

INVESTIGATION OF SENSORY AND INSTRUMENTAL METHODS TO  
PREDICT SHELF- LIFE OF JELLY GUMS

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PREDICT SHELF-LIFE OF JELLY GUMS**

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## **ABSTRACT**

### **INVESTIGATION OF SENSORY AND INSTRUMENTAL METHODS TO PREDICT SHELF-LIFE OF JELLY GUMS**

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The main aim of this study was to correlate sensorial changes with instrumental techniques to predict shelf life during storage of jelly gums.

Effects of glucose syrup:sucrose ratio, starch and gelatine content on quality and sensorial properties of jellies were investigated. Glucose syrup: sucrose ratio, starch and gelatine contents affected hardness significantly. Moisture content, water activity, total soluble solids, pH, springiness and gumminess was influenced by only glucose syrup:sucrose ratio.

Moisture content changes after a week at 30°C were similar to those after 8 weeks at 15-22°C. At 30°C, the rate of change in water activity was more than double as compared to 10°C and 20°C. Formulations with glucose syrup:sucrose ratio of 1.5 were more stable in terms of pH and colour. Glass transition temperatures increased after storage. Formulation with glucose syrup:sucrose ratio of 1.5, no starch and 6% gelatine was the most stable formulation in terms of hardness and gumminess. Moisture diffusion coefficients were determined to be between  $5.93 \times 10^{-10} \text{m}^2/\text{s}$  and  $5.46 \times 10^{-10} \text{m}^2/\text{s}$ .

In sensory analysis, starch content was more effective on appearance and gelatine was more effective on texture of jellies. Storage of about 8 weeks at 30°C was equivalent to storage of 52 weeks at 15-22°C when sensory taste liking was correlated with sugar profile. Therefore, 30°C could be selected for accelerated shelf life testing.

Jellies with no starch, 6% gelatine and glucose syrup:sucrose ratio of 1.5 had the most stable formulation in terms of critical parameters with high sensory scores and shelf life was determined as 52 weeks.

**Keywords:** Shelf-life, confectionery, sensory testing

## ÖZ

### ŞEKERLEME ÜRÜNLERİNİN RAF ÖMRÜ TAHMİNİNDE DUYUSAL VE ENSTRÜMENTAL METOTLARIN İLİŞKİLENDİRİLMESİ

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Bu tezin ana amacı, enstrümental teknikler ile jöle şekerlemelerin depolanması boyunca gerçekleşen duysal değişimleri ilişkilendirerek raf ömrünün tahmin edilmesidir.

Glukoz şurubu:şeker oranı, nişasta ve jelatin miktarlarının jöle şekerlemelerin kalite ve duysal özelliklerine olan etkileri incelenmiştir. Glukoz şurubu:şeker oranı, nişasta ve jelatin jöle şekerlemelerin sertliğini önemli derecede etkilemiştir. Nem içeriği, su aktivitesi, toplam çözünabilir kuru madde, pH, esneklik ve sakızimsılık sadece glukoz şurubu:şeker oranından etkilenmiştir.

30°C’de bir hafta sonraki nem içeriği değişimi 15-22°C’de 8 hafta sonraki değişime benzer bulunmuştur. 30°C’de su aktivitesindeki değişim oranı 10°C ve 20°C’ye kıyasla iki kattan fazla olmuştur. Glukoz şurubu:şeker oranı 1.5 olan formülasyonlar pH ve renk açısından daha stabil olmuştur. Depolama sonrası camsı geçiş sıcaklıkları artmıştır. Sertlik ve sakızimsılık açısından depolama boyunca en stabil formülasyon glukoz şurubu:şeker oranı 1.5 olan ve %6 jelatin içeren nişastasız şekerleme

olmuştur. Nem difüzyon katsayıları  $5.93 \times 10^{-10} \text{m}^2/\text{s}$  ve  $5.46 \times 10^{-10} \text{m}^2/\text{s}$  olarak bulunmuştur.

Duyusal analizde, nişasta miktarı görünümde ve jelatin miktarı yapıda daha etkili olmuştur. Duyusal tat beğenisi şeker profili ile ilişkilendirildiğinde 8 haftalık 30°C depolama 52 haftalık 15-22°C depolamaya eşdeğer olduğu görülmüştür. Bu yüzden, hızlandırılmış raf ömrü testi için 30°C uygun bir sıcaklık olarak seçilebilir.

Duyusal değerlendirmede yüksek puan alan nişastasız, %6 jelatin ve 1.5 oranında glukoz şurubu:şeker içeren jöle şekerlemeler depolama boyunca kritik parametreler açısından en stabil formülasyon olmuştur ve raf ömrü 52 hafta olarak saptanmıştır.

**Anahtar Sözcükler:** Raf ömrü, şekerleme, duyusal test

*To My Family*

*Şükran Tireki, Mehmet Ali Tireki, Murat Tireki, Gülsüm Tireki*





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## LIST OF ABBREVIATIONS

$a_w$	Water activity
G	Glucose syrup
m.c.	Moisture content (%)
$R^2$	Coefficient of determination
RH	Relative humidity (%)
S	Sucrose
t	Time
Tg	Glass transition temperature
TSS	Total soluble solids (%)
$\Delta C$	Change in chroma
$\Delta E$	Total colour difference
$\Delta H$	Change in hue
$\Delta L$	Change in lightness





