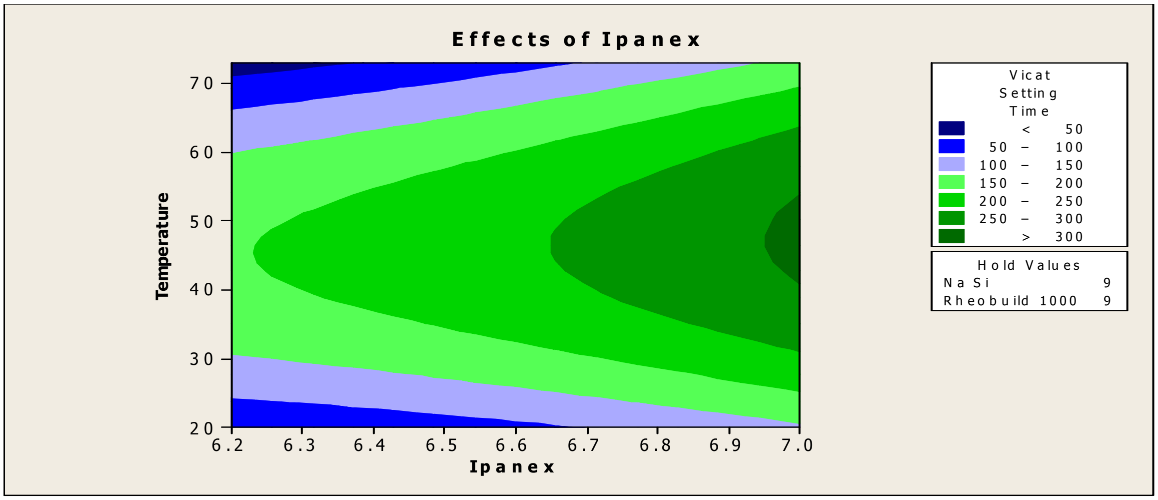


Figure C7: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

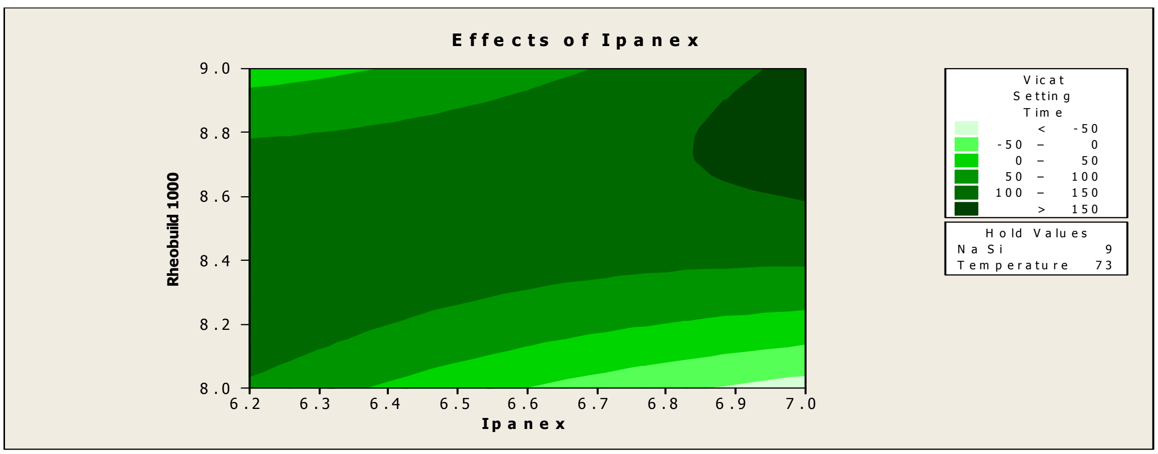


Figure C8: Contour Plot of Vicat Setting Time for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

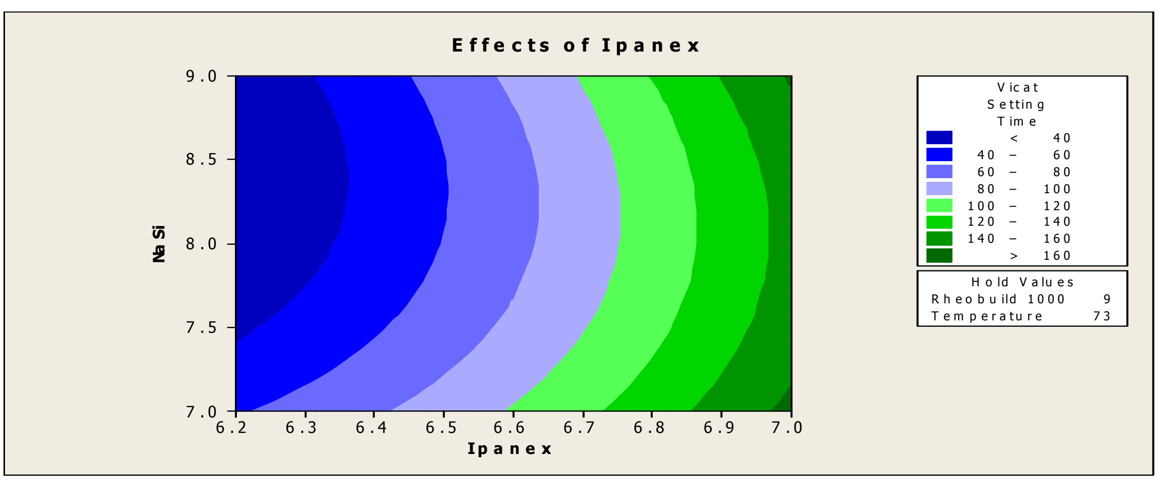


Figure C9: Contour Plot of Vicat Setting Time for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

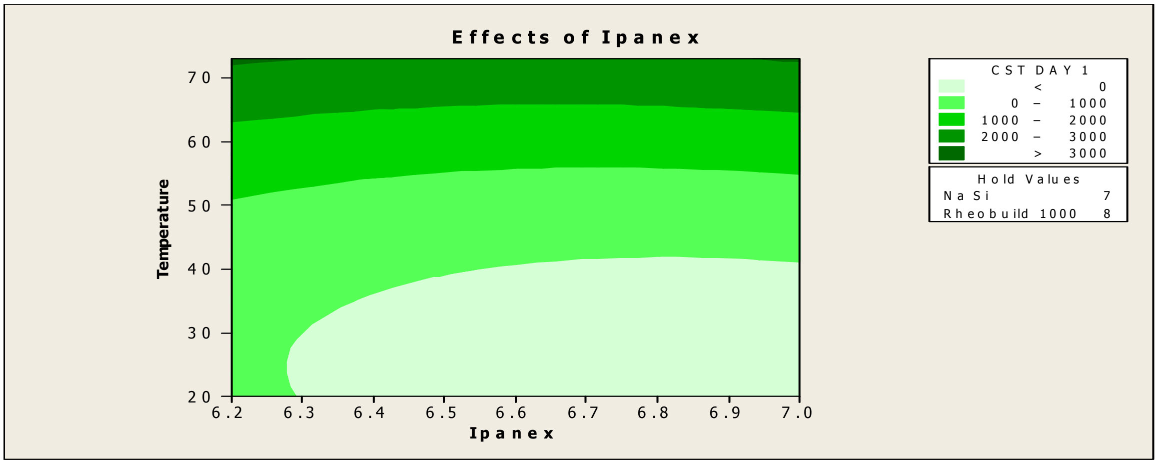


Figure C10: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

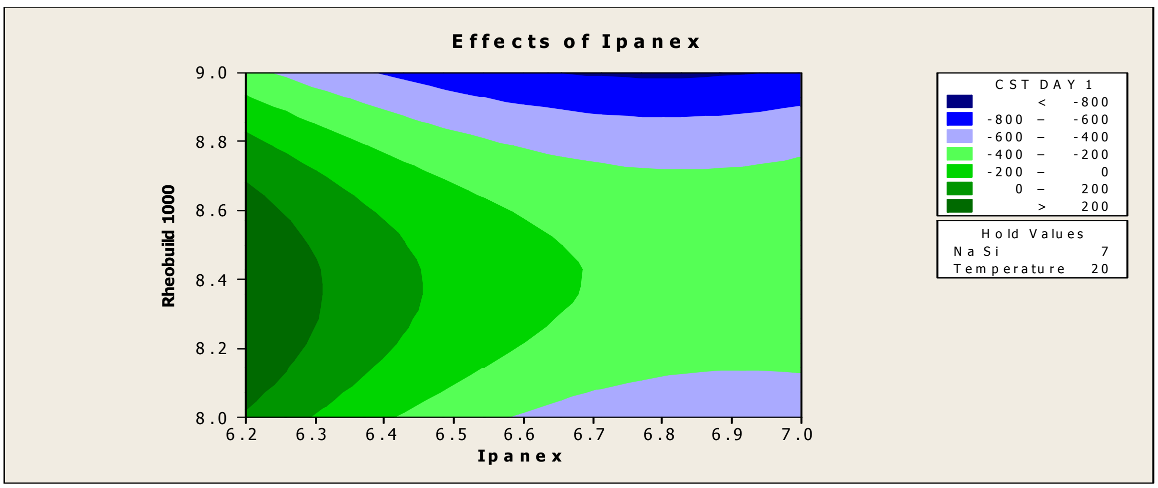


Figure C11: Contour Plot of CST Day 1 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

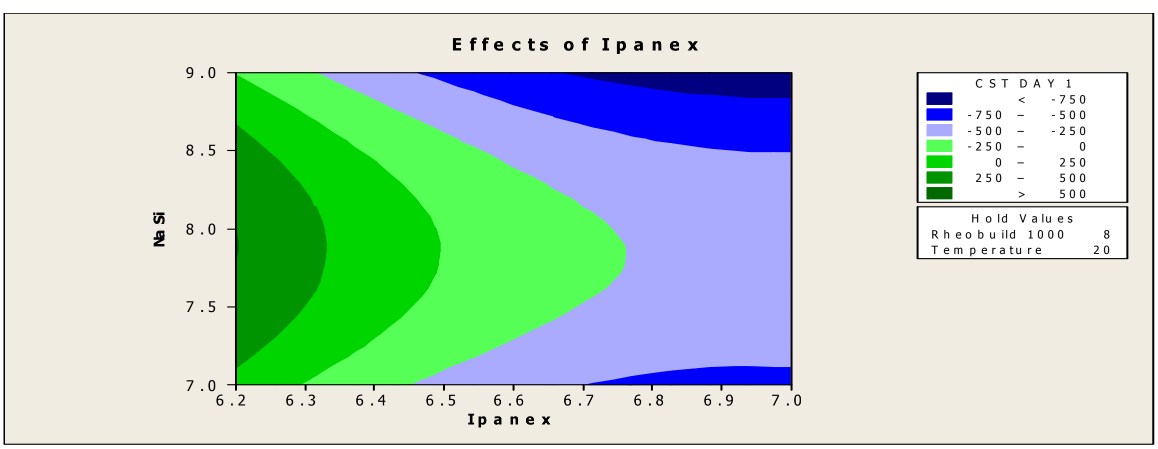


Figure C12: Contour Plot of CST Day 1 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

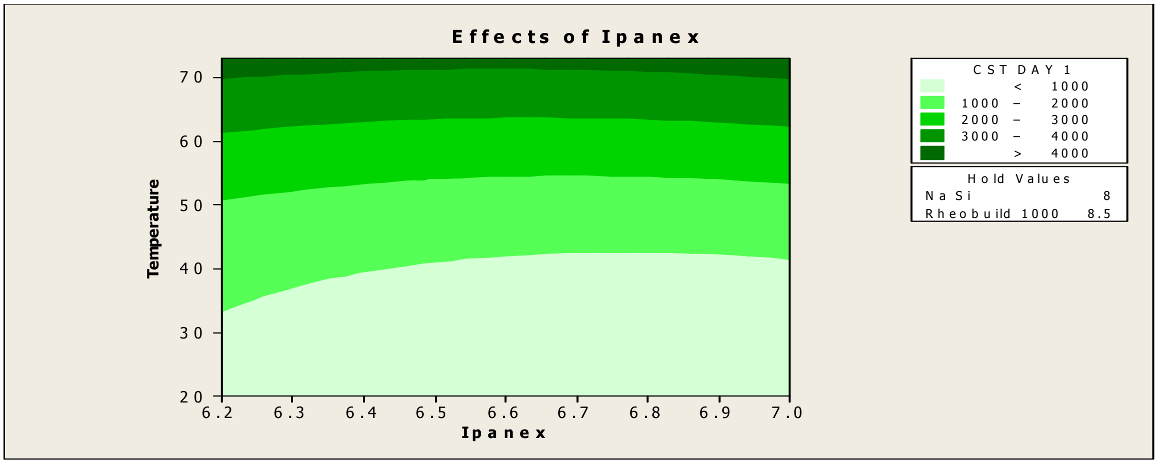


Figure C13: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

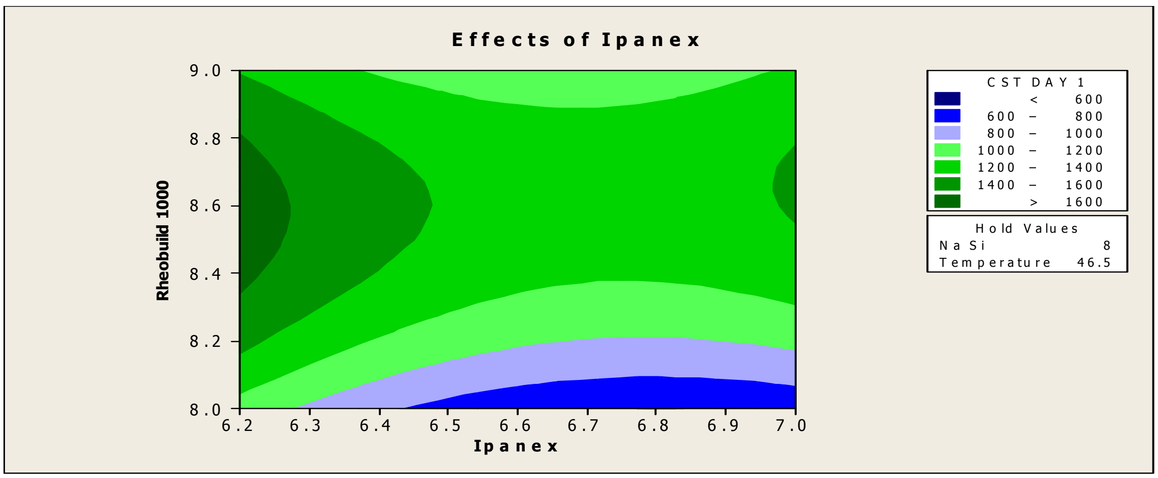


Figure C14: Contour Plot of CST Day 1 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

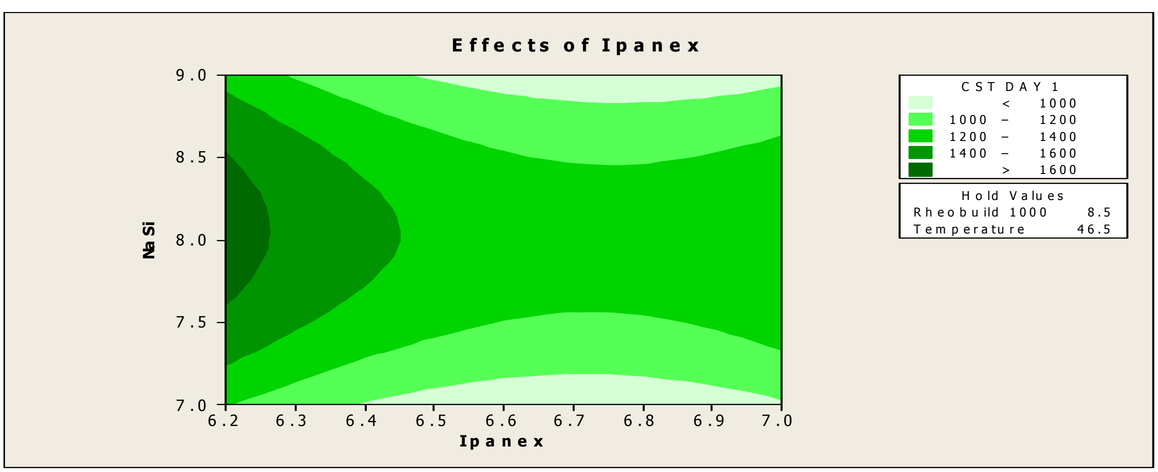


Figure C15: Contour Plot of CST Day 1 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

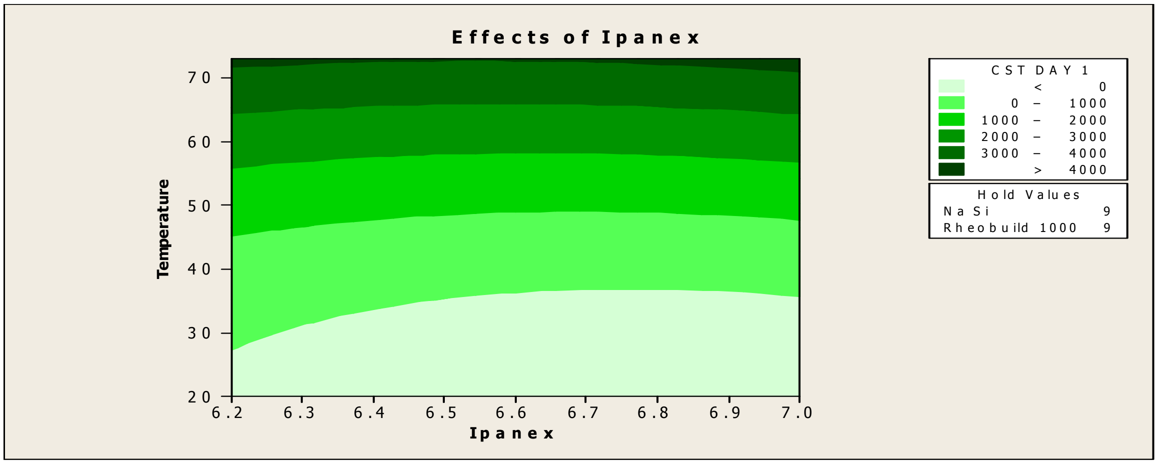


Figure C16: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

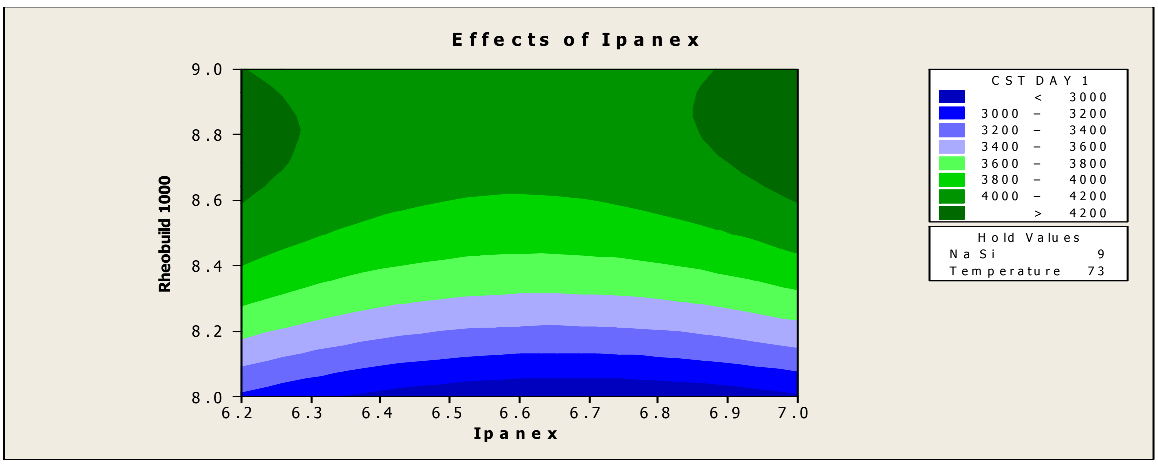


Figure C17: Contour Plot of CST Day 1 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

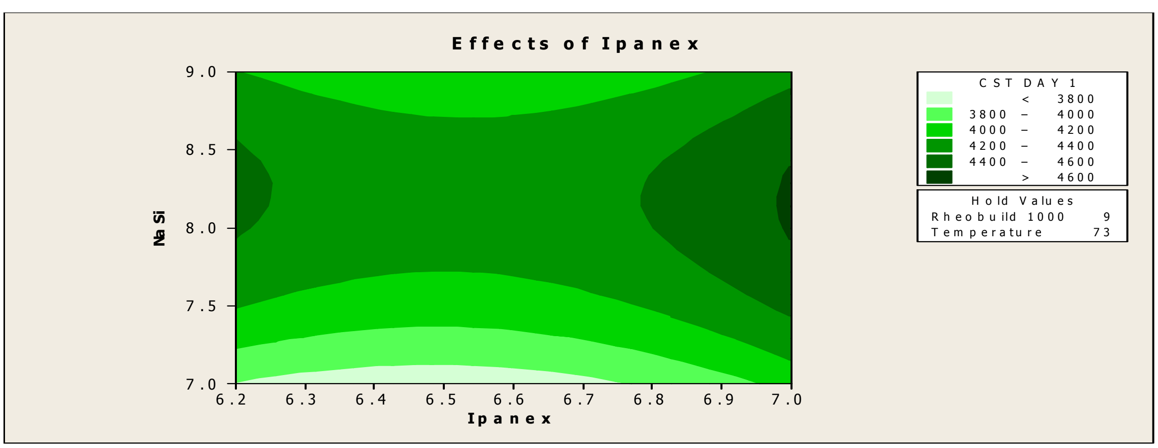


Figure C18: Contour Plot of CST Day 1 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

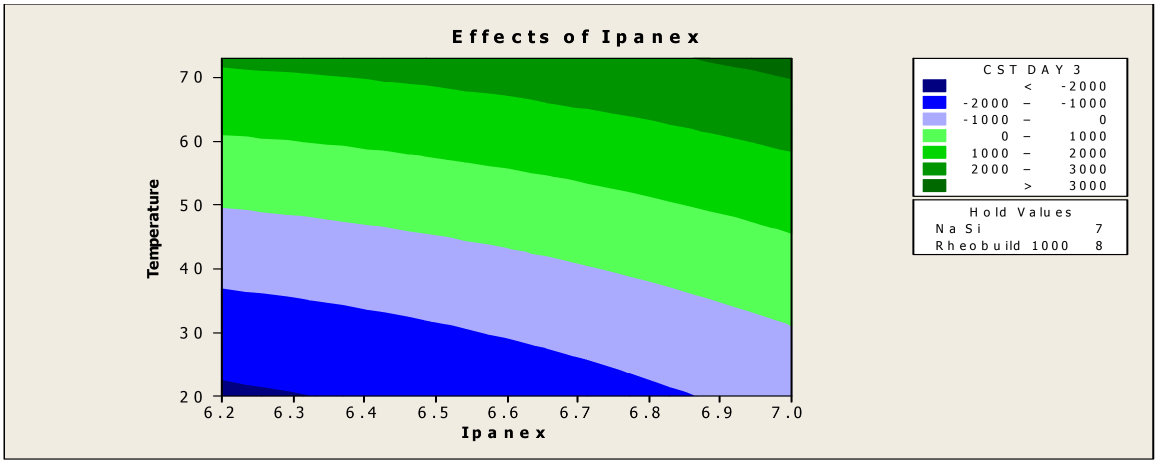


Figure C19: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

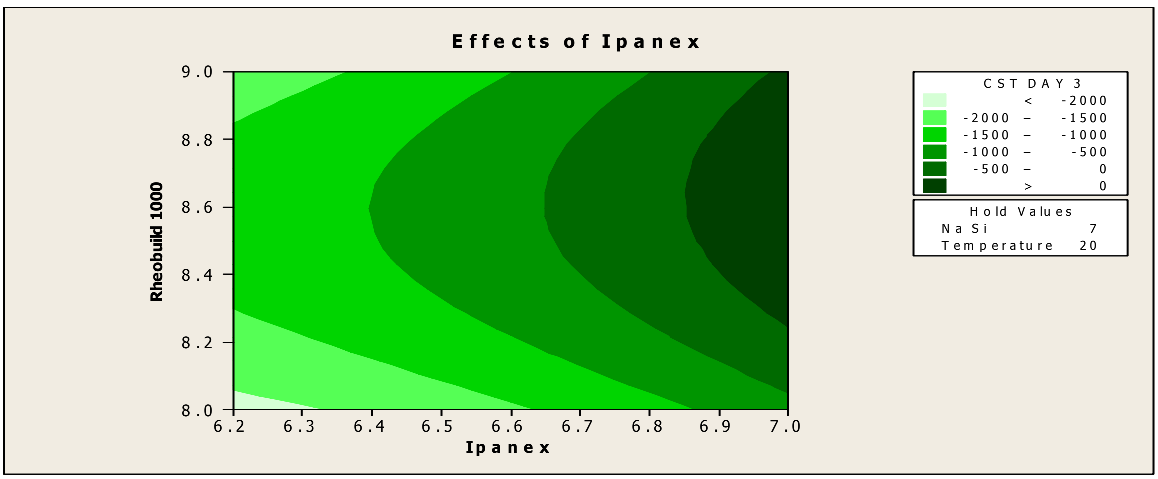


Figure C20: Contour Plot of CST Day 3 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

