# DETERMINATION OF THE BEST DRYING CONDITIONS FOR GELATIN BASED CANDIES

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**DETERMINATION OF THE BEST DRYING CONDITIONS FOR GELATIN BASED CANDIES**

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# ABSTRACT

## DETERMINATION OF THE BEST DRYING CONDITIONS FOR GELATIN BASED CANDIES

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In gelatin-based candies, consumers prefer hard and less sticky products. Stickiness and melting of the candies and hardening observed in the packages can bring out many consumer complaints. In order to serve desirable products to the consumers, stability of the moisture content during the shelf life is a significant parameter. And moisture content is controlled through the drying and conditioning step in candy manufacturing. Manufacturers usually use the same drying times for the candies of different weight that are produced for different consumer groups. And the same drying times could lead to some undesirable changes in the products. In this study, the objective was to determine the best drying and conditioning times for achieving the desired quality for jelly candies of different weights. Drying time (12, 16, 20, 24 h) in oven, conditioning time (0, 24, 48, 72 h), unit weight of the candies (2.5 g, 3 g, 6 g) were the variable parameters investigated. Physical parameters of the candies affected by the drying and conditioning time were investigated. For the candies, TPA (Texture Profile Analysis), moisture content, water activity, total soluble solid content (TSS), TD-NMR relaxometry experiments were performed. Drying time, conditioning time and unit weight were all found to be significant factors on textural properties ($p<0.05$). Also, harder texture was obtained when unit weight decreased under same conditions. The best drying time was found as 20 h for 2.5 g and 24 h for both 3 g and 6 g. Following drying, the best conditioning time with respect to unit weight was determined as 72 h for 2.5 g and 48 h for 3 g and 6 g candies. In addition, it was found that moisture content and total soluble solid content were correlated with the textural properties. It was concluded that moisture content, texture analysis and total soluble solid content could be used to check the quality of the products in routine production. Total soluble solid content as being strongly correlated with other parameters could solely be used as it is the quickest method among the others. It was also recommended that products could be taken out from the drying oven after % total soluble solid content reached and moisture reached approximately %79 and %18 respectively. Relaxation times of $T_1$ and $T_2$ were also found correlated with the physical parameters. Texture analysis provided an insight for the desirable texture profile before packaging and recommended hardness value was found between 500-550 N.The results of the study suggested that to achieve and keep the desired quality parameters during shelf life each unit weight candy should be dried separately.

Keywords: Keywords: Gelatin based soft candy; texture; moisture; drying; conditioning

# ÖZ

## JELATİN BAZLI YUMUŞAK ŞEKERLERİN KURUTMA KOŞULLARININ OPTİMİZASYONU

Kaya, Damla
Yüksek Lisans, Gıda Mühendisliği
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Jelatin bazlı şekerlerde en önemli kalite sorunları çoğunlukla su kaybından kaynaklanan sertleşme problemi, erime ve yapışma problemleridir. Paket içerisinde yapışık ürün, sertleşmiş şeker bu problemlerle ilgili alınan tüketici şikayetlerine örnek verilebilir. Şekerleme ürünlerinde tüketici beklentisini karşılayabilmenin en önemli koşulu nem dengesinin korunmasıdır. Jelatin bazlı yumuşak şekerlerde, nem dengesinin sağlandığı en önemli proses adımı fırın kurutma ve şartlandırma adımlarıdır. Üreticiler, farklı gramajlardaki ürünlerde aynı kurutma sürelerini kullanırlar. Ancak, kurutma sürelerinin aynı olması ürünlerde beklenmedik değişikliklere sebep olabilir. Projenin asıl amacı, şekerlemelerin istenilen yapıya ulaşılması için gereken minimum düzeydeki kurutma ve şartlandırma sürelerinin belirlenmesidir. Bu projede fırında kurutma zamanı (12,16,20,24 saat), şartlandırılmış odada bekleme süresi (0,24,48,72 saat) ve birim ağırlık (2.5 g, 3 g, 6 g) takip edilen değişken parametrelerdir. TPA (Doku Profil Analizi), nem analizi, su aktivitesi, TD-NMR, toplam kuru madde analizleri yapılarak bu değişken parametrelerin birbiri üzerine etkileri araştırılmıştır. Fırında kurutma zamanı, şartlandırılmış alanda bekleme süresi ve birim ağırlık, şekerin yapısal özellikleri üzerinde farkedilir etkiye sahiptir

($p<0.05$). Aynı kurutma koşullarında, birim ağırlık küçüldükçe daha sert yapısal özelliklerde şeker elde edilmektedir. Sonuçlar analiz edildiğinde, en optimum fırın kurutma süreleri 2.5 g jelli şekerler için 20 saat iken; 3 g ve 6 g şekerler için 24 saat olabilir. Şartlandırılmış odada bekleme süresi ile ilgili olarak ise 2.5 g şekerler 72 saate kadar ilgili koşullarda bekletilebilirken 3 g ve 6 g şeker numunelerinde 48 saat bekletildikten sonra yapısal özelliklerinde farkedilebilir değişiklik olduğu belirlenmiştir. Buna ek olarak, nem analizi ve toplam kuru madde sonuçlarının, yapısal özellikler ile ilişkili olduğu tespit edilmiştir. Sonuç olarak; korelasyon verilerine bakıldığında; normal üretimler esnasında toplam kuru madde, nem analizi veya tekstür analizleri kontrol metodu olarak kullanılabilir. Ancak, toplam kuru madde analizinin diğer parametrelerle olan korelasyonu daha iyi olması ile birlikte diğer methodlarla kıyaslandığında en hızlı metottur. Jelli şekerler, fırından çıkarıldığında yaklaşık %79 toplam kuru madde ve %18 nem değerlerine sahip olduklarında yapısal özelliklerinin istenilen değerler aralığında olduğu görülmüştür. T1 ve T2 değerleri de fiziksel parametrelerle ilişkilidir. Buna ek olarak, şartlandırılmış ortamın etkilerini etkili bir şekilde analiz etmek için ise tekstür analizi kullanılabilir ve sonuçlar analiz edildiğinde şekerlerin 500- 550 N aralığındaki sertlik değerlerini yakalamış olması tavsiye edilmektedir. Bu çalışmanın sonucunda, farklı gramajlara sahip şekerlerin, beklenen kalite parametrelerine ulaşabilmesi için gramaj bazında farklılaşan kurutma süreleri kullanılmalıdır.

Anahtar Kelimeler: Jelatin bazlı yumuşak şeker; tekstür, nem, kurutma, şartlandırma

To my beloved family

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# INTRODUCTION

## 1.1. Confectionery

Confectionary word is used for boiled sweets (hard candy, fondant, caramels, toffee, and fudge), aerated confections (marshmallows, nougats), gelled candies (toffee, gums, licorice, and pastilles), coatings (panned confections) and chocolate confectionary. The main ingredients of whole confectionary products are sugar and sugar derivatives. With changing confectionary types, ingredients are specified. Sucrose, glucose syrup, hydrocolloids (edible gums, gelatins, starches), cocoa butter, whipping agents (egg albumin, gelatins), starches are other ingredients that are used in confectionary (Edwards, 2000).

## 1.2. Candy

Candies are generally made of sugar, corn syrup, flavor, coloring agents. Also, gelling agents (starch, pectin gum arabic, and gelatin) can be used to make soft candies. Gelling agent is the most effective ingredient on textural properties of candies.

Most sugar confectionary is made by dissolving sugar in water and boiling glucose and sugar syrup in order to increase the concentration of the mixture (Edwards, 2000). In Table 1.1, different confectionery products are classified based on their moisture content and water activity values (Ergun, Lietha & Hartel, 2010). In the following section, different confectionery products will be discussed.

Table 1.1. *Moisture content and water content of different candy categories* (Ergun, Lietha, & Hartel, 2010)

| **Category** | ***Moisture (%)*** | $a_w$ |
|---|---|---|
| Hard Candy | 2-5 | 0.25-0.40 |
| Caramel, fudge, toffee | 6-18 | 0.45-0.60 |
| Chewy candies | 6-10 | 0.45-0.60 |
| Nougat | 5-10 | 0.40-0.65 |
| Marshmallow | 12-20 | 0.60-0.75 |
| Gummies and jellies | 8-22 | 0.50-0.75 |
| Jam | 30-40 | 0.80-0.85 |
| Fondants and creams | 10-18 | 0.65-0.80 |
| Chewing gum | 3-6 | 0.40-0.65 |
| Soft panned coating | 3-6 | 0.40-0.65 |
| Hard panned coating | 0-1 | 0.40-0.75 |
| Tablets and lozenges | 0-1 | 0.40-0.75 |

### 1.2.1. Hard Candy

Hard candy is a glassy product (Lees & Jackson, 2012). Lollipops, candy canes are the example of hard candies. The main ingredients are glucose (corn) syrup, sucrose and other sugars, colors, flavors and acids. Hard candy production involves dissolving the sugar and glucose syrup in water first and then boiling the mixture to high temperatures (147 – 152 ◦C) to achieve a low moisture content (2- 5%) (Ergun et al., 2010). Glucose syrup is the most important ingredient for the hard candy formulation and is responsible for the hygroscopic properties of the product. Moisture can be trapped in the product after boiling and vacuum can be applied in order to remove the excess moisture. Flavors, colors and acids are added to candy before molding (Lees & Jackson, 2012). Following molding, candies are cooled to transform into the glassy state. Glass transition temperature is related with the stability of the candy. High molecular weight sweeteners decrease moisture content and increase the $T_g$ values. Glass transition temperature determines the hardness of the candies. When $T_g$ value increases, candy becomes harder, sharper and more brittle. So, optimization of glass transition temperature and water content are the two important parameters of hard candy's quality and also the shelf life (Ergun et al., 2010).

An important problem affecting the quality of the hard candies is the crystallization which is also known as 'graining' in confectionery industry. This problem is observed when low amount of glucose syrup and high sucrose concentrations are used and also when the candies are stored at a temperature higher than $T_g$.

### 1.2.2. Jelly Candy Ingredients

#### 1.2.2.1. Sucrose

Sucrose is also named as the table sugar and known as the most common food sweetener. Sugar beet and sugar cane are the main sources of sucrose (Edwards, 2000). Sucrose is a disaccharide that is composed of glucose and fructose (Figure1.2).

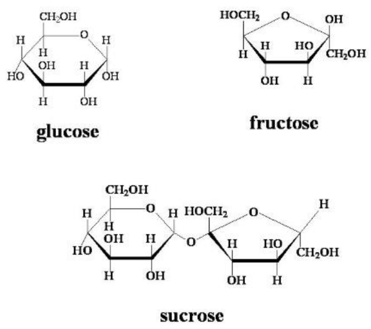


*Figure 1.1.* Chemical structure of sucrose ("*Sucrose," 2016*)

Sucrose is a non-reducing sugar because it does not have a free carbonyl group. Sweetness of sucrose could be considered as 1 and other sweetener's sweetness value are reported relative to sucrose. Relative sweetness values for other sweeteners are given in Table 1.3 (IFIC Foundation, 2018).

*Table 1.2*. Comparative sweetness value of sweetener ((Confectionery Products Handbook , 2013)

| **Sweetener** | ***Sweetness compared to sucrose*** |
|---|---|
| Dextrose | 0.7 |
| Glucose syrup | 0.51 |
| Fructose | 1.2 |
| Invert Sugar | 0.9 |
| Maltose | 0.5 |
| Aspartame | 100-400 |
| Sorbitol | 0.5 |
| Xylitol | 1 |
| Mannitol | 0.7 |

Solubility of sucrose is 2.0047 g sucrose/g water at 20 °C and molecular weight (MW) is 342.297 g/mol (IFIC Foundation, 2018; Mathlouthi & Reiser, 1995). Due to its high solubility and molecular weight, it has significant effect on decreasing the water activity. Increasing sucrose concentration decreases water activity. That is why sucrose is also known as the *water activity depressor* (Ergun et al., 2015).

Sucrose can be hydrolyzed into invert sugar with the presence of acids and high temperatures. The inversion process yields same amounts of glucose and fructose and these are more soluble than sucrose. Invert sugar is desirable in food industry because of increasing solubility (Vaclavik & Christian, 2007).

During hard candy production, increasing cooking duration causes inversion of sucrose into glucose and fructose because of having lower $T_g$ values than sucrose. Inversion is known to lower the $T_g$ and reduce the stability of the candy (Ergun et al., 2010).

Sucrose also helps in the gelation of pectin and starch-based jellies (Mathlouthi, 2001). Also, gelatin gels' chain association can increase in the presence of sugar. Sugar helps to destabilize polysaccharide gel networks (Burey et al., 2009).

#### 1.2.2.2. Corn Syrup

Corn syrup is called as glucose syrup and obtained also basically from corn starch. Caloric value on dry basis is 4 cal/g (Varzakas, n.d.).

In confectionary products, glucose (corn) syrup is the one of the main ingredients with sucrose. Glucose syrup is obtained by hydrolysis of starch in the presence of acid. This syrup is specified in terms of *dextrose equivalence* (DE) which shows the efficiency of the hydrolysis. Glucose syrup can be obtained from many different sources of carbohydrate; but, mostly maize, potato or wheat starch are used. Generally, 42 DE glucose syrup is used in confectionary products. However, 42 DE syrup's sweetness is less as compared to sucrose (Edwards, 2000). Sweetness, viscosity and nutritional value are related with the DE value. Sweetness value of 42 DE syrup is 50; whereas for 63 DE glucose syrup sweetness value is 70. Viscosity becomes higher with lower DE values and consequently this effects the products' taste and texture (Varzakas, n.d.).

Also, in food industry, high fructose corn syrup can be used. This is obtained by conversion of dextrose to fructose by glucose isomers by using enzymes (Edwards, 2000).

Corn syrup (especially high DE corn syrups and high fructose corn syrup), invert sugar, fructose, glucose, and polyols (sorbitol, isomalt etc.) are also known as humectants which helps to keep confectionary moist. High water content or high DE corn syrup content can cause stickiness that is related with moisture gain (Ergun et al., 2010).

#### 1.2.2.3. Gelatin

Gelatin consists of 14% moisture and 2% ash and 84% protein that composed several amino acids (mostly glycine, proline and hydroxyproline). It is obtained from collagen and protein polymer with acid or alkaline hydrolysis. Collagen could be obtained from hide, bones, skin. Bovine and porcine bones are mostly used. However, some consumers avoid bovine and porcine based gelatin. Poultry and fish are new raw materials for gelatin and they can be preferred more (Burey et al., 2009).

*Acid- conditioned type A* and *alkaline-conditioned high-Bloom type B* are the types of gelatin. Gel strength is the most important parameter for gelatin. Bloom is a kind of test for measuring gel strength. Bloom values range from 50 to 300 gr. Types of gelatin can be classified as high-Bloom (200 g-300 g), medium-Bloom (100 g-200 g) and low-Bloom (50 g-100 g). From low-bloom gelatin to high bloom gelatin, gelation time decreases, gel strength becomes stronger (Schrieber & Gareis, 2007).

Gelatin is a thermoreversible gel and there are two important steps during gelatin gelation, setting and ageing. In setting step, network formation occurs and gel strength is developed in ageing step, the typical gelatin gelation is shown in Figure 1.4 (Burey et al., 2009). Thermal history (rate of cooling or heating) of the process is important parameter during gelatin gelation. Stronger gels can be obtained with low cooling rates (Fonkwe, et al., 2003).

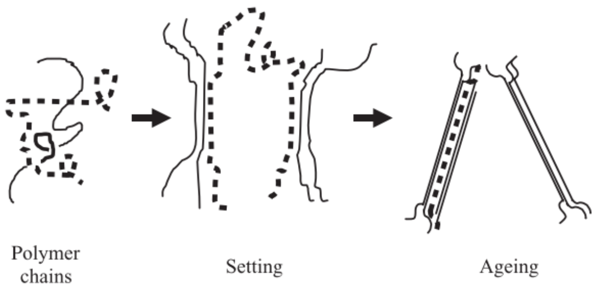


*Figure 1.2.* Gelatin gelation formation (Burey et al., 2009)

Gelatin is used as mainly the gelling agent on jellies, fruit gums etc. Besides gelling agent, texturizing, foaming, stabilization, thickening, water binding are the other different properties of gelatin (Reinhard Schrieber and Herbert Gareis, 2007). In Table 1.3, comparison of gelling agents' physical properties is shown.

Table 1.3. *Physical Properties of Gelling Agents Used in Confectionery Products* (Reinhard Schrieber and Herbert Gareis, 2007)

| Gelling Agent | Gel Formation | Thickening Effect | Transparency | pH Stability |
|---|---|---|---|---|
| **Gelatin** | High | High | High | Middle |
| **Modified Starch** | High | High | Low | Middle |
| **Native Starch** | High | High | Low | Low |
| **Pectin** | High | Middle | High | Low |
| **Gelatin** | High | High | High | Middle |

### 1.2.3. Jelly/ Soft Candy

Jellies, pastilles and gums belong to the same classification in confectionary products. They have around 20% moisture content (Lees & Jackson, 2012). Jelly candies have gelling agents such as gelatin, pectin, starch, gum arabic, sucrose and glucose syrup and as the additional ingredients; food acids, flavoring, coloring (Burey, Bhandari, Rutgers, Halley, & Torley, 2009).

In Table 1.2, effect of various gelling agents on the properties of soft candies are summarized (Lees & Jackson, 2012). Textural properties of soft candies are affected from the type of the gelling agent significantly (Ergun et al., 2010). To make jellies, sugar, glucose syrup and gelling agent are dissolved in water and then the mixture is boiled. After boiling, concentrated mixture (slurry) is deposited, dried, coated and packaged as explained in Figure 1.1 (Edwards, 2000). Especially, jelly candies are deposited in starch molds that helps to candy to lose its moisture and creates a skin on the surface of candy. Formation of the skin prevents deformation of the candy when removed from starch mold (Ergun et al., 2010; Edwards, 2000). After depositing, the filled trays are taken to oven. Time that products are kept in the oven is called as *stoving time.* After stoving cycle, samples could be waited in *Work In Progress (WIP)* area before packaging to gain textural properties. Actually, drying parameters (stoving time, temperature and Rh) can change according to selected gelling agent (pectin, starch, gelatin etc.). Drying time on oven changes from 24 h to 72 h depending on the type of the candy, size of the candy, type of the gelling agent and the desired moisture content (Ergun et al., 2010). Stoving time and the temperature for gelatin-based candies should be lower than the starch based or pectin based jelly candies to prevent gelatin browning and let gelatin based candies obtain the desired texture properties faster. Also, the rate of drying affect the texture of the candy (Edwards, 2000). Rate of drying have a direct effect on the skin formation. When the skin forms very fast, the surface of the candy becomes too hard.

Table 1.4. Effect of different gelling agents on the physical properties for soft candies (Lees & Jackson, 2012).

| **Agents** | ***Gum Arabic*** | ***Starch*** | ***Gelatin*** | **Pectin** |
|---|---|---|---|---|
| Percent Use in Confectionery (%) | 35-45 | 9-12 | 5-12 | 1-11 |
| Temperature of Solution ($^{o}$C) | 25 | 71-82 | 60-65 | 93-100 |
| Sweetener Ratio Sucrose/Glucose Syrup Ratio | 66/33-50/50 | 66/33-50/50 | 66/33-50/50 | 60/40-50/50 |
| Setting Temperature ($^{o}$C) | 20-37 | 20-37 | 20-37 | 71-82 |
| Time in Molds (hour) | 36-72 | 12-36 | 12-24 | 6-12 |
| Total Solids (%) | 68-70<br>85+ | 72-78<br>78+ | 72-78<br>78+ | 76-78<br>78+ |
| Texture | Smooth, Hard, Bite | Short | Tough-long | Clean Bite |
| pH during Cooking (Recommended) | 5.0-6.0 | 5.0-6.0 | 5.0-6.0 | 4.0-5.0 |
| Percent Acid for Flavoring (%) | 0.3-0.45 | 0.2-0.4 | 0.2-0.3 | 0.4-0.7 |
| Final pH of Product | 4.2-5.0 | 4.2-5.0 | 4.4-5.0 | 3.2-3.5 |

During drying in oven, moisture migration, occurs between starch - candy and the air as described below.

(1) Migration of moisture from the candy into the starch bed;

(2) Migration of moisture from the candy into the air;

(3) Migration of the moisture from the starch bed into the air, depending on the %RH of the air (Troutman, Mastikhin, Balcom, Eads, & Ziegler, 2001).

Dehydration is a crucial parameter in candy textural profile. Highest dehydration rate of candy is usually the beginning of the drying in starch mold. As stated above, water inside a candy is transferred and consequently increases the solid content (Delgado & Bañón, 2015).