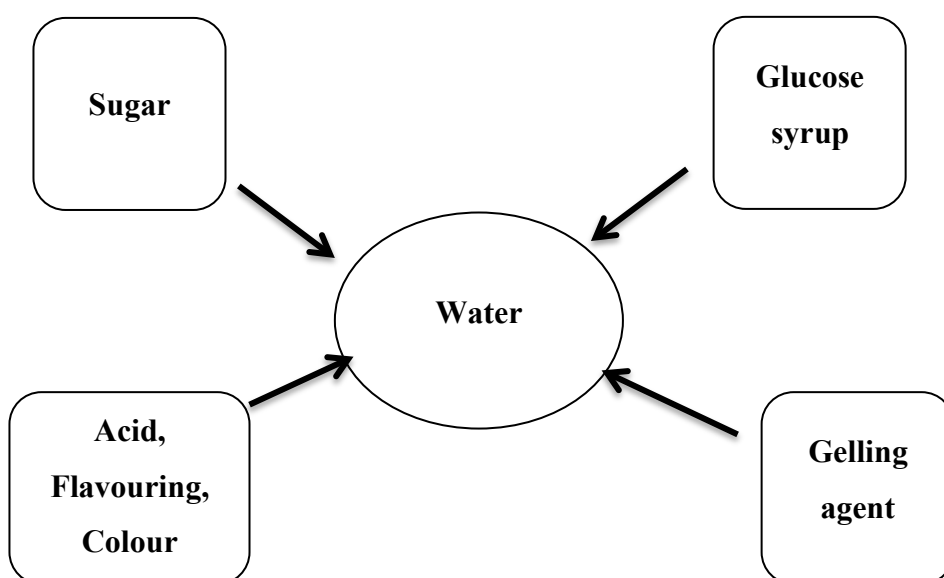
## CHAPTER I

### INTRODUCTION

#### 1.1 Production of Jelly Gum

The confectionery industry categorises confectionery products into three groups: chocolate confectionery, flour confectionery and sugar confectionery (Edwards, 2000). Jelly gums are popular sugar confectionery products, which range from hard to soft texture depending on the type of gelling agent used and glucose syrup grade (Hull, 2010).

According to structural formulae of various confectionery products obtained by the application of structure theory, structure formula of jelly gum is given in Figure 1.1 (Mohos, 2010).



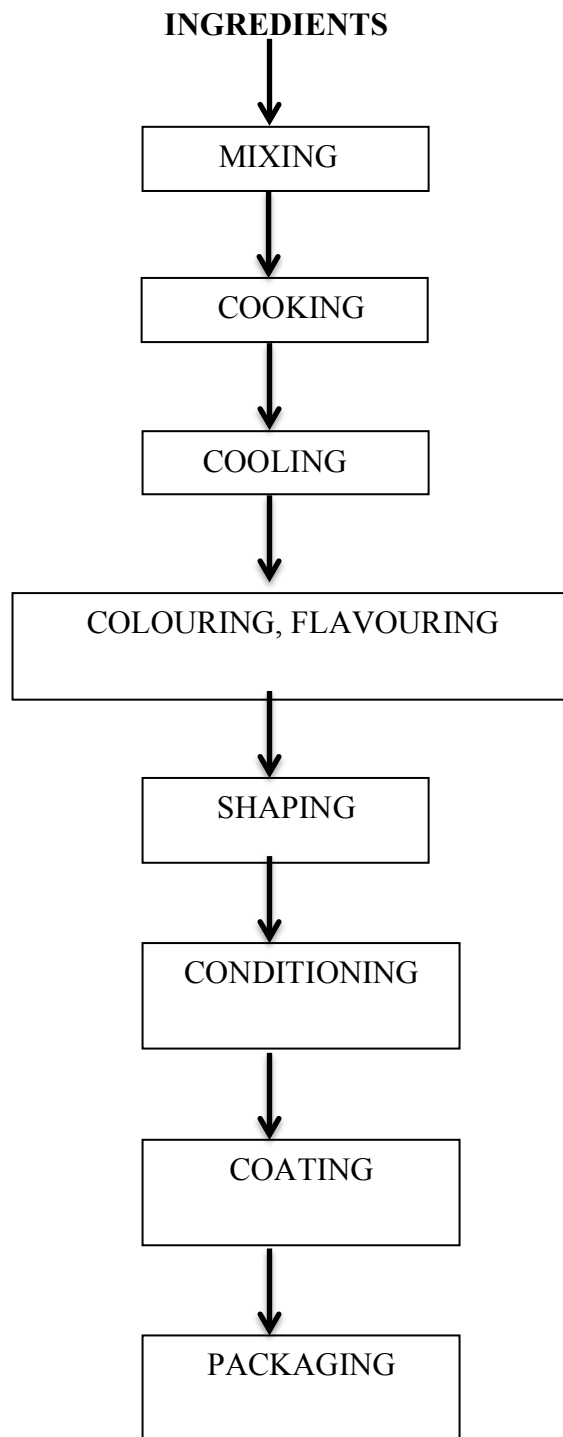
**Figure 1.1** Structural formula of jelly gum (Mohos, 2010).

According to their characteristic phase conditions, confectionery products are classified with the help of a 3x3 cartesian product given in Table 1.1 (Mohos, 1982). The mentioned table represents a combination of hydrophobic solutions (1), hydrophilic solutions (2) and hydrophilic solids (3). The first factor in an element of this Cartesian product shows the dominant (continuous) phase, and the second factor shows the dispersed phase. With respect to Table 1.1 jelly gums are classified as 2x3.

**Table 1.1** Cartesian product of phases (Mohos, 2010).

<b>1x1</b> Fat melts	<b>1x2</b> (Water-in-oil) Emulsions	<b>1x3</b> Chocolate, compounds
<b>2x1</b> (Oil-in-water) Toffee, ice cream, fudge	<b>2x2</b> Hard- boiled candies, Soft- boiled candies	<b>2x3</b> Jelly gums, foams, wafers
<b>3x1</b> Cocoa/chocolate powders, Pudding powders	<b>3x2</b> Dragees, tablets, lozenges	<b>3x3</b> Biscuits, crackers

Jelly gum production includes the stages of mixing, cooking, cooling, shaping (starch moulding), conditioning, coating and packaging (Mohos, 1982; Jackson 1990). A general flow diagram of jelly gum is shown in Figure 1.2.