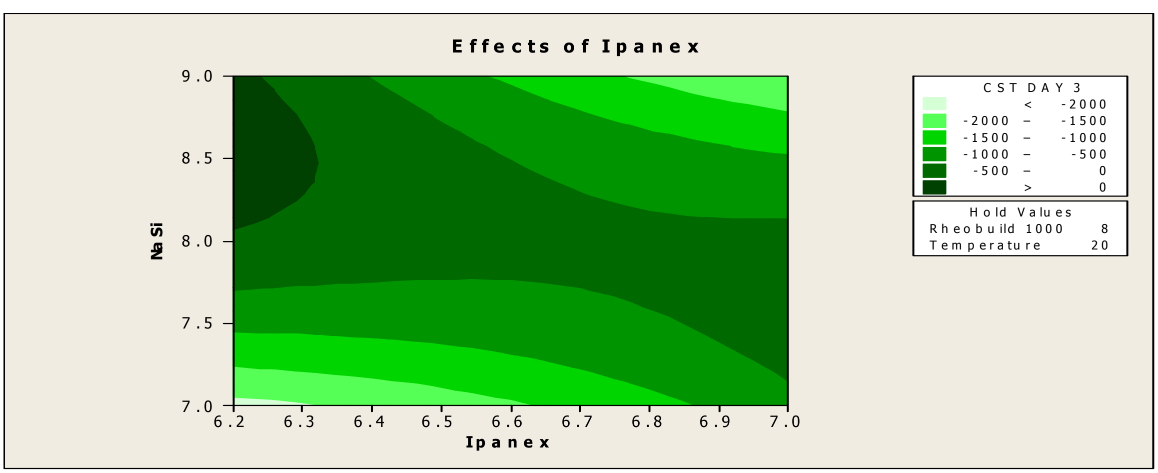


Figure C21: Contour Plot of CST Day 3 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

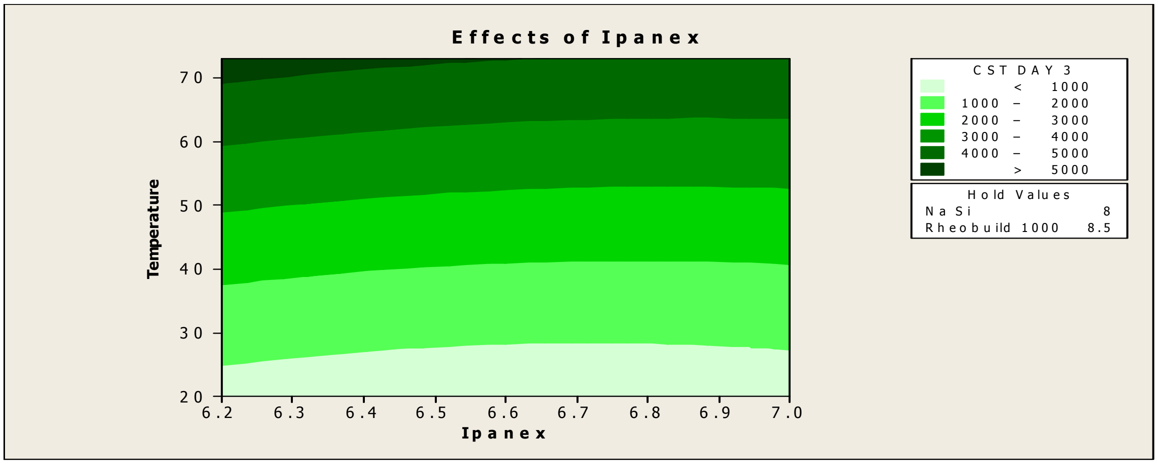


Figure C22: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

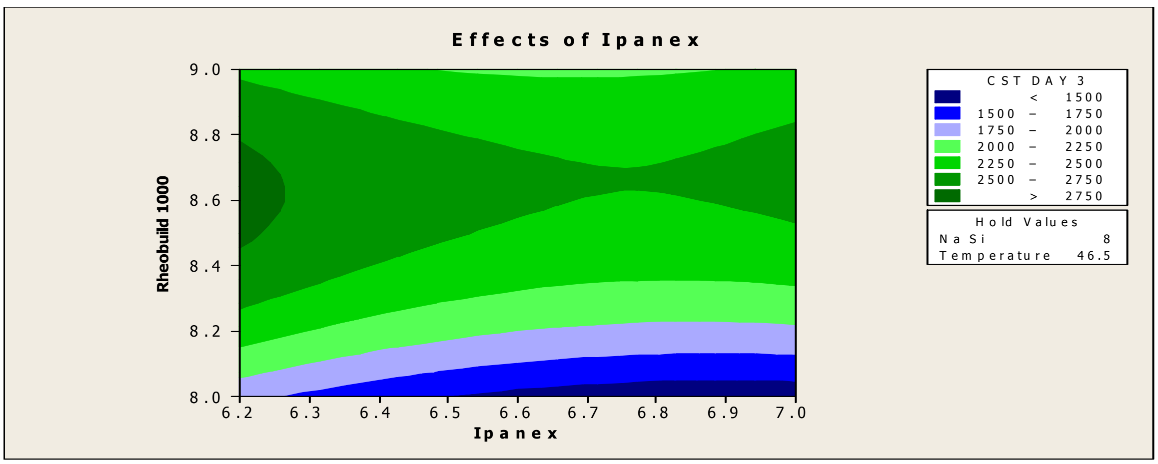


Figure C23: Contour Plot of CST Day 3 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

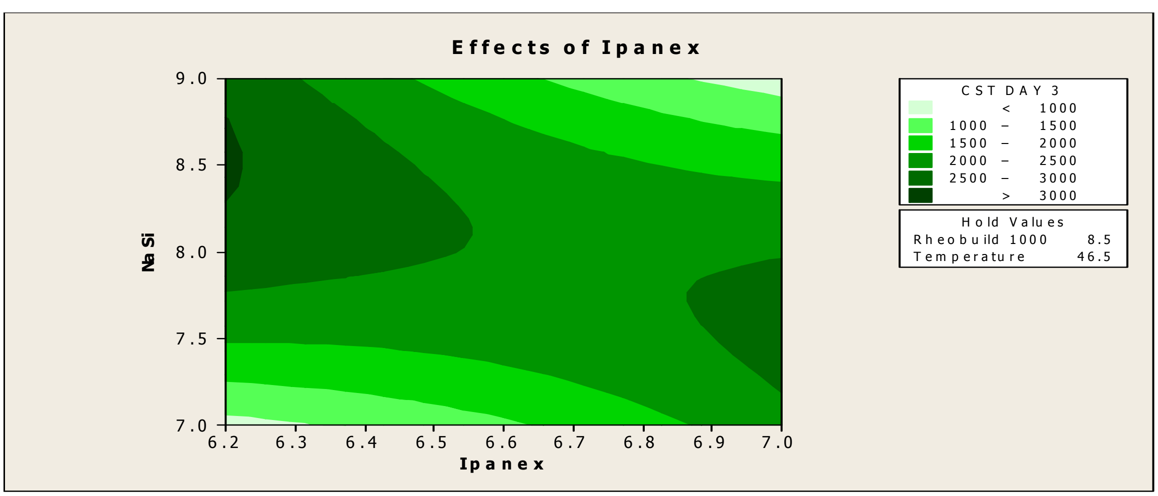


Figure C24: Contour Plot of CST Day 3 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

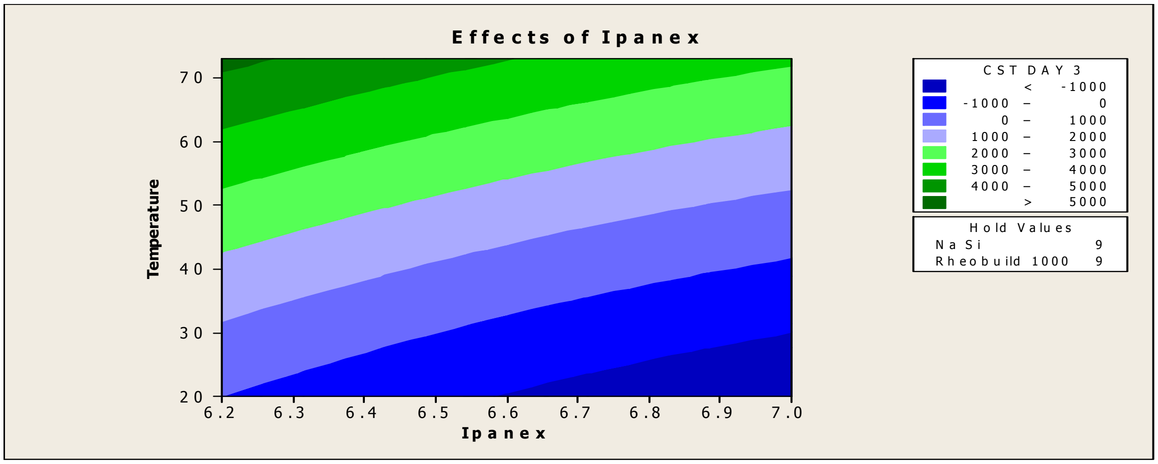


Figure C25: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

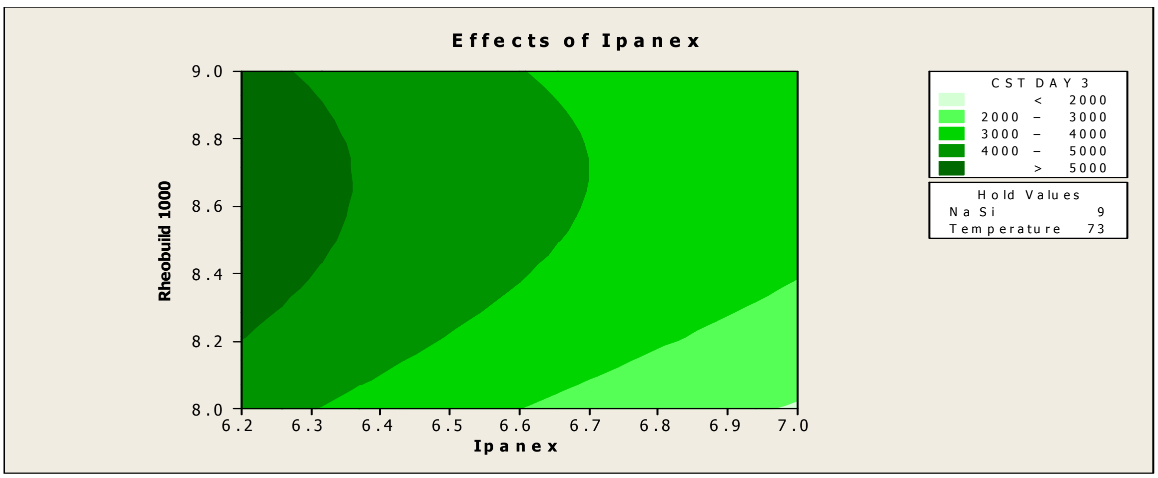


Figure C26: Contour Plot of CST Day 3 for Rheobuilde vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

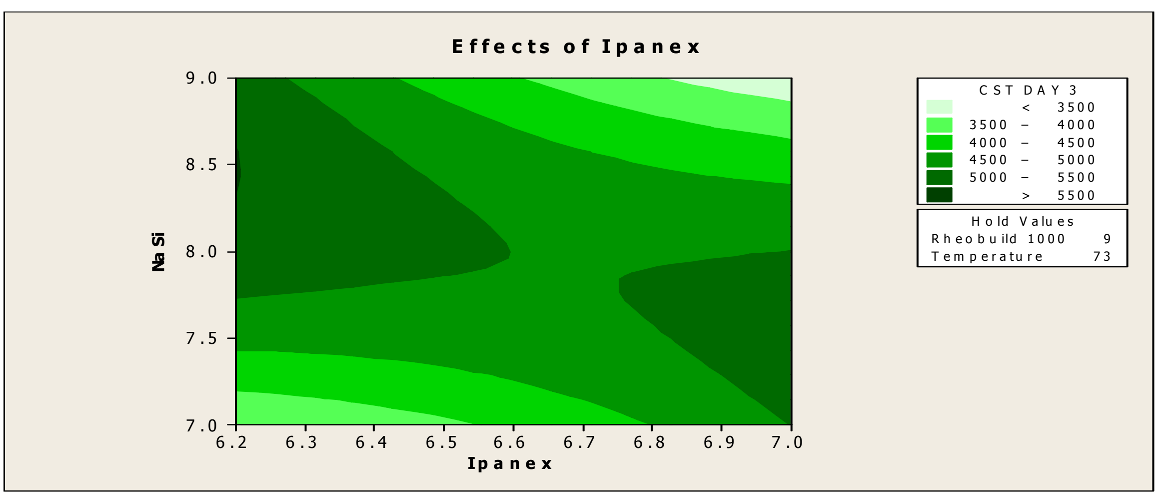


Figure C27: Contour Plot of CST Day 3 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

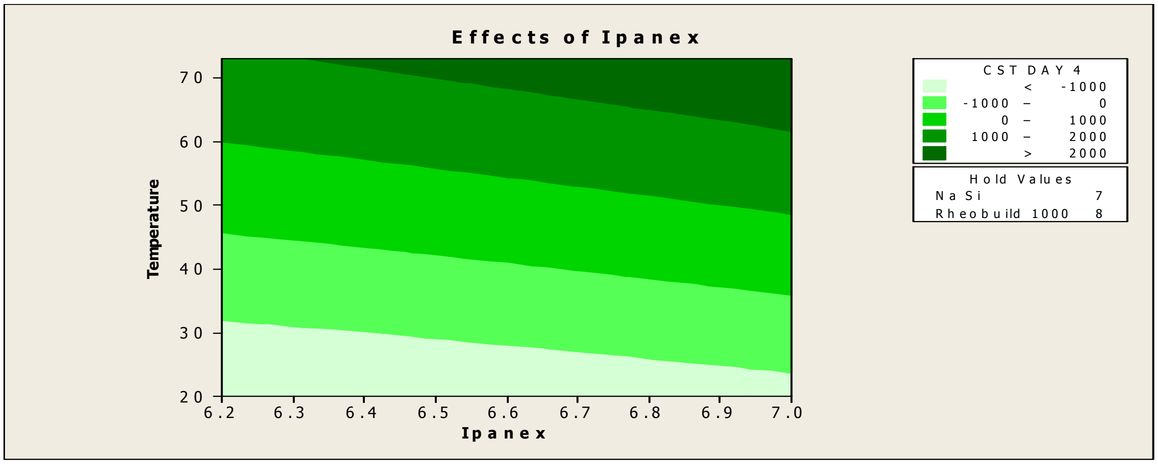


Figure C28: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

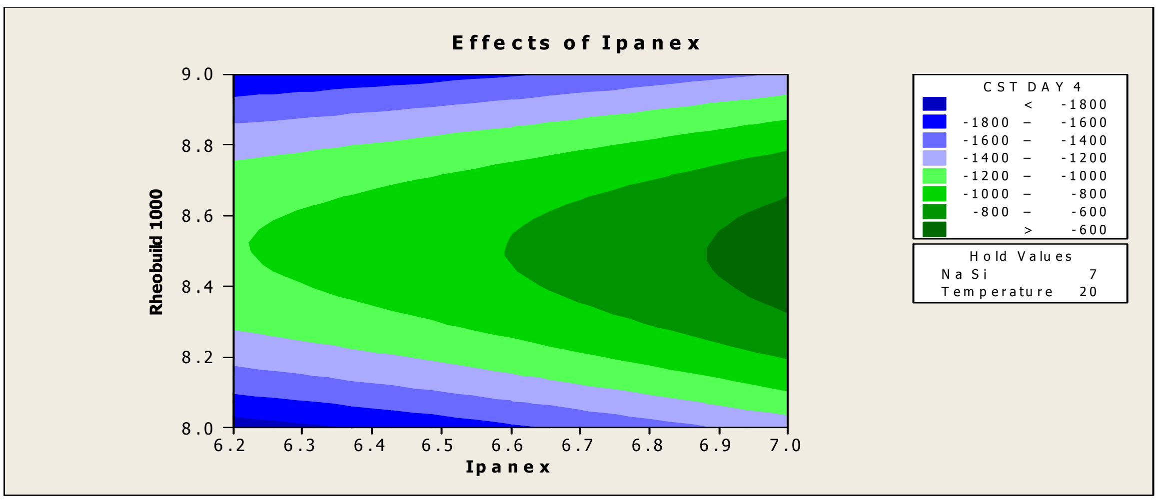


Figure C29: Contour Plot of CST Day 4 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

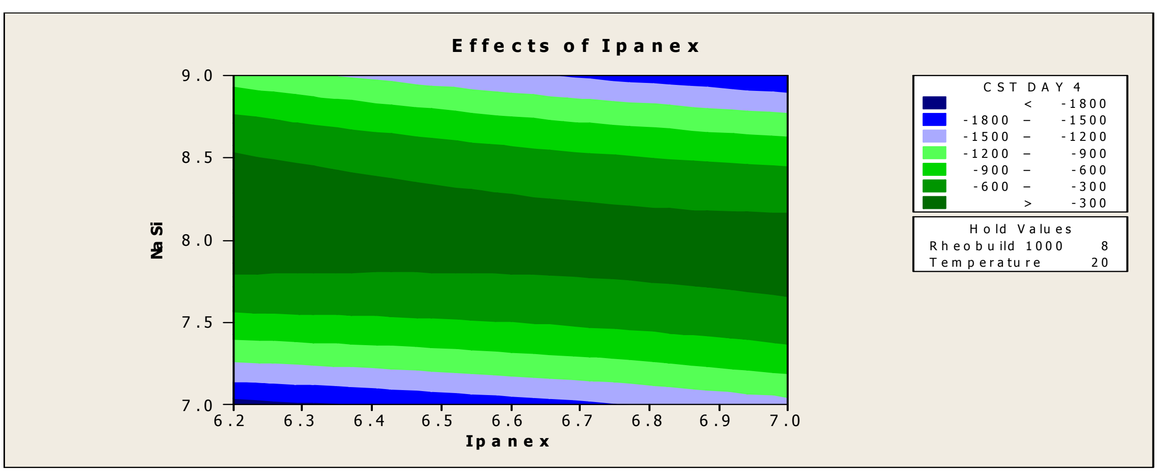


Figure C30: Contour Plot of CST Day 4 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their low settings.

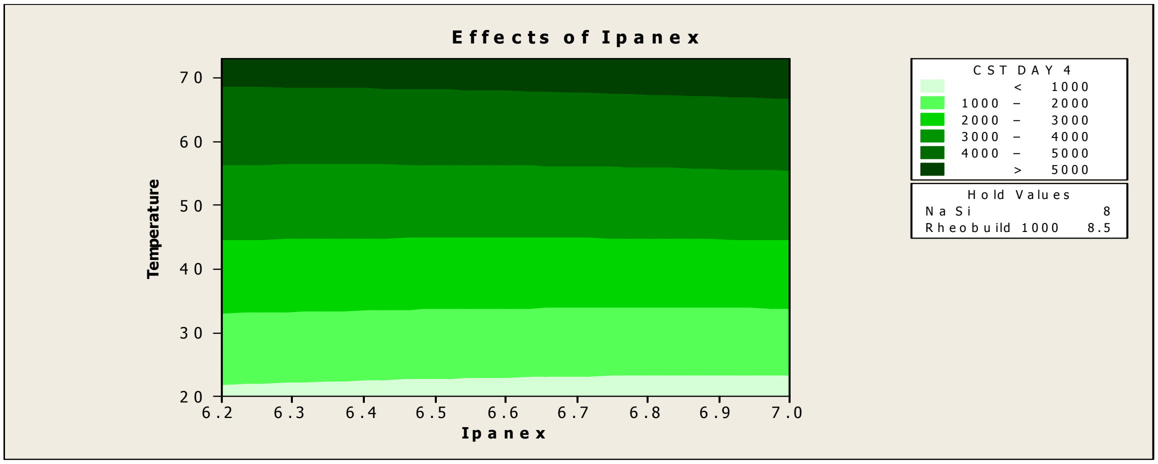


Figure C31: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

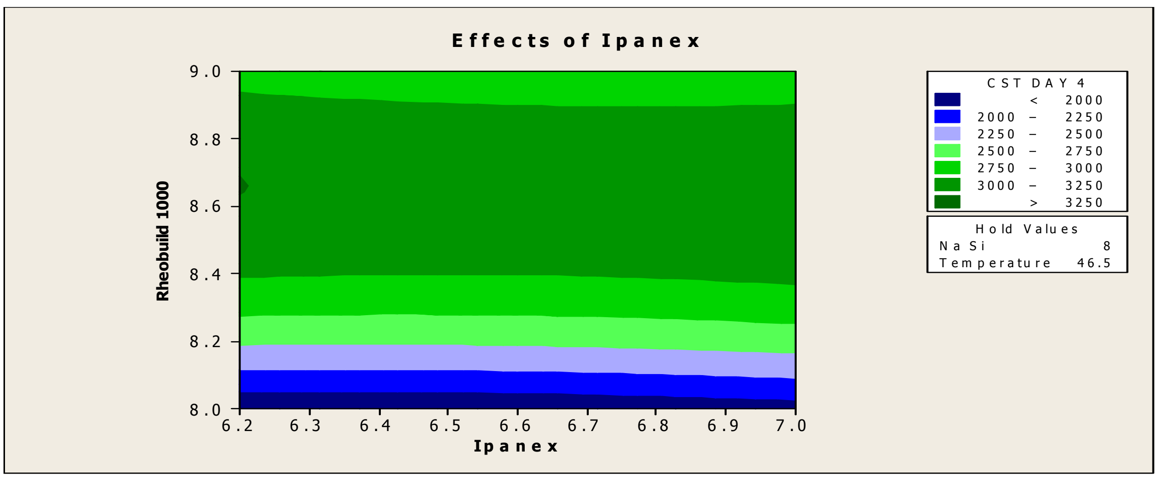


Figure C32: Contour Plot of CST Day 4 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

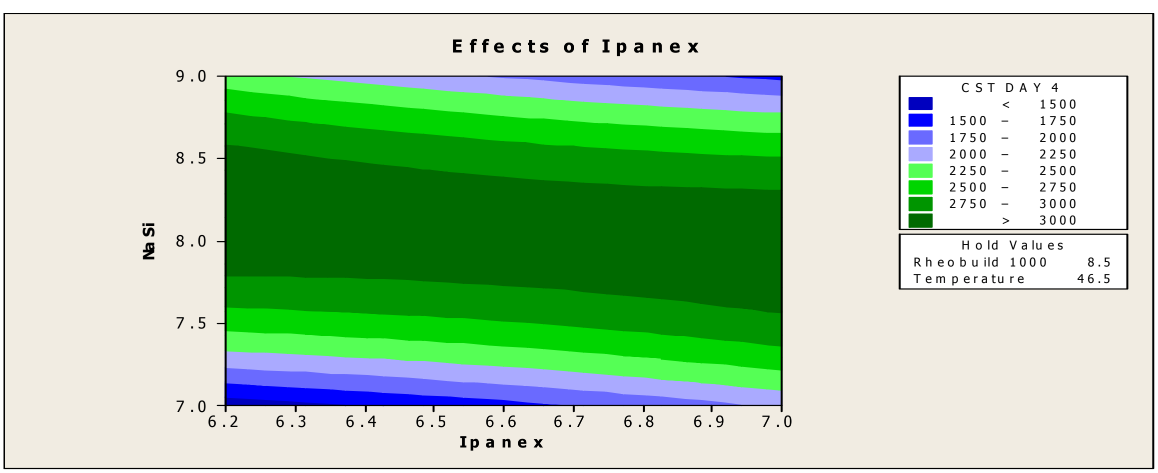


Figure C33: Contour Plot of CST Day 4 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their medium settings.