Mostly, jelly candies' water activity changes between 0.5 and 0.7 and the final product should achieve at least 75% total soluble solids to prevent mold growth. (Ergun et al., 2010)

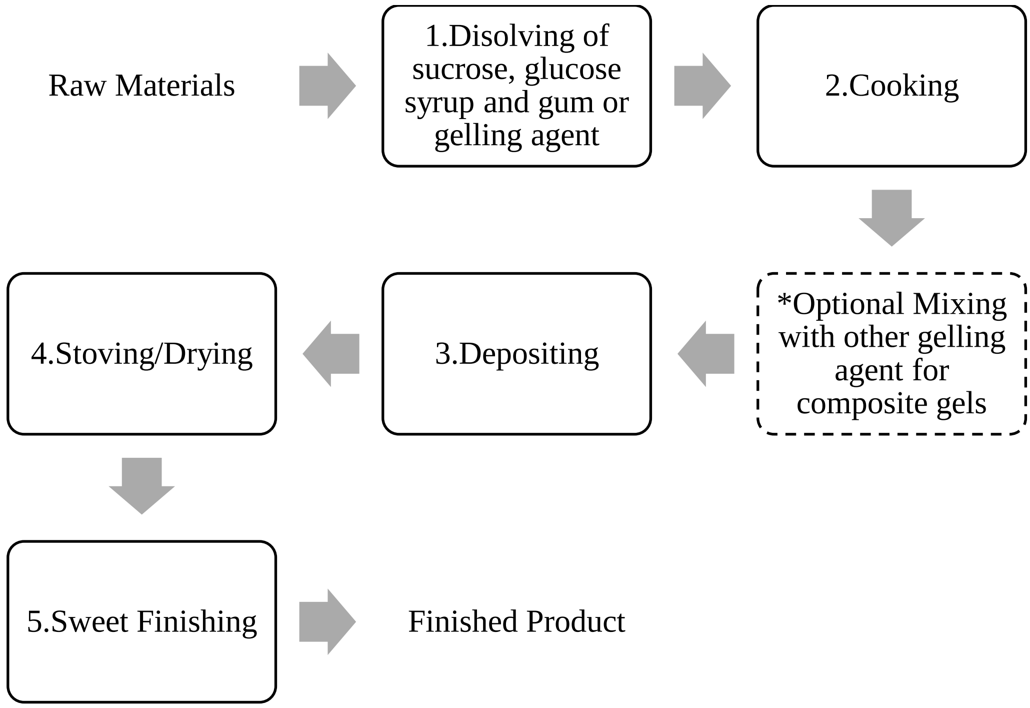


*Figure 1.3.* Basic flow chart of jelly candy process (Burey et al., 2009)

As stated before the main ingredients of jelly candies are sucrose, glucose syrup, gelatin and with food acids, flavoring, colorings (Burey et al., 2009).

### 1.2.4. Physical Properties of Soft Candies & Quality Characteristics

#### 1.2.4.1. Total Solid Content

Total soluble solid content shows the total soluble solids amount and can be measured with a refractometer which reports the refractive index (Delgado & Bañón, 2015). Substance refractive index is the ratio of velocity of light in vacuum to velocity of light in the sample (Edwards, 2000)

For pure sucrose measurement, refractive index is proportional to concentration directly. However, confectionary products have mixture of sucrose and other sweeteners. So, the refractive index of the solution depends on ingredients the relative ratios. This means that reading of refractometer that is used in confectionary can give slightly erroneous result. In most confectionary application, Brix is accepted to understand total soluble solid concentration without using a correction factor (Ergun et al., 2010)

#### 1.2.4.2. Moisture Content

Moisture content is the amount of bound water in material. Moisture content is related with boiling point relationship of sugars. Sorption isotherm indicates the moisture content at constant temperature and nature of the water, free or bound (Ergun et al., 2010).

Moisture is very important parameter in order to gain textural properties and to determine confectionary's shelf life and quality parameters. Confectionery has lower moisture contents as compared to other food products (Ergun et al., 2010). Variation in moisture content causes quality variation such as stickiness, lack of body, chewiness, hardness, poor handling on cutting machines (Burey et al., 2009). For example, high water content in hard candies may cause stickiness, softer texture, and faster flavor loss; however, low moisture content can give hard and brittle texture. Moisture content ranges are shown in Table 1.4.

Table 1.4. *Moisture content and water content of different candy categories*

| **Category** | ***Moisture (%)*** | $a_w$ |
|---|---|---|
| Hard Candy | 2-5 | 0.25-0.40 |
| Caramel, fudge, toffee | 6-18 | 0.45-0.60 |
| Chewy candies | 6-10 | 0.45-0.60 |
| Nougat | 5-10 | 0.40-0.65 |
| Marshmallow | 12-20 | 0.60-0.75 |
| Gummies and jellies | 8-22 | 0.50-0.75 |
| Jam | 30-40 | 0.80-0.85 |
| Fondants and creams | 10-18 | 0.65-0.80 |
| Chewing gum | 3-6 | 0.40-0.65 |
| Soft panned coating | 3-6 | 0.40-0.65 |
| Hard panned coating | 0-1 | 0.40-0.75 |
| Tablets and lozenges | 0-1 | 0.40-0.75 |

There are lots of numerous methods to measure amount of water in food. Loss of drying, Karl Fischer titration, distillation and refractometer are mainly used in the confectionery industry (Ergun et al., 2010).

#### 1.2.4.3. Water Activity

Water activity is an indicator of the relative vapor pressure of the food and is used to understand micro stability, texture and water migration during storage. Relative vapor pressure could be defined as the ratio of materials vapor pressure to pure water vapor pressure.

Water activity is based on the number and the size of molecules dissolved in water. It is affected by presence of dissolved sugar, other sweeteners (polyols), humectants in confections. When the ingredients have low molecular weights and high solubility, water activity decreases. In confectionery products, proteins and polysaccharides (starch, gums) can be used. These ingredients are high molecular weights and have little effect on decreasing $a_w$. However, humectants such as corn syrup, invert syrup, fructose, glucose polyols such as isomalt, sorbitol, maltitol, glycol can reduce the water activity of a product (Ergun et al., 2010).

Also, water activity helps to understand microbiological susceptibility of the food products. In Table 1.6, confectionary products have been classified based on their water activities (Ergun et al., 2010).

Table 1.5. *Water activity range of confectionery products*

| ***Water activity range*** | ***Microorganisms that can grow*** | ***Confections*** |
|---|---|---|
| > 0.88 | Normal bacteria and pathogens, many yeasts | Ganache, very soft fondant |
| 0.80-0.88 | Normal molds, some yeasts | Soft fondant, soft jellies, etc. |
| 0.70-0.80 | Molds, yeasts | Fondant, fudge, jellies, grained nougats, marshmallow, etc. |
| 0.60-0.70 | Osmophilic yeasts, some molds | Fudge, fondant, hard jellies, nougat, soft caramel, etc. |
| <0.60 | None | Caramel, toffee, jellies, gum, hard candy, chocolate, etc. |

#### 1.2.4.4. Texture

Texture is an important factor in consumer sensory acceptance. Food texture can be evaluated with sensory and instrumental methods of analysis. Sensory methods of analysis are subject to a wide variability as expected, though this variability can be reduced by using trained assessors. It is sometimes preferable to use instrumental methods of assessing food texture rather than sensory analysis because they can be carried out under more strictly defined and controlled conditions. In instrumental methods, mechanical characteristics of food are divided to hardness, cohesiveness, viscosity, elasticity, adhesiveness, chewiness, gumminess, stickiness (Figiel & Tajner-Czopek, 2006). Types of texture analyses are compression, puncture/penetration, tension/extension, shearing cutting, adhesion, extrusion and bending. Compression test and penetration are generally used in confectionery products.

Penetration tests use small cylinder/ball/cone probes. Samples have a small surface area for testing and penetration tests are more affected by the non-uniform product structures. A penetration test produces a hole in the product and does not allow the product to recover like a small deformation compression test. However, in compression test, larger cylinder probes are used. TPA (Texture Profile Analysis) test is two cycle compression test; but, size and the irregular shape of confectionery gels may affect their TPA values thus it is important to prepare standardized samples (Delgado & Bañón, 2015).

In a penetration test, depth of penetration (or the time required to reach a certain depth) is measured under a constant load. This test causes irreversible changes in the sample and is commonly used in the testing of fresh fruits and vegetables, cheese, confectionery and the spreadability of butter and margarine. Penetration tests have also been used extensively for testing the rigidity of gels.

As a working principle for penetration, once the probe triggers on the surface it then proceeds to penetrate to a depth of 2 mm within the sample. At this point the force value is recorded and taken as a measure of 'hardness' of the sample. The probe then withdraws from the sample at which point the maximum force to withdraw or 'stickiness' is recorded. The curvature of the plot appears to indicate flow of the sample as the probe is penetrating to the required depth. An increase in hardness of the sample would give an increase in the relative force values with an anticipated change in curvature as flow (or 'chewiness') decreases.

Stickiness and hardness affect product quality directly. More sticky and harder surface of the candies can be related with moisture and water activity relationship. Water activity difference cause moisture migration in products. When water activity’s difference is large, moisture migration may be rapid. If the water activity of the product is greater than the environment’s water activity, moisture migrates from candy to package or environment, so water activity decreases and increases hardness. On the contrary, candies that have low $a_{w,}$ candy takes moisture from environment and surface of the candy becomes wetness, increases stickiness (Ergun et al., 2010).

**1.2.5. Time Domain NMR Relaxometry (TD-NMR)**

Magnetic resonance imaging (MRI) and Nuclear Magnetic Resonance (NMR) are non-destructive characterization techniques which are utilized to determine food quality (Kirtil, Cikrikci, McCarthy, & Oztop, 2017). Magnetic resonance imaging (MRI) is a more advanced approach of NMR relaxometry and with the help of MRI, images of the internal structure could be obtained without any disruption to the sample leading to utilization of MRI in analysis of soft tissues for medical purposes (Kirtil & Oztop, 2016a). MRI enables researchers to visualize the interior of the samples without destruction on a macroscopic scale (Kirtil, Cikrikci, et al., 2017). The images in MRI are attained with the help of spatial encoding (Kirtil & Oztop, 2016a). On the other hand, spatial information is not obtained for the NMR Relaxometry and signal comes from the whole sample although it enables to differentiate signals which comes from different compartments with changing protons such as cellular organelles (Kirtil & Oztop, 2016a).

Since foods are good examples to the chemically and structurally heterogeneous systems, various contributions to the NMR signal is possible due to the changes in molecular mobility (Kirtil, Cikrikci, et al., 2017). These changes in NMR signal could be explained with two main variable: longitudinal ($T_1$) and transverse ($T_2$) relaxation time (Kirtil, Cikrikci, et al., 2017).

For the $^1$H NMR measurements, firstly sample is placed in external magnetic field than this field is disturbed with another magnetic signal which is perpendicular to the primary one (Kirtil & Oztop, 2016b). Afterwards, relaxation of the signal that comes from the sample to its initial state is measured (Kirtil & Oztop, 2016b). Longitudinal relaxation time ($T_1$) is also called as spin–lattice relaxation time and it refers to the time which is necessary for spins to realign themselves along the axis of the external magnetic field (Kirtil & Oztop, 2016a). On the other hand, transverse relaxation time

($T_2$) is also called as spin-spin relaxation time and it refers to the time that is necessary for transverse magnetization to decay to the equilibrium value and reach zero (Kirtil & Oztop, 2016a).

NMR is valuable tool since it makes possible the characterization of mobility of protons and variations of conformations through the biopolymer chains through the time constants longitudinal relaxation time ($T_1$) and transverse relaxation time ($T_2$) (Ozel, Cikrikci, Aydin, & Oztop, 2017).

For instance, from the previous studies, it was known that $T_1$ (spin-lattice relaxation time) is highly rely on the mobility of protons which comes from the water component of the gel matrices (Pocan, Ilhan, & Oztop, 2019b). For this reason, it could be concluded that, $T_1$ relaxation time might be considered as valuable tool to detect the moisture distribution of food samples (Pocan et al., 2019b). Longitudinal relaxation time ($T_1$) was utilized in many studies for the analysis of food systems such as effect of microwave heating on starch-water interactions (Ozel, Dag, Kilercioglu, Sumnu, & Oztop, 2017), impact of pectin methyl esterase and $CaCl_2$ infusion on mangoes (Kirtil et al., 2014), effect of D-Psicose addition on gelatin based soft candies (Pocan et al., 2019b) and moisture migration in soft-panned confections during aging (Troutman et al., 2001).

$T_2$ (spin-lattice) relaxation time is also important parameter to deduce water content, interaction of water with surrounding molecules and physical properties of water (Kirtil et al., 2014). Both multi-exponential and mono-exponential approach could be utilized to interpret the transverse ($T_2$) relaxation times (Pocan et al., 2019b). For example, $T_2$ relaxation time of food products having multi-compartment nature such as gluten free cakes (Yildiz, Guner, Sumnu, Sahin, & Oztop, 2018), thawed and frozen mangoes (Kirtil et al., 2014) and gelatin based soft candies (Pocan et al., 2019b) were analyzed with the help of multi-exponential approach while emulsion stabilization properties of some gums like gum tragacanth (Pocan, Ilhan, & Oztop, 2019a) and characterization of capsaicin emulsions (Akbas, Soyler, & Oztop, 2016) were explored with the help of T2 relaxation times that expressed as mono-exponential.

### 1.3. Objective of Study

**Problem definition:**

Consumers prefer hard and less sticky jelly products. Stickiness of the soft candies in the packages and also melting of the candies bring many consumer complaints. To understand the reason of that problem, processing conditions of the jelly candies should be examined and adjustments be done if necessary. In this study, drying process conditions, in which the stickiness and melting problems could initiate from, were explored.

**Hypothesis:**

Moisture content, water activity and total soluble solid content are important parameters affecting the textural properties of candies. Stickiness and hardness strongly depend on these parameters. Since stoving time and conditioning time effect drying, and drying rate is significantly affected from the surface area, testing these 3 parameters at different levels for different surface area products (these also have different weights) can provide insight on selecting the desired quality candies that will not suffer from softness, hardness or stickiness inside the package.

**Objective of the Study:**

In this study, the objective is to determine the best stoving and conditioning times for achieving the desired quality for soft candies of different weights. Moisture content, water activity, total soluble solid content, textural, and NMR Relaxometry experiments were performed for samples stored at different stoving and conditioning times.

# CHAPTER 2

# MATERIALS AND METHODS

## 2.1. Materials

In soft candy production; sucrose, glucose syrup (DE: 42), gelatin (Bloom:240), coloring agent, flavoring, citric acids are the ingredients.

## 2.2. Methods

All candies were produced the facilities of Mondelez International plant in Gebze, Kocaeli,Turkey.

### 2.2.1. Gelatin Based Soft Candy Production

Firstly, gelatin (240 bloom) solution was prepared around 65 $^{o}$ C in the gelatin preparation tank. In weighing tank, gelatin solution, glucose syrup and sucrose were mixed according to the recipe while heating to dissolve sugar crystals. After, slurry (mixed solution) was cooked at approximately 100 $^{o}$ C, it was fed into vacuum chamber to adjust solid content, remove air bubbles and eliminate excess amount of water. Solid content is generally between 75 to 80 %. Flavor, acid and coloring agent were added into slurry immediately prior to molding to minimize time & temperature effect on volatile flavor components and avoid inversion of the sucrose. Then, the slurry was molded into starch trays. The filled trays were taken to oven at approximately 30 ºC and 45 % RH to condition and gain the desired texture. Time that products were kept on oven is called *stoving time*. When the products achieved desired total soluble solid content and texture, drying was completed. After drying, the trays were inverted to remove the jellies from the molds and were brushed gently to eliminate starch powder. Finally, the jellies were coated with oil using a drum to avoid a sticky surface. After coating, products were transferred to cases and kept on conditioning area before packaging. Conditioning room temperature was around 20 ºC and <65 % RH. In jelly candy technology, *conditioning* is termed as WIP *(Work In Process).* In following sections, *WIP* term is used instead of conditioning. Production flow chart is also given in Fig. 3.1.

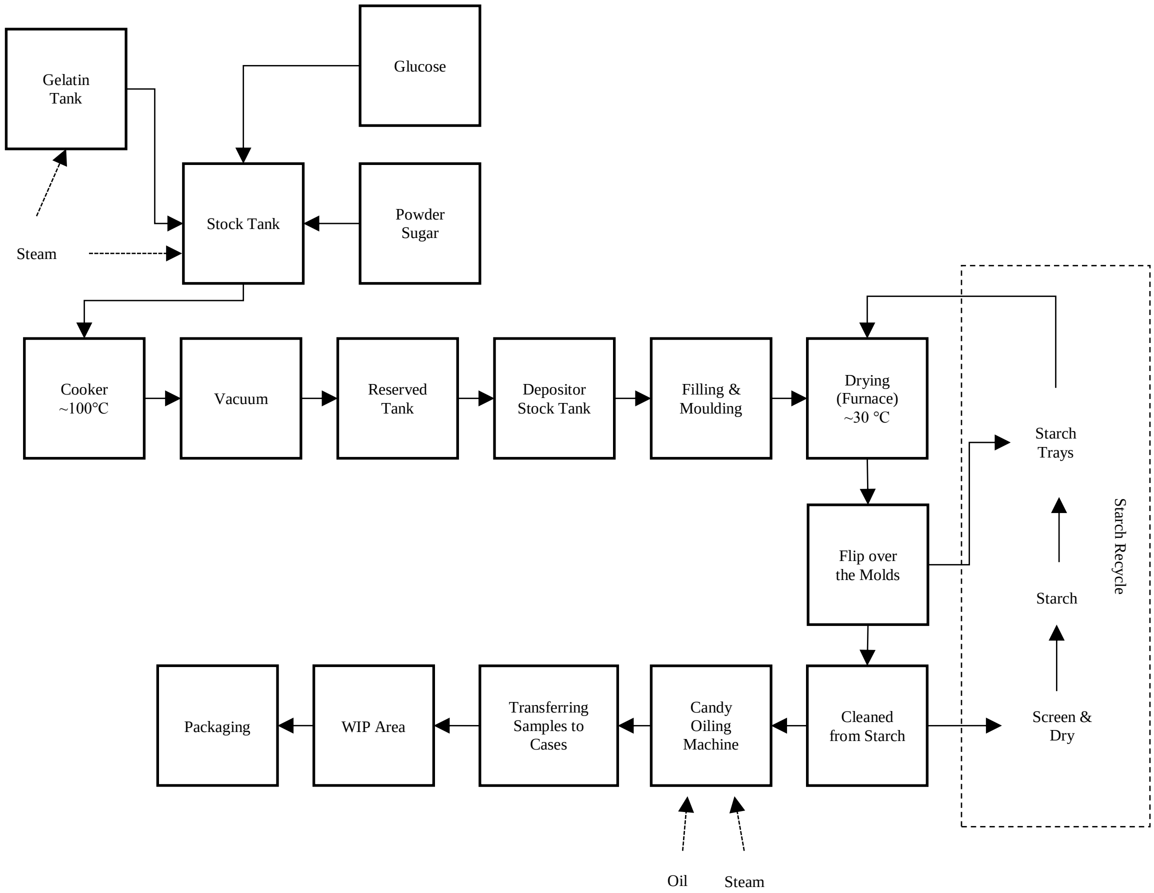


*Figure 2.1.* Flow chart of jelly candy production

In this study, 3 different types of candies whose recipes, raw materials, flavors and pH values were totally same, were used. The only difference between samples was unit weight and accordingly surface area. Unit weight levels were 2.5 g, 3 g and 6 g. Surface area of samples were increasing while increasing unit weight.

*Figure 2.2.* Samples (2.5 g, 3 g and 6 g respectively)

Samples molded into starch molds and put into oven for drying process. Effect of four levels of stoving time (12, 16, 20, 24 h) were studied. Moisture content, water activity, total soluble solid content, texture analysis and NMR experiments were performed. After stoving cycle was completed, samples were transferred to coating section. In this study, samples that were hold 12 h and 16 h sample on oven were not coated with oil, only 20 h and 24 h samples were coated; since, this trial was made in candy plant and products that were hold 12 h and 16 h on oven were not saleable products due to textural properties' of these candies not being appropriate for the consumer (too sticky and too soft). So, 12 and 16 h products were not coated for trial purposes because of the big batch size. In order to analyze the effect of stoving time on the moisture content, total solid content and water activity, non-coated samples were used.

Following the coating, samples were transferred to conditioning room for further drying. Conditioning (WIP) time was another factor studied. Coated samples that were held 20 and 24 hours in the oven were kept in conditioning room for 24 h, 48 h and 72 h. The above-mentioned physical measurements were also repeated for these samples. Effect of conditioning time was only studied for the coated samples of which 20 and 24 h stoving cycle was completed due to the same reasons explained above.

In routine production, stoving and conditioning (WIP) time are same for all different unit weight. Stoving time is 20 h and conditioning takes 72 h or more than 72 h for all gelatin based soft candies (for all different unit weight). In this study, 20 h stoving time is taken as reference.