**Figure 1.2** Flow diagram of jelly gum production.

Manufacturing processes of jelly gum can vary from plant to plant (Desrosier, 1977). The first production step is the mixing of main ingredients namely, glucose syrup, sugar, gelatine solution and water. Gelatine solution is prepared by hydration of

gelatine in the water generally at 60°C for two hours (Hull, 2010). After the mixing stage, cooking of the slurry is the next step. Cooking provides the reduction of the moisture content of a mixture of ingredients. Besides from moisture content reduction of a mixture formulated for a confectionery, there are other reasons for cooking such as to melt, solubilize, caramelize and invert (Alikonis, 1979).

Cooking is performed in three ways, which are batch, semi-continuous and continuous. In batch cooking, cooking may be either under vacuum, under steam pressure or at atmospheric pressure. In semi-continuous operation, cooking is carried out under vacuum, and continuous cooking takes place under either vacuum or atmospheric conditions. In batch operations; direct-fire cookers, steam-jacketed kettles, pressure kettles and vacuum cookers are involved equipments in the batch cooking of confectionery. Direct-fire cooking takes place under atmospheric conditions. Direct-fire cookers are conventional and old fashioned cooking equipment among candy manufacturers. However, improvements have been made in this cooker, like, sturdier stainless steel pans are used instead of copper pans, full draft air supplies and automatic flame controls (Alikonis, 1979). Steam-jacketed kettles are involved for open air cooking and for maintaining the product at fairly constant processing temperatures. In these equipments, pressures from 40 to 125 psi are employed. If needed, additional heat transfer surfaces are provided by putting booster coils in a direct way into a steam-jacketed kettle (Alikonis, 1979). In pressure kettles, application of high pressures increases the boiling point achieving higher temperature and faster cooking. Equipments for this aim are designed to operate at pressures up to 100 psi, which are generally equipped with a tightly fitted cover with openings for steam, pressure gauge, pressure release and safety valves (Alikonis, 1979). Batch type vacuum cookers are with jackets made of steel welded to 350-lb capacity copper pans and the pressures are up to 150 psi (Desrosier, 1977). Semicontinuous equipments cook under vacuum. In order to minimize heat transfer surface for removal of moisture under vacuum, the water content of a confectionery syrup is decreased prior to being fed into the vacuum chamber. This is done by batch heating a syrup mix under atmospheric pressure up to 110°C. Preheated slurry is then charged into the vacuum chamber in a continuous way. In semicontinuous processes,

two cookers are used for preheating a syrup mix. While one batch is being preheated in a cooker, a precook is discharged from another cooker and fed into the cooker's vacuum chamber. In addition, heat boosters may be added additionally in some cookers to accelerate precooking (Alikonis, 1979).

Continuous atmospheric cookers, continuous vacuum cookers and continuous pressure cookers are used as continuous cookers used in confectionery manufacturing. Continuous atmospheric cookers are falling film type of cookers, which are successful for concentrating confectionery syrups in a short time. Baker-Perkins (Microfilm), Groen and Buflovak types are the main brands used in confectionery industry. Baker- Perkins cooker consists of an agitator- equipped kettle, steam pressure jacketed evaporator tube and a water jacketed cooler in a conical shape. It cooks up to 160 °C and able to decrease the moisture content down to 1.5%. Groen's cooker is similar to Baker- Perkins except that in former agitation is not used, in which slurry is fed at one end. The slurry then falls along the inner surfaces of the concentrator tube and discharges at the opposite end (at temperatures up to 160 °C and moisture content of 1.5%). Buflovak type differs from Baker- Perkins and Groen in that it has a series of smaller diameter tubes in order to increase the heat transfer efficiencies. In Buflovak equipment, confectionery slurry is also top-fed and flows downward in a film form inside small steam-jacketed tubes, and cooked slurry then continuous to a unit that separates it from the cooker (Desrosier, 1977).

Muller cooker, Turba-Film processor and Roto-Vak cooker are continuous vacuum cooker equipments used in confectionery production. Muller cooker was the first continuous vacuum thin film concentrator, consisting of a vertical evaporator tube surrounded with a steam jacket. In Muller cooker, a separator is placed mounted above the tube and a rotor (placed on a vertical axis) passes through the separator and evaporator tube. Blades of the equipment extend from rotor shaft in order to form a thin layer of high turbulence product. Droplets carried over with the vapor are removed via separator and concentrated product discharges through a conical outlet at the bottom. The other continuous vacuum cooker type, Turba- Film processor uses

the same mechanically assisted thin film technique as does Muller type. Most of the caramel and jelly gum producers prefer this type of cooker. Regarding with the Roto-Vak continuous vacuum cooker, a thin film of product is redistributed and agitated with a rotor running through the evaporator tube. Liquid and vapor moving down in the tube, are separated in an external unit which is connected with the evaporation part of the cooker. Concentrated product is discharged below via a pump.

In jelly gum processing, in which gelatine is involved in the formulations, cooking stage is performed at 113-121°C and after cooking syrup is cooled down to 100 °C (Mohos, 2010). After cooling, flavours and colours are added to the cooled syrup with acids. A pH value of 3.0- 3.5 is optimum for flavor and gelling quality (Desrosier, 1977).

After addition of flavourings, colours and acids jelly gum syrup is deposited into starch shapes or moulds in starch trays via a starch moulding machine like given in Figure 1.3.



**Figure 1.3** Automated high speed starch moulding line (N.I.D. PTY. Ltd., N. S. W., Australia; <http://nid.com.au/products-services/mogul-lines/m3000/>)

Starch trays, with dimensions of 81.28 cm x 40.64 cm x 5.08 cm, are filled with corn starch, commercially known as moulding starch. Better impressions are stated in

literature with oiled starch containing 0.05-0.10 % of purified mineral oil (Minifie, 1970). The filled starch tray is printed with a metal plate to which are fixed rows of protrusions representing shape to be made in the starch and finally the shape of the jelly gum product.

Moulding starch has three purposes in the moulding step, which are,

- (a) it receives the jelly gum fluid deposited within its impressions and gives the desired shapes to them;
- (b) it retains the deposited jelly gums during subsequent drying, cooling and setting in conditioning stage;
- (c) it absorbs moisture from deposited jelly gums during conditioning.