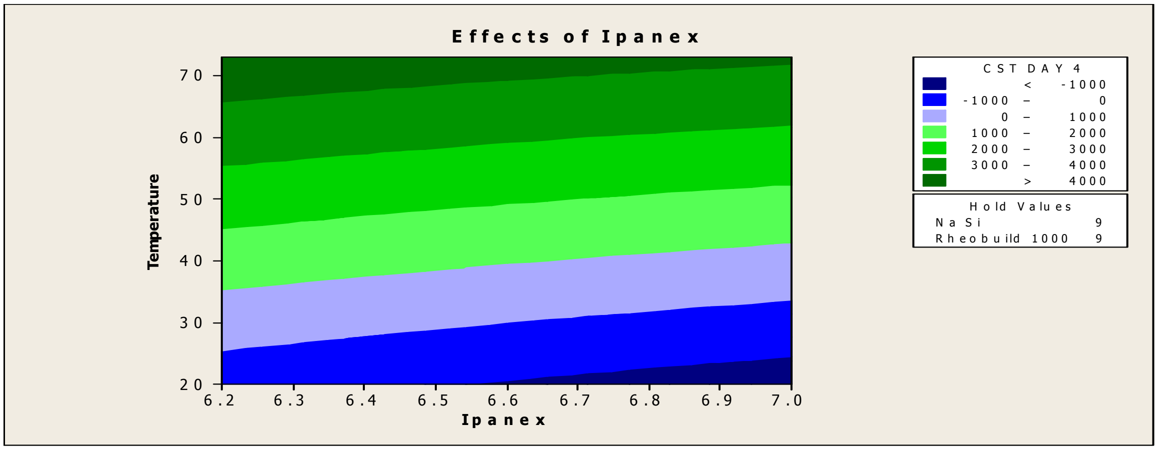


Figure C34: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

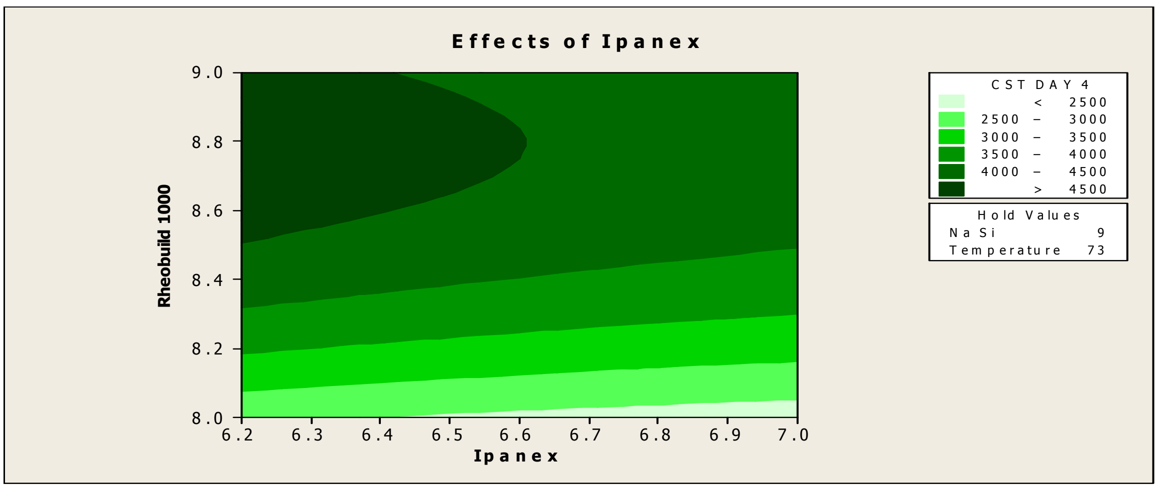


Figure C35: Contour Plot of CST Day 4 for Rheobuild 1000 vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

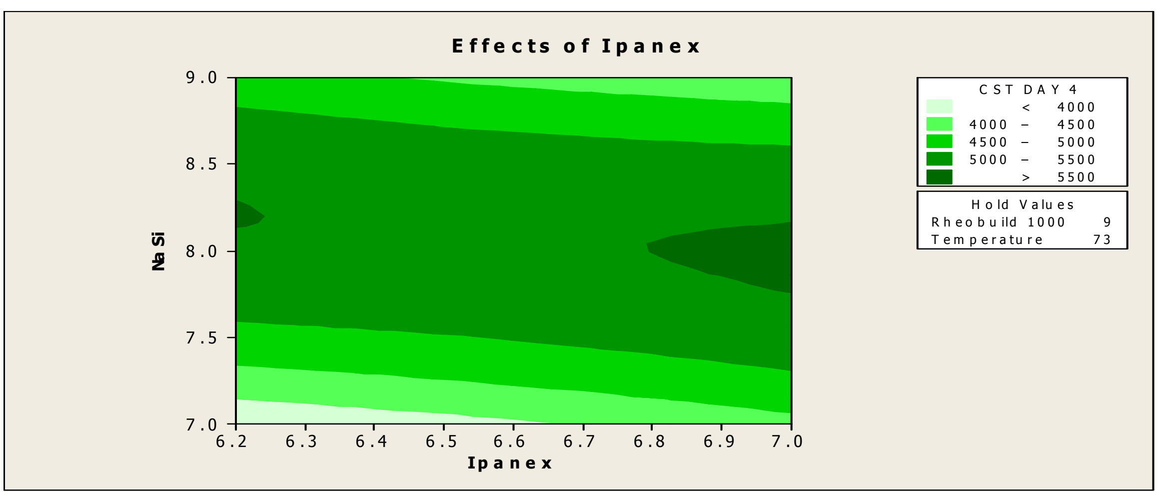


Figure C36: Contour Plot of CST Day 4 for Na Silicate vs Ipanex for the Mix 1 keeping all the other factors at their high settings.

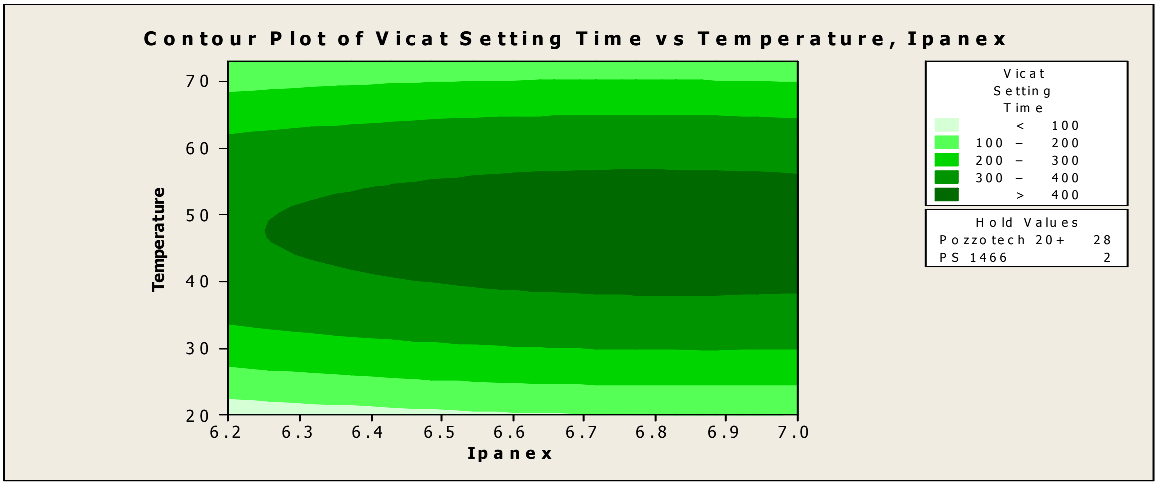


Figure C37: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

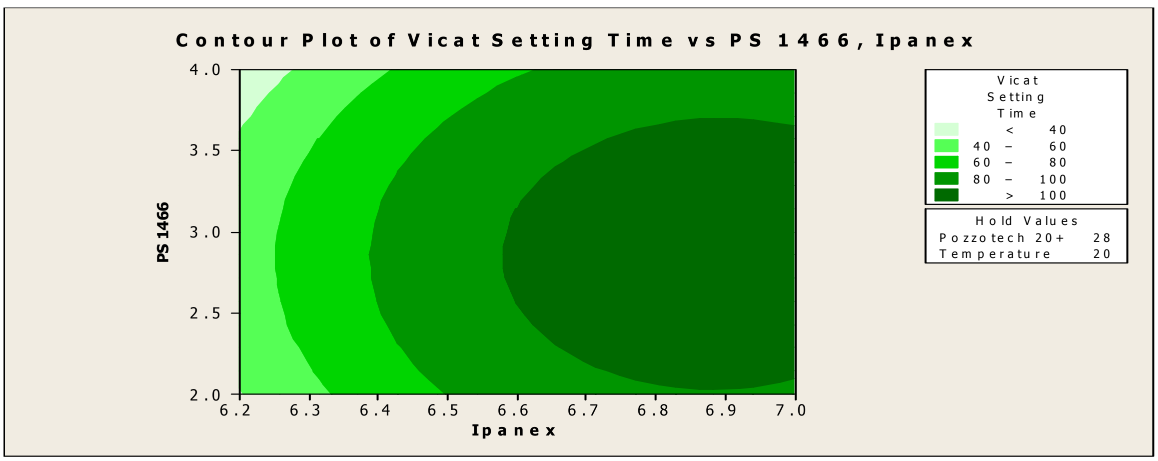


Figure C38: Contour Plot of Vicat Setting Time for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

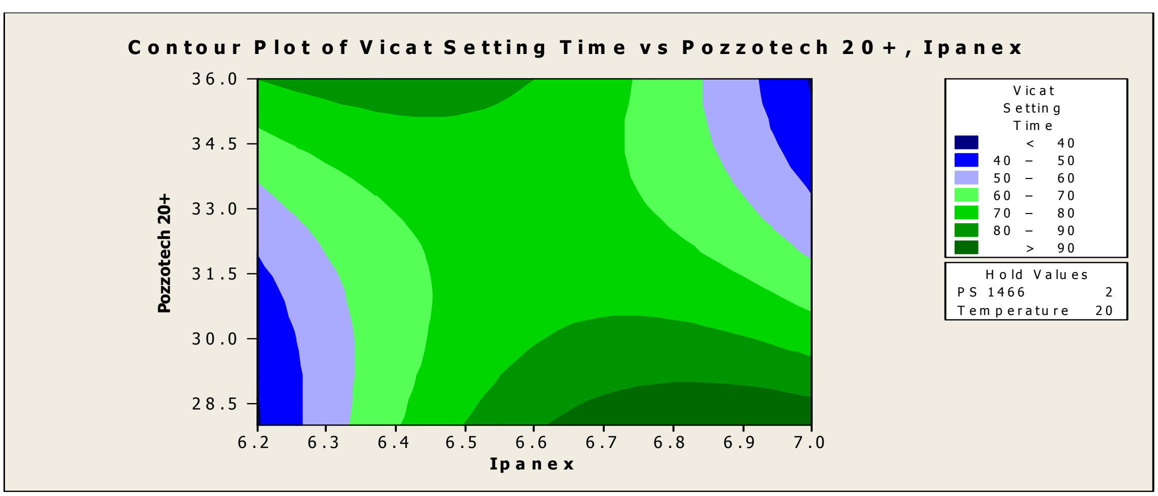


Figure C39: Contour Plot of Vicat Setting Time for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

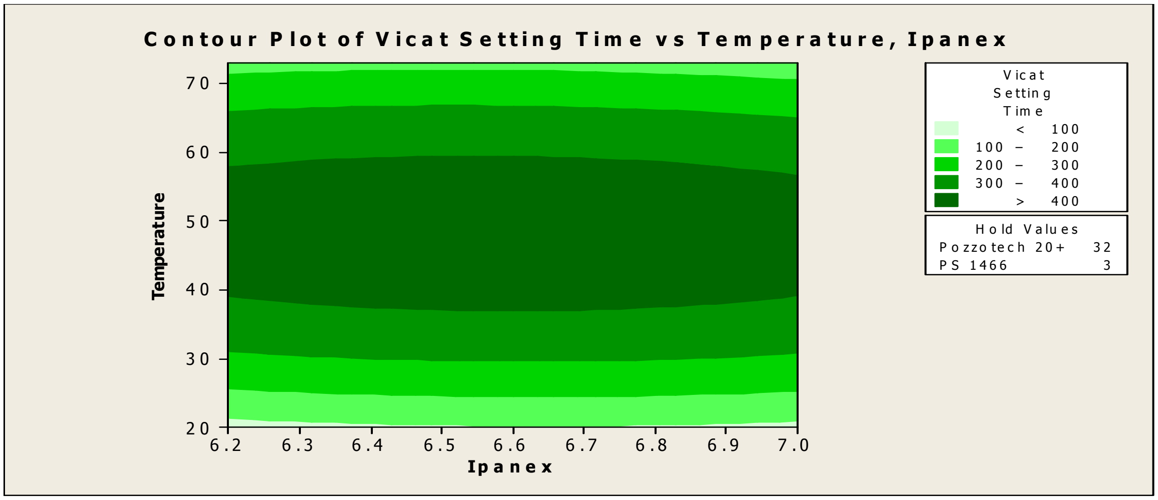


Figure C40: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

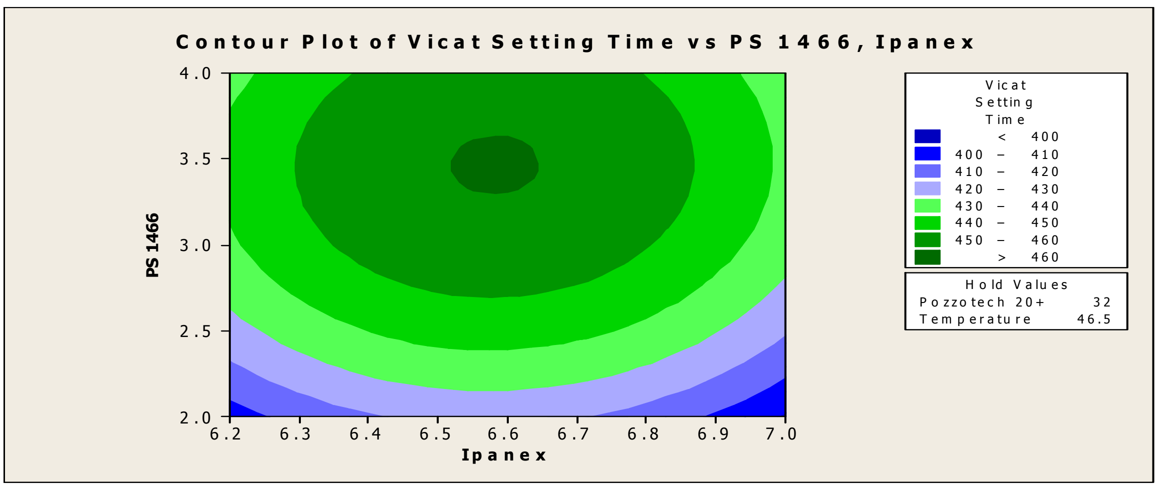


Figure C41: Contour Plot of Vicat Setting Time for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

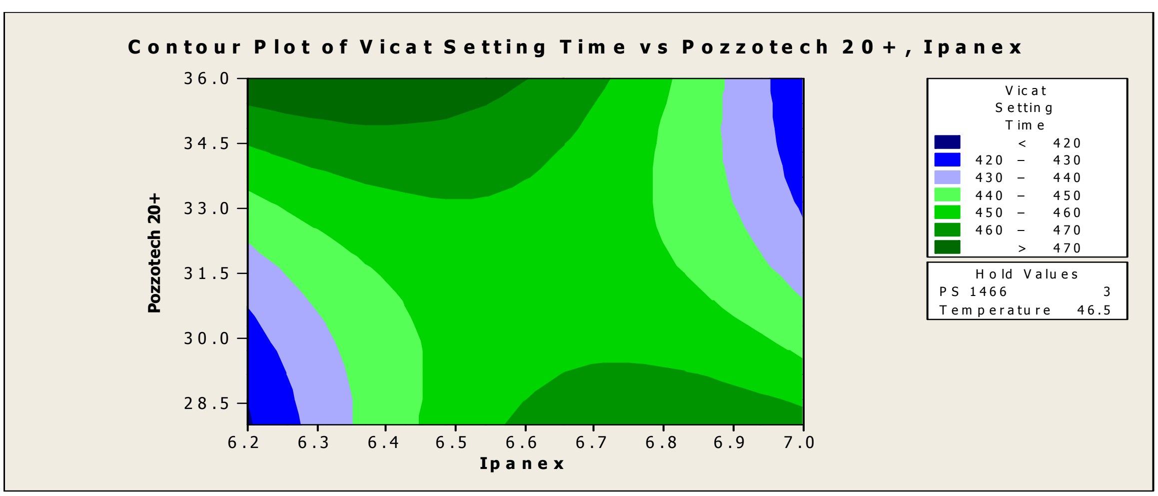


Figure C42: Contour Plot of Vicat Setting Time for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their medium settings

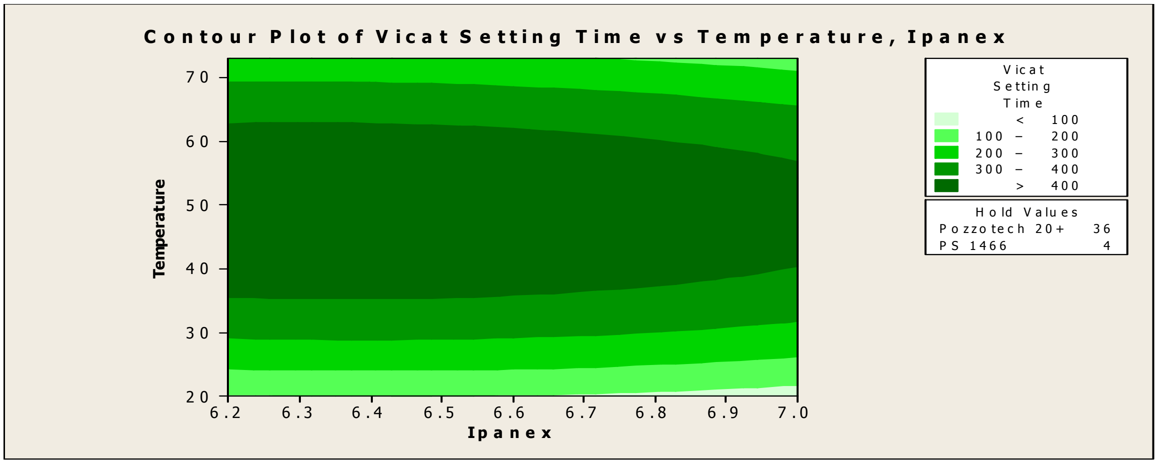


Figure C43: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

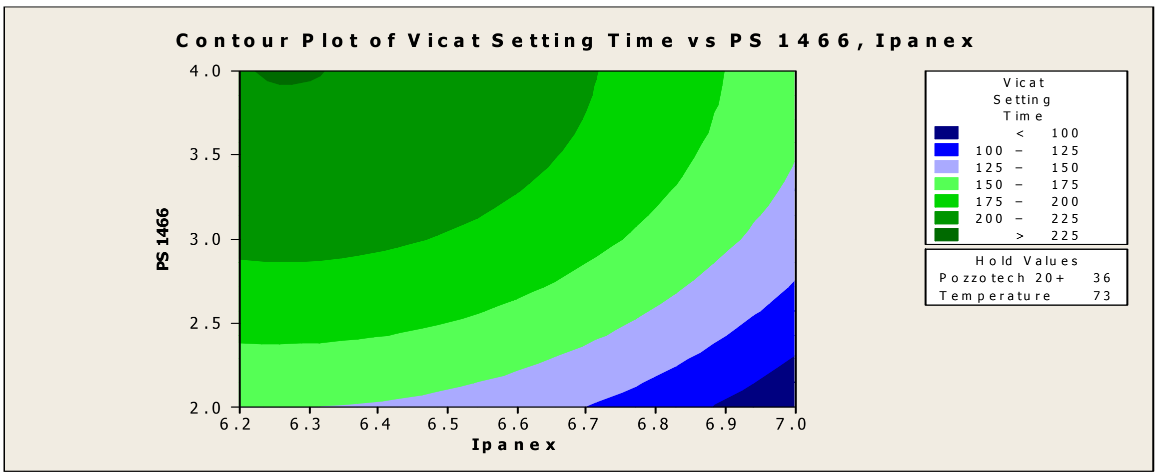


Figure C44: Contour Plot of Vicat Setting Time for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

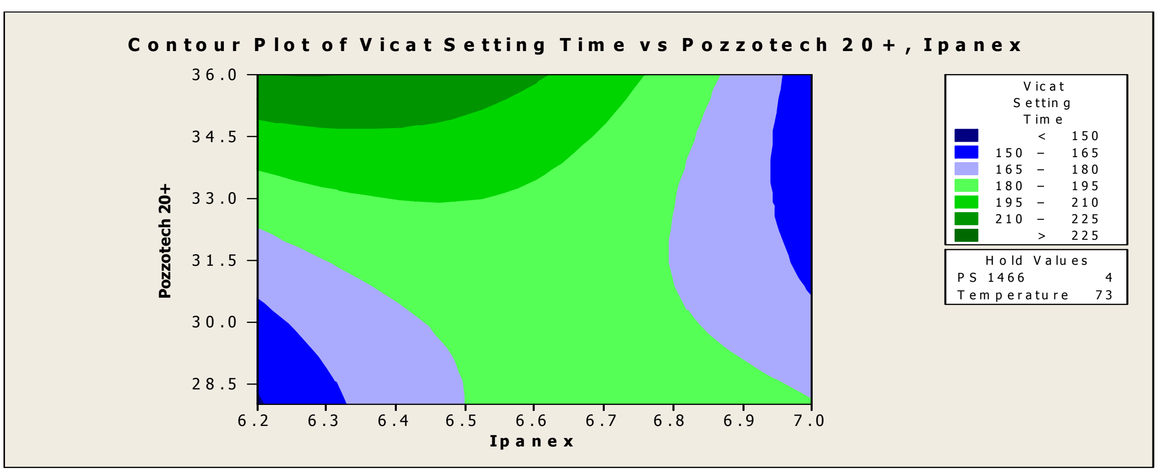


Figure C45: Contour Plot of Vicat Setting Time for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