### 2.2.2. Moisture Analysis – Vacuum Oven Method

Moisture content was determined by the vacuum oven method. This method was referenced from Mondelez International KJS 110 (Total solids in products by sea sand hot air-drying method under vacuum) and CRTM 011 (Determination of moisture in jelly candy and sugar syrups by vacuum oven drying) approved methods.

20 g sea sand and glass pieces were put on aluminum dish and dishes were put in oven at 105 ºC for 1 hour. Then, the dish was held in a desiccator at room temperature to cool down. One piece of sample is added to the dish and 1-2 ml hot water is then added and mixed into sea sand and glass pieces. This mixture is placed on drying oven at 60 º C for half an hour to evaporate excess water. After that, dishes are put into vacuum oven and kept for 18 hours. After 18 hrs, dishes are cooled in the desiccator and weighed. Moisture content was calculated as follows:

### 2.2.3. Determination of Total Soluble Solids

Total soluble solids (expressed as g of total soluble solids contained in 100 g or °Brix) are measured using refractometer (Atago Co. Ltd. RX- 5000) with an accuracy of 0.01 °Brix. This method is referenced from Mondelez International approved methods GRTM-518 (Determination of Total Solids by Refractometer in Gummy Candy).

Firstly, device is adjusted to 30º C and calibrated three times with water. One piece of sample is taken and cut in size of quarter of a sample. Inner part of sample is spread into the prism and cap is closed. The important point is that sample that is put into prism should be the inner part of sample not the surface section. Calculation is made as below;

### 2.2.4. Texture Analysis

Penetration test measurements were made using Stable Micro Systems (SMS) Texture Analyzer Plus with a 5 kg load cell. Also, this method is referenced from Mondelez International approved methods (Texture Analysis in jellies Penetration Test). The penetration test used for this analysis was performed with a 3mm cylindrical probe which penetrated to 80% of the full depth of a standard unit, and then retracted. Instrument plots the force-time curve during the analysis and ten measurements were recorded:

- *Force of penetration: "Hardness"* - the maximum force recorded as the probe penetrates the sample. This is measured in grams (g) and the value is positive as the probe is entering the sample at this stage
- *Work of penetration* – the area under the force-time curve recorded as the probe penetrates the sample. This is measured in gram-seconds (g. sec) and the value is positive as the probe has entered the sample at this stage
- *Force of probe withdrawal: "Stickiness"* – the maximum negative force measured as the probe withdraws from the sample. This is measured in grams (g) and the reading is negative as the probe is withdrawing from the sample at this stage

- *Work of probe withdrawal* – the negative area under the force-time graph recorded as the probe withdraws from the sample. This is measured in gram-seconds (g. sec) and the value is negative as the probe is withdrawing from the sample at this stage

Table 2.1 shows the settings for the SMS TA.XT Plus instrument for this analysis.

Table 2.1. *Texture analyzer settings for penetration test*

| ***Sample orientation*** | ***Tooling*** | ***Trigger mass (g)*** | ***Pre-speed (mm/sec)*** | ***Test speed (mm/sec)*** | ***Post speed (mm/sec)*** | ***%Strain*** |
|---|---|---|---|---|---|---|
| Mold side up | P3 probe | 20 | 5 | 0.5 | 10 | 80 |

Analysis was made at room temperature (25 ºC) with minimum 10 replicates. A representative plot is given in Fig. 2.2.

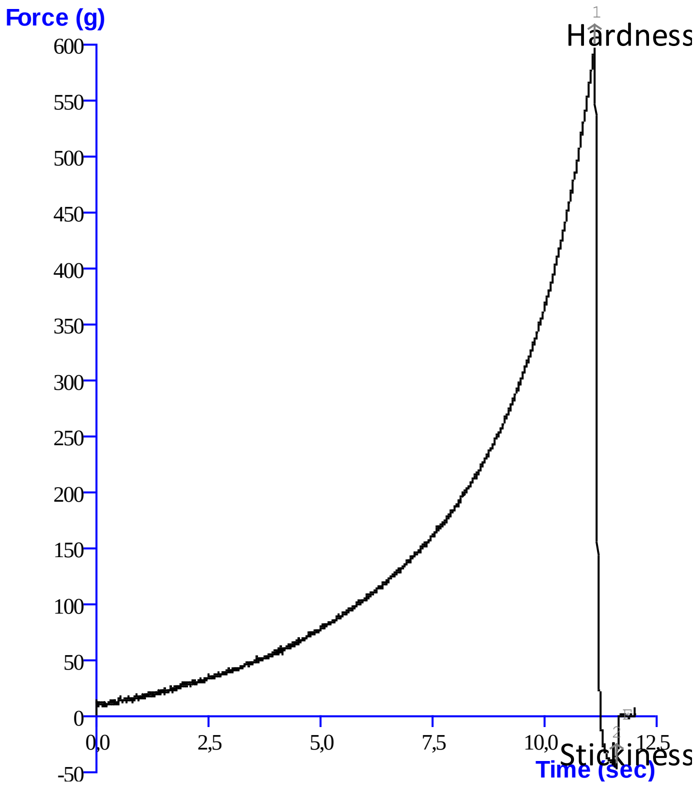


*Figure 2.3.* 2.5 g sample texture profile example (ST: 24 h & WIP =72 h)

### 2.2.5. Water Activity Determination

Gelatin based soft cadies' water activity was measured by water activity meter (Novasina, ms1 Set Aw). Small pieces of jellies were put in a chamber and kept until equilibrium. Experiments were conducted at 25$^0$C as triplicates.

### 2.2.6. Time Domain NMR Relaxometry (TD-NMR) Experiments

NMR relaxometry experiments were performed by using the parameters that was mentioned in the study Pocan et al. (2019) with some modifications.

TD NMR relaxometry experiments were carried out using 0.5 T (20.34 MHz) system (Spin Track, Russia). For T1 measurements, saturation-recovery sequence was used with a 300 ms relaxation period (TR) and 400 ms observation time. For T2 measurements, the Carr–Purcell–Meiboom–Gill sequence was utilized with parameters of 40 us echo time, 500 echoes, and four scans. T1 and T2 measurements were performed for all samples.

### 2.2.7. Statistical Analysis

All measurements except texture measurements were conducted with three replicates. Texture measurements were carried out at least 10 replicates. Data were analyzed using Minitab 17 (Minitab Inc., Penn State, USA) at %5 significance level. One-way ANOVA was conducted for the analysis of stoving time on non- coated samples. Other analysis were made with two or three way ANOVA. In order to determine difference between samples, Tukey's comparison tests were used at %95 confidence interval. In the Results & Discussion section, all results were represented mean of replicates with ± standard error. Also, detailed ANOVA analysis can be found in Appendix A1-A12.

### 2.2.8. Experimental Design

Table 2.2. Factors levels and responses used in the study

| Factors | *Levels* | *Responses* |
|---|---|---|
| Jelly Type | 2.5 g<br>3 g<br>6 g | 1. Moisture Content<br>2. Water Activity<br>3. Total Solid Content<br>4. Texture/Hardness<br>5. Texture/Stickiness<br>***6.*** NMR Relaxation Times |
| Stoving Time | 12 h<br>16 h<br>20 h<br>24 h | |
| WIP (Work in Progress & Conditioning) Time | 24 , 48, 72 h | |

# CHAPTER 3

# RESULTS AND DISCUSSIONS

## 3.1. Total Soluble Solid (TSS) Content

In these experiments, refractometer was used to investigate % total soluble solid (TSS) content. $^{o}$Brix values which represent the equivalent sucrose concentration of a syrup with that refractive index were reported (Ergun, Lietha, & Hartel, 2010). $^{o}$Brix value is a fast and adequate parameter that is used to understand TSS content in confectionery products (Lees & Jackson, E., 1973). In gelatin-based candies, total soluble solid content should be between % 75-82 to prevent graining (Lees & Jackson, 2012).

As mentioned on experimental design Table 2.2, 3 gelatin-based soft candies of different weights (2.5 g, 3 g and 6 g) were investigated for different stoving and WIP times. Also, ANOVA results related with TSS content was shown in Table A.1 and Table A.2.

When the results were examined, 2.5 g oil coated candy (Stoving time: 24 h, WIP: 72 h) had the highest % TSS content and 6 gr oil coated candy (Stoving time: 20 h, WIP time: 0 h) had the lowest % TSS content as it is seen Table 3.1. TSSC increased and moisture content reduced with drying (Vieira et al., 2008). So, as expected increasing stoving and WIP time caused increasing % TSS content.

As it was shown in Table A.1 and A.2, stoving time, WIP time and the weight had significant effects on the results ($p<0.05$). Moreover, there was a significant interaction between stoving time, WIP time and type of candy ($p<0.05$). 2.5 g samples that were kept for 24 h in the oven were significantly different from 12, 16 and 20 h stoving time ($p<0.05$) as it was shown in Table A.2.1 and 3 g and 6 g samples had lower % TSS content results as expected even if there was no significant difference (Table 3.1).

Table 3.1. *Total Solid Content Results for 2.5,3,6 g before coating*

| Stoving Time (h) | Type (g) | %Total Solid |
|---|---|---|
| 12 h | 2.5 | $78.60^{c}\pm0.18$ |
| 16 h | 2.5 | $78.83^{c}\pm0.02$ |
| 20 h | 2.5 | $79.49^{b}\pm0.00$ |
| 24 h | 2.5 | $82.26^{a}\pm0.00$ |
| 12 h | 3 | $78.92^{a}\pm0.36$ |
| 16 h | 3 | $79.05^{a}\pm0.25$ |
| 20 h | 3 | $79.09^{a}\pm0.12$ |
| 24 h | 3 | $79.45^{a}\pm0.15$ |
| 12 h | 6 | $78.98^{a}\pm0.01$ |
| 16 h | 6 | $79.10^{a}\pm0.01$ |
| 20 h | 6 | $78.85^{a}\pm0.00$ |
| 24 h | 6 | $79.70^{a}\pm0.31$ |

For WIP factors, % TSS content was increasing while increasing WIP time and all factors were significantly different from each other ($p<0.05$) shown in Table A.1.1. Products that were hold for 72 hours at approximately at 20 ºC and <65 %RH had the highest % TSS content.

Unit weight basis analysis showed more indicative results on the effects of storage conditions. For 6 g samples, WIP time was found to be effective on TSS content ($p<0.05$) shown in Table A.1.2. After 48 h, TSS content did not change.

For 3 g samples, WIP time effected TSS content significantly ($p<0.05$) and significant difference was found between 0 and 72 h samples (Table A.1.3).

For 2.5 g samples, it was found increasing trend for both stoving time and WIP time ($p<0.05$) shown in Table A.1.4.

Therefore, % TSS content increased unit weight decreased. TSS content result increased from 2.5 g to 6 g samples respectively. Moreover, 6 g, 3 g and 2.5 g candies were significantly different from each other regarding % TSS content (Table A.1.1). 2.5 g candy type had highest % TSS content.

In that study, it was hypothesized that stoving time and WIP time could be different as changing unit weight because for different weights and different surface areas on the starch bed. Larger samples had larger surface area in starch bed molds. So, time to reach equilibrium takes longer time as compared to smaller samples. Results proved that hypothesis.

*Table 3.2. Total Soluble Solid Content Results for 2.5, 3 and 6 g oil coated jelly candies*

| Stoving time (h) | WIP Time (h) | Type (g) | TSS% |
|---|---|---|---|
| 20 | 0 | 2.5 | 79.49±0.01 |
| 20 | 24 | 2.5 | 80.74±0.08 |
| 20 | 48 | 2.5 | 82.32±0.03 |
| 20 | 72 | 2.5 | 82.34±0.03 |
| 24 | 0 | 2.5 | 82.26±0.01 |
| 24 | 24 | 2.5 | 82.29±0.02 |
| 24 | 48 | 2.5 | 82.38±0.03 |
| 24 | 72 | 2.5 | 86.97±0.04 |
| 20 | 0 | 3 | 78.68±0.21 |
| 20 | 24 | 3 | 81.54±0.11 |
| 20 | 48 | 3 | 80.52±0.15 |
| 20 | 72 | 3 | 81.27±0.04 |
| 24 | 0 | 3 | 79.42±0.06 |
| 24 | 24 | 3 | 82.28±0.03 |
| 24 | 48 | 3 | 80.56±0.21 |
| 24 | 72 | 3 | 81.63±0.05 |
| 20 | 0 | 6 | 78.60±0.03 |
| 20 | 24 | 6 | 80.20±0.01 |
| 20 | 48 | 6 | 81.50±0.01 |
| 20 | 72 | 6 | 81.30±0.28 |
| 24 | 0 | 6 | 79.30±0.08 |
| 24 | 24 | 6 | 79.60±0.11 |
| 24 | 48 | 6 | 80.70±0.35 |
| 24 | 72 | 6 | 81.40[a]±0.10 |

*ANOVA results were shown in Appendix part.

## 3.2. Moisture Content

Moisture content (% wb) was determined using vacuum oven method. ANOVA results related with moisture content are shown in Table A.3 and Table A.4.

Stoving time, WIP time and candy weight were found to have significant effects on % moisture content results separately ($p<0.05$). The highest moisture content was 6 g sample that was hold for 20 h on the oven without WIP time as expected. Moreover, 2.5 g sample that was exposed to 24 h stoving cycle and 72 h WIP time had the lowest moisture content as it was shown in Table 3.2. When comparing total soluble solid content results, 6g (20 h stoving time & 0 h WIP time) had lowest % TSS content and 2.5 g (24 h stoving time &72 h WIP time) had the highest % TSS content.

Stoving time levels (12, 16, 20, 24 hour) were found effective on moisture content for non-coated samples ($p<0.05$). In Table 3.3, moisture decreased from 12 h to 24 h. According to Delgano, & Banon's study (2015), total solid content increased when moisture content decreased from 12 h to 24 h for gummy products (Delgado & Bañón, 2015). So, this descending trend was expected.

Generally, gelatin candies are rested in starch molds during 12 to 24 hours. Typical moisture migration in starch-candy-air system occurs with three way. The first one is migration of moisture from candy into starch bed; second one moisture from candy into air and moisture migration from starch bed into air (Sudharsan, Ziegler, & Duda, 2004). If moisture loss is rapid, hard skin and undesirable textural properties could be observed (Ergun et al., 2010). For this reason, drying condition (air flow, air temperature and humidity and oven design) should be specified depending on product type (type of gelation agent) and product size (Burey et al., 2009).

Table 3.3. *%Moisture Content Results for 2.5,3 and 6 g samples before coating*

| Stoving Time (h) | Type (g) | %Moisture C. |
|---|---|---|
| 12 h | 2.5 | $18.76^{a}\pm0.13$ |
| 16 h | 2.5 | $18.37^{ab}\pm0.38$ |
| 20 h | 2.5 | $17.72^{bc}\pm0.15$ |
| 24 h | 2.5 | $17.55^{c}\pm0.11$ |
| 12 h | 3 | $19.17^{a}\pm0.05$ |
| 16 h | 3 | $19.04^{a}\pm0.06$ |
| 20 h | 3 | $18.96^{a}\pm0.02$ |
| 24 h | 3 | $18.64^{b}\pm0.14$ |
| 12 h | 6 | $19.79^{a}\pm0.09$ |
| 16 h | 6 | $19.49^{a}\pm0.11$ |
| 20 h | 6 | $19.34^{a}\pm0.22$ |
| 24 h | 6 | $18.46^{b}\pm0.07$ |

After stoving time, samples were coated with oil and kept on the WIP area. In a general perspective, results showed that when samples were held on WIP area for longer time, moisture content decreased (Table A.4.1) as expected ($p<0.05$). According to literature search, soft panning candies tended to lose moisture from the coating of the jelly to the environment during aging (Troutman et al., 2001).

When the effect of stoving time ($p<0.05$) on 6 g samples were checked, 24 hours stoving time was found to be different from other time levels (Table A.3.4) Moisture content significantly decreased after 20 h stoving cycle ($p<0.05$). Moreover, WIP time had a significant effect ($p<0.05$) on 6 gr unit weight samples (Table A.4.2). As the stoving time and WIP time were increased, moisture content decreased that were shown in Table 3.4 in a detailed way.

24 h stoving time was found to be different from other time levels for 3 g non-coated samples ($p<0.05$) like 6 g samples (Table A.3.3). After stoving cycle, coated samples were affected significantly from WIP conditions ($p<0.05$). As the stoving time and WIP time increased, moisture content decreased. Samples that were held 72 h on WIP area were significantly different from others as shown in Table A.4.2.

2.5 g non-coated samples' moisture content decreased as stoving time increased. The difference between time levels were not significant ($p>0.05$) as can be seen Table.A.3.2. WIP time did not affect 2.5 g oil coated samples ($p>0.05$) and results can be seen in Table A.4.4.

In general, stoving time was found to be effective on jelly candies and 24 hours made difference as compared to other time levels for 2.5 g, 3g and 6 g non-coated samples (Table A.2.1). Samples, that were held on conditioning (WIP) room, tended to lose moisture and moisture content decreased as time increased. For 3 g and 6 g coated samples, moisture content was decreased significantly when waiting 72 h on conditioning room although there was not observed significant change until 72 h. However, WIP time was not effective on 2.5 g samples and these had the lowest moisture content (Table A.4). Moisture content results showed descending trend from 6 g to 2.5 g.

In summary, moisture content did not change significantly until 20 h; but, it decreased slowly for all non-coated samples from 20 to 24 h (Table A.3.1). Also, coated samples lost moisture after 48 h waiting on WIP area; however, only 3 g and 6 g samples were generally affected from being kept in the WIP area. 2.5 g oil coated samples were not affected from the WIP area, so it was confirmed that they were able to lose the whole free water inside and reached equilibrium totally during stoving. Stoving is important to remove the excess moisture from the candy. It is normal to lose moisture with increasing stoving time and WIP time.

*Table 3.4. Moisture Content Results for 2.5,3 and 6 g oil coated samples*

| Stoving time (h) | WIP Time (h) | Type (g) | % Moisture C |
|---|---|---|---|
| 20 | 0 | 2.5 | 18.74±0.08 |
| 20 | 24 | 2.5 | 18.67±0.13 |
| 20 | 48 | 2.5 | 18.66±0.13 |
| 20 | 72 | 2.5 | 17.05±0.10 |
| 24 | 0 | 2.5 | 18.57±0.06 |
| 24 | 24 | 2.5 | 18.54±0.13 |
| 24 | 48 | 2.5 | 18.53±0.08 |
| 24 | 72 | 2.5 | 18.50±0.16 |
| 20 | 0 | 3 | 19.22±0.08 |
| 20 | 24 | 3 | 19.35±0.05 |
| 20 | 48 | 3 | 19.55±0.06 |
| 20 | 72 | 3 | 19.03±0.07 |
| 24 | 0 | 3 | 19.31±0.21 |
| 24 | 24 | 3 | 18.97±0.05 |
| 24 | 48 | 3 | 18.57±0.07 |
| 24 | 72 | 3 | 18.38±0.09 |
| 20 | 0 | 6 | 19.85±0.04 |
| 20 | 24 | 6 | 19.62±0.12 |
| 20 | 48 | 6 | 19.42±0.14 |
| 20 | 72 | 6 | 18.75±0.06 |
| 24 | 0 | 6 | 19.38±0.11 |
| 24 | 24 | 6 | 18.80±0.05 |
| 24 | 48 | 6 | 18.79±0.08 |
| 24 | 72 | 6 | 18.69±0.03 |

*ANOVA results were shown in Appendix part.

### 3.3. Water Activity (aw)

Water activity could be defined as the ratio of partial pressure of water vapor ratio to pressure of pure water at a specified temperature (Mathlouthi, 2001).

When analyzing results before coating (Table A.5), stoving time and unit weight had significant effect on the water activity ($p<0.05$). Water activity remained constant after 20 hours. ANOVA results related with water activity was shown in Table A.5 and Table A.6.

When water activity as around 0.40-0.50 from 12 h to 16 h on oven, moisture content did not change significantly and remained almost constant. Around 20 h, moisture content decreased distinguishably (Table A.3.1) in the same time aw values did not change so much. This behavior is similar to Type II, intermediate moisture products that is shown in Figure 3.1 (Ergun et al., 2010). Also, according to literature, study there was no further dehydration after 20 hours in the oven (Delgado & Bañón, 2015). Products reached almost equilibrium after 20 h with respect to moisture and $a_w$.