Starch moulding process has below steps;

- (1) stacks of wooden trays containing starch and moulded jelly gums are received and are then fed to the moulding unit;
- (2) trays are emptied, starch and formed jelly gums removed from the trays;
- (3) jelly gums are separated from starch automatically;
- (4) jelly gums are removed from starch and they are transferred onto a conveyor belt;
- (5) separated starch is delivered to a starch conditioning system for cleaning, drying and cooling, and then the conditioned starch is returned to the starch moulding machines for reuse;
- (6) empty trays are filled with conditioned starch, then starch is flush- leveled to the top of the trays and excess starch is brushed off the trays;
- (7) starch is machine printed with male moulds to create desired shapes into which the jelly gum stock is to be deposited;
- (8) jelly gum fluid is deposited in the starch cavities;
- (9) trays are removed with starch and jelly gums, trays are stacked and then transferred into conditioning rooms for setting;
- (10) cycle is repeated.

After shaping of the jelly gums, the trays of starch and products are conditioned in a warm room for 24 hours or more until the correct texture is obtained. It is important to control the moisture content of starch and the time the jelly gums in starch in conditioning stage of the production (Minifie, 1970).

In conditioning stage, moulding starch pulls moisture out of the jelly gum piece and creates a skin on the surface, and this skin helps in preventing the deformation of jelly gums during removing from starch (Lees, 1979). Conditioning time may change from 24 to 72 hours depending on the product type and desired final moisture content. Drying speed during conditioning should be controlled in the manufacturing of jelly gums. If skin formation is too rapid, the surface of the product may become too hard and trap moisture inside; which can cause sweating on the surface during storage (Sudharsan et. al., 2004).

After conditioning stage, coating stage is performed for jelly gums with application of glazing agents in tumblers mostly continuously. Several waxes like beeswax, carnauba wax have applications in jelly gum industry. Glazing agents are applied with one of the waxes or they may be applied as an emulsion, sometimes combined with glyceryl monostearate (Alikonis, 1979).

## **1.2 Role of Ingredients in Jelly Gum Production**

### **1.2.1 Gelling Agents**

#### **1.2.1.1 Gelatine**

Gelatine (Figure 1.4) is used in the food industry due to its unique physical properties rather than for its nutritional properties as a protein. Jelly gums prepared with gelatine are unique in their easy reversibility, the closeness of the setting point to the melting point, and the ease of their preparation (Ward and Courts, 1977). Gelatine is a refined extract of collagenous tissue and is an amphoteric protein

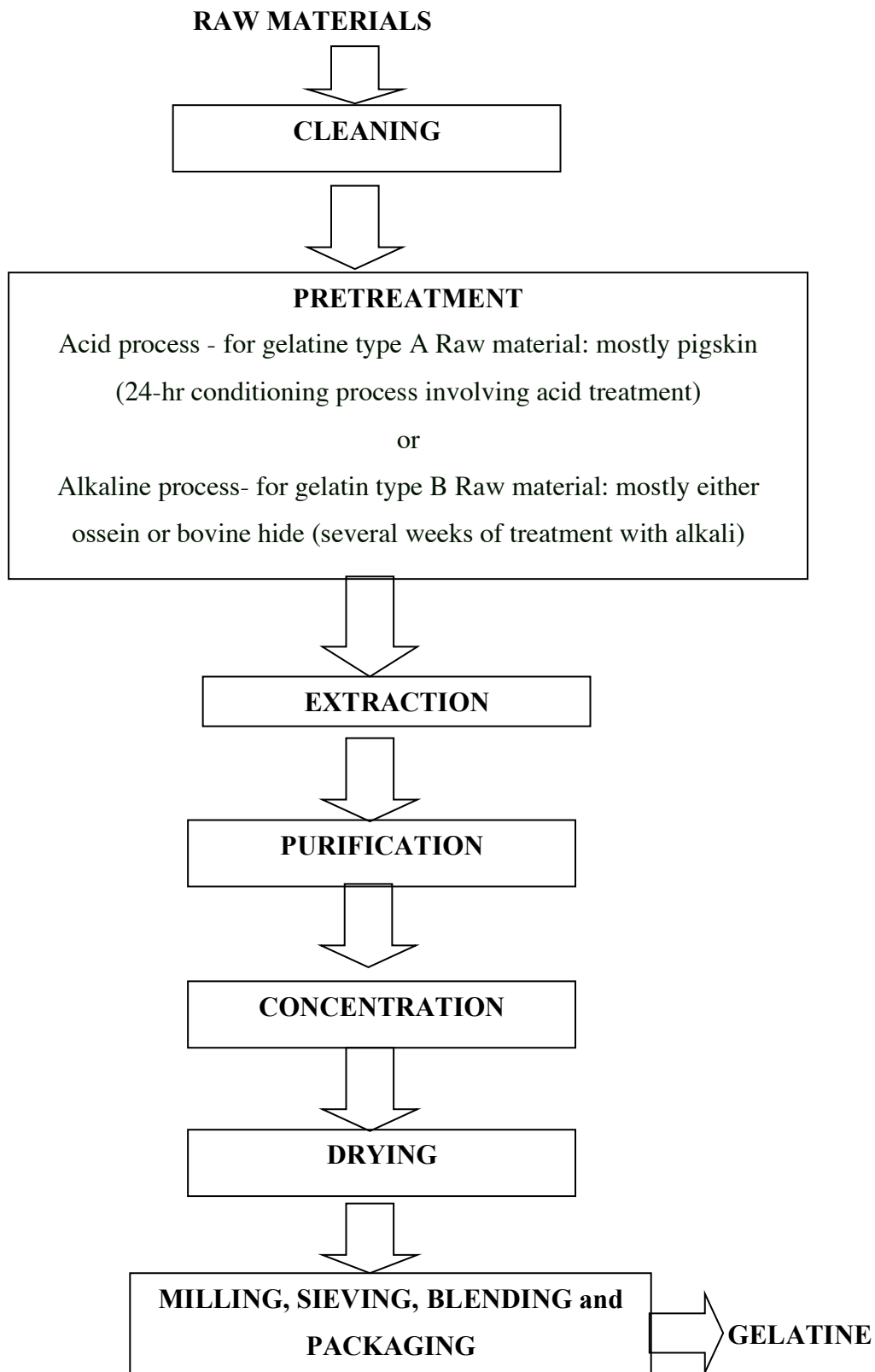


having both basic (amino and guanidine) and acidic (carboxyl) groups and is prepared by processes, involving destruction of the tertiary, secondary and to some extent the primary of native collagens, by the partial hydrolysis of collagen driven from the skin, white connective tissue and bones of animals. Overall charge of gelatine molecule depends on the solution pH and other ions present in the solution (Alikonis, 1979; Fernandez-Diaz et al., 2001; Morrison et al., 1999).