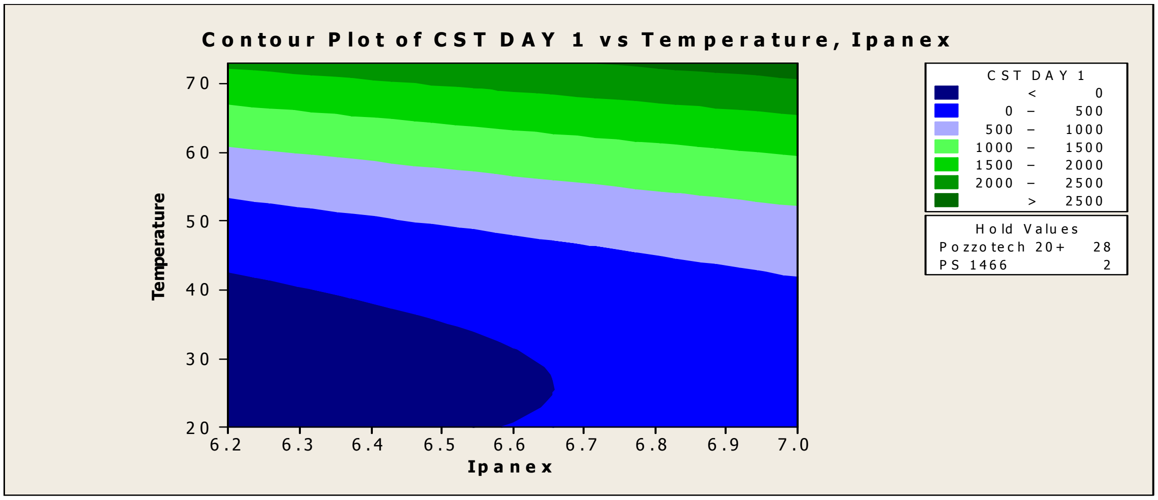


Figure C46: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

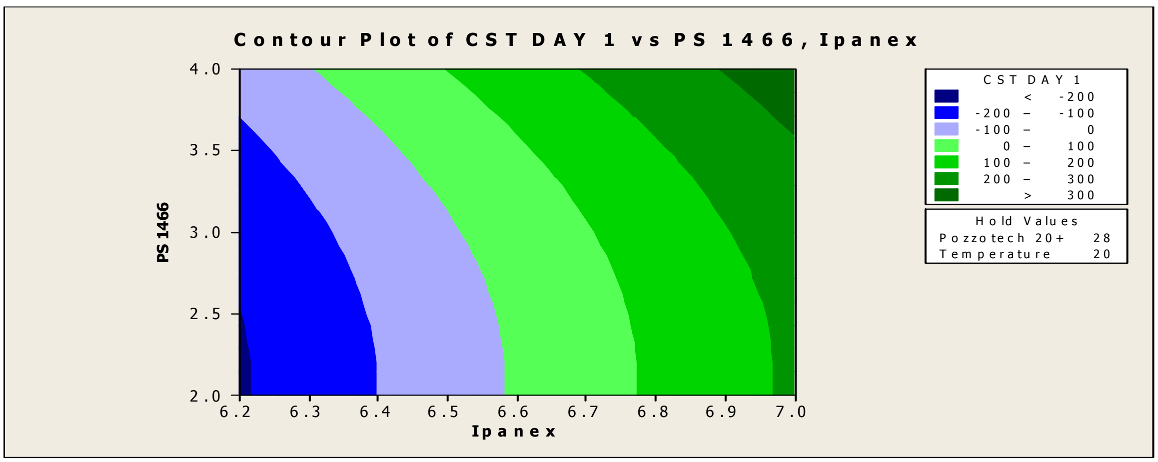


Figure C47: Contour Plot of CST Day 1 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

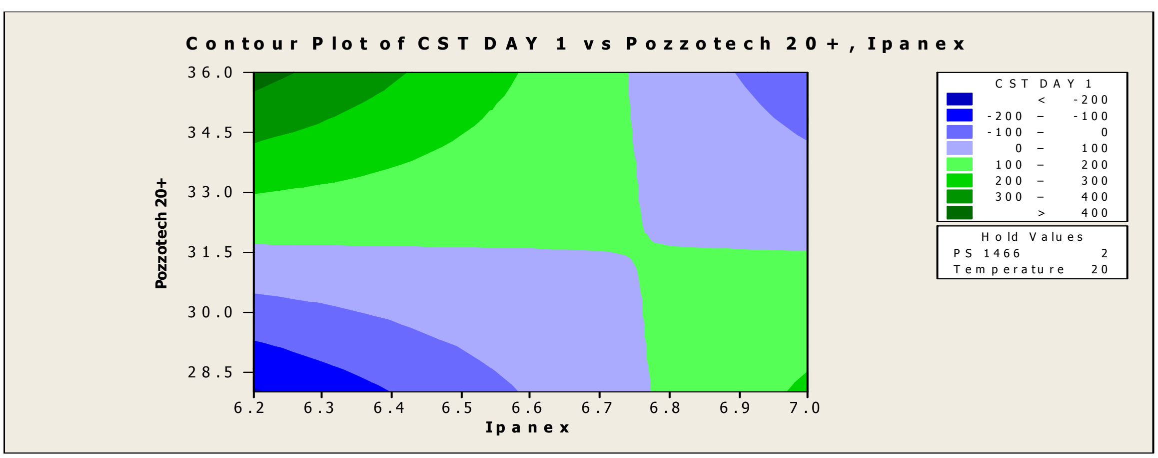


Figure C48: Contour Plot of CST Day 1 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

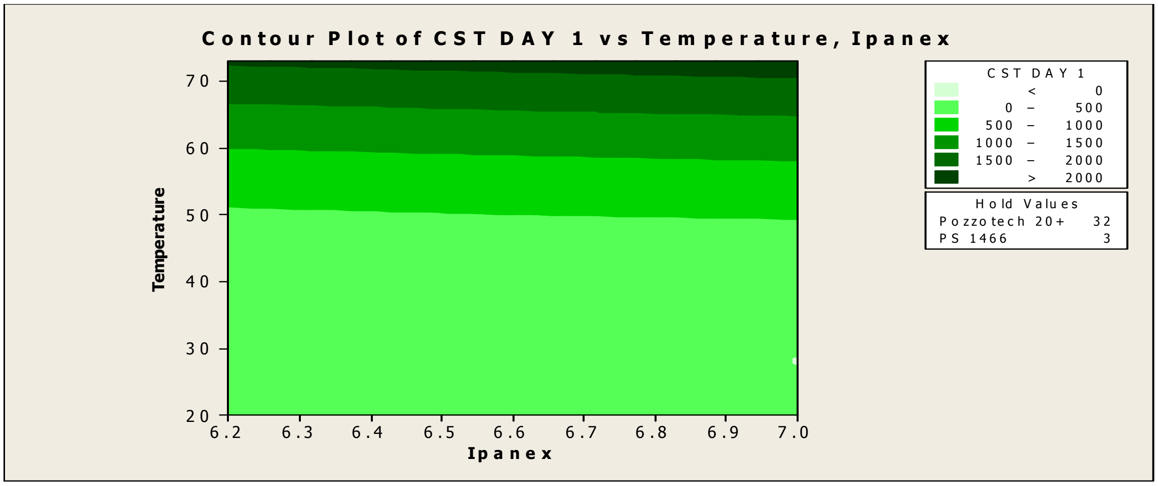


Figure C49: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

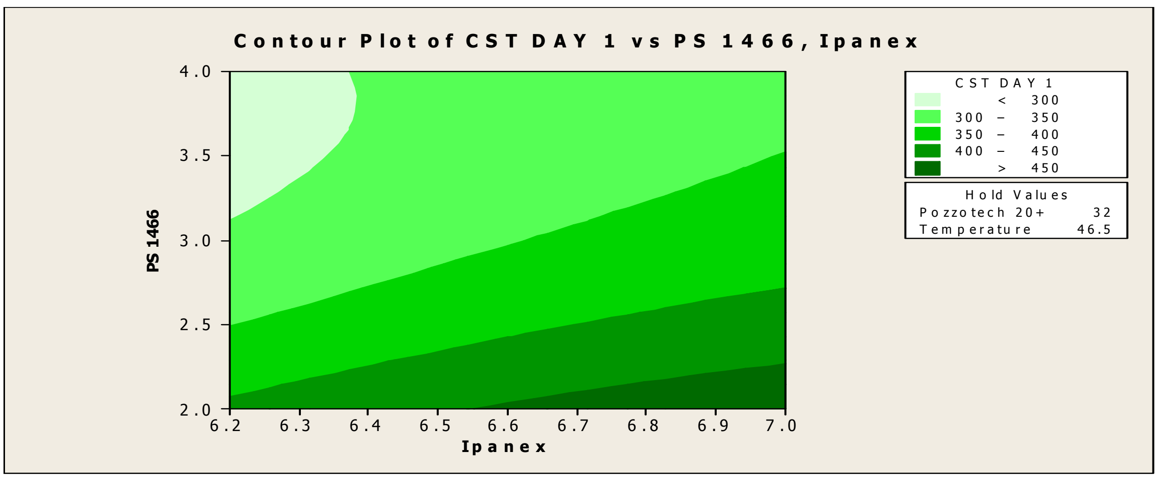


Figure C50: Contour Plot of CST Day 1 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

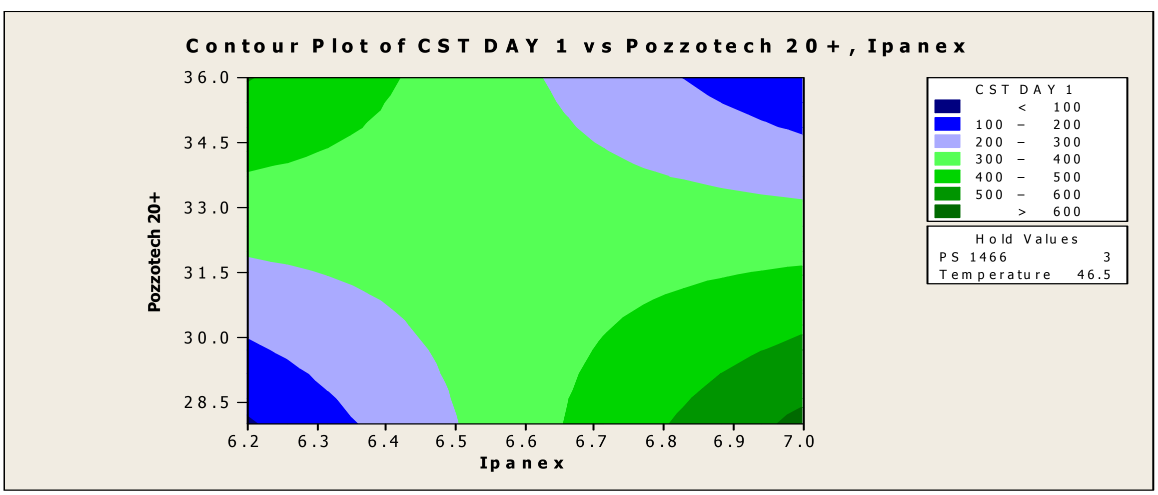


Figure C51: Contour Plot of CST Day 1 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

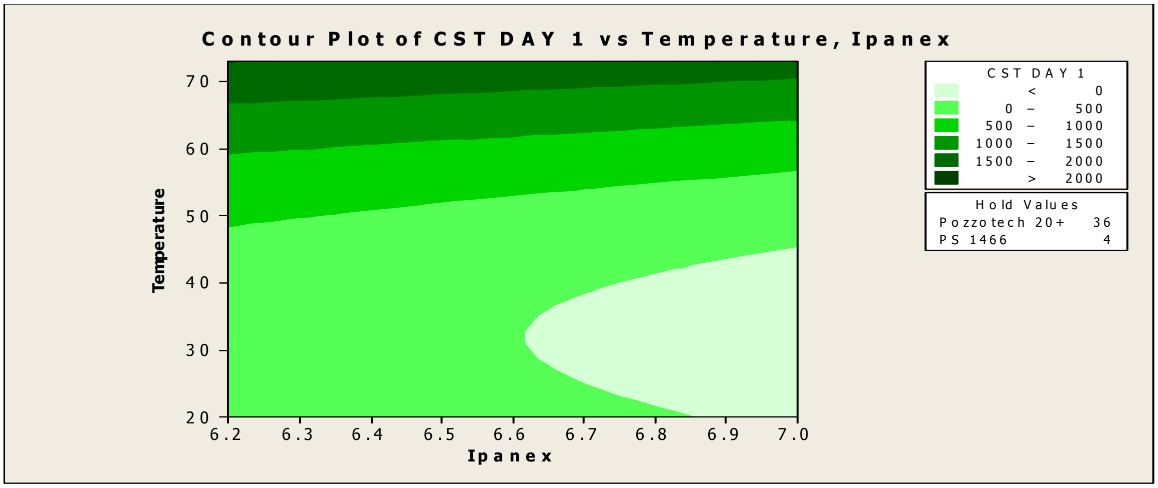


Figure C52: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

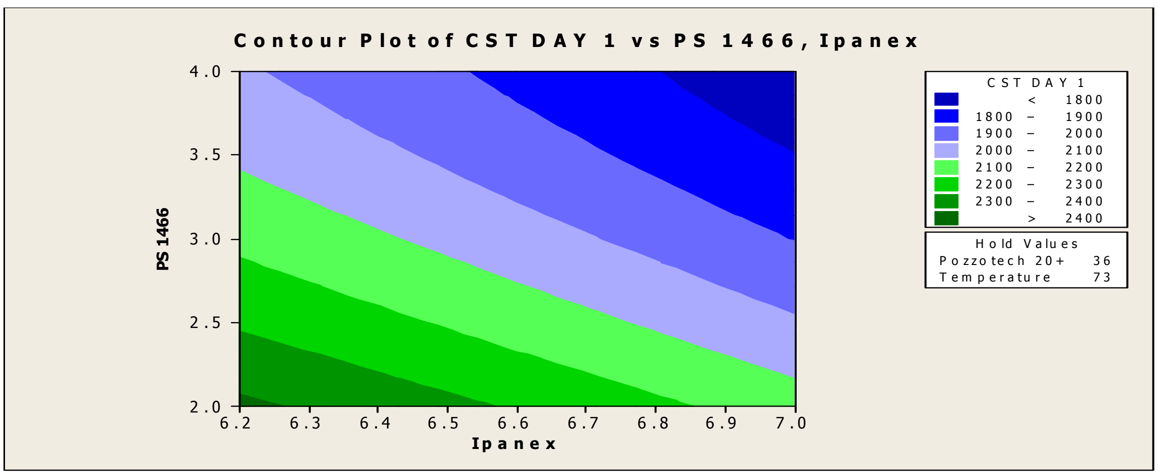


Figure C53: Contour Plot of CST Day 1 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

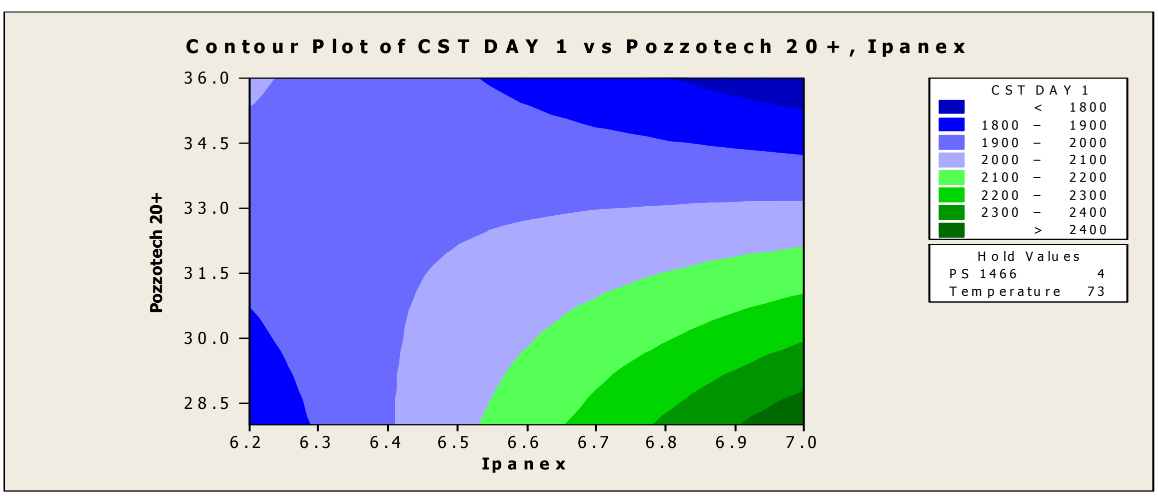


Figure C54: Contour Plot of CST Day 1 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

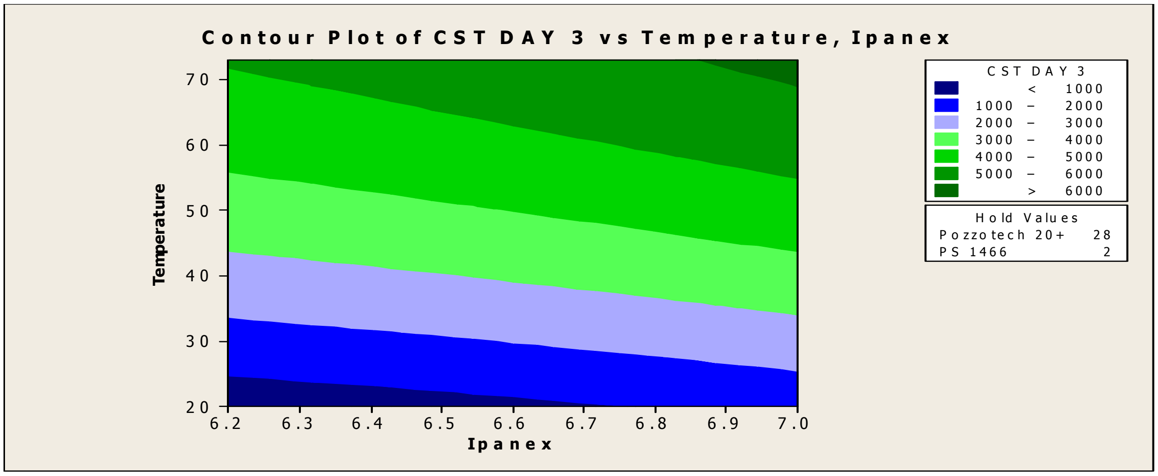


Figure C55: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

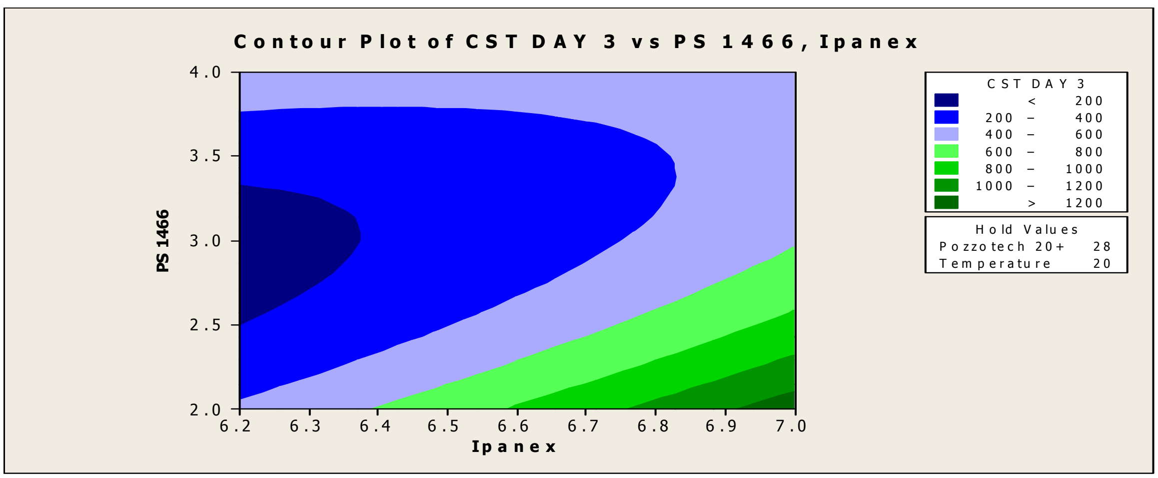


Figure C56: Contour Plot of CST Day 3 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

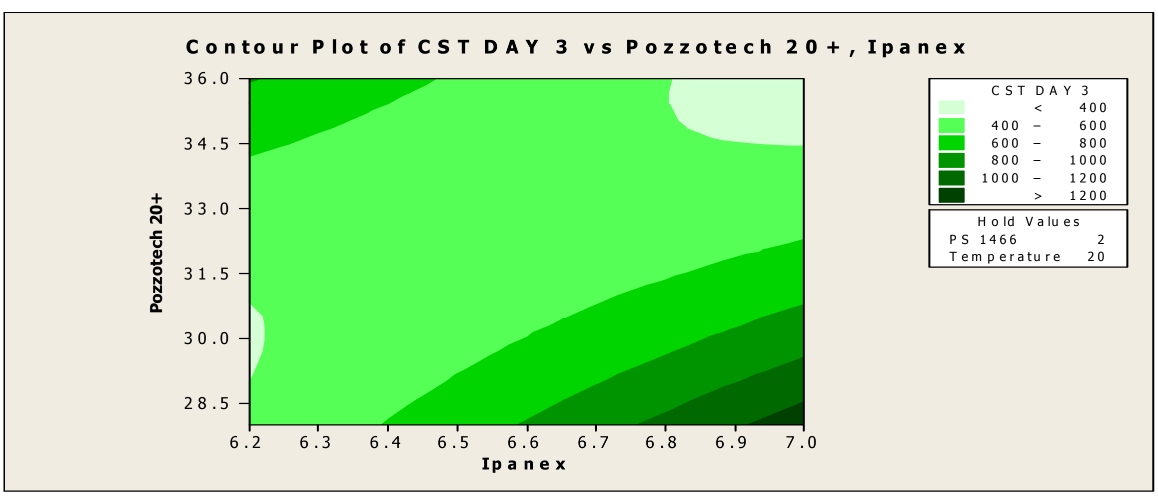


Figure C57: Contour Plot of CST Day 3 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