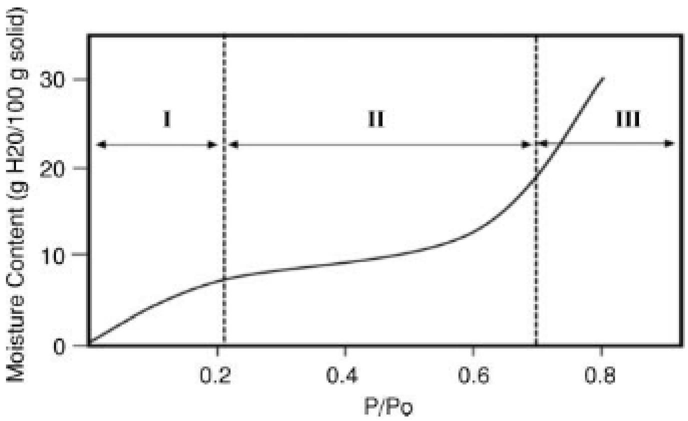


*Figure 3.1.* Typical moisture sorption curve for food that shows different regions of water (I: bound water, II: intermediate; III: free water) (Ergun et al., 2010)

After coating, samples were kept conditioning area. According to Table A.6.1., it was observed that water activity did not change significantly after 24 hour. On the other hand, changing water activity on conditioning room could be associated with the oil coating. $A_w$ could change after coating. Small increase in $a_w$ could be related with moisture transfer from coating to center of candy since oil has higher water activity compared to the product. This hypothesis associated with Troutman et al. study, they found moisture migration from coating to center initially because of oil water activity of panning syrup 0.8, when product's water activity were between 0.54 to 0.60 (Troutman et al., 2001).

When analyzing unit weight effect for coated products (Table A.6), different unit weight did not have an effect on water activity while waiting WIP area. According to literature search, raw materials that have high molecular weight and solubility (proteins, gums) cannot affect significantly $a_w$ (Ergun et al., 2015). Normally, humectants that are corn syrup, fructose, glucose, polyols typically reduce water activity. The reason not to observe significant change on water activity could be related with stable gel matrixes and although gelatin, as being a protein, had the little effect on water activity, $a_w$ (Pocan et al., 2019b).

Table 3.5. *Water activity results before coating*

| **Stoving Time (h)** | **Type (g)** | **Water activity ($a_w$)** |
|---|---|---|
| 12 h | 2.5 | 0.45±0.03 |
| 16 h | 2.5 | 0.53±0.01 |
| 20 h | 2.5 | 0.53±0.01 |
| 24 h | 2.5 | 0.56±0.01 |
| 12 h | 3 | 0.41±0.01 |
| 16 h | 3 | 0.40±0.01 |
| 20 h | 3 | 0.48±0.01 |
| 24 h | 3 | 0.53±0.01 |
| 12 h | 6 | 0.44±0.02 |
| 16 h | 6 | 0.49±0.01 |
| 20 h | 6 | 0.54±0.01 |
| 24 h | 6 | 0.44±0.01 |

*ANOVA results were shown in Appendix part.

Table 3.6. *Water activity Results for 2.5,3,6 g coated samples*

| **Stoving Time (h)** | **WIP Time (h)** | **Type (g)** | **Water activity ($a_w$)** |
|---|---|---|---|
| 20 | 0 | 2.5 | 0.53±0.01 |
| 20 | 24 | 2.5 | 0.60±0.01 |
| 20 | 48 | 2.5 | 0.57±0.02 |
| 20 | 72 | 2.5 | 0.60±0.03 |
| 24 | 0 | 2.5 | 0.56±0.01 |
| 24 | 24 | 2.5 | 0.62±0.01 |
| 24 | 48 | 2.5 | 0.42±0.25 |
| 24 | 72 | 2.5 | 0.58±0.01 |
| 20 | 0 | 3 | 0.45±0.01 |
| 20 | 24 | 3 | 0.59±0.01 |
| 20 | 48 | 3 | 0.60±0.02 |
| 20 | 72 | 3 | 0.63±0.01 |
| 24 | 0 | 3 | 0.53±0.01 |
| 24 | 24 | 3 | 0.59±0.01 |
| 24 | 48 | 3 | 0.59±0.01 |
| 24 | 72 | 3 | 0.60±0.01 |
| 20 | 0 | 6 | 0.51±0.01 |
| 20 | 24 | 6 | 0.48±0.01 |
| 20 | 48 | 6 | 0.54±0.01 |
| 20 | 72 | 6 | 0.53±0.01 |
| 24 | 0 | 6 | 0.50±0.02 |
| 24 | 24 | 6 | 0.56±0.01 |
| 24 | 48 | 6 | 0.63±0.01 |
| 24 | 72 | 6 | 0.59±0.01 |

*ANOVA results were shown in Appendix part.

## 3.4. Textural Properties

### 3.4.1. Hardness

Hardness is directly proportional to the maximum force when analyzing deformation in the first bite (Delgado & Bañón, 2015). In experimental design table, 3 different types gelatin based soft candy (2.5 g, 3 g and 6 g) were investigated on different stoving and WIP times. Hardness was determined with penetration test.

It was found that stoving time, WIP time and unit weight had significant effects on hardness results separately ($p<0.05$). The detailed ANOVA results were given in Table A.7. 6 g samples that were held 24 h in oven and 72 h in WIP area had highest hardness values as expected as seen in Table 3.7.

In texture analyses, it was not logical to use 12 and 16 h stoving time data. Since, after stoving cycle, samples' surface was covered with fully starch and it was very hard to analyze texture profile because of the sticky surface (inadequate drying). 12 h and 16 h samples could not coat with oil like 20 h and 24 h because of the same reason explained in Material & Methods part. As explained in experimental design part, samples were taken after 20 h and 24 h separately from oven and coated with oil; then kept in the WIP area. In order to understand stoving time effect on hardness, oil coated samples were used and it was found that stoving time (20 h and 24 h) affected hardness value significantly ($p<0.05$).

As it was stated above, WIP time affected hardness significantly ($p<0.05$) shown in Table A.7.1. As time increased, hardness values increased significantly like total solid content. In Delgado and Banon's study (2015), it was stated that hardness increased as total solid content increased in gummy candies.

For 2.5 g samples, hardness values increased as stoving time increased ($p<0.05$). 2.5 g samples' texture profile were not influenced from conditioning area although total solid content had increasing trend with time (Table A.7.3).

3 g samples' hardness value increased when stoving time increased like 2.5 g samples ($p<0.05$). Also, WIP time was effective on the texture for 3 g samples ($p<0.05$). Until 72 h, hardness did not affect significantly from conditioning (Table A.7.2).

Similar to 2.5 g and 3 g samples, hardness value increased as stoving time increased for 6 g samples ($p<0.05$). WIP time was effective on hardness value significantly for 6 g samples ($p<0.05$). Hardness value increased as WIP time increased as expected. This result had a similar trend with total solid content (Table A.7.4).

When investigating unit weight differences, samples were different from each other ($p<0.05$) as shown in Table A.7.1. However, this result was not directly the same with total soluble solid content. In general, 6 g samples were influenced by being kept in conditioning area. It could be related with that moisture migration was still going on because of larger surface area and having more free water molecules.

When analyzing with moisture results, hardness increased when moisture decreased. Drying reduces moisture and increases the solids content, and this causes increasing hardness and change on other textural properties. (Vieira et al., 2008). Pearson's correlation results are shown in Table 3.7. Hardness value was correlated with the moisture content, total solid content ($p<0.05$).

Table 3.7. *Pearson's correlation between moisture content, total soluble solids, water activity and textural properties*

| | | ***TSS*** | ***Moisture*** | ***AW*** | ***Hardness*** | ***Stickiness*** | ***T1*** |
|---|---|---|---|---|---|---|---|
| **Moisture** | R | -0.890 | | | | | |
| | p | 0.000 | | | | | |
| **AW** | R | 0.211 | -0.137 | | | | |
| | p | 0.322 | 0.522 | | | | |
| **Hardness** | R | 0.701 | -0.701 | 0.201 | | | |
| | p | 0.000 | 0.000 | 0.346 | | | |
| **Stickiness** | R | -0.885 | 0.903 | -0.220 | -0.770 | | |
| | p | 0.000 | 0.000 | 0.303 | 0.000 | | |
| **T1** | R | 0.401 | -0.406 | -0.125 | 0.250 | -0.360 | |
| | p | 0.052 | 0.049 | 0.562 | 0.240 | 0.084 | |
| **T2** | R | 0.0145 | -0.021 | 0.020 | 0.051 | -0.064 | -0.453 |
| | p | 0.498 | 0.924 | 0.925 | 0.811 | 0.768 | 0.026 |

Increasing hardness value during drying could be related with gelatin and dehydration. Gelling agent (gelatin), sugar, water and other components cause firm and chewy structure. Also, dehydration is an important factor for jelly texture. Jellies' dehydration rate is higher at the beginning of drying in starch mold. To increase dehydration rate, higher temperature and lower RH would be required; but, drying conditions and circulating air should be optimized to prevent gel formation delay and increase the risk of surface crusting. In oven, water inside the product transfer to air so moisture content decreases and solid content increases. Hardness is found to be more correlated with solid content (Delgado & Bañón, 2015).

Table 3.8. *Hardeness (N) Results for 2.5,3,6 g oil coated samples*

| Stoving time (h) | WIP Time (h) | Type (g) | Hardness (N) |
|---|---|---|---|
| 20 | 0 | 2.5 | 560.64±10.59 |
| 20 | 24 | 2.5 | 556.77±19.62 |
| 20 | 48 | 2.5 | 550.80±10.18 |
| 20 | 72 | 2.5 | 516.54±15.78 |
| 24 | 0 | 2.5 | 582.67±7.41 |
| 24 | 24 | 2.5 | 557.88±17.63 |
| 24 | 48 | 2.5 | 563.56±14.99 |
| 24 | 72 | 2.5 | 581.50±13.35 |
| 20 | 0 | 3 | 507.34±8.30 |
| 20 | 24 | 3 | 510.58±10.38 |
| 20 | 48 | 3 | 485.17±17.11 |
| 20 | 72 | 3 | 497.69±10.14 |
| 24 | 0 | 3 | 512.03±10.46 |
| 24 | 24 | 3 | 529.49±17.61 |
| 24 | 48 | 3 | 512.34±14.02 |
| 24 | 72 | 3 | 528.55±18.35 |
| 20 | 0 | 6 | 445.72±24.22 |
| 20 | 24 | 6 | 458.99±20.52 |
| 20 | 48 | 6 | 534.87±21.24 |
| 20 | 72 | 6 | 627.72±20.10 |
| 24 | 0 | 6 | 458.59±20.79 |
| 24 | 24 | 6 | 498.06±18.52 |
| 24 | 48 | 6 | 538.06±18.63 |
| 24 | 72 | 6 | 635.78±24.01 |

*ANOVA results were shown in Appendix part.

### 3.4.2. Stickiness

Stickiness is a very important parameter for jelly candies. *Stickiness is* the force necessary to overcome the attractive forces between the surface of the product and the surface of the material (the probe) with which the product comes in contact. Stickiness can be defined as having the property of adhering or sticking to a surface (Jiamjariyatam, 2018). In jelly products, it could be faced with quality defects, such as excessive stickiness / hardness, poor crusting, poor chewiness in the case moisture deviations (Ergun et al., 2010).

In general, unit weight, stoving time and WIP time had an effect on stickiness values ($p<0.05$). ANOVA results are given to Table A.8.

12 h and 16 h stoving time could not be evaluated with respect to stickiness like hardness. 20 h and 24 h results were used to evaluate stoving time effects on stickiness. Stoving time was effective on stickiness value significantly ($p<0.05$). Also, stickiness decreased as holding time on oven increased (Table A.8.1).

In general, stickiness was influenced by WIP time ($p<0.05$). So, stickiness decreased from 0 h to 72 h WIP time as general results were shown in Table A.8.1.

For 2.5 g, 3 g and 6 g samples, stickiness value decreased as stoving time increased ($p<0.05$). WIP time was also effective on texture ($p<0.05$) and there were significant changes after 48 hours for all oil coated samples according to Table A.8.2&3&4.

Moreover, while investigating unit weight effect on stickiness, it was observed that 2.5 g samples were different than others similar to the moisture content ($p<0.05$).

According to Table 3.7, stickiness values were correlated with moisture content and TSS like hardness value ($p<0.05$). Also, stickiness and hardness values were correlated at the same time (R:-0.770; $p<0.05$). Moisture caused a sticky feeling in products. As expected, stickiness decreased when moisture content decreased. Similar results were found in Ergun et al.'s study. In that study, stickiness increased with moisture content (Ergun et al., 2010).

Table 3.9. *Stickiness Results for 2.5,3 and 6 g coated samples*

| Stoving time (h) | WIP Time (h) | Type (g) | Stickiness (N) |
|---|---|---|---|
| 20 | 0 | 2.5 | -38.60±3.26 |
| 20 | 24 | 2.5 | -38.30±2.93 |
| 20 | 48 | 2.5 | -37.14±1.94 |
| 20 | 72 | 2.5 | -38.45±1.88 |
| 24 | 0 | 2.5 | -37.18±1.32 |
| 24 | 24 | 2.5 | -36.89±2.22 |
| 24 | 48 | 2.5 | -39.65±1.23 |
| 24 | 72 | 2.5 | -44.45±2.58 |
| 20 | 0 | 3 | -29.56±2.90 |
| 20 | 24 | 3 | -30.96±5.21 |
| 20 | 48 | 3 | -31.98±2.17 |
| 20 | 72 | 3 | -32.97±2.78 |
| 24 | 0 | 3 | -33.04±2.92 |
| 24 | 24 | 3 | -33.39±5.30 |
| 24 | 48 | 3 | -33.13±3.09 |
| 24 | 72 | 3 | -37.23±2.95 |
| 20 | 0 | 6 | -29.43±6.67 |
| 20 | 24 | 6 | -28.43±5.11 |
| 20 | 48 | 6 | -33.80±2.22 |
| 20 | 72 | 6 | -38.74±3.27 |
| 24 | 0 | 6 | -30.99±5.28 |
| 24 | 24 | 6 | -33.68±2.74 |
| 24 | 48 | 6 | -34.42±3.44 |
| 24 | 72 | 6 | -35.40±8.18 |

*ANOVA results were shown in Appendix part.

### 3.5. Time Domain NMR Relaxometry Results

#### 3.5.1. $T_2$ (Spin-Spin Relaxation Time) Measurements

Time domain NMR Relaxometry (TD-NMR) is a non-destructive and promising tool to analyze various food systems such as oil in water emulsions (Pocan et al., 2019a), whey protein hydrogels (Ozel, Aydin, Grunin, & Oztop, 2018), green tea loaded lecithin based liposomes (Kirtil, Dag, Guner, Unal, & Oztop, 2017) and gluten-free cakes (Yildiz et al., 2018). In recent studies, TD-NMR was also utilized to analyze confectionary systems to explore the effect of allulose substitution on gelatin based soft candies (Pocan et al., 2019b) and effect of different sweeteners such as maltitol, stevia, isomalt on gelatin based low calorie soft candies (Efe, Bielejewski, Tritt-Goc, Mert, & Oztop, 2019). T2 relaxation time is also known as the spin-spin relaxation time and the changes in this relaxation time could be attributed to the various proton related alterations such as change in moisture content, exchange of protons between compartments in food systems (Pocan et al., 2019b). Therefore, in this study, T2 values of oil coated gelatin based soft candies with 2.5 g, 3g and 6 unit weights which exposed to different stoving and WIP times were determined and results were shown in Table 3.10. In order to examine correlations between textural properties (hardness, stickiness), Relaxometry data (T2 and T1) was only represented for the oil coted ones like previous experiments.

Considering the relaxation times of samples (Table 3.10), very short T2 relaxation times which vary in the range of 0.58-0.83 ms were observed for all samples. This was an expected outcome since such a short relaxation times for gelatin based soft candies were also observed in the study of Pocan et al. (2019). In their study, this phenomena was explained with the utilization of very short echo time (40 us) enabling us to get an idea about the solid-solid interactions which is very important to analyze gel systems such as jelly candies (Pocan et al., 2019b). This reason is also valid for our study since same echo time (40 us) was used to analyze gelatin based soft cadies that exposed to different stoving and WIP times and small T2 values were found as expected. T2 relaxation times were expressed by using mono-exponential model.

Referring back to the T2 relaxation time data that was illustrated in Table 3.10, for the 2.5 g samples that expose to 20 hour stoving time, gradual and significant decrease in T2 relaxation times was observed as the WIP time was increased in the range between 24-72 hours ($p<0.05$). Similar case was also observed for the 3 g samples that waited at constant 20 hour stoving time. As the WIP time was increased in the range of 0-72 hours, T2 values were decreased similar to samples that have unit weight of 2 g ($p<0.05$). Normally, T1 and T2 relaxation times show increasing trend as the moisture content increases due to the enhancement of water mobility in the samples (Cikrikci, Mert, & Oztop, 2018). However, in our case, different situation was observed. For the samples that mentioned above, T2 values were decreased although their moisture content remained same. Therefore, decrease in T2 values might be explained with different factors rather than moisture. For example, for the same samples, it was demonstrated that total solid content increased as the WIP time increased from 0 to 72 hours ($p<0.05$). At this point, it was hypothesized that, increasing total solid content might have enhanced solid-solid interactions of gelatin based soft candies leading to decrease in overall T2 values. This was an expected result since short relaxation times are related with the enhanced solid-solid interactions as it was found for the starch based soft candies. (Ilhan E. 2019).

For the samples having 6 g unit weight that expose 20 hour WIP time, scenario is different. For these specimens, total solid content again increased gradually and significantly ($p<0.05$) similar to previous samples. However, this time their T2 values remained same and significant alterations were not observed ($p>0.05$). When their moisture content was examined, it was revealed that detectable decrease was observed while passing through 48 hour WIP time to 72 hour WIP time as discussed in previous sections. Therefore, at this point different scenario is valid. As indicated ANOVA Table A.1, total solid content of 6 g samples is obviously smaller than its counterparts ($p<0.05$) meaning that solid-solid interactions are less dominant for this sample.

Relatively smaller solid-solid interactions might have led to increase in overall T2 relaxation time since compartments where solid-solid interactions become dominant generally have lower T2 relaxation time. On the other hand, decrease in moisture content while passing through 48 to 72 h WIP might have led to decrease in mobility of water in sample thereby causing decrease in overall T2. To sum up, increase in T2 because of less solid-solid interactions might have compensated with decrease in moisture content leading overall T2 relaxation time to remain constant.

*Table 3.10.* T2 (Spin-spin relaxation time) Results for 2.5,3 and 6 g coated samples

| Stoving time (h) | WIP Time (h) | Type (gr) | T2(ms) |
|---|---|---|---|
| 20 | 0 | 2.5 | $0.74^{bc}$±0.01 |
| 20 | 24 | 2.5 | $0.77^{ab}$±0.01 |
| 20 | 48 | 2.5 | $0.71^{cd}$±0.02 |
| 20 | 72 | 2.5 | $0.61^{f}$±0.00 |
| 24 | 0 | 2.5 | $0.72^{cd}$±0.00 |
| 24 | 24 | 2.5 | $0.67^{de}$±0.01 |
| 24 | 48 | 2.5 | $0.64^{ef}$±0.00 |
| 24 | 72 | 2.5 | $0.79^{a}$±0.00 |
| 20 | 0 | 3 | $0.83^{a}$±0.02 |
| 20 | 24 | 3 | $0.75^{b}$±0.02 |
| 20 | 48 | 3 | $0.76^{ab}$±0.01 |
| 20 | 72 | 3 | $0.74^{bc}$±0.01 |
| 24 | 0 | 3 | $0.72^{bcd}$±0.00 |
| 24 | 24 | 3 | $0.67^{cde}$±0.02 |
| 24 | 48 | 3 | $0.66^{de}$±0.01 |
| 24 | 72 | 3 | $0.64^{e}$±0.01 |
| 20 | 0 | 6 | $0.66^{ab}$±0.01 |
| 20 | 24 | 6 | $0.58^{b}$±0.04 |
| 20 | 48 | 6 | $0.62^{ab}$±0.03 |
| 20 | 72 | 6 | $0.66^{ab}$±0.00 |
| 24 | 0 | 6 | $0.75^{a}$±0.03 |
| 24 | 24 | 6 | $0.58^{b}$±0.03 |
| 24 | 48 | 6 | $0.67^{ab}$±0.02 |
| 24 | 72 | 6 | $0.62^{ab}$±0.02 |

### 3.5.2. T1 (Spin-Lattice Relaxation) Time Measurements

T1 (Spin-Lattice Relaxation) time is also known as longitudinal relaxation time and it refers the time which is necessary for spins to give back the energy that they obtained from the radio frequency pulse for turning their initial state (Ozel, Dag, et al., 2017). From previous studies, it was known that, T1 relaxation time is strongly depend on mobile protons that come from the free water (Ozel, Dag, et al., 2017). Therefore, it is worth to mention that, T1 (spin-lattice relaxation time) is a great tool to detect the moisture distribution of food samples (Pocan et al., 2019b). Therefore, in this study, T1 relaxation times of oil coated gelatin based soft candies with 2.5 g, 3g and 6 g unit weights which exposed to different stoving and WIP times were determined and results were shown in Table 3.11. T1 relaxation times were expressed by using mono-exponential model like previous section.