**Figure 1.4** Granulated industrial gelatine (Schrieber and Gareis, 2007)

Gelatine is extracted from animal tissues containing high amount of collagen, like, bones, skins and tendons. It can be extracted from these tissues by boiling water directly; but extraction and gelatine quality are improved by adjusting pH in an acid solution or by pretreating of the collagenous tissue in a prolonged soak in saturated lime. Gelatines may behave differently, depending on their processing i.e. whether they are acid-processed or lime-processed. Acid-processed gelatines are classified as type A and alkali-treated gelatines as type B according to the U.S. Pharmacopoeia (Ward and Courts, 1977). Gelatine manufacturing process is given Figure 1.5, which is explained in the website of Gelita, one of the world's leading collagen supplier (<http://www.gelita.com/knowledge/gelatine/what-is-gelatine/manufacture>).



**Figure 1.5** Gelatine manufacturing process

Human body can produce all the proteins needed from amino acids. However, there are nine amino acids that human body can not produce itself, which should be regularly present in human's diet. These amino acids are called essential amino acids. Gelatine is comprised of 18 amino acids with 9 essential amino acids ([http://www.gelatine.org/fileadmin/user\\_upload/downloads/press/publications\\_downloads/GME\\_all\\_about\\_gelatine\\_en.pdf](http://www.gelatine.org/fileadmin/user_upload/downloads/press/publications_downloads/GME_all_about_gelatine_en.pdf)). Table 1.2 shows various amino acids that can be obtained from some gelatines by complete hydrolysis (De Clerq and Quanten, 1986; Eastoe, 1955; Eastoe and Leach, 1958; Newman, 1949 and Rose, 1987).

**Table 1.2** Amino acid composition of some gelatines (grams/100 grams of dry gelatine) (\*: essential amino acid)

Amino acid	Type A (Porkskin)	Type B (Calf Skin)	Type B (Bone)
Alanine	8.6	9.3	10.1
Arginine*	8.3	8.55	5.0
Aspartic Acid	6.2	6.6	4.6
Glutamic Acid	11.3	11.1	8.5
Glycine	26.4	26.9	24.5
Histidine*	0.9	0.74	0.4
Hydroxylysine	1.0	0.91	0.7
Hydroxyproline	13.5	14.0	11.9
Isoleucine*	1.4	1.7	1.3
Leucine*	3.1	3.1	2.8
Lysine*	4.1	4.5	4.4
Methionine*	0.8	0.8	0.6
Phenylalanine*	2.1	2.2	1.3
Proline	16.2	14.8	13.5
Serine	2.9	3.2	3.4
Threonine*	2.2	2.2	2.0
Tyrosine	0.4	0.2	0.2
Valine *	2.5	2.6	2.4

Ability to form gels in water solution is the most important function of gelatine. The most important physical property of gelatine is the gel strength or bloom. Bloom is a function of gelatine's molecular weight which is generally between 50 and 300. Typical properties of edible gelatines are given in Table 1.3.

**Table 1.3** Properties of edible gelatines Type A and Type B

Property	Type A	Type B
pH	3.8- 5.5	5- 7.5
Isoelectric Point	7.0- 9.0	4.7- 5.4
Gel Strength (Bloom)	50- 300	50- 300
Viscosity (mps)	15- 75	20- 75
Ash (%)	0.3- 2.0	0.5- 2.0

Bloom value is used to determine the gelling strength and firmness of gelatine, the higher the bloom value the greater the gelling strength of the gelatine. The bloom test is empirical, however it is acceptable and is used to determine the gelatine commercial grade. Different blooms are used for different food applications (Table 1.4). (Johnston-Banks, 1990; Ward and Courts, 1977; Wittich, 2005).

Gelatine is compatible with a wide variety of foods and ingredients. It is mentioned that gelatine has been used to keep incompatible ingredients together. Some of the general nutritional information of gelatines is given in Table 1.5. The values given in the stated table are typical values and vary greatly depending on the raw material type and manufacturing method (Gelatine Manufacturers Institute of America, 2012).

**Table 1.4** Gelatine as a food ingredient

<b>Application</b>	<b>Use Level (%)</b>	<b>Bloom</b>
Dairy Products	0.2- 1.0	150- 250
Frozen Foods	0.1- 0.5	225- 250
Gelatine Desserts	7.0- 9.0	175- 275
Confectionery- Gummy Bears	7.0- 9.0	200- 250
Confectionery- Marshmallows	1.7- 2.5	225- 275
Confectionery- Circus Peanuts	2.0- 2.5	225- 250
Confectionery- Lozenges	0.5- 1.0	50- 100
Confectionery- Wafers	0.5- 1.0	50- 100
Bakery Fillings and Icings	1.0- 2.0	225- 250
Meat Products	1.0- 5.0	175- 275
Wine, Beer and Juices	0.002- 0.015	100- 200