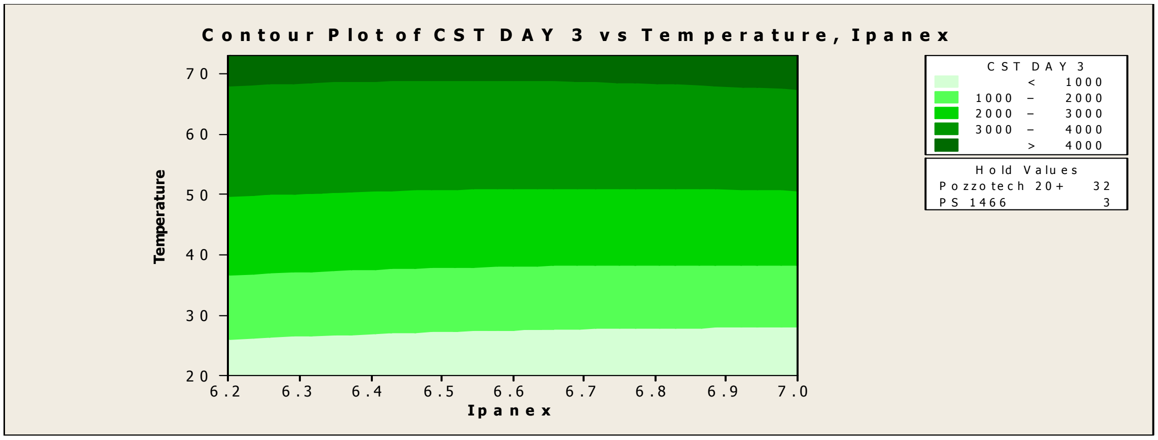


Figure C58: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

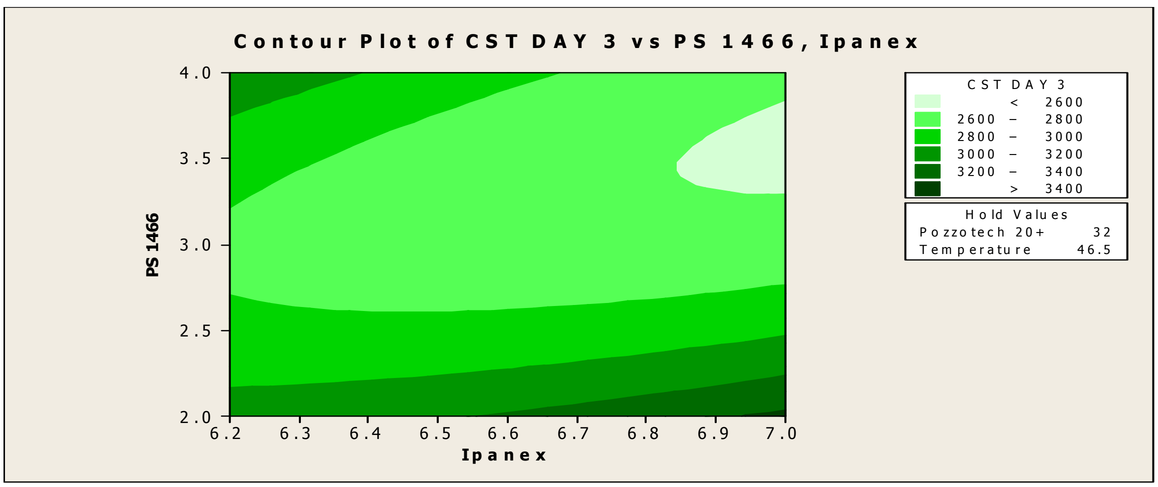


Figure C59: Contour Plot of CST Day 3 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

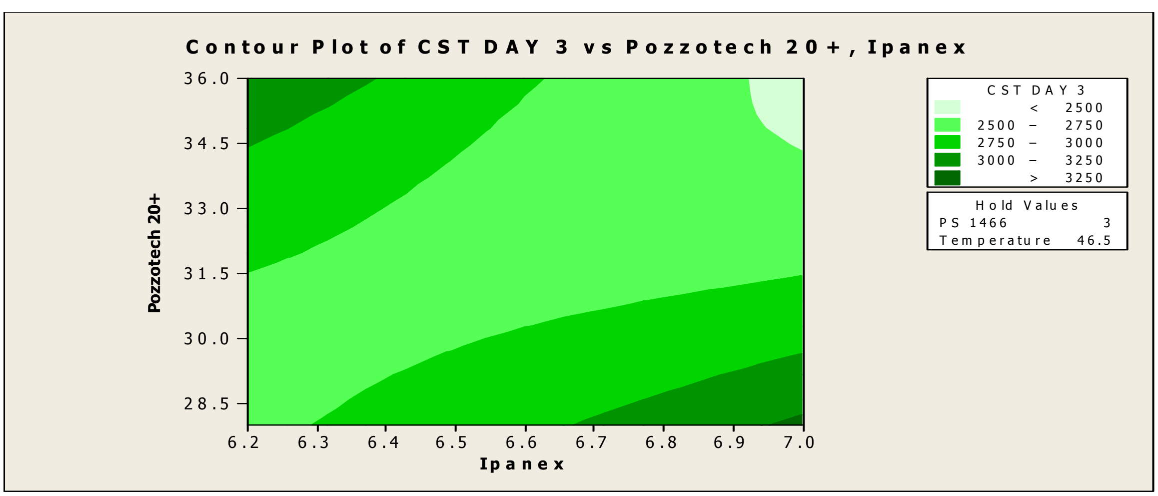


Figure C60: Contour Plot of CST Day 3 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

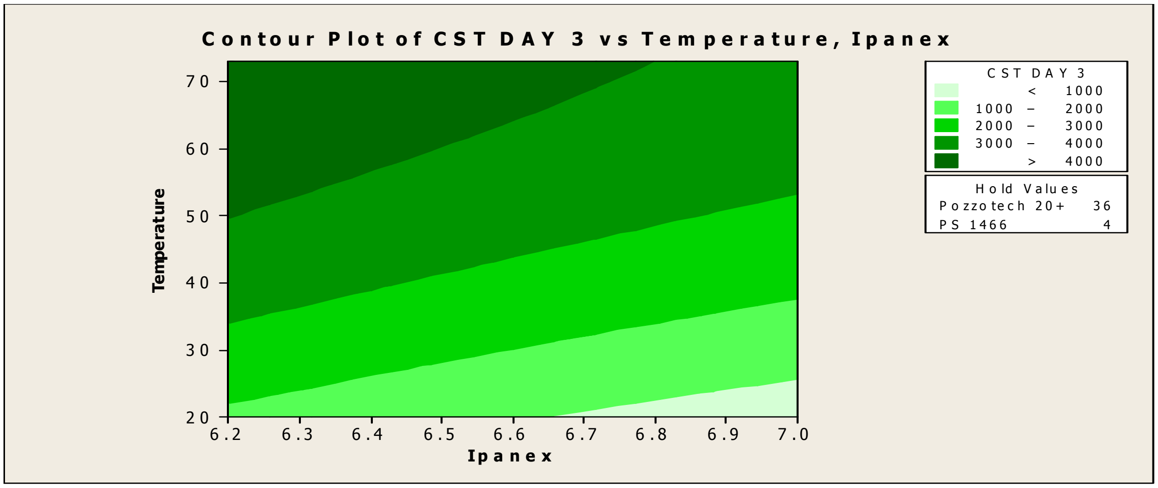


Figure C61: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

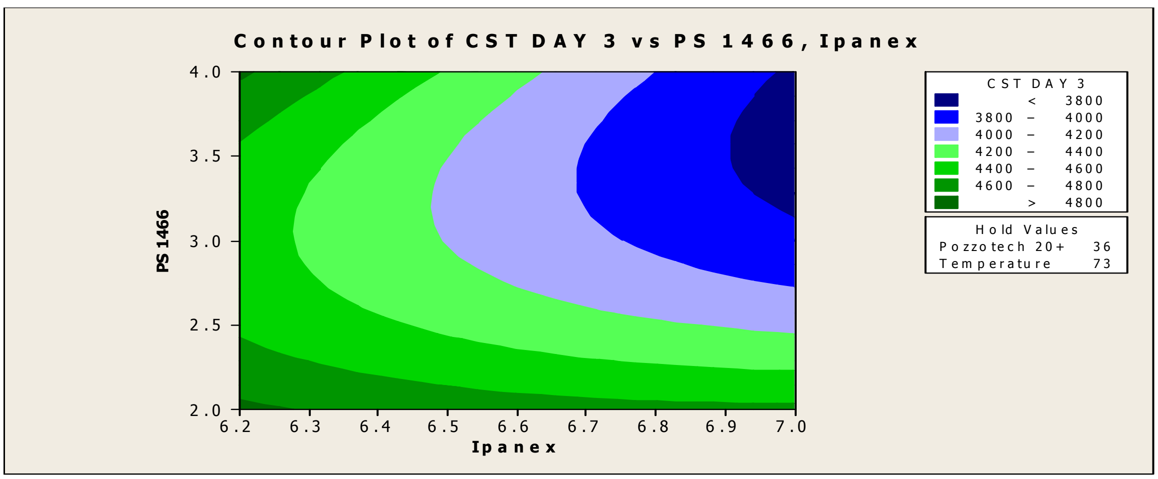


Figure C62: Contour Plot of CST Day 3 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

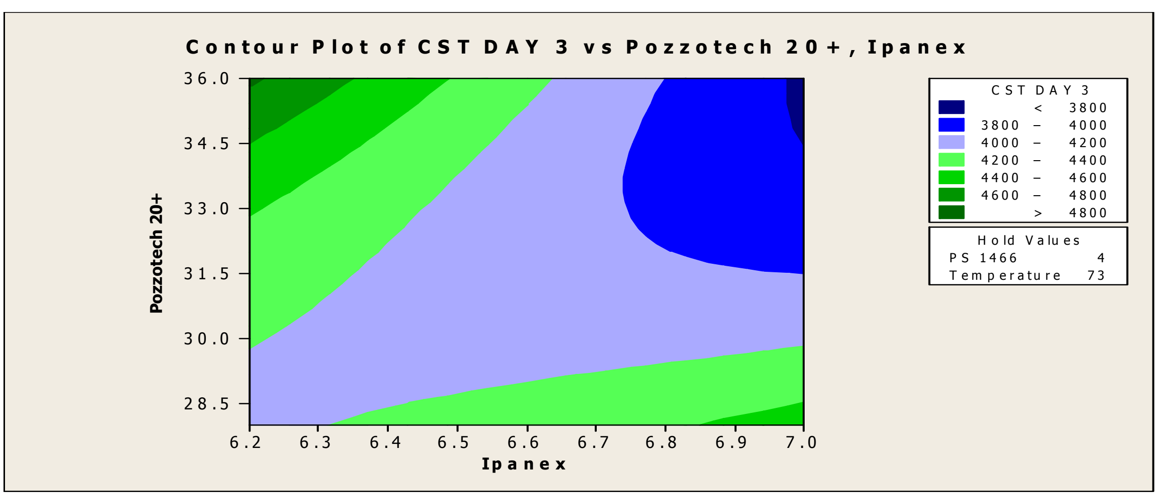


Figure C63: Contour Plot of CST Day 3 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

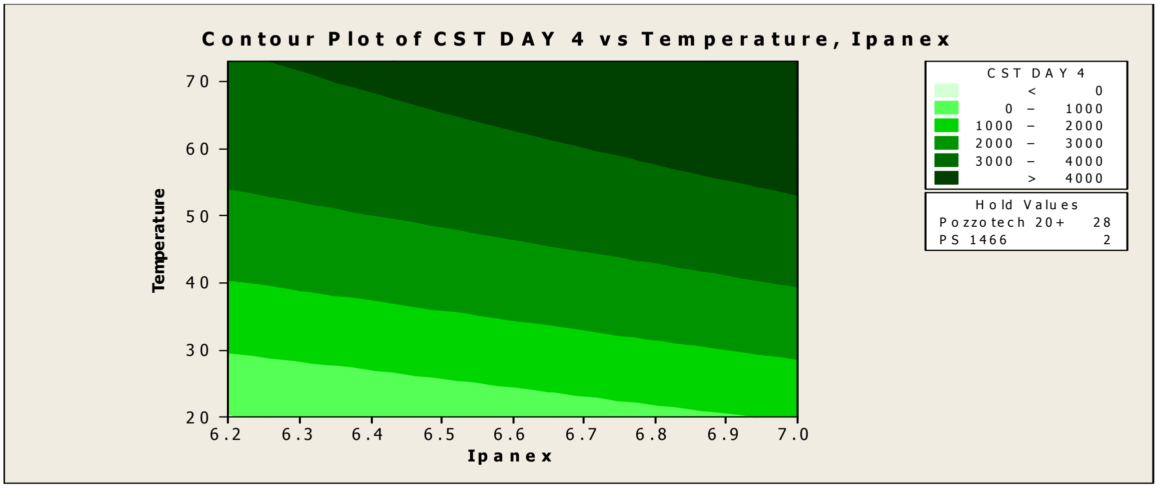


Figure C64: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

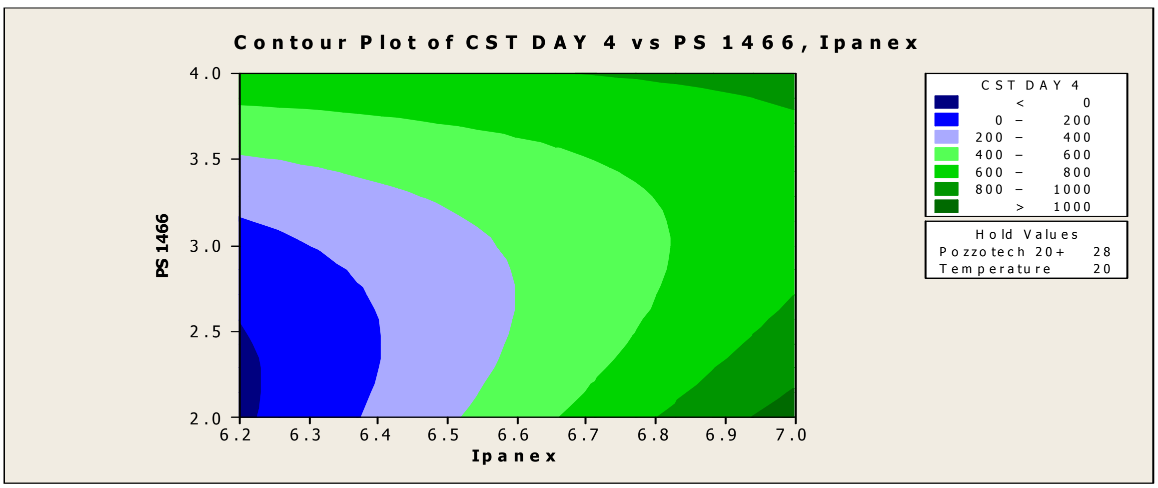


Figure C65: Contour Plot of CST Day 4 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

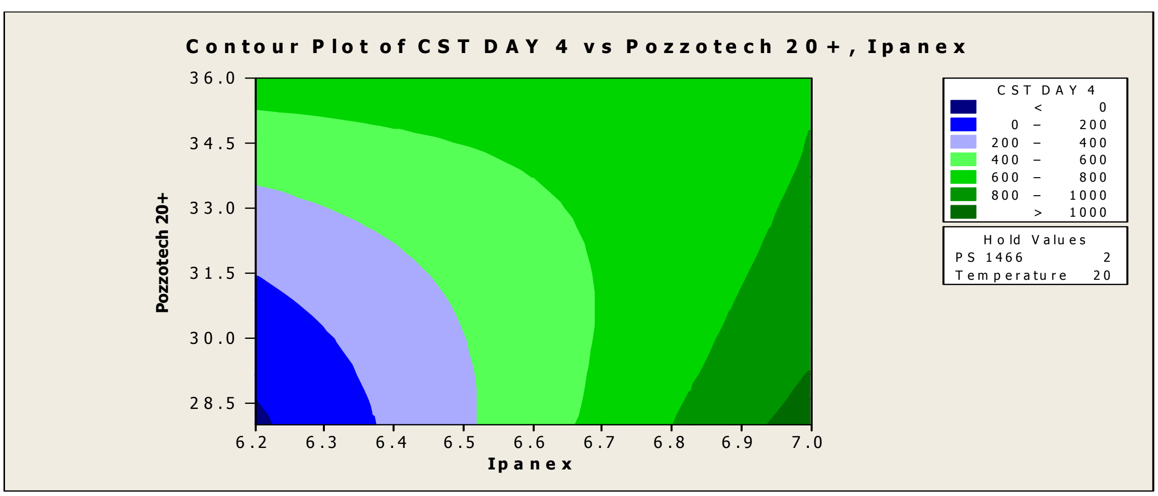


Figure C66: Contour Plot of CST Day 4 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their low settings.

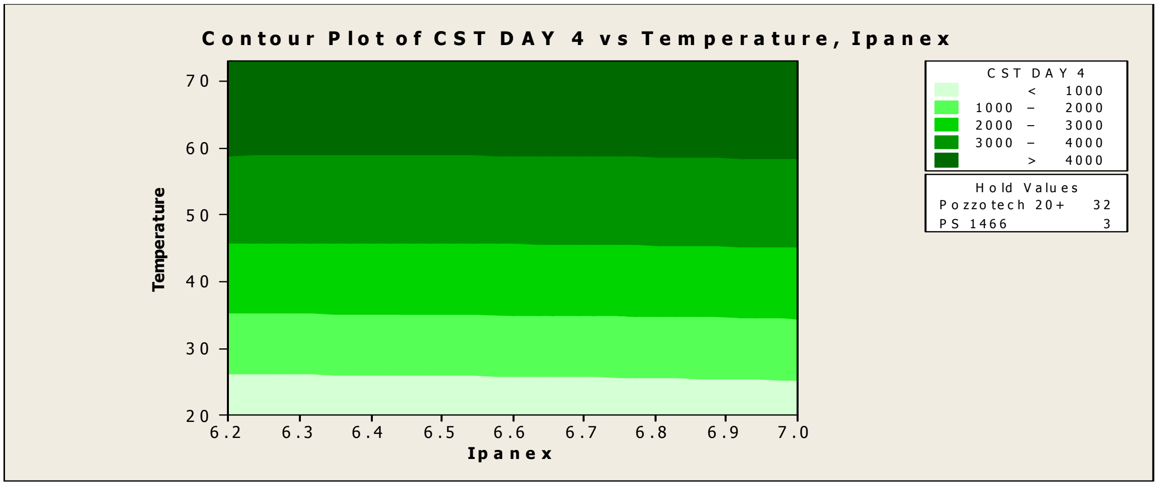


Figure C67: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

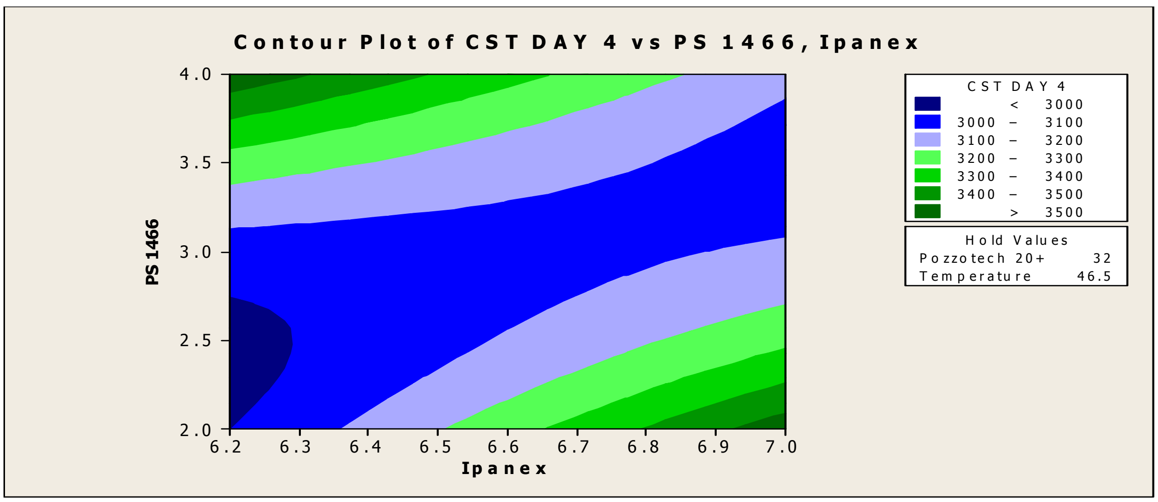


Figure C68: Contour Plot of CST Day 4 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

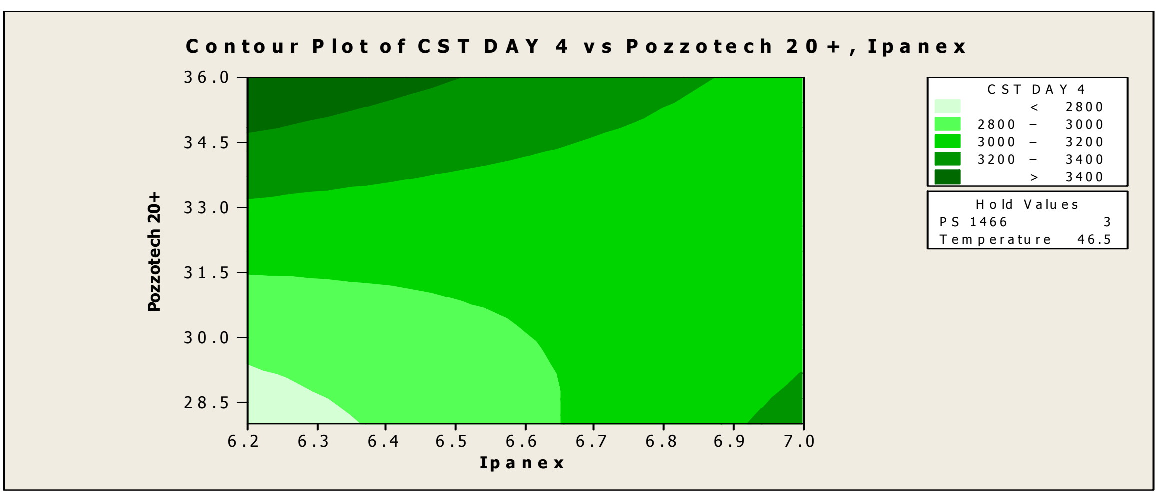


Figure C69: Contour Plot of CST Day 4 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their medium settings.