Referring back to the T1 relaxation time data that was illustrated in Table 3.11, for the 2.5, 3 and 6 g samples that expose to 20 hour stoving time, steadiness in T1 (spin-lattice) relaxation times were observed as the WIP time increased. Similar steadiness was also observed in moisture content results for the same samples. This point is important to mention since it is known that NMR Relaxometry is a valuable tool to detect the moisture distribution of food products and strong correlations were found between T1 and moisture contents of sponge cakes in previous studies (Botosoa, Chèné, Blecker, & Karoui, 2015). In addition to this study, TD-NMR Relaxometry was also utilized for the analysis of gelatin based soft candies with different formulations and similar T1-moisture content correlations were also found in this study (Pocan et al., 2019b). Pocan et al. (2019) indicated that as the moisture content of the samples with different formulations increased, T1 relaxation time also increased and they mentioned that T1 (spin-lattice relaxation) time is directly related with the mobility of water. In another study, similar results were also found. Maltitol containing gelatin based soft candies was found to be highest moisture content leading to highest T1 relaxation time (Efe et al., 2019). Although our study is related with drying conditions of soft candies, similar moisture content-T1 relaxation time relation is also valid for this study. Considering whole data set (regarding the effect of both stoving and WIP time), Pearson correlation coefficients were found as -0.96 and 0.72 for the samples having unit weight 3 gr and 6 gr respectively in our study as shown in Table A.12.

In literature, in addition to formulation studies, T1 relaxation was also utilized to visualize moisture migration profiles of starch molded confectionaries by using magnetic resonance imaging (Ziegler, MacMillan, & Balcom, 2003). In their study, T1 relaxation times were found by using inversion recovery sequence differently from ours and similarly moisture content was well correlated with the T1 relaxation times.

It is worth to mention that, although effect of moisture content is dominant in T1 relaxation times, information related with the crystallinity could be also obtained by utilizing spin-lattice relaxation times. For instance, when the data was examined in Table 3.11, it was observed that for the 6gram samples that exposed constant 24 hour stoving time but not waited WIP area (0 hour WIP time) have significantly higher moisture content compared to samples exposing 24 hour stoving and 72 hour WIP time ($p<0.05$). In terms of moisture content, it was an expected result. Increasing drying resulted in reduction in the moisture content since it was known that small amount of moisture was lost to starch and candy-air surface was dominant factor during drying process (Ziegler et al., 2003). However, it was revealed that, T1 values of these samples did not change significantly ($p>0.05$). According to the previous studies, longer T1 (spin-lattice) relaxation time resulted in more crystalline region (Le Botlan, Casseron, & Lantier, 1998). Crystallinity studies were not performed for our study but it was hypothesized that, increased WIP time might have resulted in “hard skin” formation on candies’ surface leading to enhanced crystallinity.

In addition to moisture content results, T1 relaxation time was also correlated with some textural properties such as hardness in order to investigate quality of food products such as sponge cakes (Botosoa et al., 2015) , gluten free cakes (Yildiz et al., 2018) and gelatin based soft candies (Pocan et al., 2019b) in previous studies. In our study, only for the 3 g samples such a hardness-T1 correlation was found ($r=0.715$, $p<0.05$). For the stickiness results, again for the 3 g samples similar correlation was also found ($r= -0.78$, $p<0.05$).

Table 3.11. *T1 (Spin-lattice Relaxation Time) Results for 2.5,3 and 6 g coated samples*

| Stoving time (h) | WIP Time (h) | Type (g) | T1(ms) |
|---|---|---|---|
| 20 | 0 | 2.5 | $47.42^{ab}\pm0.11$ |
| 20 | 24 | 2.5 | $48.10^{ab}\pm0.31$ |
| 20 | 48 | 2.5 | $47.57^{ab}\pm0.16$ |
| 20 | 72 | 2.5 | $48.16^{a}\pm0.26$ |
| 24 | 0 | 2.5 | $47.73^{ab}\pm0.21$ |
| 24 | 24 | 2.5 | $46.86^{b}\pm0.18$ |
| 24 | 48 | 2.5 | $47.18^{ab}\pm0.13$ |
| 24 | 72 | 2.5 | $47.66^{ab}\pm0.26$ |
| 20 | 0 | 3 | $44.07^{cd}\pm0.48$ |
| 20 | 24 | 3 | $44.19^{cd}\pm0.22$ |
| 20 | 48 | 3 | $43.80^{d}\pm0.10$ |
| 20 | 72 | 3 | $44.76^{bcd}\pm0.22$ |
| 24 | 0 | 3 | $44.22^{cd}\pm0.12$ |
| 24 | 24 | 3 | $45.62^{abc}\pm0.29$ |
| 24 | 48 | 3 | $45.88^{ab}\pm0.22$ |
| 24 | 72 | 3 | $46.27^{a}\pm0.26$ |
| 20 | 0 | 6 | $47.85^{a}\pm0.26$ |
| 20 | 24 | 6 | $48.16^{a}\pm0.75$ |
| 20 | 48 | 6 | $48.39^{a}\pm0.35$ |
| 20 | 72 | 6 | $47.70^{a}\pm0.36$ |
| 24 | 0 | 6 | $47.57^{a}\pm0.17$ |
| 24 | 24 | 6 | $47.19^{a}\pm0.14$ |
| 24 | 48 | 6 | $47.40^{a}\pm0.43$ |
| 24 | 72 | 6 | $47.07^{a}\pm0.22$ |

# CHAPTER 4

# CONCLUSIONS

In soft candy production drying time, the so-called stoving time and WIP time or in other words conditioning time are important process parameters that could affect the final quality of the products.

Manufacturers would like to determine the minimum drying time while attaining the best quality parameters since time and energy are very important for cost. Following stoving, samples are kept in a conditioning area (WIP) before packaging to save time before packaging and also to let samples achieve their final moisture content. In WIP area, products' moisture migration could continue, so WIP room could also be used to obtain the desired moisture content and textural properties.

In this study different unit weight candies (2.5, 3 and 6 g) were exposed to different stoving and conditioning times. When unit weight was small, harder texture was obtained under same conditions. 2.5 g jelly candies were found harder compared to 3 g and 6 g jelly candies as moisture migration was directly related with length and thickness of candy into starch bed (Troutman et al., 2001).

It was found that, 6 g and 3 g jelly candies' stoving time could be set to 24 h. On the other hand, 2.5 g jelly candies' stoving time could be kept on 20 h since, moisture content of candy did not change significantly until 24 h. For 24 h stoving time, 3 g and 6 g candies' moisture content dropped below % 19. On the other hand, 2.5 g candy reached % 18 moisture content after 20 h stoving cycle. Thus moisture content results showed that %18-19 moisture content could reach around 24 h stoving for 3 g and 6 g; but around 20 h stoving for 2.5 g samples.

Also, it was observed that 2.5 g samples was not affected from conditioning on the WIP area. 2.5 g samples could be kept for 72 hours on WIP area until packaging since

2.5 g samples' textural properties was not affected from WIP area as they reached equilibrium before coating on the oven. Moreover, it was recommended not to keep more than 48 h on WIP area for 3 g and 6 g jelly candy; since, textural properties became undesirable. NMR T1 relaxation results also confirmed this recommendation.

In that study, it was found that moisture content and total solid content were correlated with textural properties. T1 and T2 relaxation times showed increasing trend as the moisture content increased due to the enhancement of water mobility in the samples (Cikrikci et al., 2018). However, in our case, a different situation was observed, T1 and T2 were not directly correlated with the textural properties. Solid-solid interactions and crystallinity changes were thought to be the reason of that.

In manufacturing, performing a fast and accurate analysis method is very important during production. According to the correlation results, moisture content, texture analysis and total soluble solid content could be used to control the quality of the products. However, moisture content analysis takes long time and making decision is very hard based on just moisture content while products are in oven because of the process time. Texture analysis is fast but very difficult method to make decision just after completing stoving cycle since products have a sticky surface. So, total soluble solid content can be used to control the quality of products with acceptance of small error and variation. It is recommended that products can be taken out from the oven after % total soluble solid content reached %79 generally. 3 g and 6 g samples reached this value after 24 h stoving while 2.5 g samples obtained this value after 20 h stoving. After products coating and waiting in the WIP area, texture analysis provided an insight for the desirable texture profile before packaging. When texture results were investigated, it was observed that 3 g and 6 g samples' texture results were affected significantly in a negative way after 48 h conditioning (WIP), while 2.5 g samples were not affected so much even if kept for 72 h on the conditioning room. When texture results at this WIP time were checked, it was found that the recommended texture value could be between 500-550 N.

To sum up, 20 h stoving time was applied before starting this study and 20 h was accepted as reference in the beginning of this study. Then, stoving time was extended 24 h in order to reach better textural properties while this study was in progress. In the light of this study, it is recommended that 2.5 g samples' stoving cycle can be decreased 20 h again. Thus, 4 h stoving time and accordingly energy can be saved. In addition to that, all samples (2.5 g, 3 g and 6g) are kept 72 h on conditioning area before packaging in manufacturing plant. It is recommended that, 3 g and 6 g samples' texture can be better if they are not waited more than 48 h in conditioning area.

## CHAPTER 5

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# APPENDICES

## **A.** ANOVA TABLES

Table A.1. Analysis of variance of gelatin based confectionery. Effect of WIP, Stoving time (ST) and unit weigh (jelly type / jt) on Total Soluble Solid (TSS) Content (for oil coated samples)

### (2) General Linear Model: Total Soluble Solid versus ST; WIP; jt

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | Fixed | 2 | 20; 24 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| jt | Fixed | 3 | 2.5; 3.0; 6.0 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST | 1 | 13.356 | 13.3558 | 578.00 | 0.000 |
| WIP | 3 | 74.921 | 24.9736 | 1080.78 | 0.000 |
| jt | 2 | 55.490 | 27.7449 | 1200.72 | 0.000 |
| ST *WIP | 3 | 10.495 | 3.4984 | 151.40 | 0.000 |
| ST *jt | 2 | 18.539 | 9.2693 | 401.15 | 0.000 |
| WIP*jt | 6 | 25.284 | 4.2140 | 182.37 | 0.000 |
| ST *WIP*jt | 6 | 8.968 | 1.4946 | 64.68 | 0.000 |
| Error | 48 | 1.109 | 0.0231 | | |
| Total | 71 | 208.161 | | | |

### Comparisons for TSS, Term= ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 36 | 81.5644 | A | |
| 20 | 36 | 80.7031 | | B |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 72 | 18 | 82.4872 | A | | | |
| 48 | 18 | 81.3244 | | B | | |
| 24 | 18 | 81.1039 | | | C | |
| 0 | 18 | 79.6194 | | | | D |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= jt

Grouping Information Using the Tukey Method and 95% Confidence

| jt | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 2.5 | 24 | 82.3508 | A | | |
| 3.0 | 24 | 80.7375 | | B | |
| 6.0 | 24 | 80.3129 | | | C |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= ST*WIP

Grouping Information Using the Tukey Method and 95% Confidence

| ST*WIP | N | Mean | Grouping | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 24 72 | 9 | 83.3478 | A | | | | | | |
| 20 72 | 9 | 81.6267 | | B | | | | | |
| 20 48 | 9 | 81.4500 | | B | C | | | | |
| 24 24 | 9 | 81.3967 | | | C | D | | | |
| 24 48 | 9 | 81.1989 | | | | D | | | |
| 20 24 | 9 | 80.8111 | | | | | E | | |
| 24 0 | 9 | 80.3144 | | | | | | F | |
| 20 0 | 9 | 78.9244 | | | | | | | G |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= ST*jt

Grouping Information Using the Tukey Method and 95% Confidence

| ST*jt | N | Mean | Grouping | | | | |
|---|---|---|---|---|---|---|---|
| 24 2.5 | 12 | 83.4775 | A | | | | |
| 20 2.5 | 12 | 81.2242 | | B | | | |
| 24 3.0 | 12 | 80.9717 | | | C | | |
| 20 3.0 | 12 | 80.5033 | | | | D | |
| 20 6.0 | 12 | 80.3817 | | | | D | E |
| 24 6.0 | 12 | 80.2442 | | | | | E |

Means that do not share a letter are significantly different.

### Comparisons for TSS, Term= WIP*jt

Grouping Information Using the Tukey Method and 95% Confidence

| WIP*jt | N | Mean | Grouping | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 72 2.5 | 6 | 84.6550 | A | | | | | | | | |
| 48 2.5 | 6 | 82.3533 | | B | | | | | | | |
| 24 3.0 | 6 | 81.9067 | | | C | | | | | | |
| 24 2.5 | 6 | 81.5167 | | | | D | | | | | |
| 72 3.0 | 6 | 81.4517 | | | | D | | | | | |
| 72 6.0 | 6 | 81.3550 | | | | D | E | | | | |
| 48 6.0 | 6 | 81.0783 | | | | | E | F | | | |
| 0 2.5 | 6 | 80.8783 | | | | | | F | | | |
| 48 3.0 | 6 | 80.5417 | | | | | | | G | | |
| 24 6.0 | 6 | 79.8883 | | | | | | | | H | |
| 0 3.0 | 6 | 79.0500 | | | | | | | | | I |
| 0 6.0 | 6 | 78.9300 | | | | | | | | | I |

## (3) General Linear Model: Total Soluble Solid Content (TSS) versus WIP (WIP_6) and Stoving time (ST_6) for 6 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP_6 | Fixed | 4 | 0; 24; 48; 72 |
| ST_6 | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP_6 | 3 | 22.5872 | 7.52906 | 177.15 | 0.000 |
| ST_6 | 1 | 0.1134 | 0.11344 | 2.67 | 0.122 |
| WIP_6*ST_6 | 3 | 2.1329 | 0.71096 | 16.73 | 0.000 |
| Error | 16 | 0.6800 | 0.04250 | | |
| Total | 23 | 25.5135 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.206155 | 97.33% | 96.17% | 94.00% |

### Comparisons forTSS, Term= WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_6 | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 72 | 6 | 81.3550 | A | | |
| 48 | 6 | 81.0783 | A | | |
| 24 | 6 | 79.8883 | | B | |
| 0 | 6 | 78.9300 | | | C |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6 | N | Mean | Grouping |
|---|---|---|---|
| 20 | 12 | 80.3817 | A |
| 24 | 12 | 80.2442 | A |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= WIP*ST

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_6*ST_6 | N | Mean | Grouping | | | | |
|---|---|---|---|---|---|---|---|
| 48 20 | 3 | 81.5067 | A | | | | |
| 72 24 | 3 | 81.4433 | A | | | | |
| 72 20 | 3 | 81.2667 | A | | | | |
| 48 24 | 3 | 80.6500 | | B | | | |
| 24 20 | 3 | 80.1567 | | B | C | | |
| 24 24 | 3 | 79.6200 | | | C | D | |
| 0 24 | 3 | 79.2633 | | | | D | |
| 0 20 | 3 | 78.5967 | | | | | E |

## (4) General Linear Model: Total Soluble Solid Content (TSS) versus WIP (WIP_3) and Stoving time (ST_3) for 3 g samples

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP_3 | 3 | 28.5780 | 9.52598 | 382.76 | 0.000 |
| ST | 1 | 1.3160 | 1.31602 | 52.88 | 0.000 |
| WIP_3*ST | 3 | 0.5057 | 0.16856 | 6.77 | 0.004 |
| Error | 16 | 0.3982 | 0.02489 | | |
| Total | 23 | 30.7979 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.157758 | 98.71% | 98.14% | 97.09% |

## Comparisons for TSS, Term= WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_3 | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 6 | 81.9067 | A | |
| 72 | 6 | 81.4517 | | B |

| | | | |
|---|---|---|---|
| 48 | 6 | 80.5417 | C |
| 0 | 6 | 79.0500 | D |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping |
|---|---|---|---|
| 24 | 12 | 80.9717 | A |
| 20 | 12 | 80.5033 | B |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term= WIP*ST

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_3*ST | N | Mean | Grouping |
|---|---|---|---|
| 24 24 | 3 | 82.2767 | A |
| 72 24 | 3 | 81.6300 | B |
| 24 20 | 3 | 81.5367 | B |
| 72 20 | 3 | 81.2733 | B |
| 48 24 | 3 | 80.5633 | C |
| 48 20 | 3 | 80.5200 | C |
| 0 24 | 3 | 79.4167 | D |
| 0 20 | 3 | 78.6833 | E |

Means that do not share a letter are significantly different.

## (5) General Linear Model: Total Soluble Solid Content (TSS) versus WIP (WIP_2.5) and Stoving time (ST_2.5) for 2.5 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP_2.5 | Fixed | 4 | 0; 24; 48; 72 |
| ST_2.5 | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP_2.5 | 3 | 49.0397 | 16.3466 | 8455.12 | 0.000 |
| ST_2.5 | 1 | 30.4651 | 30.4651 | 15757.79 | 0.000 |
| WIP_2.5*ST_2.5 | 3 | 16.8243 | 5.6081 | 2900.74 | 0.000 |
| Error | 16 | 0.0309 | 0.0019 | | |
| Total | 23 | 96.3600 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0439697 | 99.97% | 99.95% | 99.93% |

## Comparisons for Total Solid Content, Term= WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_2.5 | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 72 | 6 | 84.6550 | A | | | |
| 48 | 6 | 82.3533 | | B | | |
| 24 | 6 | 81.5167 | | | C | |
| 0 | 6 | 80.8783 | | | | D |

Means that do not share a letter are significantly different.

## Comparisons for Total Solid Content, Term= ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5 | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 12 | 83.4775 | A | |
| 20 | 12 | 81.2242 | | B |

Means that do not share a letter are significantly different.

## Comparisons for Total Solid Content, Term= WIP*ST

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_2.5*ST_2.5 | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 72 24 | 3 | 86.9700 | A | | | |
| 48 24 | 3 | 82.3833 | | B | | |
| 72 20 | 3 | 82.3400 | | B | | |
| 48 20 | 3 | 82.3233 | | B | | |
| 24 24 | 3 | 82.2933 | | B | | |
| 0 24 | 3 | 82.2633 | | B | | |
| 24 20 | 3 | 80.7400 | | | C | |
| 0 20 | 3 | 79.4933 | | | | D |

Table A.2. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST) and unit weigh (jelly type /jt ) on total soluble solid content (TSS) ( for non- oil coated)

### (13) General Linear Model: TSS versus Type; ST

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| JT | Fixed | 3 | 2.5; 3.0; 6.0 |
| ST | Fixed | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| JT | 2 | 3.429 | 1.7146 | 14.18 | 0.000 |
| ST | 3 | 15.213 | 5.0710 | 41.95 | 0.000 |
| Type*ST | 6 | 12.138 | 2.0230 | 16.74 | 0.000 |
| Error | 24 | 2.901 | 0.1209 | | |
| Total | 35 | 33.682 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.347680 | 91.39% | 87.44% | 80.62% |

## Comparisons for TSS ; Term = JT

Grouping Information Using the Tukey Method and 95% Confidence

| Type | N | Mean | Grouping | |
|---|---|---|---|---|
| 2.5 | 12 | 79.7967 | A | |
| 6.0 | 12 | 79.1574 | | B |
| 3.0 | 12 | 79.1275 | | B |

Means that do not share a letter are significantly different.

## Comparisons for TSS, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 9 | 80.4700 | A | |
| 20 | 9 | 79.1467 | | B |

```
16  9  78.9922         B
12  9  78.8332         B
```

Means that do not share a letter are significantly different.

## Comparisons: TSS, Term = jt*ST

Grouping Information Using the Tukey Method and 95% Confidence

| JT | *ST | N | Mean | Grouping | |
|---|---|---|---|---|---|
| 2.5 | 24 | 3 | 82.2633 | A | |
| 6.0 | 24 | 3 | 79.7000 | B | |
| 2.5 | 20 | 3 | 79.4933 | B | C |
| 3.0 | 24 | 3 | 79.4467 | B | C |
| 6.0 | 16 | 3 | 79.1000 | B | C |
| 3.0 | 20 | 3 | 79.0933 | B | C |
| 3.0 | 16 | 3 | 79.0467 | B | C |
| 6.0 | 12 | 3 | 78.9764 | B | C |
| 3.0 | 12 | 3 | 78.9233 | B | C |
| 6.0 | 20 | 3 | 78.8533 | B | C |
| 2.5 | 16 | 3 | 78.8300 | B | C |
| 2.5 | 12 | 3 | 78.6000 | | C |

Means that do not share a letter are significantly different.

* NOTE * Cannot draw the interval plot for the Tukey procedure. Interval plots for
comparisons are illegible with more than 45 intervals.

## (13) One-way ANOVA: TSS versus Stoving time ( ST_3) for 3 g sample

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| ST_3 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_3 | 3 | 0.4538 | 0.15125 | 1.77 | 0.230 |
| Error | 8 | 0.6823 | 0.08528 | | |
| Total | 11 | 1.1360 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.292033 | 39.94% | 17.42% | 0.00% |

Means

| ST_3 | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| 12 | 3 | 78.923 | 0.443 | ( 78.535; 79.312) |
| 16 | 3 | 79.047 | 0.305 | ( 78.658; 79.435) |
| 20 | 3 | 79.0933 | 0.1419 | (78.7045; 79.4821) |
| 24 | 3 | 79.447 | 0.178 | ( 79.058; 79.835) |

Pooled StDev = 0.292033

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| ST_3 | N | Mean | Grouping |
|---|---|---|---|
| 24 | 3 | 79.447 | A |
| 20 | 3 | 79.0933 | A |
| 16 | 3 | 79.047 | A |
| 12 | 3 | 78.923 | A |

Means that do not share a letter are significantly different.