**Table 1.5** Nutritional Information of Gelatine

<b>Nutritional information</b>	<b>Type A</b>	<b>Type B</b>
Moisture (%)	10.5± 1.5	10.5± 1.5
Fat (%)	0	0
Carbohydrates (%)	0	0
Ash (%)	0.5± 0.4	1.5± 0.5
Sodium (ppm)	500±200	3600±1400
Phosphorous (ppm)	200±1	-
Iron (ppm)	4±2	15±10
Lead (ppm)	0.002 ± 0.0002	0.005± 0.0002
Zinc (ppm)	1.5± 0.5	5 ± 3
Nitrogen (%)	16.2 ± 0.3	16.2± 0.3
Calcium (ppm)	90± 30	900±100
Potassium (ppm)	125± 50	330± 50
Calories/ 100 grams	360	360

Gelatine has a unique hydrocolloidal nature, which enables gelatine to find many applications in the food industry. Basically, there are four main groups of gelatines; confectionery and jelly desserts, dairy products, meat products and hydrolysed gelatine applications. Gelatine is used for providing chewiness, texture, and foam stabilization in confectionery. There are two types of confectionery gelatines, which are quick setter and slow setter. Quick setter gives chocolate coated marshmallows a tender and short breaking body. Slow setter provides a more stretchy texture.

In jelly deserts, gelatine is involved in the recipes to provide creaminess, fat reduction and mouth feel. Gelatine is employed in dairy products to provide stabilization and texturization and in meat products to provide water binding (Alikonis, 1979; Nishimoto et al., 2005, Karim and Bhat, 2009). Further food applications of gelatine include fruit toppings for pastry, instant sauces and soups and edible films for confectionery products. In addition, it is used as a stabilizer in ice cream, cream cheese and cottage cheese, in foams and fruit salads (Karim and Bhat, 2008; McWilliams, 2001). In order to reduce colour deterioration, gelatine is used as coating in meat products and gelatine coating improved colour in dark and light storage (Antoniewski et al., 2007; Tyburcy, 2010). In canned meat products, gelatine is employed in order to hold juices lost and to provide a good heat transfer medium during cooking (Sams, 2001). Because of gelatine's functional properties; it is well suited for many food applications, some of which are mentioned priorly. Some of the examples are listed in Table 1.6 (Mariod and Adam, 2013; Turner, 1988).

As mentioned previously, jelly gums consist of sugars, gelling agents and other ingredients. Jellies are described by a firm structure with chewiness and softness provided by the help of gelatine, starch or pectin (Burey et al., 2009). It was seen that most of the studies of jelly gums containing gelatine generally contained another gelling agents, like starch (Nicholas, 2009; Marfil et al., 2012; Delgado and Banon, 2015), pectin (DeMars and Ziegler, 2001), agar (Gramza- Michalowska and Regula, 2007) and xanthan gum (Kim et. al., 2014)

**Table 1.6** Functional properties of gelatine in foods (Turner, 1988).

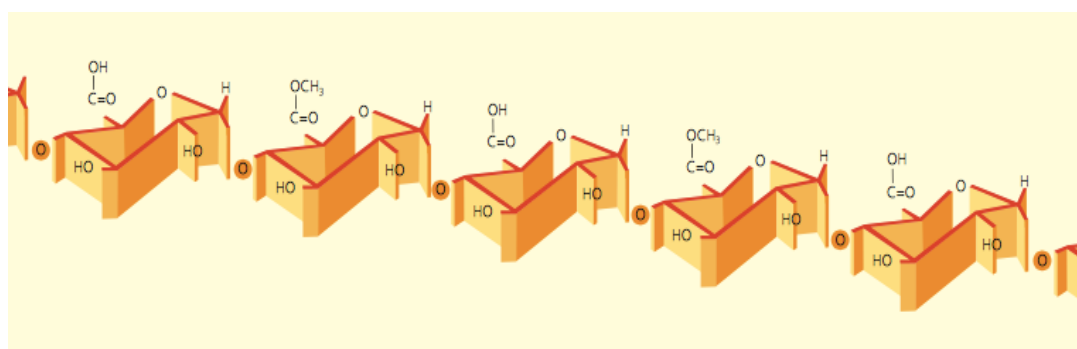
Function	Application
Gel former	gelled desserts, lunch meats, confectionery, pate, consommé, aspics
Whipping agent	marshmallows, nougats, mousses, souffles, chiffons, whipped cream
Protective colloid	confectionery, icings, ice creams, frozen desserts and confections
Binding agent	meat rolls, canned meats, confectionery, cheeses, dairy products
Clarifying agent	beer, wine, fruit juices, vinegar
Film former	coating for fruits, meats
Thickener	powdered drink mixes, bouillon, gravies, sauces, soups, puddings, jellies, syrups, dairy products
Process aid	microencapsulation of colours, flavours, oils, vitamins
Emulsifier	cream soups, sauces, flavourings, meat pastes, whipped cream, confectionery, dairy products
Stabilizer	cream cheese, chocolate milk, yogurt, icings, cream fillings, frozen desserts
Adhesive agent	to affix nonpareils, coconut and other items to confections, to bond layered confections together, to bind frostings to baked goods, to bind seasonings to meat products.

#### **1.2.1.2 Pectin**

Pectin, which is a gelling agent natural to fruits, is produced commercially from the peel of citrus fruits and from apple in crushed and pressed residue form called as pomace. In citrus fruit peel, 20- 40% of the dry matter content is pectin and in apple pomace this percentage is 10- 20%. Pectin, widely distributed in plants, is extracted, standardized and sometimes modified by chemical or enzyme treatment and becomes one of the most valuable gelling agents in confectionery business. Extraction is

performed at a pH range of 1.5- 3.0 at temperatures between 60 °C and 100 °C (Minifie, 1970; Mohos 2010).

Pectic substances are the compounds that are present in plant tissues and derived from colloidal carbohydrates associated with hemicelluloses and lignins. These are composed of mainly of  $\alpha$ -D-galacturonic acid chains linked by 1  $\rightarrow$ 4 linkages (Figure 1. 6). Other sugars are also present in main chain and in side chains (Minifie, 1970).