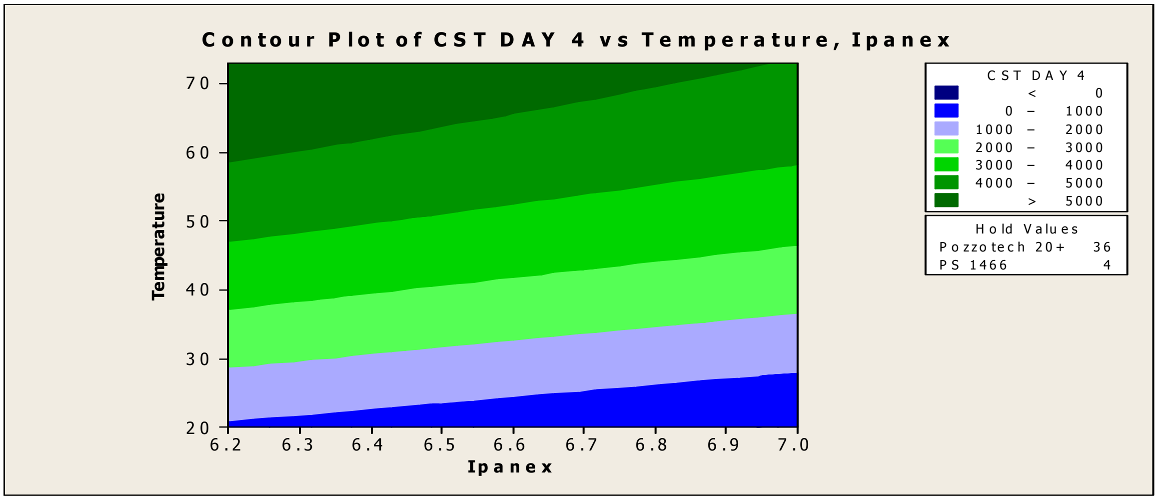


Figure C70: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

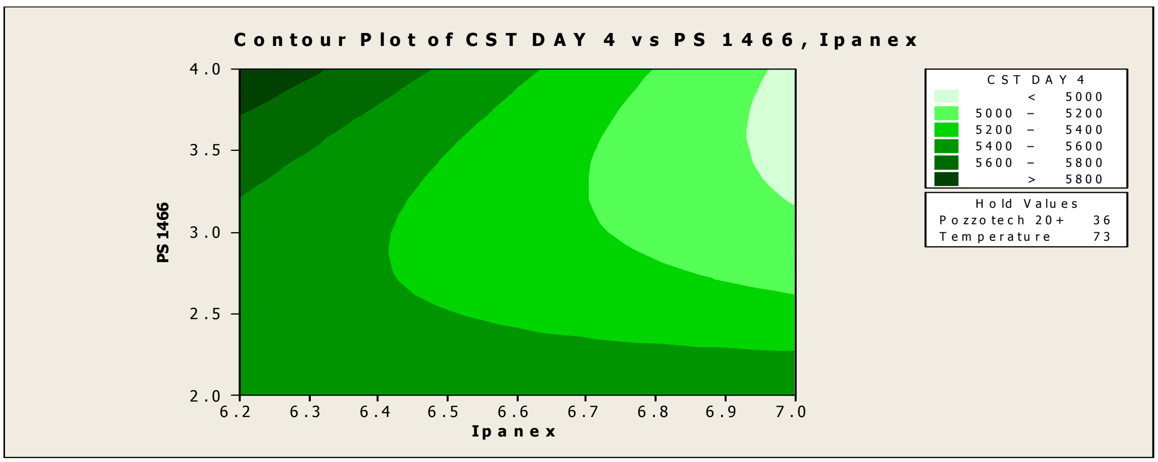


Figure C71: Contour Plot of CST Day 4 for PS1466 vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

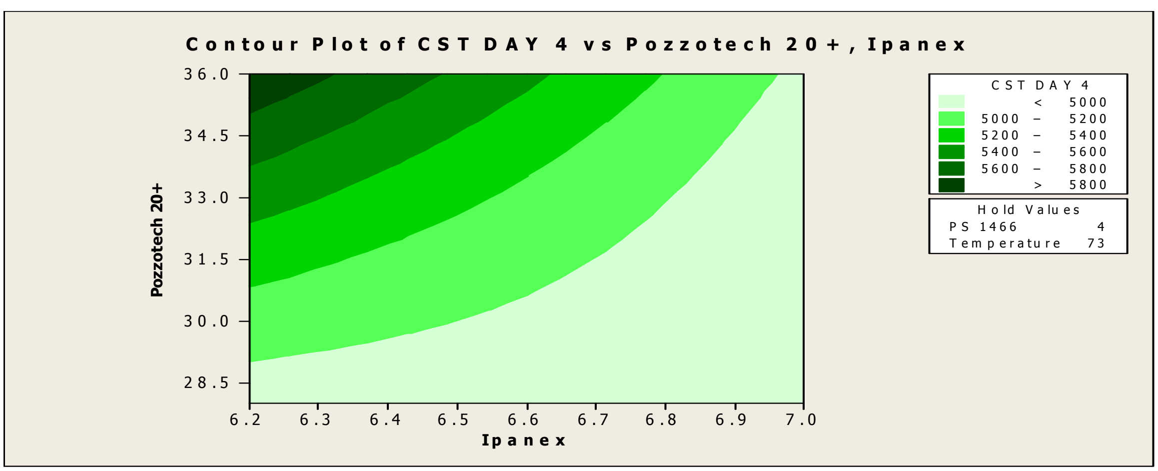


Figure C72: Contour Plot of CST Day 4 for Pozzutec 20+ vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

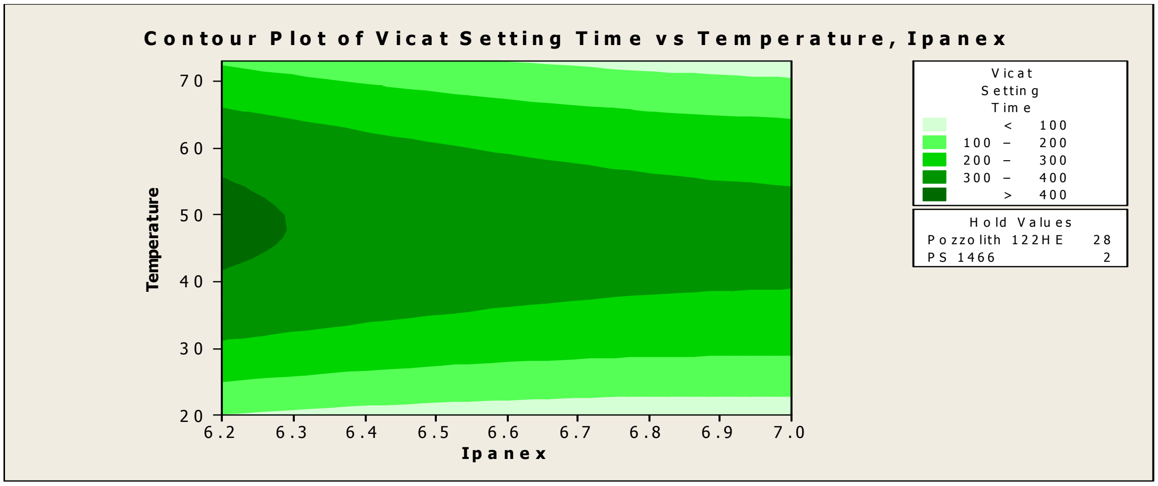


Figure C73: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

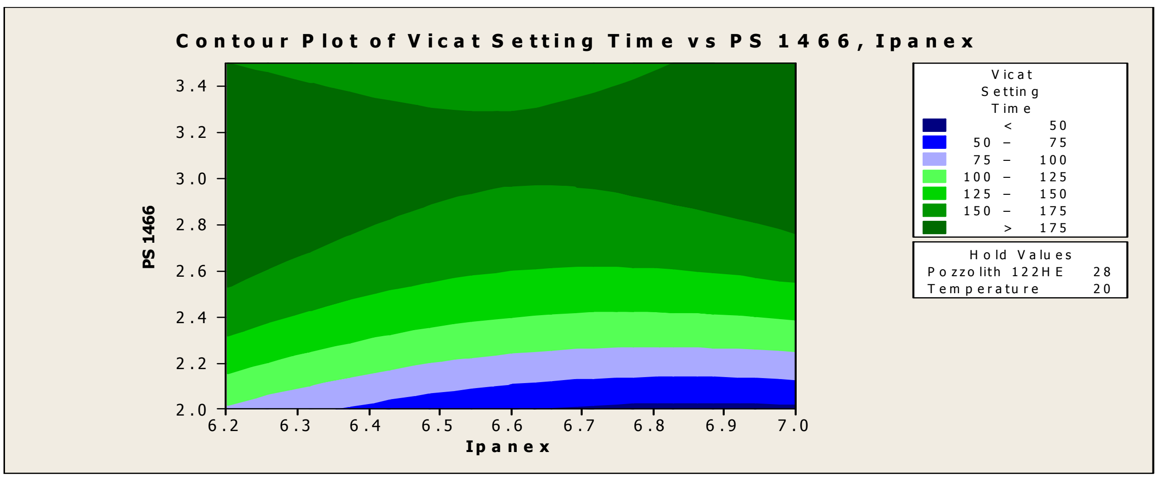


Figure C74: Contour Plot of Vicat Setting Time for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

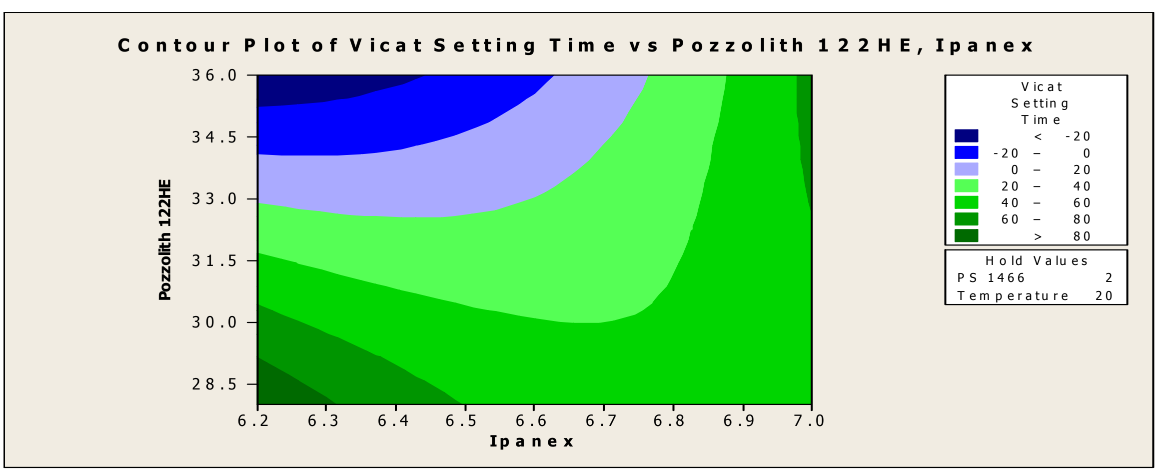


Figure C75: Contour Plot of Vicat Setting Time for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

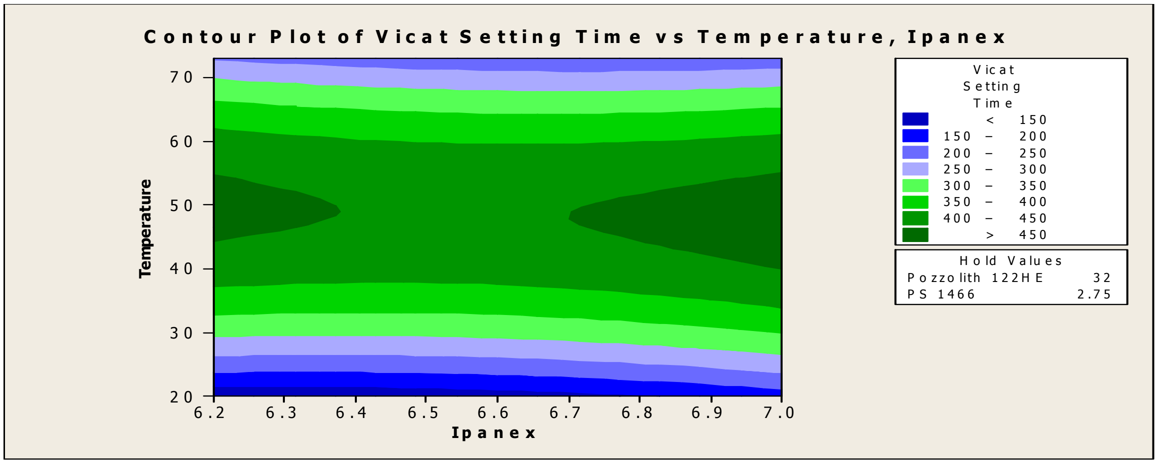


Figure C76: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

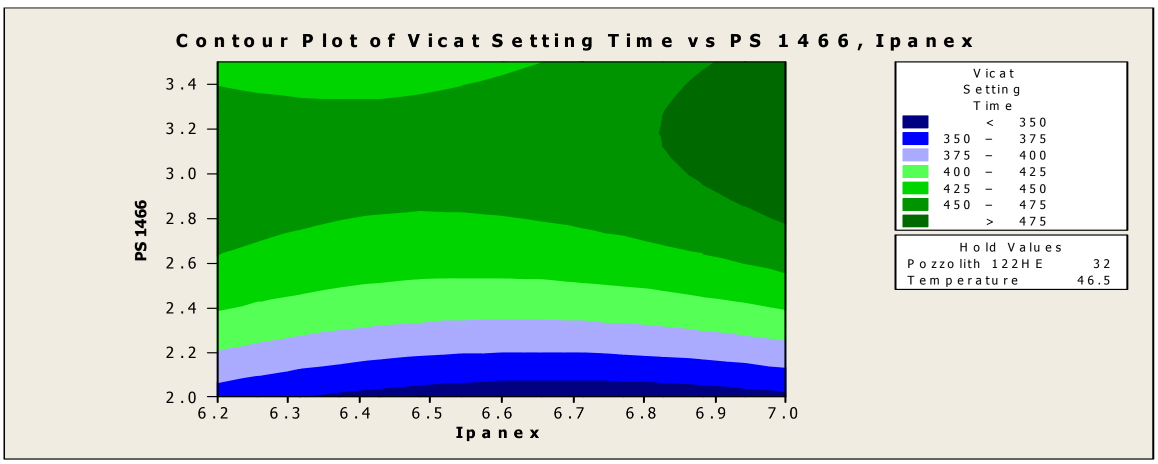


Figure C77: Contour Plot of Vicat Setting Time for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

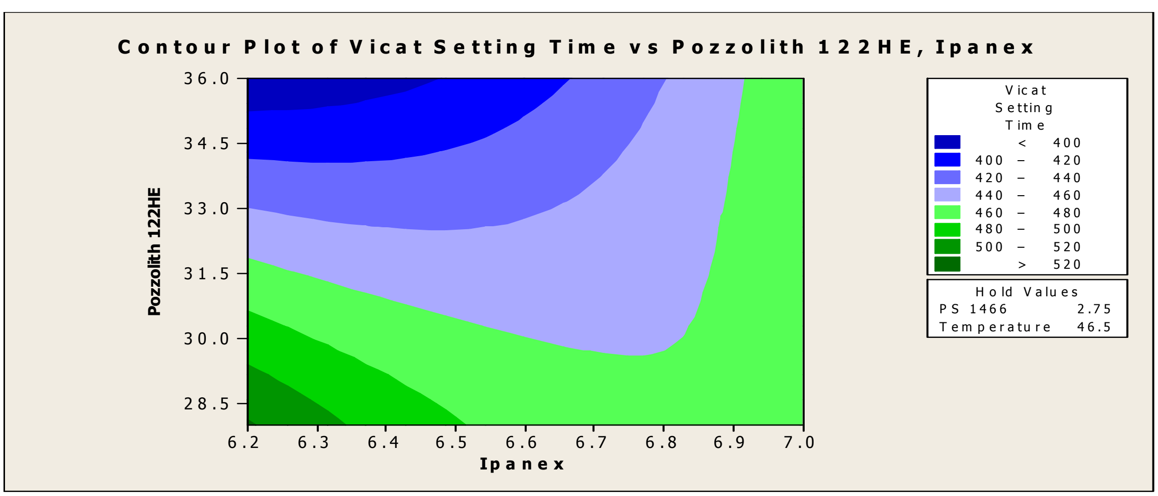


Figure C78: Contour Plot of Vicat Setting Time for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their medium settings

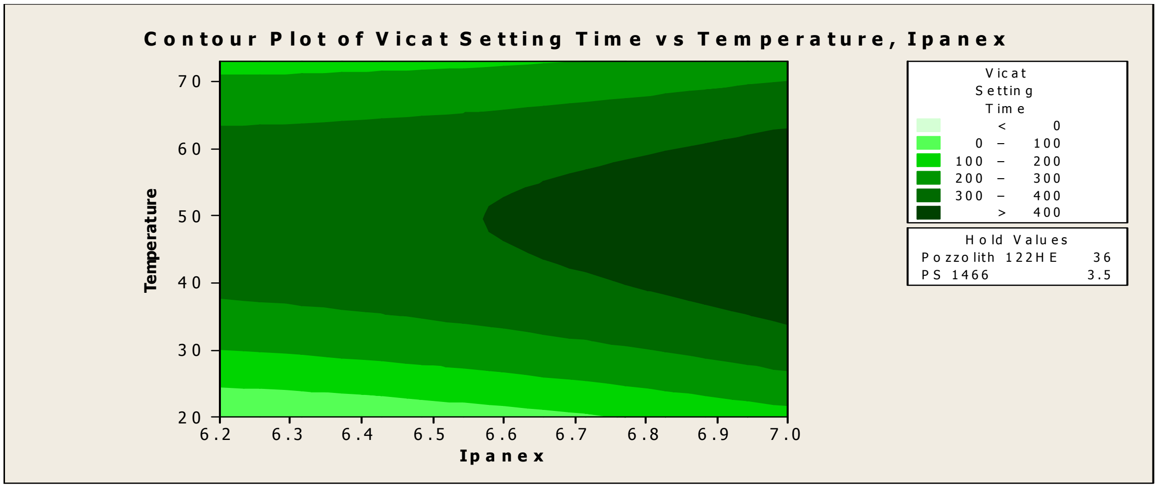


Figure C79: Contour Plot of Vicat Setting Time for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

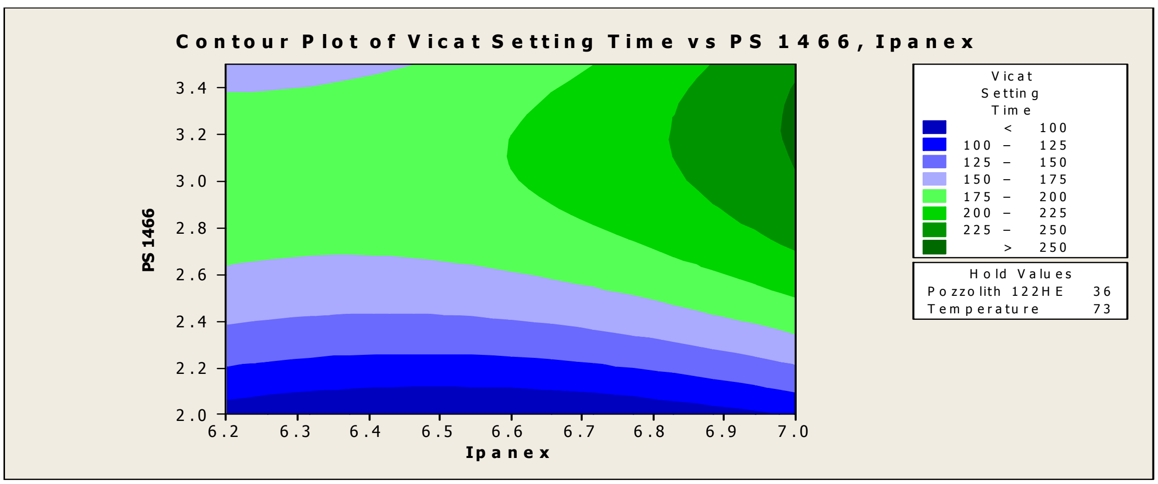


Figure C80: Contour Plot of Vicat Setting Time for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

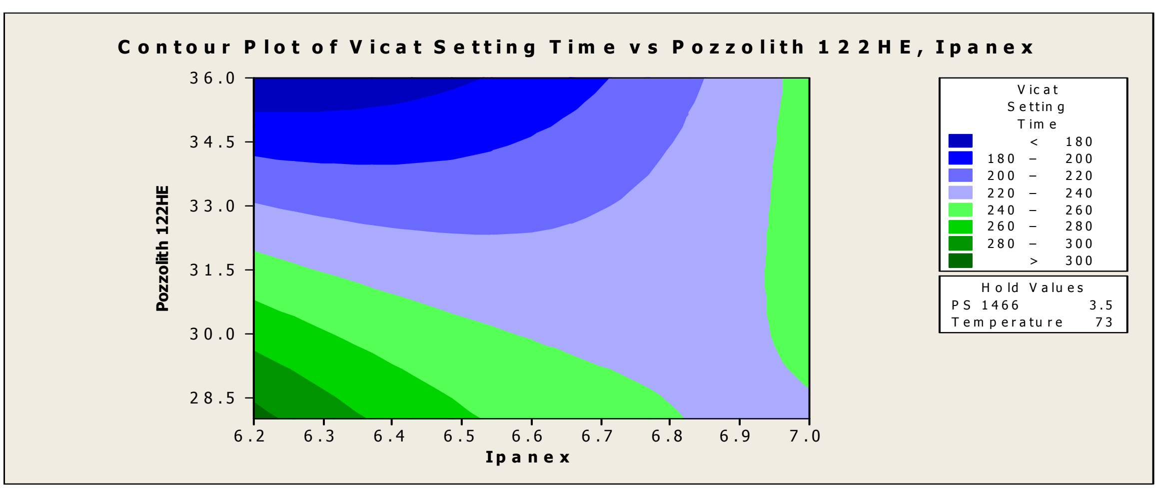


Figure C81: Contour Plot of Vicat Setting Time for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

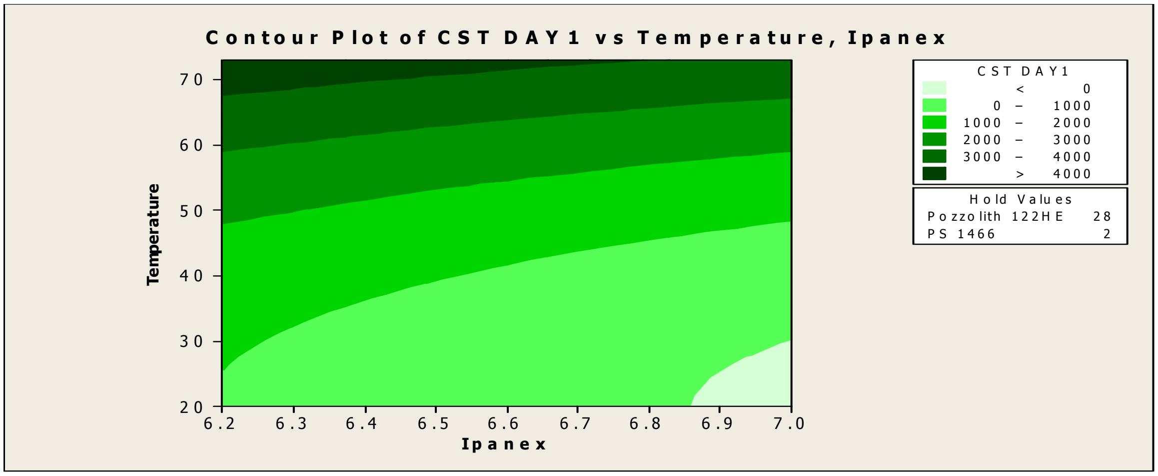


Figure C82: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

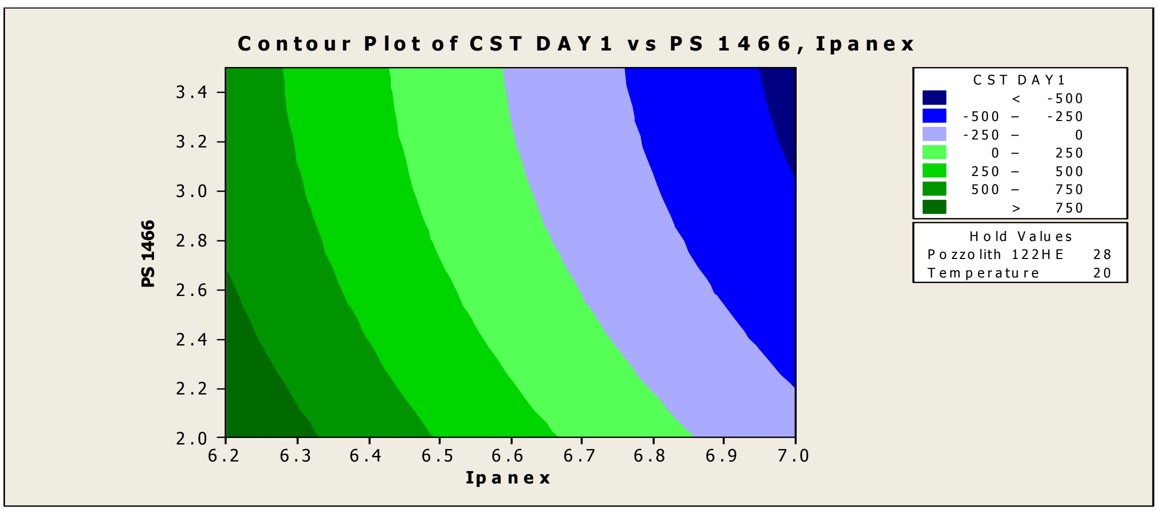


Figure C83: Contour Plot of CST Day 1 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