### (14) One-way ANOVA: TSS versus Stoving time (ST_6) for 6 g sample

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| ST_6 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_6 | 3 | 1.269 | 0.4229 | 1.57 | 0.271 |
| Error | 8 | 2.156 | 0.2694 | | |
| Total | 11 | 3.424 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.519080 | 37.05% | 13.45% | 0.00% |

Means

| ST_6 | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| 12 | 3 | 78.976 | 0.965 | ( 78.285; 79.667) |
| 16 | 3 | 79.1000 | 0.0100 | (78.4089; 79.7911) |
| 20 | 3 | 78.8533 | 0.0058 | (78.1622; 79.5444) |
| 24 | 3 | 79.700 | 0.383 | ( 79.009; 80.391) |

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6 | N | Mean | Grouping |
|---|---|---|---|
| 24 | 3 | 79.700 | A |
| 16 | 3 | 79.1000 | A |
| 12 | 3 | 78.976 | A |
| 21 | 3 | 78.8533 | A |

## (15) One-way ANOVA: TSS versus Stoving time (ST_2.5) for 2.5 g sample

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| ST_2.5 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_2.5 | 3 | 25.6287 | 8.54291 | 1079.10 | 0.000 |
| Error | 8 | 0.0633 | 0.00792 | | |
| Total | 11 | 25.6921 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0889757 | 99.75% | 99.66% | 99.45% |

Means

| ST_2.5 | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| 12 | 3 | 78.600 | 0.177 | ( 78.482; 78.718) |
| 16 | 3 | 78.8300 | 0.0173 | (78.7115; 78.9485) |
| 20 | 3 | 79.4933 | 0.0058 | (79.3749; 79.6118) |
| 24 | 3 | 82.2633 | 0.0058 | (82.1449; 82.3818) |

Pooled StDev = 0.0889757

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 24 | 3 | 82.2633 | A |
| 20 | 3 | 79.4933 | B |
| 16 | 3 | 78.8300 | C |
| 12 | 3 | 78.600 | C |

Table A.3. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST) and unit weigh (jelly type / jt) on Moisture Content (MC) ( for non-coated sample)

### (1) General Linear Model: Moisture Content versus JT; ST

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| JT | Fixed | 3 | 2.5; 3.0; 6.0 |
| ST | Fixed | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| JT | 2 | 9.3594 | 4.67972 | 133.89 | 0.000 |
| ST | 3 | 4.2811 | 1.42703 | 40.83 | 0.000 |
| JT*ST | 6 | 1.6278 | 0.27130 | 7.76 | 0.000 |
| Error | 24 | 0.8389 | 0.03495 | | |
| Total | 35 | 16.1072 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.186957 | 94.79% | 92.40% | 88.28% |

## Comparisons for Moisture Content, Term= JT

Grouping Information Using the Tukey Method and 95% Confidence

| JT | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 6.0 | 12 | 19.2692 | A | | |
| 3.0 | 12 | 19.0650 | | B | |
| 2.5 | 12 | 18.1000 | | | C |

Means that do not share a letter are significantly different.

## Comparisons for Moisture Content, Term= ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 12 | 9 | 19.2389 | A | | |
| 16 | 9 | 19.0067 | A | | |
| 20 | 9 | 18.6744 | | B | |
| 24 | 9 | 18.3256 | | | C |

## Comparisons for Moisture Content, Term= JT*ST

Grouping Information Using the Tukey Method and 95% Confidence

| JT*ST | N | Mean | Grouping | | | | | |
|---|---|---|---|---|---|---|---|---|
| 6.0 12 | 3 | 19.7900 | A | | | | | |
| 6.0 16 | 3 | 19.4867 | A | B | | | | |
| 6.0 20 | 3 | 19.3400 | A | B | | | | |
| 3.0 12 | 3 | 19.1667 | | B | C | | | |
| 3.0 16 | 3 | 19.1667 | | B | C | | | |
| 3.0 20 | 3 | 18.9633 | | B | C | D | | |
| 3.0 24 | 3 | 18.9633 | | B | C | D | | |
| 2.5 12 | 3 | 18.7600 | | | C | D | E | |
| 6.0 24 | 3 | 18.4600 | | | | D | E | |
| 2.5 16 | 3 | 18.3667 | | | | | E | |
| 2.5 20 | 3 | 17.7200 | | | | | | F |
| 2.5 24 | 3 | 17.5533 | | | | | | F |

## (2) One-way ANOVA: Moisture Content versus Stoving Time ( ST_2.5) for 2.5 g samples

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| ST_2.5 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_2.5 | 3 | 2.8499 | 0.94996 | 13.15 | 0.002 |
| Error | 8 | 0.5781 | 0.07227 | | |
| Total | 11 | 3.4280 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.268825 | 83.13% | 76.81% | 62.05% |

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 12 | 3 | 18.7600 | A |
| 16 | 3 | 18.367 | A B |
| 20 | 3 | 17.720 | B C |
| 24 | 3 | 17.5533 | C |

## (3) One-way ANOVA: Moisture Content versus Stoving Time ( ST_3) for 3 g samples

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| ST_3 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_3 | 3 | 0.4460 | 0.14865 | 11.09 | 0.003 |
| Error | 8 | 0.1073 | 0.01341 | | |
| Total | 11 | 0.5532 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.115794 | 80.61% | 73.34% | 56.37 |

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| ST_3 | N | Mean | Grouping |
|---|---|---|---|
| 12 | 3 | 19.1667 | A |
| 16 | 3 | 19.0367 | A |
| 20 | 3 | 18.9633 | A |
| 24 | 3 | 18.643 | B |

## (4) One-way ANOVA: Moisture Content versus Stoving Time ( ST_6) for 6 g samples

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| ST_6 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_6 | 3 | 2.9350 | 0.97834 | 32.07 | 0.000 |
| Error | 8 | 0.2441 | 0.03051 | | |
| Total | 11 | 3.1791 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.174666 | 92.32% | 89.44% | 82.73% |

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6 | N | Mean | Grouping |
|---|---|---|---|
| 12 | 3 | 19.7900 | A |
| 16 | 3 | 19.4867 | A |
| 20 | 3 | 19.340 | A |
| 24 | 3 | 18.4600 | B |

Table A.4. Analysis of variance of gelatin based confectionery. Effect of WIP, Stoving time (ST) and unit weigh (jelly type / jt) on Moisture Content (oil coated sample)

## (1) General Linear Model: Moisture Content versus WIP; ST; jt

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| ST | Fixed | 2 | 20; 24 |
| jt | Fixed | 3 | 2.5; 3.0; 6.0 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST | 1 | 2.0436 | 2.0436 | 44.91 | 0.000 |
| WIP | 3 | 3.5923 | 1.1974 | 26.31 | 0.000 |
| jt | 2 | 42.5919 | 21.2960 | 467.97 | 0.000 |
| WIP*ST | 3 | 0.8410 | 0.2803 | 6.16 | 0.001 |
| ST *jt | 2 | 0.8123 | 0.4061 | 8.92 | 0.001 |
| WIP*jt | 6 | 0.4511 | 0.0752 | 1.65 | 0.154 |
| WIP*ST *jt | 6 | 1.0288 | 0.1715 | 3.77 | 0.004 |
| Error | 48 | 2.1843 | 0.0455 | | |
| Total | 71 | 53.5453 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.213324 | 95.92% | 93.97% | 90.82% |

## Comparisons for Moisture

## Tukey Pairwise Comparisons: Response = Moisture, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 | 36 | 18.7308 | A | |
| 24 | 36 | 18.3939 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Moisture, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 0 | 18 | 18.8372 | A | | |
| 24 | 18 | 18.6522 | A | B | |
| 48 | 18 | 18.5372 | | B | |
| 72 | 18 | 18.2228 | | | C |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Moisture, Term = jt

Grouping Information Using the Tukey Method and 95% Confidence

| jt | N | Mean | Grouping | |
|---|---|---|---|---|
| 6.0 | 24 | 19.1625 | A | |
| 3.0 | 24 | 19.0479 | A | |
| 2.5 | 24 | 17.4767 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Moisture, Term = ST*WIP

Grouping Information Using the Tukey Method and 95% Confidence

| ST | *WIP | N | Mean | Grouping | | |
|---|---|---|---|---|---|---|
| 20 | 0 | 9 | 18.9278 | A | | |
| 20 | 48 | 9 | 18.8678 | A | | |
| 20 | 24 | 9 | 18.8522 | A | | |
| 24 | 0 | 9 | 18.7467 | A | B | |
| 24 | 24 | 9 | 18.4522 | | B | C |
| 20 | 72 | 9 | 18.2756 | | | C |
| 24 | 48 | 9 | 18.2067 | | | C |
| 24 | 72 | 9 | 18.1700 | | | C |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Moisture, Term = ST *jt

Grouping Information Using the Tukey Method and 95% Confidence

| ST | *jt | N | Mean | Grouping | | |
|---|---|---|---|---|---|---|
| 20 | 6.0 | 12 | 19.4100 | A | | |
| 20 | 3.0 | 12 | 19.2875 | A | | |
| 24 | 6.0 | 12 | 18.9150 | | B | |
| 24 | 3.0 | 12 | 18.8083 | | B | |
| 20 | 2.5 | 12 | 17.4950 | | | C |
| 24 | 2.5 | 12 | 17.4583 | | | C |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Moisture, Term = WIP*jt

Grouping Information Using the Tukey Method and 95% Confidence

| WIP*jt | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 0 6.0 | 6 | 19.6133 | A | | | |
| 0 3.0 | 6 | 19.2617 | A | B | | |
| 24 6.0 | 6 | 19.2133 | A | B | | |
| 24 3.0 | 6 | 19.1600 | | B | | |
| 48 6.0 | 6 | 19.1050 | | B | C | |
| 48 3.0 | 6 | 19.0633 | | B | C | |
| 72 6.0 | 6 | 18.7183 | | | C | |
| 72 3.0 | 6 | 18.7067 | | | C | |
| 0 2.5 | 6 | 17.6367 | | | | D |
| 24 2.5 | 6 | 17.5833 | | | | D |
| 48 2.5 | 6 | 17.4433 | | | | D |
| 72 2.5 | 6 | 17.2433 | | | | D |

Means that do not share a letter are significantly different.

## (2) General Linear Model: Moisture Content (MC) versus Stoving time (ST_6); WIP_6 time for 6 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST_6 | Fixed | 2 | 20; 24 |
| WIP_6 | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_6 | 1 | 1.4702 | 1.47015 | 40.88 | 0.000 |
| WIP_6 | 3 | 2.4385 | 0.81285 | 22.60 | 0.000 |
| ST_6*WIP_6 | 3 | 0.4780 | 0.15932 | 4.43 | 0.019 |
| Error | 16 | 0.5754 | 0.03596 | | |
| Total | 23 | 4.9621 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.189638 | 88.40% | 83.33% | 73.91% |

### Tukey Pairwise Comparisons: Response =MC , Term = WIP_6

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_6 | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 0 | 6 | 19.6133 | A | | |
| 24 | 6 | 19.2133 | | B | |
| 48 | 6 | 19.1050 | | B | |
| 72 | 6 | 18.7183 | | | C |

### Tukey Pairwise Comparisons: Response = MC , Term = ST_6

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6 | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 | 12 | 19.410 | A | |
| 24 | 12 | 18.915 | | B |

### Tukey Pairwise Comparisons: Response = MC, Term = ST_6*WIP_6

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6*WIP_6 | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 0 | 3 | 19.8467 | A | |
| 20 24 | 3 | 19.6233 | A | |
| 20 48 | 3 | 19.4233 | A | |
| 24 0 | 3 | 19.3800 | A | |
| 24 24 | 3 | 18.8033 | | B |
| 24 48 | 3 | 18.7867 | | B |
| 20 72 | 3 | 18.7467 | | B |
| 24 72 | 3 | 18.6900 | | B |

Means that do not share a letter are significantly different.

### (3) General Linear Model: Moisture Content versus Stoving time(ST_3); WIP_3 time for 3 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | Fixed | 2 | 20; 24 |
| WIP_3 | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST | 1 | 1.3776 | 1.37760 | 32.31 | 0.000 |
| WIP_3 | 3 | 1.0496 | 0.34988 | 8.21 | 0.002 |
| ST*WIP_3 | 3 | 0.9245 | 0.30816 | 7.23 | 0.003 |
| Error | 16 | 0.6823 | 0.04264 | | |
| Total | 23 | 4.0340 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.206499 | 83.09% | 75.69% | 61.95% |

## Tukey Pairwise Comparisons: Response = MC, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 | 12 | 19.2875 | A | |
| 24 | 12 | 18.8083 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = MC, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_3 | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 | 6 | 19.2617 | A | |
| 24 | 6 | 19.1600 | A | |
| 48 | 6 | 19.0633 | A | |
| 72 | 6 | 18.7067 | | B |

## Tukey Pairwise Comparisons: Response = MC, Term = ST_3*WIP_3

Grouping Information Using the Tukey Method and 95% Confidence

| ST_3*WIP_3 | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 20 48 | 3 | 19.5533 | A | | |
| 20 24 | 3 | 19.3467 | A | | |
| 24 0 | 3 | 19.3067 | A | | |
| 20 0 | 3 | 19.2167 | A | | |
| 20 72 | 3 | 19.0333 | A | B | |
| 24 24 | 3 | 18.9733 | A | B | |
| 24 48 | 3 | 18.5733 | | B | C |
| 24 72 | 3 | 18.3800 | | | C |

Means that do not share a letter are significantly different.

## (4) General Linear Model: Moisture Content versus Stoving time(ST_2.5); WIP_2.5 time for 2.5 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST_2.5 | Fixed | 2 | 20; 24 |
| WIP_2.5 | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_2.5 | 1 | 0.00807 | 0.008067 | 0.14 | 0.714 |
| WIP_2.5 | 3 | 0.55520 | 0.185067 | 3.20 | 0.052 |
| ST_2.5*WIP_2.5 | 3 | 0.46740 | 0.155800 | 2.69 | 0.081 |
| Error | 16 | 0.92667 | 0.057917 | | |
| Total | 23 | 1.95733 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.240659 | 52.66% | 31.94% | 0.00% |

## Tukey Pairwise Comparisons: Response = MC, Term = ST_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 20 | 12 | 17.4950 | A |
| 24 | 12 | 17.4583 | A |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response =MC , Term = WIP_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 0 | 6 | 17.6367 | A |
| 24 | 6 | 17.5833 | A |
| 48 | 6 | 17.4433 | A |
| 72 | 6 | 17.2433 | A |

## Tukey Pairwise Comparisons: Response =MC, Term = ST_2.5*WIP_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5*WIP_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 20 0 | 3 | 17.7200 | A |
| 20 48 | 3 | 17.6267 | A |
| 20 24 | 3 | 17.5867 | A |
| 24 24 | 3 | 17.5800 | A |
| 24 0 | 3 | 17.5533 | A |
| 24 72 | 3 | 17.4400 | A |
| 24 48 | 3 | 17.2600 | A |
| 20 72 | 3 | 17.0467 | A |

Table A.5. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST) and unit weigh (jelly type / jt) on water activity (for non- oil coated)

### (1) General Linear Model: Water activity (aw) versus JT; ST

Method

Factor coding  (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| Jelly type | Fixed | 3 | 2.5; 3.0; 6.0 |
| Stoving time | Fixed | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Jelly type | 2 | 0.025393 | 0.012697 | 50.05 | 0.000 |
| Stoving time | 3 | 0.037808 | 0.012603 | 49.68 | 0.000 |
| Jelly type*Stoving time | 6 | 0.033033 | 0.005505 | 21.70 | 0.000 |
| Error | 24 | 0.006089 | 0.000254 | | |
| Total | 35 | 0.102323 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0159278 | 94.05% | 91.32% | 86.61% |

### Tukey Pairwise Comparisons: Response = aw, Term = jt

Grouping Information Using the Tukey Method and 95% Confidence

| Jt | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 2.5 | 12 | 0.517083 | A | | |
| 6.0 | 12 | 0.477000 | | B | |
| 3.0 | 12 | 0.452667 | | | C |

### Tukey Pairwise Comparisons: Response = aw, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 20 | 9 | 0.515333 | A | | |
| 24 | 9 | 0.505889 | A | | |
| 16 | 9 | 0.475556 | | B | |
| 12 | 9 | 0.432222 | | | C |

### (2) One-way ANOVA: Water Activity versus Stoving time (ST_6) for 6 g samples

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| St_6 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| St_6 | 3 | 0.019299 | 0.006433 | 26.27 | 0.000 |
| Error | 8 | 0.001959 | 0.000245 | | |
| Total | 11 | 0.021258 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0156498 | 90.78% | 87.33% | 79.26% |

Grouping Information Using the Tukey Method and 95% Confidence

| St_6 | N | Mean | Grouping |
|---|---|---|---|
| 20 | 3 | 0.53567 | A |
| 16 | 3 | 0.49233 | B |
| 12 | 3 | 0.4430 | C |
| 24 | 3 | 0.43700 | C |

### (3) **One-way ANOVA: Water Activity versus Stoving time (ST_3) for 3 g samples**

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | $\alpha = 0.05$ |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| St_3 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| St_3 | 3 | 0.031353 | 0.010451 | 126.29 | 0.000 |
| Error | 8 | 0.000662 | 0.000083 | | |
| Total | 11 | 0.032015 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0090967 | 97.93% | 97.16% | 95.35% |

Grouping Information Using the Tukey Method and 95% Confidence

| St_3 | N | Mean | Grouping |
|---|---|---|---|
| 24 | 3 | 0.52500 | A |
| 20 | 3 | 0.47667 | B |
| 12 | 3 | 0.40567 | C |
| 16 | 3 | 0.40333 | C |

## (4) **One-way ANOVA: Water Activity versus Stoving time (ST_2.5) for 2.5 g samples**

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| St_2.5 | 4 | 12; 16; 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Stoving | 3 | 0.020190 | 0.006730 | 15.53 | 0.001 |
| Error | 8 | 0.003467 | 0.000433 | | |
| Total | 11 | 0.023657 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0208187 | 85.34% | 79.85% | 67.02% |

Grouping Information Using the Tukey Method and 95% Confidence

| St_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 24 | 3 | 0.55567 | A |
| 20 | 3 | 0.53367 | A |
| 16 | 3 | 0.53100 | A |
| 12 | 3 | 0.4480 | B |

Table A.6. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST), WIP Time and unit weigh (jelly type / jt) on water activity (oil coated)

### (1) General Linear Model: Water activity versus Stoving time (ST), WIP Time and unit weigh (jelly type / jt)

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | Fixed | 2 | 20; 24 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| jt | Fixed | 3 | 2.5; 3.0; 6.0 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST | 1 | 0.001786 | 0.001786 | 0.44 | 0.510 |
| WIP | 3 | 0.053559 | 0.017853 | 4.41 | 0.008 |
| jt | 2 | 0.011173 | 0.005586 | 1.38 | 0.261 |
| ST*WIP | 3 | 0.010068 | 0.003356 | 0.83 | 0.484 |
| ST*jt | 2 | 0.021016 | 0.010508 | 2.60 | 0.085 |
| WIP*jt | 6 | 0.073494 | 0.012249 | 3.03 | 0.014 |
| ST*WIP*jt | 6 | 0.039020 | 0.006503 | 1.61 | 0.166 |
| Error | 48 | 0.194214 | 0.004046 | | |
| Total | 71 | 0.404330 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0636092 | 51.97% | 28.95% | 0.00% |

### Tukey Pairwise Comparisons: Response = AW, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping |
|---|---|---|---|
| 24 | 36 | 0.562989 | A |
| 20 | 36 | 0.553028 | A |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | |
|---|---|---|---|---|
| 72 | 18 | 0.586889 | A | |
| 24 | 18 | 0.573444 | A | |
| 48 | 18 | 0.557311 | A | B |
| 0 | 18 | 0.514389 | | B |

## Tukey Pairwise Comparisons: Response = AW, Term = jt

Grouping Information Using the Tukey Method and 95% Confidence

| jt | N | Mean | Grouping |
|---|---|---|---|
| 3.0 | 24 | 0.573000 | A |
| 2.5 | 24 | 0.558525 | A |
| 6.0 | 24 | 0.542500 | A |

Means that do not share a letter are significantly different.