**Figure 1.6** Poly-D-Galacturonic acid partially esterified (pectin) ([http://www.herbstreithfox.de/fileadmin/tmp/pdf/broschueren/Suesswaren\\_englisch.pdf](http://www.herbstreithfox.de/fileadmin/tmp/pdf/broschueren/Suesswaren_englisch.pdf))

Mentioned polygalacturonic acid chains can be esterified with methyl groups to convert the carboxylic acid groups (for example  $\text{COOH} \rightarrow \text{COOCH}_3$ ) and can also be neutralized by various bases. These various methylated compounds are called as one collective term, “pectin” (Minifie, 1970; Rollin and De Vries, 1996).

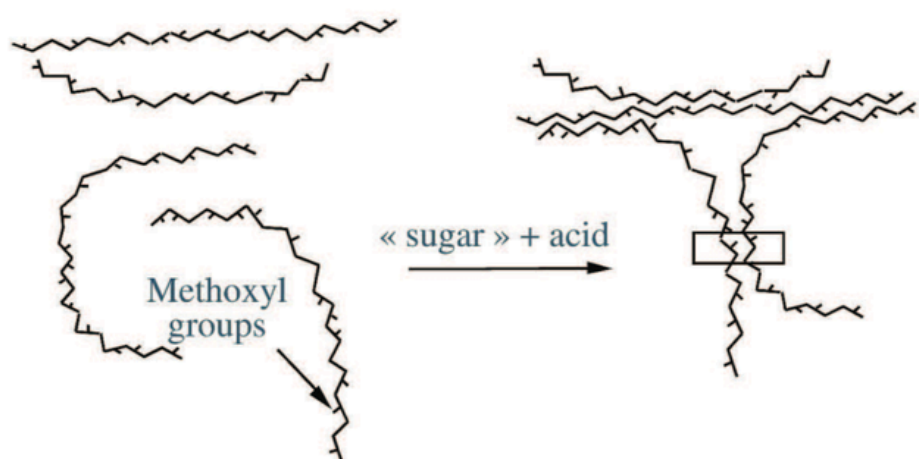
Pectic substances are classified into three; namely, protopectins, pectinic acids and pectic acids. Protopectins are the pectic substances that are not soluble in water in their natural state, however, extractable as pectinic acids at high temperatures in the presence of acid. Pectinic acids are colloidal polygalacturonic acids with some of the carboxylic groups esterified. They form gels with sugars at some certain pH. If the degree of esterification is low, they form gels with metallic salts and these are called pectinates. Pectic acids are the colloidal polygalacturonic acids, which have not been



esterified with methyl groups and the salts are called pectates (Kertesz, 1951).

Commercial pectins are classified into two main groups, high-ester pectins (high-methoxyl pectins) and low-ester pectins (low-methoxyl pectins) according to the degree of esterification (DE). Degree of esterification is the percentage of galacturonic acid subunits that are methyl esterified (Mohos, 2010). High-ester pectins or high-methoxyl pectins have 50% or more of the carboxylic groups esterified, and they require sugar and acid in order to form gels. High-ester pectins (DE > 50%) are subclassified into three; rapid-set with 70-75% DE, medium-rapid-set with 65-69% DE and slow-set with 60-64% DE. The degree of esterification correlates with texture of the gel and setting rate. For example very high-methoxyl pectins jellify faster than less high-methoxyl ones. Hence, slow-set pectins are preferred by confectionery industry as flavourings, colouring and dosing need a certain time prior to setting (Mohos, 2010).

Schematic representation of the junction zones in high-ester pectin gels is given in Figure 1.7 below (Lopes da Silva and Rao, 1995).



**Figure 1.7** Junction zones in high-ester pectin gels.

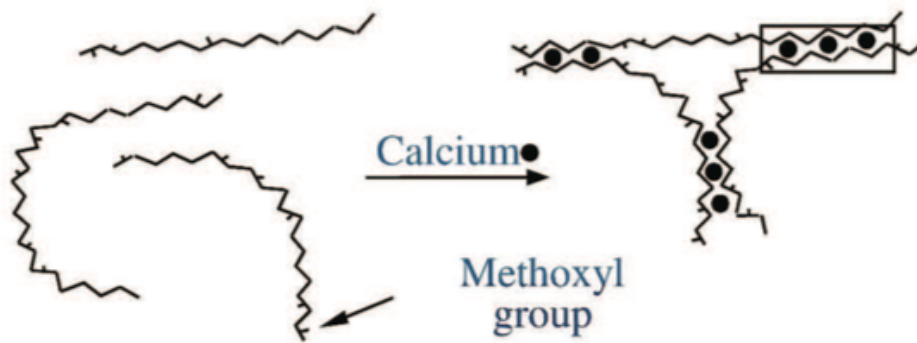
Pectin companies manufacture several grades of high-ester pectin for different formulations and processes given in Table 1.7 (Mohos, 2010).

**Table 1.7** High-ester pectins (Mohos, 2010)

Type	%	Setting	pH range	Uses
	Soluble Solids	Temperature (°C)		
Rapid set	60- 70	75- 85	3.1- 3.6	Preserves, jams, jellies- small containers
Medium set	60- 70	55- 75	3.0- 3.3	Preserves, jams, jellies- larger containers
Slow set	60- 70	45-60	2.8- 3.2	Preserves, jams, jellies where batches may be held before pouring
Buffered	75- 80	90- 95	3.2- 3.7	Confectionery jellies with or without fruit

High-ester pectins are generally standardized to USA- SAG grade 150, meaning that one part of the pectin is able to turn 150 parts of sucrose into a prepared under standard conditions (Minifie, 1970).

Low-ester pectins are the ones with a proportion of methoxylated polygalacturonic acid units, i.e. DE, less than 50%. They can jelly with  $\text{Ca}^{2+}$  ions. These type of pectins form gels not only with sugar and acids, but also with less soluble solids containing calcium ions (Mohos, 2010). On the pectin chains,  $\text{Ca}^{2+}$  ions binding to the ionized carboxyl groups mechanism is similar to egg-box model for alginate (Grant et al., 1973). In the presence of divalent ions, dimerization of polygalacturonic chains occurs. These chains have a 2-fold symmetry and form negative cavities which divalent cation can fit. This is known as egg-box binding and shown in Figure 1.8 (Lopes da Silva and Rao, 2006).