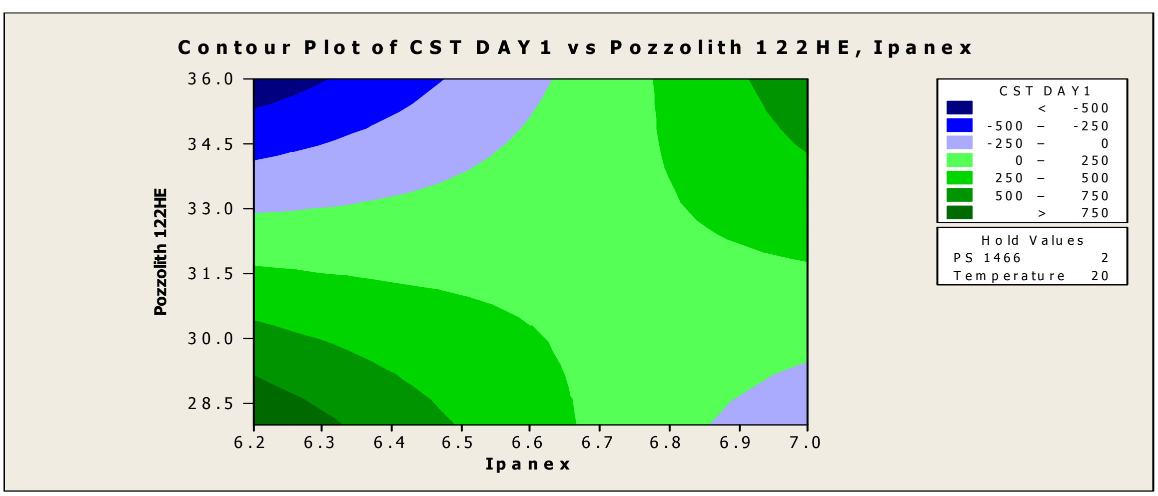


Figure C84: Contour Plot of CST Day 1 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

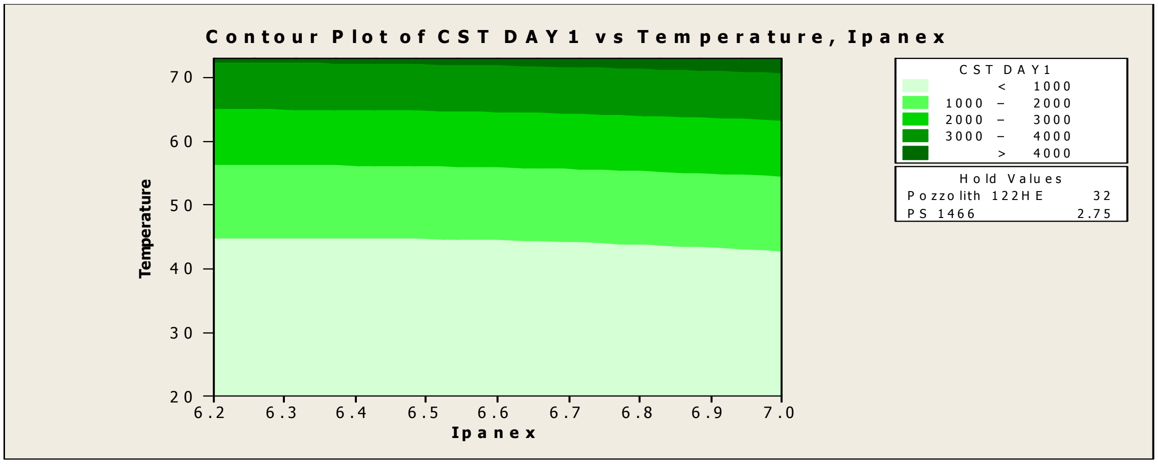


Figure C85: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

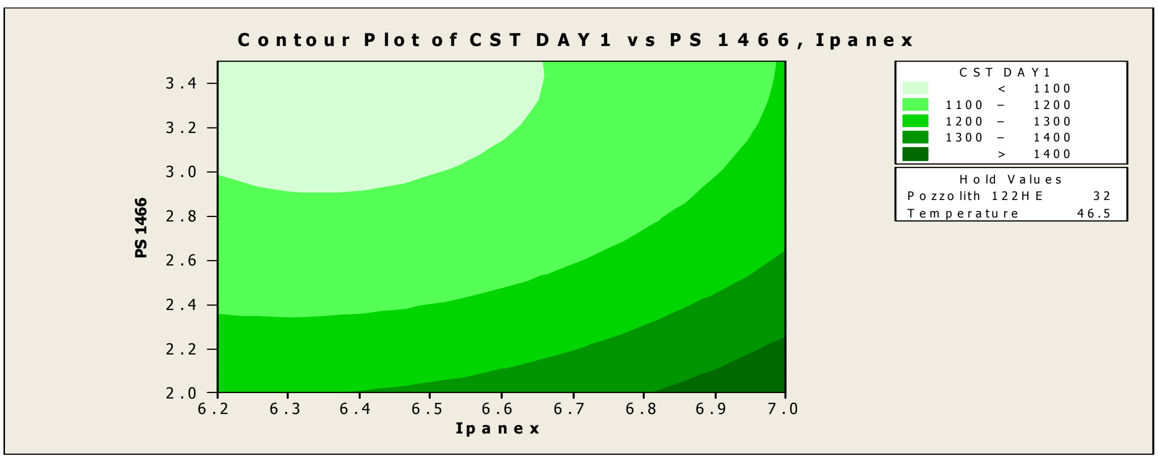


Figure C86: Contour Plot of CST Day 1 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

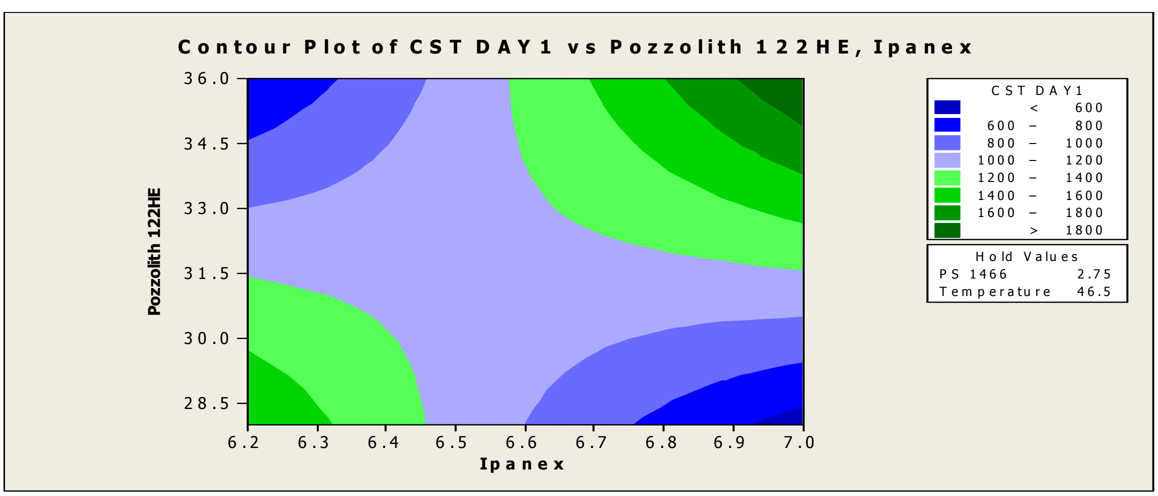


Figure C87: Contour Plot of CST Day 1 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

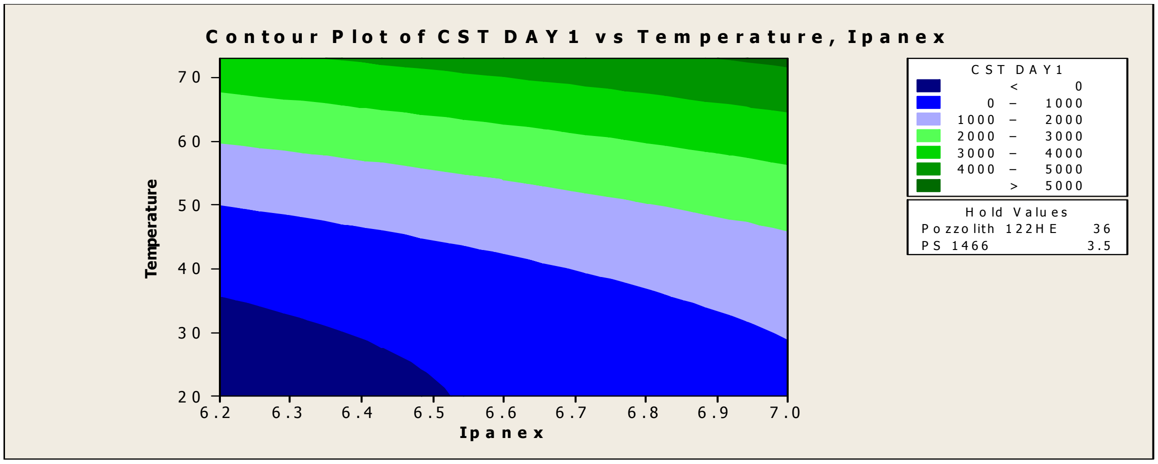


Figure C88: Contour Plot of CST Day 1 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

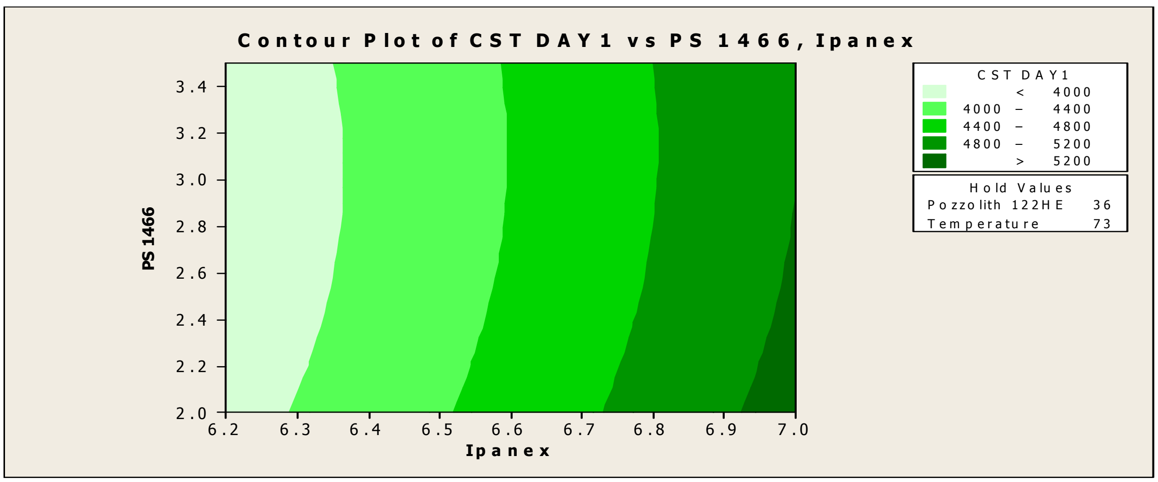


Figure C89: Contour Plot of CST Day 1 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

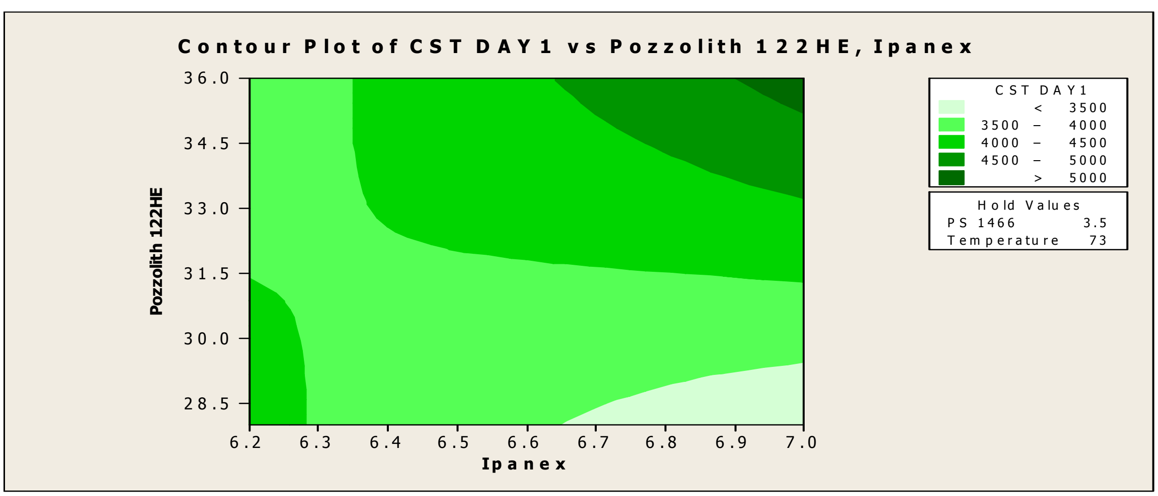


Figure C90: Contour Plot of CST Day 1 for Pozzolith 122HE vs Ipanex for the Mix 8 keeping all the other factors at their high settings.

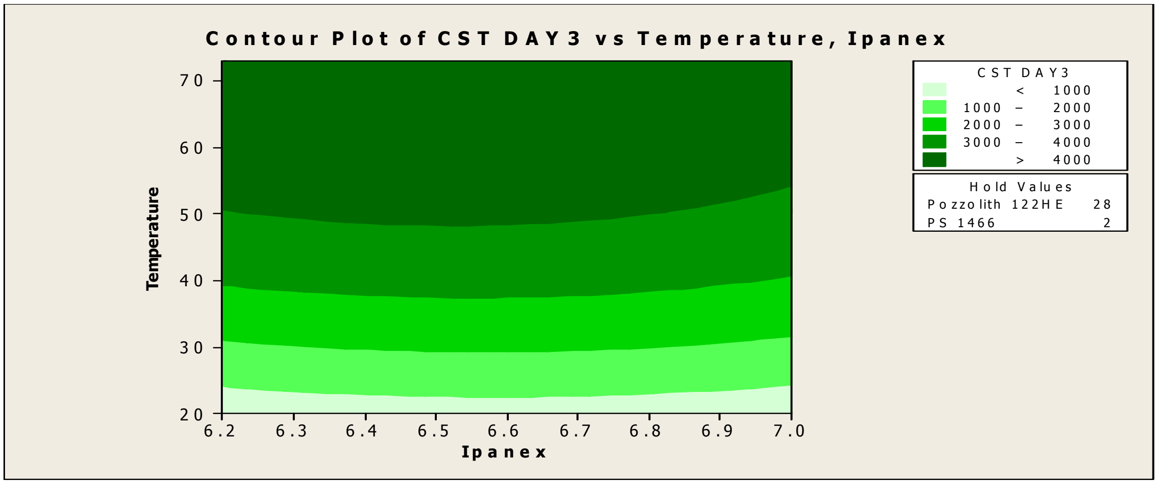


Figure C91: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

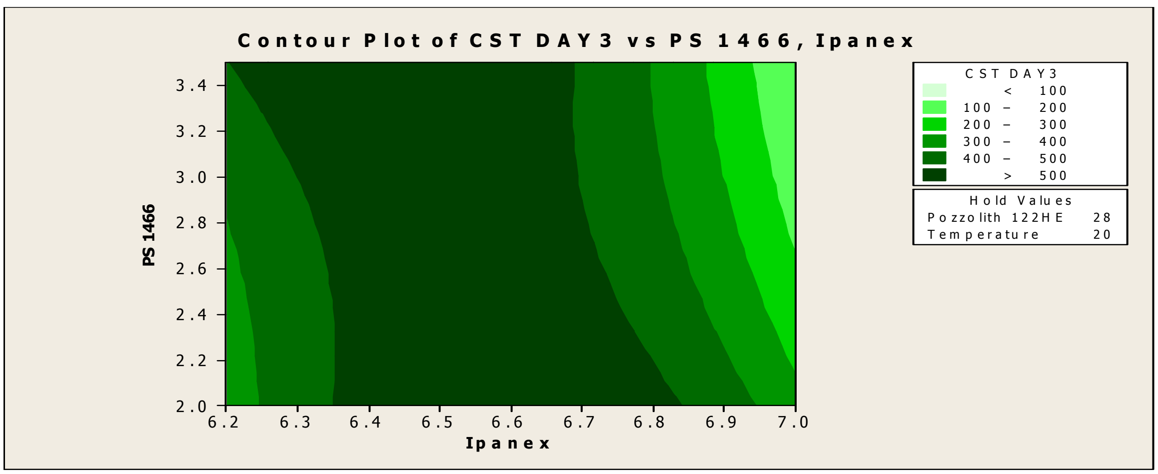


Figure C92: Contour Plot of CST Day 3 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

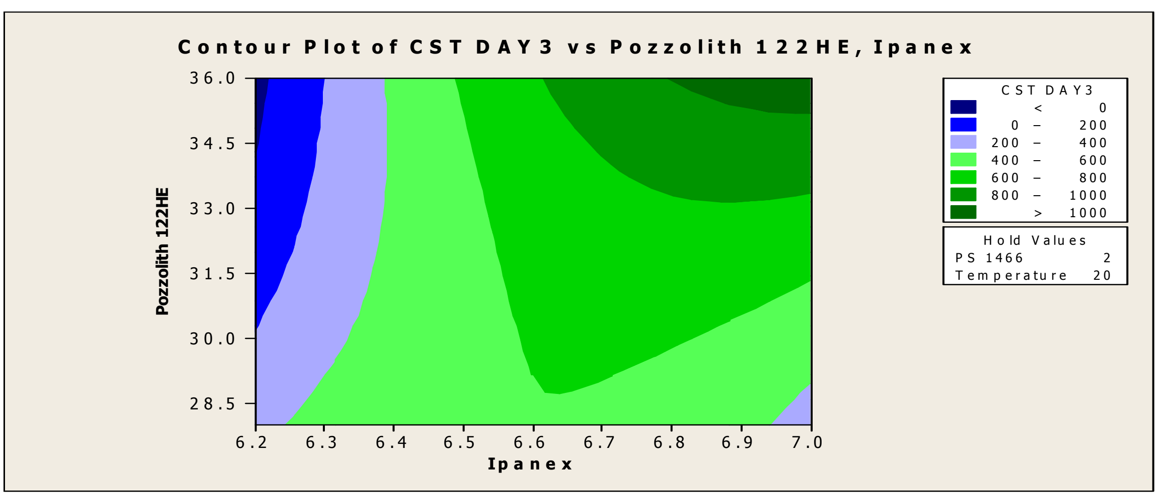


Figure C93: Contour Plot of CST Day 3 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

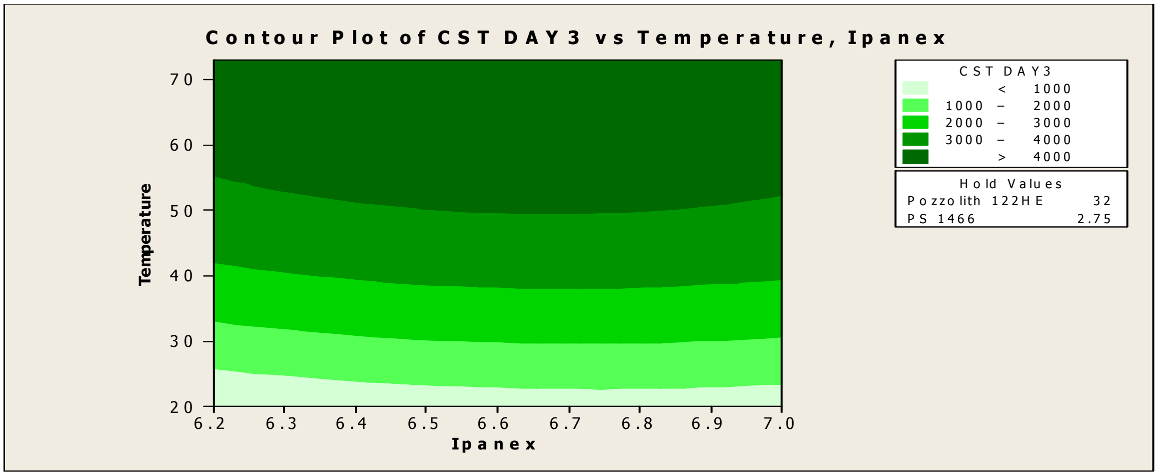


Figure C94: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

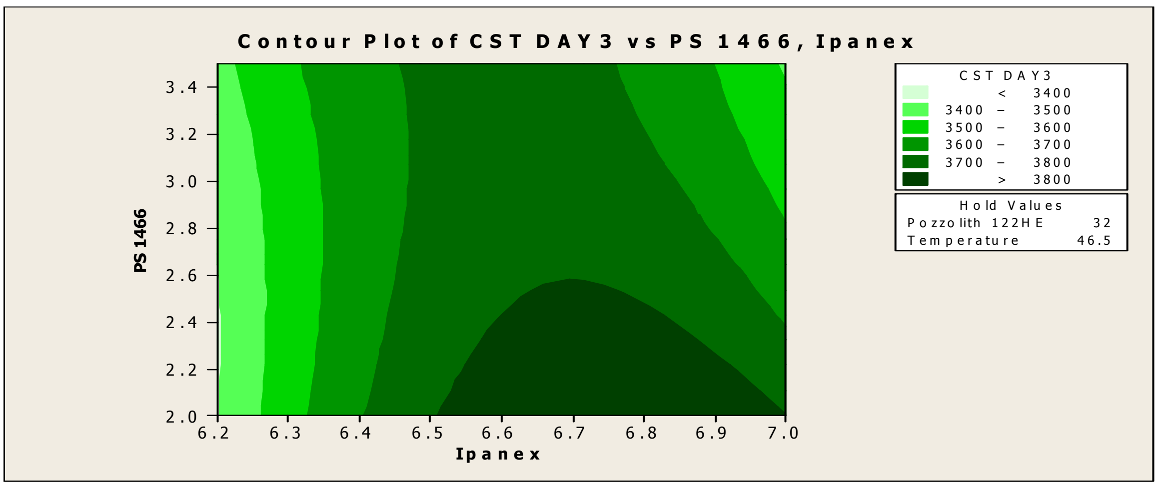


Figure C95: Contour Plot of CST Day 3 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