## (2) General Linear Model: Water activity (AW_2.5) versus ST(2.5); WIP for 2.5 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST_2.5 | Fixed | 2 | 20; 24 |
| WIP_2.5 | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_2.5 | 1 | 0.005686 | 0.005686 | 0.47 | 0.502 |
| WIP_2.5 | 3 | 0.048518 | 0.016173 | 1.34 | 0.296 |
| ST_2.5*WIP_2.5 | 3 | 0.028708 | 0.009569 | 0.79 | 0.515 |
| Error | 16 | 0.192643 | 0.012040 | | |
| Total | 23 | 0.275555 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.109728 | 30.09% | 0.00% | 0.00% |

## Tukey Pairwise Comparisons: Response = AW_2.5, Term = ST_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 20 | 12 | 0.573917 | A |
| 24 | 12 | 0.543133 | A |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW_2.5, Term = WIP_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 24 | 6 | 0.609167 | A |
| 72 | 6 | 0.588000 | A |
| 0 | 6 | 0.545333 | A |
| 48 | 6 | 0.491600 | A |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW_2.5, Term = ST_2.5*WIP_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5*WIP_2.5 | N | Mean | Grouping |
|---|---|---|---|
| 24 24 | 3 | 0.617000 | A |
| 20 24 | 3 | 0.601333 | A |
| 20 72 | 3 | 0.595000 | A |
| 24 72 | 3 | 0.581000 | A |
| 20 48 | 3 | 0.565667 | A |
| 24 0 | 3 | 0.557000 | A |
| 20 0 | 3 | 0.533667 | A |
| 24 48 | 3 | 0.417533 | A |

Means that do not share a letter are significantly different.

### (3) General Linear Model: Water activity (AW_3) versus ST_3; WIP for 3 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST_3 | Fixed | 2 | 20; 24 |
| WIP_3 | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_3 | 1 | 0.000368 | 0.000368 | 5.27 | 0.036 |
| WIP_3 | 3 | 0.054036 | 0.018012 | 257.93 | 0.000 |
| ST_3*WIP_3 | 3 | 0.010708 | 0.003569 | 51.11 | 0.000 |
| Error | 16 | 0.001117 | 0.000070 | | |
| Total | 23 | 0.066230 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0083566 | 98.31% | 97.57% | 96.20% |

## Tukey Pairwise Comparisons: Response = AW_3, Term = ST_3

Grouping Information Using the Tukey Method and 95% Confidence

| ST_3 | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 12 | 0.576917 | A | |
| 20 | 12 | 0.569083 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW_3, Term = WIP_3

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_3 | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 72 | 6 | 0.612167 | A | | |
| 48 | 6 | 0.595000 | | B | |
| 24 | 6 | 0.593000 | | B | |
| 0 | 6 | 0.491833 | | | C |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW_3, Term = ST_3*WIP_3

Grouping Information Using the Tukey Method and 95% Confidence

| ST_3*WIP_3 | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 20 72 | 3 | 0.626667 | A | | | |
| 20 48 | 3 | 0.603667 | A | B | | |
| 24 72 | 3 | 0.597667 | | B | | |
| 20 24 | 3 | 0.593667 | | B | | |
| 24 24 | 3 | 0.592333 | | B | | |
| 24 48 | 3 | 0.586333 | | B | | |
| 24 0 | 3 | 0.531333 | | | C | |
| 20 0 | 3 | 0.452333 | | | | D |

Means that do not share a letter are significantly different.

## (4) General Linear Model: Water activity (AW_6) versus ST_6; WIP for 6 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST_6 | Fixed | 2 | 20; 24 |
| WIP_6 | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST_6 | 1 | 0.016748 | 0.016748 | 590.24 | 0.000 |
| WIP_6 | 3 | 0.024498 | 0.008166 | 287.79 | 0.000 |
| ST_6*WIP_6 | 3 | 0.009671 | 0.003224 | 113.62 | 0.000 |
| Error | 16 | 0.000454 | 0.000028 | | |
| Total | 23 | 0.051372 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0053268 | 99.12% | 98.73% | 98.01% |

## Tukey Pairwise Comparisons: Response = AW_6, Term = ST_6

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6 | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 12 | 0.568917 | A | |
| 20 | 12 | 0.516083 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW_6, Term = WIP_6

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_6 | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 48 | 6 | 0.585333 | A | | | |
| 72 | 6 | 0.560500 | | B | | |
| 24 | 6 | 0.518167 | | | C | |
| 0 | 6 | 0.506000 | | | | D |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = AW_6, Term = ST_6*WIP_6

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6*WIP_6 | N | Mean | Grouping | | | | | |
|---|---|---|---|---|---|---|---|---|
| 24 48 | 3 | 0.627667 | A | | | | | |
| 24 72 | 3 | 0.590333 | | B | | | | |
| 24 24 | 3 | 0.559000 | | | C | | | |
| 20 48 | 3 | 0.543000 | | | | D | | |
| 20 72 | 3 | 0.530667 | | | | D | | |
| 20 0 | 3 | 0.513333 | | | | | E | |
| 24 0 | 3 | 0.498667 | | | | | E | |
| 20 24 | 3 | 0.477333 | | | | | | F |

Means that do not share a letter are significantly different.

Table A.7. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST), WIP and unit weigh (jelly type / jt) on Hardness ( for oil coated)

## (1) General Linear Model: Hardness versus jt; WIP; ST

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| jt | Fixed | 3 | 2.5; 3.0; 6.0 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| ST | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| jt | 2 | 94802 | 47400.9 | 150.45 | 0.000 |
| WIP | 3 | 110313 | 36771.1 | 116.71 | 0.000 |
| ST | 1 | 14805 | 14804.6 | 46.99 | 0.000 |
| jt*WIP | 6 | 190567 | 31761.2 | 100.81 | 0.000 |
| jt*ST | 2 | 359 | 179.5 | 0.57 | 0.567 |
| WIP*ST | 3 | 1861 | 620.4 | 1.97 | 0.120 |
| jt*WIP*ST | 6 | 9191 | 1531.8 | 4.86 | 0.000 |
| Error | 196 | 61750 | 315.1 | | |
| Total | 219 | 483186 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 17.7497 | 87.22% | 85.72% | 83.87% |

## Tukey Pairwise Comparisons: Response = hardness, Term = jt

Grouping Information Using the Tukey Method and 95% Confidence

| jt | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 2.5 | 72 | 560.328 | A | | |
| 6.0 | 73 | 524.725 | | B | |
| 3.0 | 75 | 509.887 | | | C |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = hardness, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 72 | 52 | 569.191 | A | | | |
| 48 | 57 | 533.808 | | B | | |
| 24 | 58 | 516.750 | | | C | |
| 0 | 53 | 506.838 | | | | D |

## Tukey Pairwise Comparisons: Response = hardness, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 111 | 540.044 | A | |
| 20 | 109 | 523.249 | | B |

## (2) General Linear Model: Hardness versus ST ; WIP for 3 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | Fixed | 2 | 20; 24 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST | 1 | 3613 | 3613.0 | 16.38 | 0.000 |
| WIP | 3 | 2947 | 982.2 | 4.45 | 0.007 |
| ST *WIP | 3 | 2763 | 921.0 | 4.18 | 0.009 |
| Error | 67 | 14778 | 220.6 | | |
| Total | 74 | 25141 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 14.8514 | 41.22% | 35.08% | 26.71% |

## Tukey Pairwise Comparisons: Response = hardness, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

ST N Mean Grouping

| | | | |
|---|---|---|---|
| 24 | 39 | 516.997 | A |
| 20 | 36 | 502.778 | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = hardness, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | |
|---|---|---|---|---|
| 72 | 23 | 517.696 | A | |
| 24 | 17 | 514.395 | A | B |
| 48 | 19 | 504.511 | | B |
| 0 | 16 | 502.948 | | B |

## Tukey Pairwise Comparisons: Response = hardness, Term = ST*WIP

Grouping Information Using the Tukey Method and 95% Confidence

| ST *WIP | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 24 72 | 14 | 528.547 | A | | |
| 24 48 | 11 | 519.608 | A | B | |
| 24 24 | 7 | 518.210 | A | B | |
| 20 24 | 10 | 510.579 | A | B | C |
| 20 72 | 9 | 506.846 | | B | C |
| 20 0 | 9 | 504.274 | | B | C |
| 24 0 | 7 | 501.621 | | B | C |
| 20 48 | 8 | 489.414 | | | C |

## (3) General Linear Model: Hardness versus WIP; ST for 2.5 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| ST | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP | 3 | 671.4 | 223.8 | 0.95 | 0.423 |
| ST | 1 | 7270.4 | 7270.4 | 30.82 | 0.000 |
| WIP*ST | 3 | 4930.0 | 1643.3 | 6.97 | 0.000 |
| Error | 64 | 15099.7 | 235.9 | | |
| Total | 71 | 27109.8 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|

15.3601  44.30%     38.21%      30.04%

### Tukey Pairwise Comparisons: Response =Hardness_2.5, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 0 | 16 | 565.409 | A |
| 48 | 18 | 560.449 | A |
| 72 | 17 | 558.127 | A |
| 24 | 21 | 557.327 | A |

Means that do not share a letter are significantly different.

### Tukey Pairwise Comparisons: Response = Hardness_2.5, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 35 | 570.510 | A | |
| 20 | 37 | 550.145 | | B |

Means that do not share a letter are significantly different.

### Tukey Pairwise Comparisons: Response =Hardness_2.5, Term = WIP*ST

Grouping Information Using the Tukey Method and 95% Confidence

| WIP*ST | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 72 24 | 10 | 581.495 | A | | |
| 0 24 | 7 | 572.560 | A | B | |
| 48 24 | 8 | 570.102 | A | B | |
| 0 20 | 9 | 558.258 | | B | C |
| 24 24 | 10 | 557.884 | | B | C |
| 24 20 | 11 | 556.769 | | B | C |
| 48 20 | 10 | 550.795 | | B | C |
| 72 20 | 7 | 534.759 | | | C |

### (4) General Linear Model: Hardness versus ST; WIP for 6 g sample

Method

Factor coding  (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | Fixed | 2 | 20; 24 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| ST | 1 | 4270 | 4269.8 | 8.71 | 0.004 |
| WIP | 3 | 276482 | 92160.6 | 187.95 | 0.000 |
| ST*WIP | 3 | 3683 | 1227.8 | 2.50 | 0.067 |
| Error | 65 | 31873 | 490.3 | | |
| Total | 72 | 316955 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 22.1438 | 89.94% | 88.86% | 87.20% |

## Tukey Pairwise Comparisons: Response = Hardness_6, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 | 37 | 532.626 | A | |
| 20 | 36 | 516.823 | | B |

## Tukey Pairwise Comparisons: Response = Hardness_6, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 72 | 12 | 631.751 | A | | | |
| 48 | 20 | 536.464 | | B | | |
| 24 | 20 | 478.528 | | | C | |
| 0 | 21 | 452.156 | | | | D |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Hardness_6, Term = ST*WIP

Grouping Information Using the Tukey Method and 95% Confidence

| ST*WIP | N | Mean | Grouping | | | |
|---|---|---|---|---|---|---|
| 24 72 | 5 | 635.782 | A | | | |
| 20 72 | 7 | 627.720 | A | | | |
| 24 48 | 10 | 538.063 | | B | | |
| 20 48 | 10 | 534.865 | | B | | |
| 24 24 | 11 | 498.065 | | | C | |
| 20 24 | 9 | 458.991 | | | | D |
| 24 0 | 11 | 458.595 | | | | D |
| 20 0 | 10 | 445.717 | | | | D |

Table 5.8. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST), WIP Time and unit weigh (jelly type / jt) on Stickiness (for oil coated)

### (1) General Linear Model: Stickiness versus jt; WIP; ST

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| jt | Fixed | 3 | 2.5; 3.0; 6.0 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| ST | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| jt | 2 | 1719.38 | 859.692 | 56.64 | 0.000 |
| WIP | 3 | 813.24 | 271.079 | 17.86 | 0.000 |
| ST | 1 | 133.17 | 133.165 | 8.77 | 0.003 |
| jt*WIP | 6 | 79.31 | 13.219 | 0.87 | 0.517 |
| jt*ST | 2 | 25.04 | 12.520 | 0.82 | 0.440 |
| WIP*ST | 3 | 9.02 | 3.008 | 0.20 | 0.898 |
| jt*WIP*ST | 6 | 285.72 | 47.620 | 3.14 | 0.006 |
| Error | 196 | 2975.07 | 15.179 | | |
| Total | 219 | 6305.25 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 3.89602 | 52.82% | 47.28% | 39.35% |

### Tukey Pairwise Comparisons: Response = Stickiness, Term = jt

Grouping Information Using the Tukey Method and 95% Confidence

| jt | N | Mean | Grouping | |
|---|---|---|---|---|
| 3.0 | 75 | -32.6514 | A | |
| 6.0 | 73 | -33.1110 | A | |
| 2.5 | 72 | -38.9304 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Stickiness, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | | |
|---|---|---|---|---|---|
| 0 | 53 | -32.9018 | A | | |
| 24 | 58 | -33.5448 | A | B | |
| 48 | 57 | -34.9586 | | B | |
| 72 | 52 | -38.1853 | | | C |

## Tukey Pairwise Comparisons: Response = Stickiness, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 | 109 | -34.1012 | A | |
| 24 | 111 | -35.6941 | | B |

### (2) General Linear Model: Stickiness versus WIP_2.5; ST_2.5 for 2.5 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP_2.5 | Fixed | 4 | 0; 24; 48; 72 |
| ST_2.5 | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP_2.5 | 3 | 239.72 | 79.906 | 13.03 | 0.000 |
| ST_2.5 | 1 | 25.02 | 25.018 | 4.08 | 0.048 |
| WIP_2.5*ST_2.5 | 3 | 144.06 | 48.022 | 7.83 | 0.000 |
| Error | 64 | 392.51 | 6.133 | | |
| Total | 71 | 845.91 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 2.47647 | 53.60% | 48.52% | 41.99% |

## Tukey Pairwise Comparisons: Response = Stickiness, Term = WIP_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_2.5 | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 | 16 | -37.3056 | A | |
| 24 | 21 | -37.5978 | A | |
| 48 | 18 | -38.7635 | A | |
| 72 | 17 | -42.0549 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Stickiness, Term = ST_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| ST_2.5 | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 | 37 | -38.3331 | A | |
| 24 | 35 | -39.5277 | | B |

## Tukey Pairwise Comparisons: Response = Stickiness, Term = WIP_2.5*ST_2.5

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_2.5*ST_2.5 | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 24 | 7 | -36.3800 | A | |
| 24 24 | 10 | -36.8920 | A | |
| 48 20 | 10 | -37.1420 | A | |
| 0 20 | 9 | -38.2311 | A | |
| 24 20 | 11 | -38.3036 | A | |
| 72 20 | 7 | -39.6557 | A | |
| 48 24 | 8 | -40.3850 | A | |
| 72 24 | 10 | -44.4540 | | B |

## (3) General Linear Model: Stickiness versus WIP_3; ST_3 for 3 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP_3 | Fixed | 4 | 0; 24; 48; 72 |
| ST_3 | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP_3 | 3 | 214.99 | 71.664 | 5.12 | 0.003 |
| ST_3 | 1 | 117.16 | 117.156 | 8.37 | 0.005 |
| WIP_3*ST_3 | 3 | 11.67 | 3.890 | 0.28 | 0.841 |
| Error | 67 | 938.10 | 14.002 | | |
| Total | 74 | 1358.50 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 3.74186 | 30.95% | 23.73% | 12.69% |

## Tukey Pairwise Comparisons: Response = Stickness, Term = WIP_3

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_3 | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 | 16 | -31.1921 | A | |
| 24 | 17 | -31.9826 | A | |
| 48 | 19 | -32.0002 | A | |
| 72 | 23 | -35.4306 | | B |

## Tukey Pairwise Comparisons: Response = Stickness, Term = ST_3

Grouping Information Using the Tukey Method and 95% Confidence

| ST_3 | N | Mean | Grouping |
|---|---|---|---|
| 20 | 36 | -31.3712 | A |
| 24 | 39 | -33.9316 | |

## Tukey Pairwise Comparisons: Response = Stickness, Term = WIP_3*ST_3

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_3*ST_3 | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 20 | 9 | -29.7000 | A | |
| 24 20 | 10 | -30.9580 | A | |
| 48 20 | 8 | -31.1912 | A | |
| 0 24 | 7 | -32.6843 | A | B |
| 48 24 | 11 | -32.8091 | A | B |
| 24 24 | 7 | -33.0071 | A | B |
| 72 20 | 9 | -33.6356 | A | B |
| 72 24 | 14 | -37.2257 | | B |

Means that do not share a letter are significantly different.

### (4) General Linear Model: Stickness versus WIP_6; ST_6 for 6 g samples

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| WIP_6 | Fixed | 4 | 0; 24; 48; 72 |
| ST_6 | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| WIP_6 | 3 | 446.06 | 148.69 | 5.88 | 0.001 |
| ST_6 | 1 | 17.92 | 17.92 | 0.71 | 0.403 |
| WIP_6*ST_6 | 3 | 141.62 | 47.21 | 1.87 | 0.144 |
| Error | 65 | 1644.47 | 25.30 | | |
| Total | 72 | 2291.68 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 5.02986 | 28.24% | 20.51% | 6.31% |

## Tukey Pairwise Comparisons: Response = Stickness, Term = WIP_6

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_6 | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 | 21 | -30.2076 | A | |
| 24 | 20 | -31.0541 | A | |
| 48 | 20 | -34.1120 | A | B |
| 72 | 12 | -37.0703 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = Stickness, Term = ST_6

Grouping Information Using the Tukey Method and 95% Confidence

| ST_6 | N | Mean | Grouping |
|---|---|---|---|
| 20 | 36 | -32.5991 | A |
| 24 | 37 | -33.6229 | A |

### Tukey Pairwise Comparisons: Response = Stickness, Term = WIP_6*ST_6

Grouping Information Using the Tukey Method and 95% Confidence

| WIP_6*ST_6 | N | Mean | Grouping | |
|---|---|---|---|---|
| 24 20 | 9 | -28.4300 | A | |
| 0 20 | 10 | -29.4280 | A | |
| 0 24 | 11 | -30.9873 | A | |
| 24 24 | 11 | -33.6782 | A | B |
| 48 20 | 10 | -33.8000 | A | B |
| 48 24 | 10 | -34.4240 | A | B |
| 72 24 | 5 | -35.4020 | A | B |
| 72 20 | 7 | -38.7386 | | B |

Means that do not share a letter are significantly different.

Table A.9. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST), WIP Time and unit weigh (jelly type / jt) on T2 Relaxation Time (oil coated)

### (1) General Linear Model: T2 versus JT; WIP; ST

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| JT | Fixed | 3 | 2.5; 3.0; 6.0 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| ST | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| JT | 2 | 0.07743 | 0.038715 | 29.61 | 0.000 |
| WIP | 3 | 0.04963 | 0.016542 | 12.65 | 0.000 |
| ST | 1 | 0.01090 | 0.010903 | 8.34 | 0.006 |
| JT*WIP | 6 | 0.02925 | 0.004874 | 3.73 | 0.004 |
| JT*ST | 2 | 0.05112 | 0.025560 | 19.55 | 0.000 |
| WIP*ST | 3 | 0.01401 | 0.004669 | 3.57 | 0.021 |
| JT*WIP*ST | 6 | 0.07573 | 0.012621 | 9.65 | 0.000 |
| Error | 48 | 0.06276 | 0.001307 | | |
| Total | 71 | 0.37081 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0361579 | 83.08% | 74.97% | 61.92% |

## Tukey Pairwise Comparisons: Response = T2, Term = JT

Grouping Information Using the Tukey Method and 95% Confidence

| JT | N | Mean | Grouping | |
|---|---|---|---|---|
| 3.0 | 24 | 0.718553 | A | |
| 2.5 | 24 | 0.705899 | A | |
| 6.0 | 24 | 0.643529 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = T2, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping | |
|---|---|---|---|---|
| 0 | 18 | 0.734678 | A | |
| 48 | 18 | 0.677203 | | B |
| 72 | 18 | 0.673517 | | B |
| 24 | 18 | 0.671911 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = T2, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping | |
|---|---|---|---|---|
| 20 | 36 | 0.701633 | A | |
| 24 | 36 | 0.677021 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = T2, Term = JT*WIP

Grouping Information Using the Tukey Method and 95% Confidence

| JT*WIP | N | Mean | Grouping | | | | |
|---|---|---|---|---|---|---|---|
| 3.0 0 | 6 | 0.771195 | A | | | | |
| 2.5 0 | 6 | 0.727500 | A | B | | | |
| 2.5 24 | 6 | 0.722300 | A | B | | | |
| 3.0 24 | 6 | 0.709328 | A | B | C | | |
| 3.0 48 | 6 | 0.706998 | A | B | C | D | |
| 6.0 0 | 6 | 0.705338 | A | B | C | D | |
| 2.5 72 | 6 | 0.697963 | | B | C | D | |
| 3.0 72 | 6 | 0.686690 | | B | C | D | |
| 2.5 48 | 6 | 0.675833 | | B | C | D | |
| 6.0 48 | 6 | 0.648779 | | | C | D | E |
| 6.0 72 | 6 | 0.635897 | | | | D | E |
| 6.0 24 | 6 | 0.584103 | | | | | E |

Means that do not share a letter are significantly different.