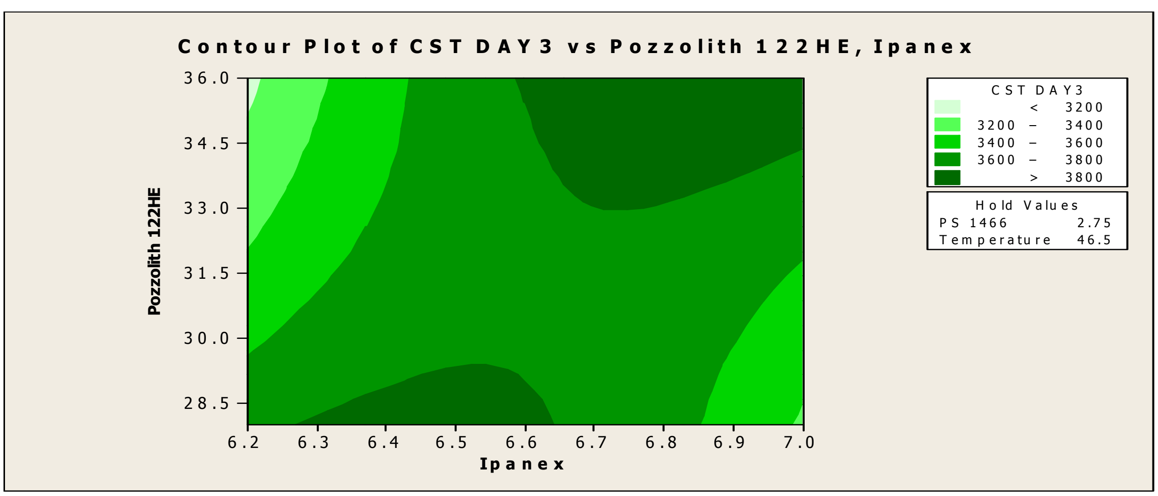


Figure C96: Contour Plot of CST Day 3 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

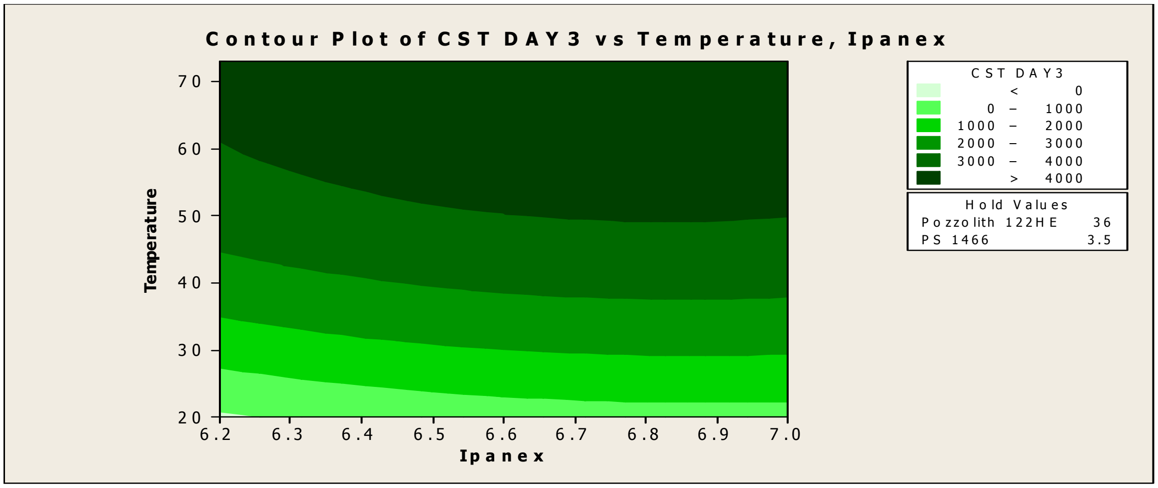


Figure C97: Contour Plot of CST Day 3 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

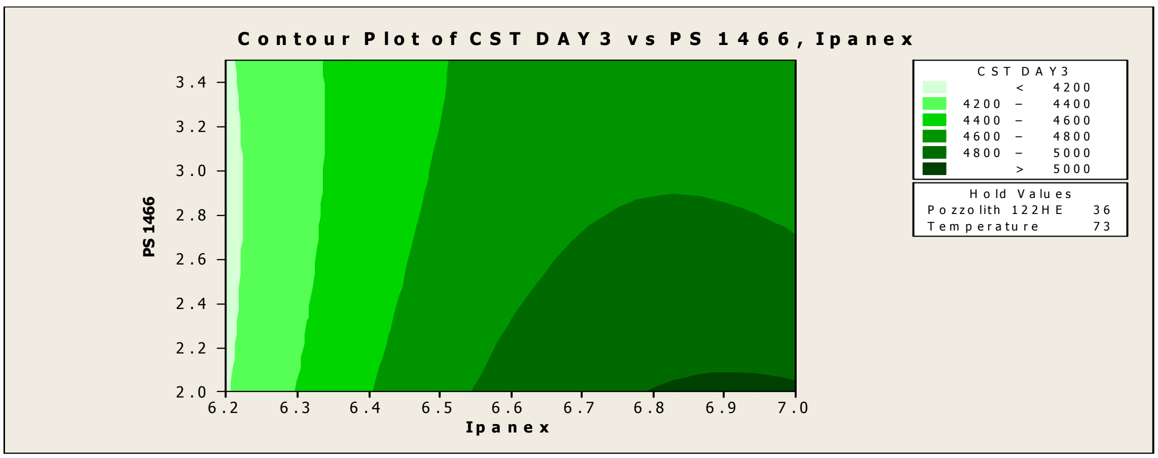


Figure C98: Contour Plot of CST Day 3 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

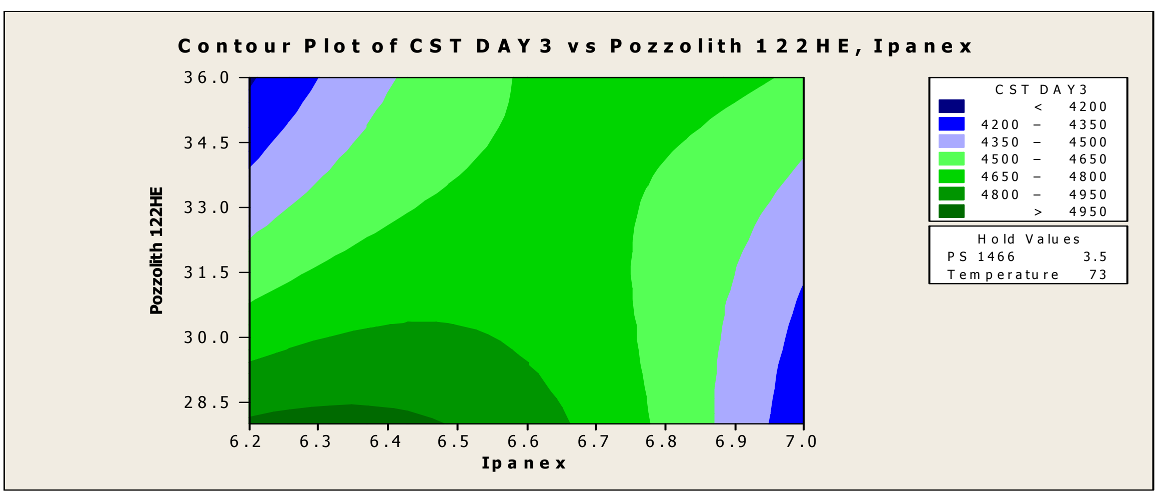


Figure C99: Contour Plot of CST Day 3 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

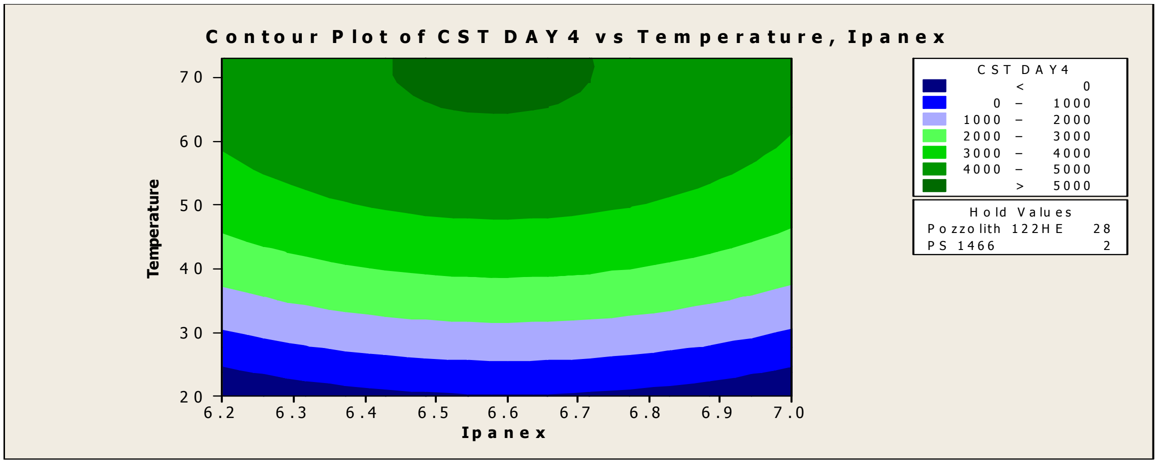


Figure C100: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

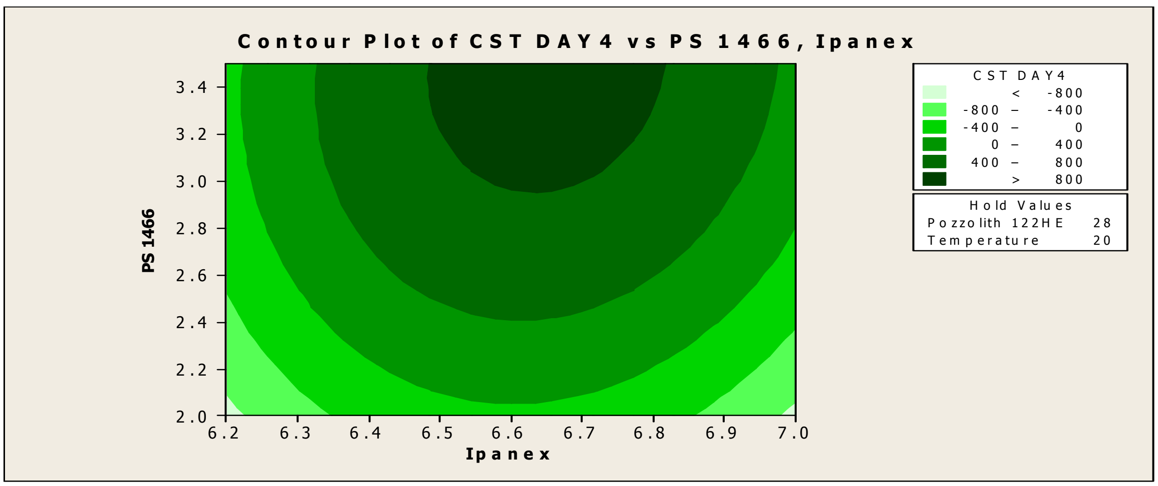


Figure C101: Contour Plot of CST Day 4 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their low settings

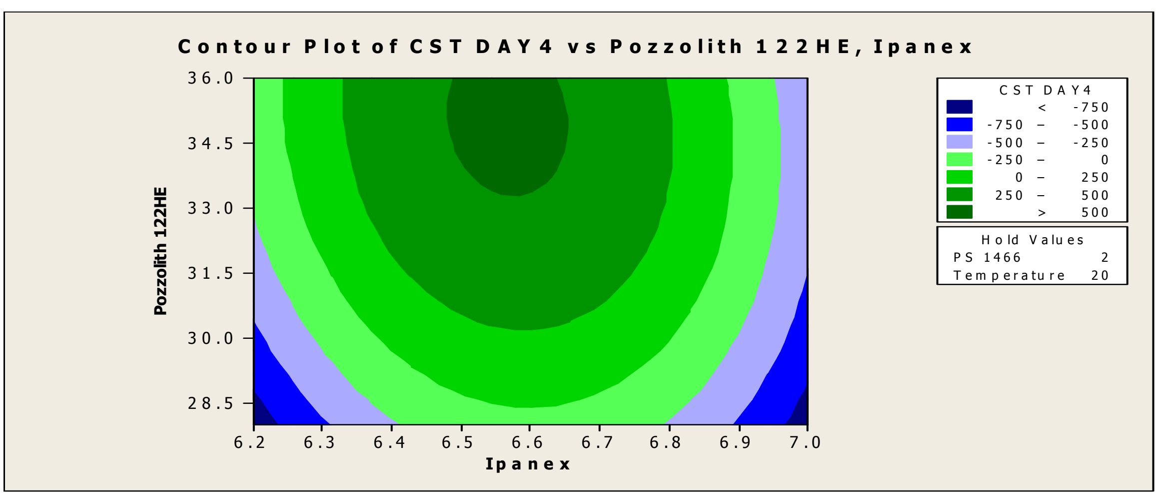


Figure C102: Contour Plot of CST Day 4 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their low settings.

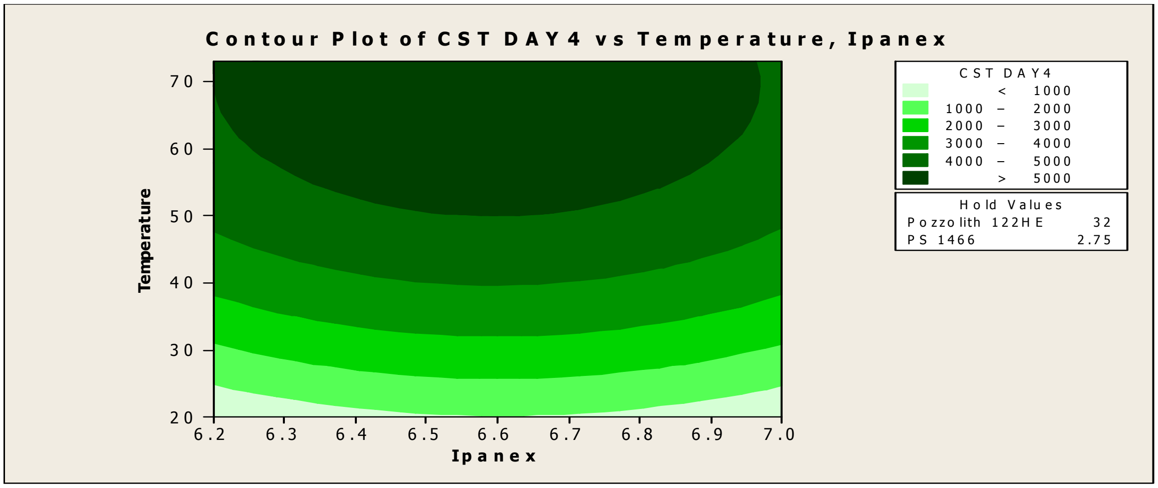


Figure C103: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

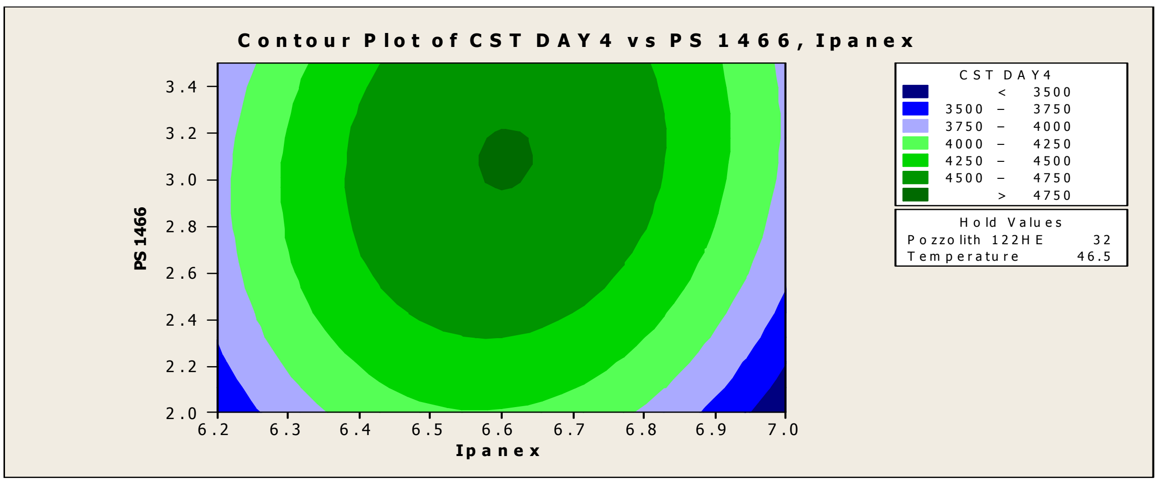


Figure C104: Contour Plot of CST Day 4 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

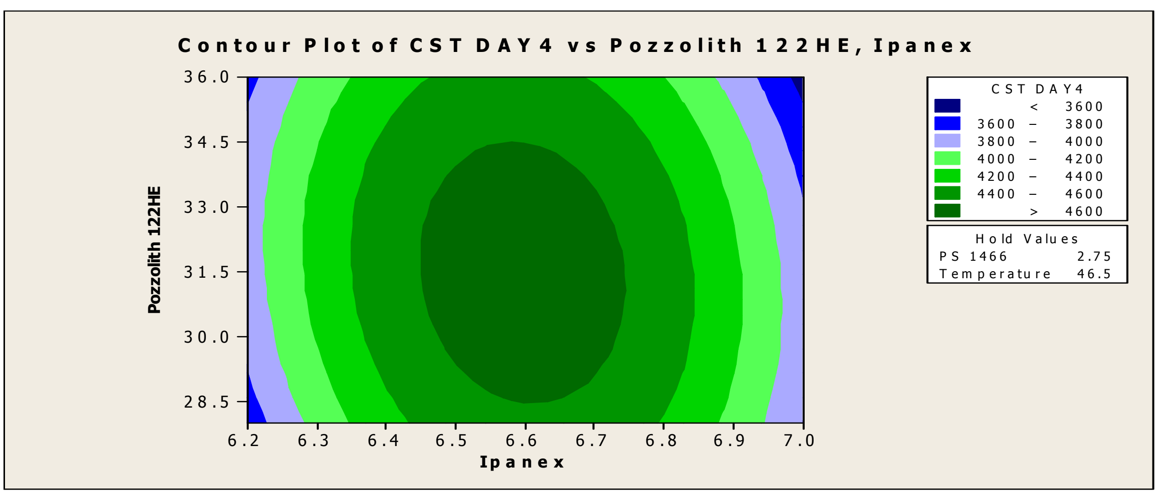


Figure C105: Contour Plot of CST Day 4 for Pozzolith 122HE vs Ipanex for the Mix 12 keeping all the other factors at their medium settings.

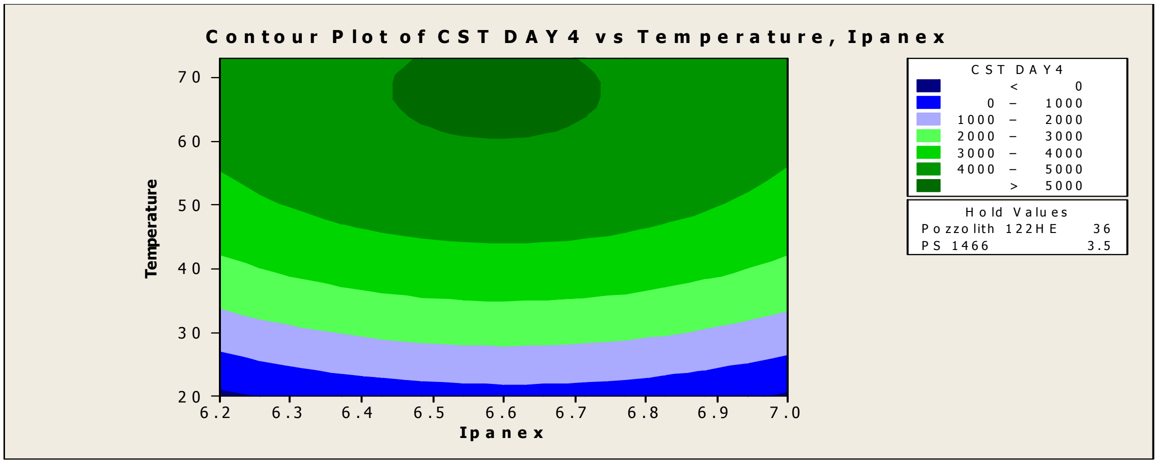


Figure C106: Contour Plot of CST Day 4 for Temperature vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

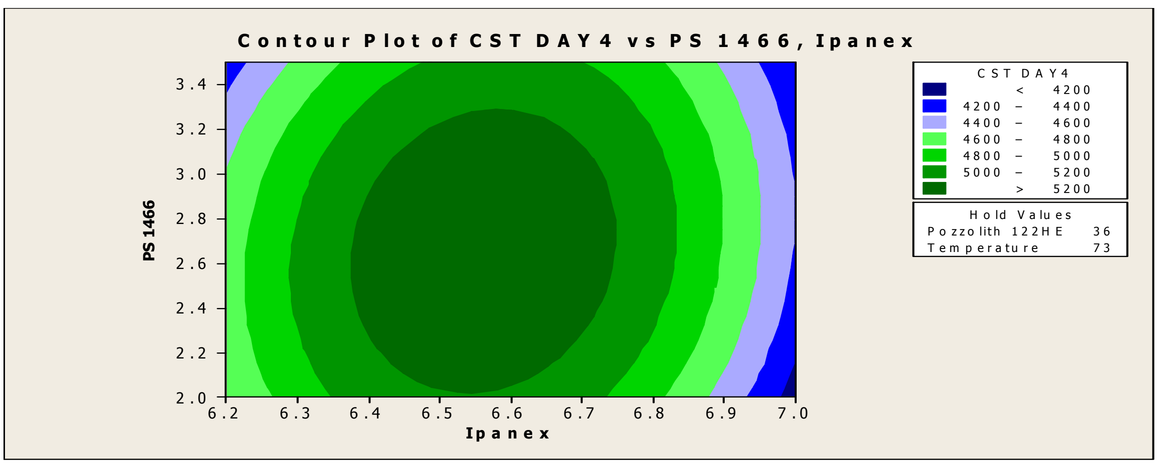


Figure C107: Contour Plot of CST Day 4 for PS1466 vs Ipanex for the Mix 12 keeping all the other factors at their high settings.

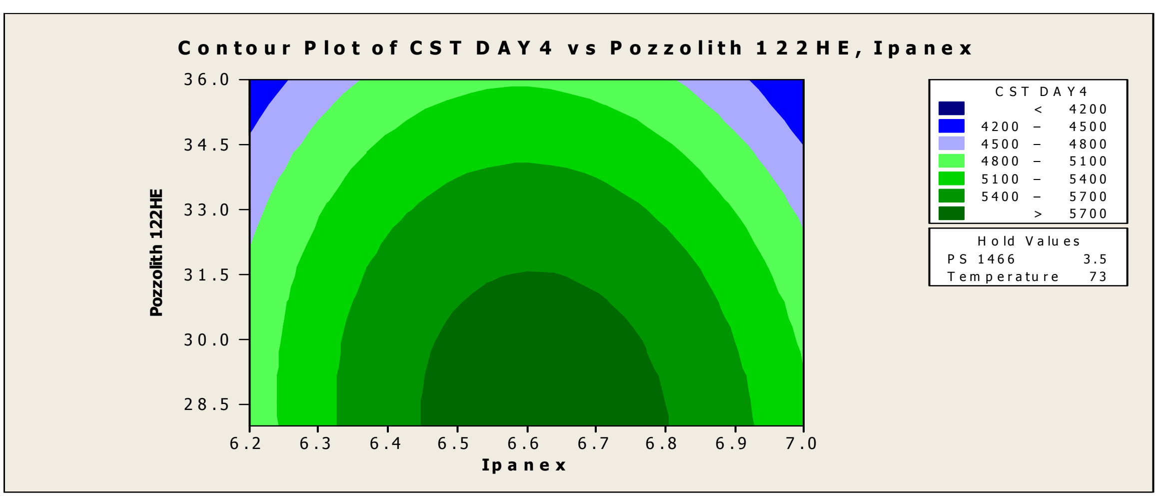


Figure C108: Contour Plot of CST Day 4 for Pozzolth 122HE vs Ipanex for the Mix 12 keeping all the other factors at their high settings.