## (2) General Linear Model: T2_2.5 versus ST; WIP for 2.5 g samples

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | fixed | 2 | 20; 24 |
| WIP | fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance for T2 2.5, using Adjusted SS for Tests

| Source | DF | Seq SS | Adj SS | Adj MS | F | P |
|---|---|---|---|---|---|---|
| ST | 1 | 0.000024 | 0.000024 | 0.000024 | 0.09 | 0.764 |
| WIP | 3 | 0.010215 | 0.010215 | 0.003405 | 13.27 | 0.000 |
| ST*WIP | 3 | 0.073167 | 0.073167 | 0.024389 | 95.06 | 0.000 |
| Error | 16 | 0.004105 | 0.004105 | 0.000257 | | |
| Total | 23 | 0.087511 | | | | |

S = 0.0160179   R-Sq = 95.31%   R-Sq(adj) = 93.26%

Unusual Observations for T2 2.5

| Obs | T2 2.5 | Fit | SE Fit | Residual | St Resid | |
|---|---|---|---|---|---|---|
| 7 | 0.684000 | 0.712333 | 0.009248 | -0.028333 | -2.17 | R |
| 9 | 0.750000 | 0.712333 | 0.009248 | 0.037667 | 2.88 | R |

R denotes an observation with a large standardized residual.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | N | Mean | Grouping |
|---|---|---|---|
| 20 | 12 | 0.7 | A |
| 24 | 12 | 0.7 | A |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 0 | 6 | 0.7 | A |
| 24 | 6 | 0.7 | A B |
| 72 | 6 | 0.7 | B C |
| 48 | 6 | 0.7 | C |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | WIP | N | Mean | Grouping |
|---|---|---|---|---|
| 24 | 72 | 3 | 0.8 | A |
| 20 | 24 | 3 | 0.8 | A B |
| 20 | 0 | 3 | 0.7 | B C |
| 24 | 0 | 3 | 0.7 | C D |
| 20 | 48 | 3 | 0.7 | C D |
| 24 | 24 | 3 | 0.7 | D E |
| 24 | 48 | 3 | 0.6 | E F |
| 20 | 72 | 3 | 0.6 | F |

Means that do not share a letter are significantly different.

## (3) General Linear Model: T2_3 versus ST; WIP for 3 g samples

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | fixed | 2 | 20; 24 |
| WIP | fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance for T2 3, using Adjusted SS for Tests

| Source | DF | Seq SS | Adj SS | Adj MS | F | P |
|---|---|---|---|---|---|---|
| ST | 1 | 0.057835 | 0.057835 | 0.057835 | 77.53 | 0.000 |
| WIP | 3 | 0.024030 | 0.024030 | 0.008010 | 10.74 | 0.000 |
| ST*WIP | 3 | 0.000742 | 0.000742 | 0.000247 | 0.33 | 0.803 |
| Error | 16 | 0.011935 | 0.011935 | 0.000746 | | |
| Total | 23 | 0.094542 | | | | |

S = 0.0273116   R-Sq = 87.38%   R-Sq(adj) = 81.85%

Unusual Observations for T2 3

| Obs | T2 3 | Fit | SE Fit | Residual | St Resid |
|---|---|---|---|---|---|
| 16 | 0.617230 | 0.668767 | 0.015768 | -0.051537 | -2.31 R |

R denotes an observation with a large standardized residual.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | N | Mean | Grouping |
|---|---|---|---|
| 20 | 12 | 0.8 | A |
| 24 | 12 | 0.7 | B |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 0 | 6 | 0.8 | A |
| 24 | 6 | 0.7 | B |
| 48 | 6 | 0.7 | B |
| 72 | 6 | 0.7 | B |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | WIP | N | Mean | Grouping |
|---|---|---|---|---|
| 20 | 0 | 3 | 0.8 | A |
| 20 | 48 | 3 | 0.8 | A B |
| 20 | 24 | 3 | 0.7 | B |
| 20 | 72 | 3 | 0.7 | B C |
| 24 | 0 | 3 | 0.7 | B C D |
| 24 | 24 | 3 | 0.7 | C D E |
| 24 | 48 | 3 | 0.7 | D E |
| 24 | 72 | 3 | 0.6 | E |

Means that do not share a letter are significantly different.

## (4) General Linear Model: T2_6 versus ST; WIP for 6 g samples

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | fixed | 2 | 20; 24 |
| WIP | fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance for T2 6, using Adjusted SS for Tests

| Source | DF | Seq SS | Adj SS | Adj MS | F | P |
|---|---|---|---|---|---|---|
| ST | 1 | 0.004164 | 0.004164 | 0.004164 | 1.43 | 0.250 |
| WIP | 3 | 0.044626 | 0.044626 | 0.014875 | 5.09 | 0.012 |
| ST*WIP | 3 | 0.015824 | 0.015824 | 0.005275 | 1.81 | 0.187 |
| Error | 16 | 0.046715 | 0.046715 | 0.002920 | | |
| Total | 23 | 0.111329 | | | | |

S = 0.0540342   R-Sq = 58.04%   R-Sq(adj) = 39.68%

Unusual Observations for T2 6

| Obs | T2 6 | Fit | SE Fit | Residual | St Resid |
|---|---|---|---|---|---|
| 5 | 0.689340 | 0.583847 | 0.031197 | 0.105493 | 2.39 R |

R denotes an observation with a large standardized residual.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | N | Mean | Grouping |
|---|---|---|---|
| 24 | 12 | 0.7 | A |
| 20 | 12 | 0.6 | A |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 0 | 6 | 0.7 | A |
| 48 | 6 | 0.6 | A B |
| 72 | 6 | 0.6 | A B |
| 24 | 6 | 0.6 | B |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | WIP | N | Mean | Grouping |
|---|---|---|---|---|
| 24 | 0 | 3 | 0.8 | A |
| 24 | 48 | 3 | 0.7 | A B |
| 20 | 0 | 3 | 0.7 | A B |
| 20 | 72 | 3 | 0.7 | A B |
| 20 | 48 | 3 | 0.6 | A B |
| 24 | 72 | 3 | 0.6 | A B |
| 24 | 24 | 3 | 0.6 | B |
| 20 | 24 | 3 | 0.6 | B |

Means that do not share a letter are significantly different.

Table 5.10. Analysis of variance of gelatin based confectionery. Effect of Stoving time (ST), WIP Time and unit weigh (jelly type / jt) on T1 Relaxation Time (oil coated)

## (1) General Linear Model: T1 versus JT; WIP; ST

Method

Factor coding (-1; 0; +1)

Factor Information

| Factor | Type | Levels | Values |
|---|---|---|---|
| JT | Fixed | 3 | 2.5; 3.0; 6.0 |
| WIP | Fixed | 4 | 0; 24; 48; 72 |
| ST | Fixed | 2 | 20; 24 |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| JT | 2 | 124.575 | 62.2875 | 160.33 | 0.000 |
| WIP | 3 | 1.758 | 0.5861 | 1.51 | 0.224 |
| ST | 1 | 0.040 | 0.0401 | 0.10 | 0.749 |
| JT*WIP | 6 | 5.174 | 0.8623 | 2.22 | 0.057 |
| JT*ST | 2 | 14.773 | 7.3865 | 19.01 | 0.000 |
| WIP*ST | 3 | 0.652 | 0.2173 | 0.56 | 0.644 |
| JT*WIP*ST | 6 | 4.769 | 0.7949 | 2.05 | 0.078 |
| Error | 48 | 18.648 | 0.3885 | | |
| Total | 71 | 170.389 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.623292 | 89.06% | 83.81% | 75.38% |

## Tukey Pairwise Comparisons: Response = T1, Term = JT

Grouping Information Using the Tukey Method and 95% Confidence

| JT | N | Mean | Grouping | |
|---|---|---|---|---|
| 6.0 | 24 | 47.6674 | A | |
| 2.5 | 24 | 47.5842 | A | |
| 3.0 | 24 | 44.8364 | | B |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = T1, Term = WIP

Grouping Information Using the Tukey Method and 95% Confidence

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 72 | 18 | 46.9169 | A |
| 48 | 18 | 46.7046 | A |
| 24 | 18 | 46.6873 | A |
| 0 | 18 | 46.4752 | A |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = T1, Term = ST

Grouping Information Using the Tukey Method and 95% Confidence

| ST | N | Mean | Grouping |
|---|---|---|---|
| 24 | 36 | 46.7196 | A |
| 20 | 36 | 46.6724 | A |

Means that do not share a letter are significantly different.

## Tukey Pairwise Comparisons: Response = T1, Term = JT*WIP

```
Grouping Information Using the Tukey Method and 95% Confidence

JT*WIP  N     Mean  Grouping
2.5 72  6  47.9100  A
6.0 48  6  47.8970  A
6.0 0   6  47.7065  A
6.0 24  6  47.6783  A
2.5 0   6  47.5733  A
2.5 24  6  47.4800  A
6.0 72  6  47.3879  A
2.5 48  6  47.3733  A
3.0 72  6  45.4527      B
3.0 24  6  44.9035      B  C
3.0 48  6  44.8435      B  C
3.0 0   6  44.1459         C

Means that do not share a letter are significantly different.

* NOTE * Cannot draw the interval plot for the Tukey procedure. Interval
plots for
         comparisons are illegible with more than 45 intervals.
```

## Tukey Pairwise Comparisons: Response = T1, Term = JT*ST

```
Grouping Information Using the Tukey Method and 95% Confidence

JT*ST    N     Mean  Grouping
6.0 20  12  48.0264  A
2.5 20  12  47.8142  A
2.5 24  12  47.3542  A
6.0 24  12  47.3084  A
3.0 24  12  45.4963      B
3.0 20  12  44.1765         C

Means that do not share a letter are significantly different.
```

## Tukey Pairwise Comparisons: Response = T1, Term = WIP*ST

```
Grouping Information Using the Tukey Method and 95% Confidence

WIP*ST  N     Mean  Grouping
72 24   9  46.9985  A
72 20   9  46.8352  A
48 24   9  46.8209  A
24 20   9  46.8194  A
48 20   9  46.5883  A
24 24   9  46.5552  A
0 24    9  46.5039  A
0 20    9  46.4466  A
Means that do not share a letter are significantly different.
```

## (2) General Linear Model: T1_2.5 versus ST; WIP for 2.5 g samples

```
Factor  Type   Levels  Values
ST      fixed       2  20; 24
WIP     fixed       4  0; 24; 48; 72


Analysis of Variance for T1 2.5, using Adjusted SS for Tests

Source  DF  Seq SS  Adj SS  Adj MS     F      P
ST       1  1.2696  1.2696  1.2696  6.10  0.025
WIP      3  0.969   5  0.9695  0.3232  1.55  0.239
ST*WIP   3  1.8199  1.8199  0.6066  2.92  0.066
Error   16  3.3280  3.3280  0.2080
Total   23  7.3870


S = 0.456070   R-Sq = 54.95%   R-Sq(adj) = 35.24%


Unusual Observations for T1 2.5

Obs   T1 2.5      Fit  SE Fit  Residual  St Resid
  4  47.3400  48.1033  0.2633   -0.7633     -2.05 R

R denotes an observation with a large standardized residual.
```

**Grouping Information Using Tukey Method and 95.0% Confidence**

```
ST   N  Mean  Grouping
20  12  47.8  A
24  12  47.4    B

Means that do not share a letter are significantly different.
```

**Grouping Information Using Tukey Method and 95.0% Confidence**

```
WIP  N  Mean  Grouping
72   6  47.9  A
 0   6  47.6  A
24   6  47.5  A
48   6  47.4  A

Means that do not share a letter are significantly different.
```

**Grouping Information Using Tukey Method and 95.0% Confidence**

```
ST  WIP  N  Mean  Grouping
20  72   3  48.2  A
20  24   3  48.1  A B
24   0   3  47.7  A B
24  72   3  47.7  A B
20  48   3  47.6  A B
20   0   3  47.4  A B
24  48   3  47.2  A B
24  24   3  46.9    B
```

Means that do not share a letter are significantly different.

## (3) General Linear Model: T1_3 versus ST; WIP for 3 g samples

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | fixed | 2 | 20; 24 |
| WIP | fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance for T1 3, using Adjusted SS for Tests

| Source | DF | Seq SS | Adj SS | Adj MS | F | P |
|---|---|---|---|---|---|---|
| ST | 1 | 10.4500 | 10.4500 | 10.4500 | 34.27 | 0.000 |
| WIP | 3 | 5.1677 | 5.1677 | 1.7226 | 5.65 | 0.008 |
| ST*WIP | 3 | 3.0955 | 3.0955 | 1.0318 | 3.38 | 0.044 |
| Error | 16 | 4.8793 | 4.8793 | 0.3050 | | |
| Total | 23 | 23.5925 | | | | |

S = 0.552228   R-Sq = 79.32%   R-Sq(adj) = 70.27%

Unusual Observations for T1 3

| Obs | T1 3 | Fit | SE Fit | Residual | St Resid |
|---|---|---|---|---|---|
| 2 | 45.2400 | 44.0733 | 0.3188 | 1.1667 | 2.59 R |

R denotes an observation with a large standardized residual.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | N | Mean | Grouping |
|---|---|---|---|
| 24 | 12 | 45.5 | A |
| 20 | 12 | 44.2 | B |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 72 | 6 | 45.5 | A |
| 24 | 6 | 44.9 | A B |
| 48 | 6 | 44.8 | A B |
| 0 | 6 | 44.1 | B |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | WIP | N | Mean | Grouping |
|---|---|---|---|---|
| 24 | 72 | 3 | 46.3 | A |
| 24 | 48 | 3 | 45.9 | A B |
| 24 | 24 | 3 | 45.6 | A B C |
| 20 | 72 | 3 | 44.6 | B C D |
| 24 | 0 | 3 | 44.2 | C D |
| 20 | 24 | 3 | 44.2 | C D |
| 20 | 0 | 3 | 44.1 | C D |
| 20 | 48 | 3 | 43.8 | D |

Means that do not share a letter are significantly different.

## (4) General Linear Model: T1_6 versus ST; WIP for 6 g samples

| Factor | Type | Levels | Values |
|---|---|---|---|
| ST | fixed | 2 | 20; 24 |
| WIP | fixed | 4 | 0; 24; 48; 72 |

Analysis of Variance for T1 6, using Adjusted SS for Tests

| Source | DF | Seq SS | Adj SS | Adj MS | F | P |
|---|---|---|---|---|---|---|
| ST | 1 | 3.0935 | 3.0935 | 3.0935 | 4.74 | 0.045 |
| WIP | 3 | 0.7949 | 0.7949 | 0.2650 | 0.41 | 0.751 |
| ST*WIP | 3 | 0.5059 | 0.5059 | 0.1686 | 0.26 | 0.854 |
| Error | 16 | 10.4404 | 10.4404 | 0.6525 | | |
| Total | 23 | 14.8347 | | | | |

S = 0.807790   R-Sq = 29.62%   R-Sq(adj) = 0.00%

Unusual Observations for T1 6

| Obs | T1 6 | Fit | SE Fit | Residual | St Resid |
|---|---|---|---|---|---|
| 4 | 49.8613 | 48.1645 | 0.4664 | 1.6968 | 2.57 R |
| 5 | 46.7240 | 48.1645 | 0.4664 | -1.4405 | -2.18 R |

R denotes an observation with a large standardized residual.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | N | Mean | Grouping |
|---|---|---|---|
| 20 | 12 | 48.0 | A |
| 24 | 12 | 47.3 | B |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| WIP | N | Mean | Grouping |
|---|---|---|---|
| 48 | 6 | 47.9 | A |
| 0 | 6 | 47.7 | A |
| 24 | 6 | 47.7 | A |
| 72 | 6 | 47.4 | A |

Means that do not share a letter are significantly different.

**Grouping Information Using Tukey Method and 95.0% Confidence**

| ST | WIP | N | Mean | Grouping |
|---|---|---|---|---|
| 20 | 48 | 3 | 48.4 | A |
| 20 | 24 | 3 | 48.2 | A |
| 20 | 0 | 3 | 47.8 | A |
| 20 | 72 | 3 | 47.7 | A |
| 24 | 0 | 3 | 47.6 | A |
| 24 | 48 | 3 | 47.4 | A |
| 24 | 24 | 3 | 47.2 | A |
| 24 | 72 | 3 | 47.1 | A |

Means that do not share a letter are significantly different.

Table A.11. General Correlation Table

## Correlation: T1; Hardness; TS; stickness; Moisture; aw; T2

| | T1 | Hardness | TS | stickness | Moisture | aw |
|---|---|---|---|---|---|---|
| Hardness | 0.939 | | | | | |
| | 0.061 | | | | | |
| TS | 0.996 | 0.922 | | | | |
| | 0.004 | 0.078 | | | | |
| stickness | -0.933 | -1.000 | -0.915 | | | |
| | 0.067 | 0.000 | 0.085 | | | |
| Moisture | -0.955 | -0.985 | -0.930 | 0.984 | | |
| | 0.045 | 0.015 | 0.070 | 0.016 | | |
| aw | 0.931 | 0.748 | 0.939 | -0.736 | -0.798 | |
| | 0.069 | 0.252 | 0.061 | 0.264 | 0.202 | |
| T2 | -0.820 | -0.596 | -0.858 | 0.583 | 0.614 | -0.941 |
| | 0.180 | 0.404 | 0.142 | 0.417 | 0.386 | 0.059 |

Cell Contents: Pearson correlation
P-Value

Table A.12. Correlation Table based on unit weight (H: Hardness; St: Stickiness; TSS: Total Solid Content; Moist : Moisture)

## Correlation: Moist 2.5; H2.5; St2.5; Aw2.5; KM2.5; T12.5; T22.5

| | Moist2.5 | H2.5 | St2.5 | Aw2.5 | TSS2.5 | T12.5 |
|---|---|---|---|---|---|---|
| H2.5 | 0.485 | | | | | |
| | 0.223 | | | | | |
| St2.5 | 0.355 | -0.267 | | | | |
| | 0.389 | 0.523 | | | | |
| Aw2.5 | 0.202 | -0.304 | 0.215 | | | |
| | 0.631 | 0.464 | 0.610 | | | |
| TS2.5 | 0.092 | 0.413 | -0.751 | 0.073 | | |
| | 0.829 | 0.309 | 0.032 | 0.864 | | |
| T12.5 | -0.312 | -0.336 | -0.096 | 0.329 | 0.368 | |
| | 0.452 | 0.415 | 0.821 | 0.426 | 0.370 | |
| T22.5 | 0.694 | 0.626 | -0.271 | 0.277 | 0.688 | 0.138 |
| | 0.056 | 0.097 | 0.516 | 0.507 | 0.059 | 0.744 |

Cell Contents: Pearson correlation
P-Value

## Correlation: TSS3; Moist3; Aw3; H3; St3; T13; T23

| | TSS3 | Moist3 | Aw3 | H3 | St3 | T13 |
|---|---|---|---|---|---|---|
| Moist3 | -0.355 | | | | | |
| | 0.388 | | | | | |
| Aw3 | 0.834 | -0.222 | | | | |
| | 0.010 | 0.598 | | | | |
| H3 | 0.363 | -0.638 | -0.084 | | | |
| | 0.377 | 0.089 | 0.843 | | | |
| St3 | -0.530 | 0.740 | -0.521 | -0.508 | | |
| | 0.177 | 0.036 | 0.186 | 0.198 | | |
| T13 | 0.548 | -0.955 | 0.363 | 0.715 | -0.783 | |
| | 0.159 | 0.000 | 0.377 | 0.046 | 0.022 | |
| T23 | -0.627 | 0.762 | -0.572 | -0.622 | 0.867 | -0.877 |
| | 0.096 | 0.028 | 0.138 | 0.100 | 0.005 | 0.004 |

Cell Contents: Pearson correlation
P-Value

## Correlation: TSS6; Nem6; Aw6; H6; St6; T16; T26

| | TSS6 | Moist6 | Aw6 | H6 | St6 | T16 |
|---|---|---|---|---|---|---|
| Moist6 | -0.567 | | | | | |
| | 0.143 | | | | | |
| Aw6 | 0.445 | -0.730 | | | | |
| | 0.269 | 0.040 | | | | |
| H6 | 0.871 | -0.821 | 0.674 | | | |
| | 0.005 | 0.012 | 0.067 | | | |
| St6 | -0.711 | 0.902 | -0.803 | -0.905 | | |
| | 0.048 | 0.002 | 0.016 | 0.002 | | |
| T16 | 0.054 | 0.721 | -0.584 | -0.350 | 0.513 | |
| | 0.900 | 0.044 | 0.128 | 0.396 | 0.193 | |
| T26 | -0.292 | 0.103 | -0.060 | -0.131 | 0.030 | -0.090 |
| | 0.483 | 0.809 | 0.888 | 0.758 | 0.943 | 0.833 |

Cell Contents: Pearson correlation p value