**Figure 1.8** Representation of egg-box model in low-ester pectin gels

For low-ester pectins, sugar and acid are not needed for gel formation in contrast to high-ester pectins, since the reticular structure of the gel is formed with calcium pectinate. Pectin companies produce different types of low-ester pectins for different uses and applications tabulated in Table 1.8. Setting temperatures of low-ester pectins can be greatly affected by calcium salt additions (Mohos, 2010).

**Table 1.8** Low-ester pectins (setting temperature can be affected by calcium salt additions) (Mohos, 2010)

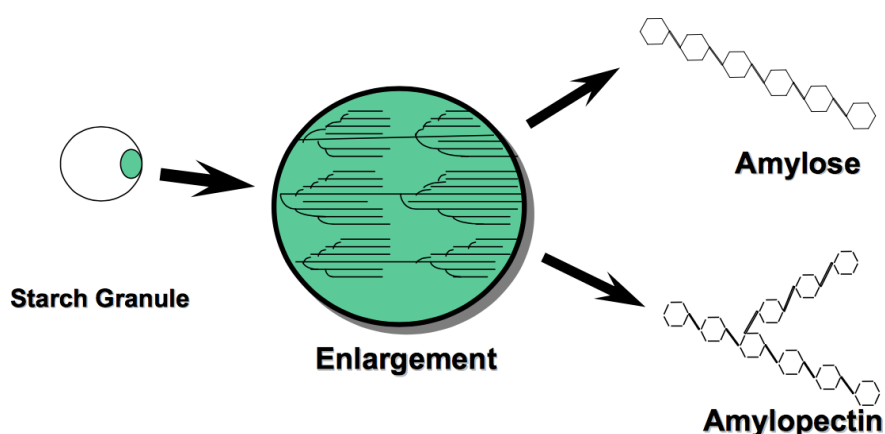
Type	% Esterification	% Soluble Solids	Setting Temperature (°C)	pH range	Uses
A	45- 53	50- 70	*	2.8- 3.3	Jams, jellies
B	40- 50	40- 65	*	2.8- 3.5	Low sugar content jams and jellies
C	40- 50	60- 70	60- 70	3.5- 4.0	Jams for tarts
		75- 80	85- 95	4.0- 5.2	High sugar confectionery jellies
D	32- 37	20- 50	*	2.8- 3.2	Low sugar jellies
		very low	-	-	Milk puddings, creams
Polygalactu- ronic acid	0	-	-	-	Pharmaceuti- cal uses

Due to the nature of jelly gum manufacturing, high methoxyl pectins are used as gelling agent of gummies as these pectins gel in the presence of sugars or other co-solutes such as sugars, polyols at low pH range of between 3.0 and 4.5 (Oakenfull and Scott, 1984; Banerjee and Bhattacharya, 2012). DeMars and Ziegler (2001) investigated texture and structure of gelatine/ pectin jellies by using 3.0%, 4.5% and

6% gelatine and 0.0%, 0.5%, 1.0% and 1.5% high methoxyl pectin. They reported the jellies they obtained as soft to firm and brittle to rubbery and they have found out that samples prepared with combination of gelatine and pectin were more fruity, sweet and tart with respect to pure gelatin samples according to the sensory results. In addition, Poppe (1995) investigated gels prepared with gelatine and high methoxyl pectins at high sugar solids and low pH and reported instrumental hardness increased when gelling agents concentration increased.

### 1.2.1.3 Starch

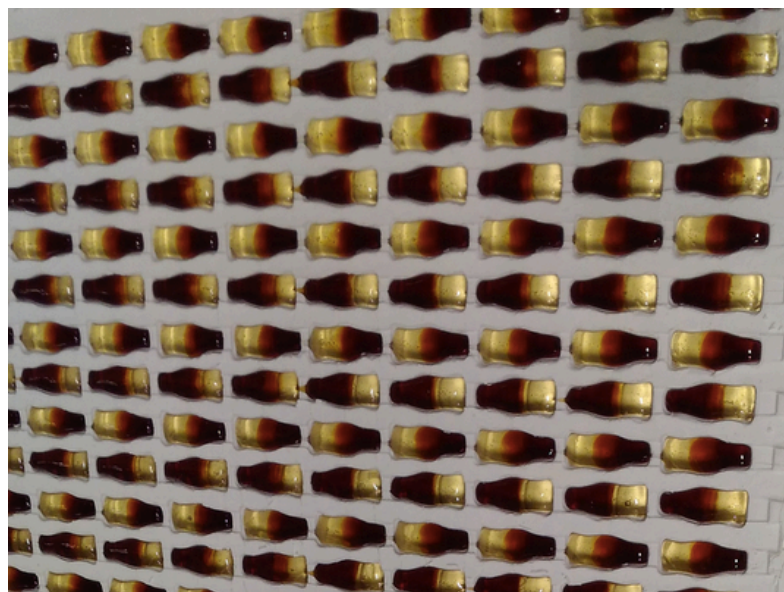
Starch is a widely distributed storage reserve carbohydrate in plants. Starch structure is formed from two glucans, amylose and amylopectin. Amylose has a linear structure and amylopectin has a branched chain structure (Figure 1.9). Most starches contain 20-39% amylose, however, new corn cultivars have been developed to have 50-80% amylose. Normal starch granules have 70-80% amylopectin, while some corn cultivars and millet contain 100% amylopectin. The physical behavior of starch granules is an indication of the molecular structure of the granules (linear or branched) (Alikonis, 1979; Mohos, 2010; Schwenk, 2001).



**Figure 1.9** Starch structure (Schwenk, 2001)

Starch is a useful polymer not only due to the ease with which its physicochemical properties, but also these properties can be changed through enzymatic or chemical modification and/or physical treatment (James et al., 2003).

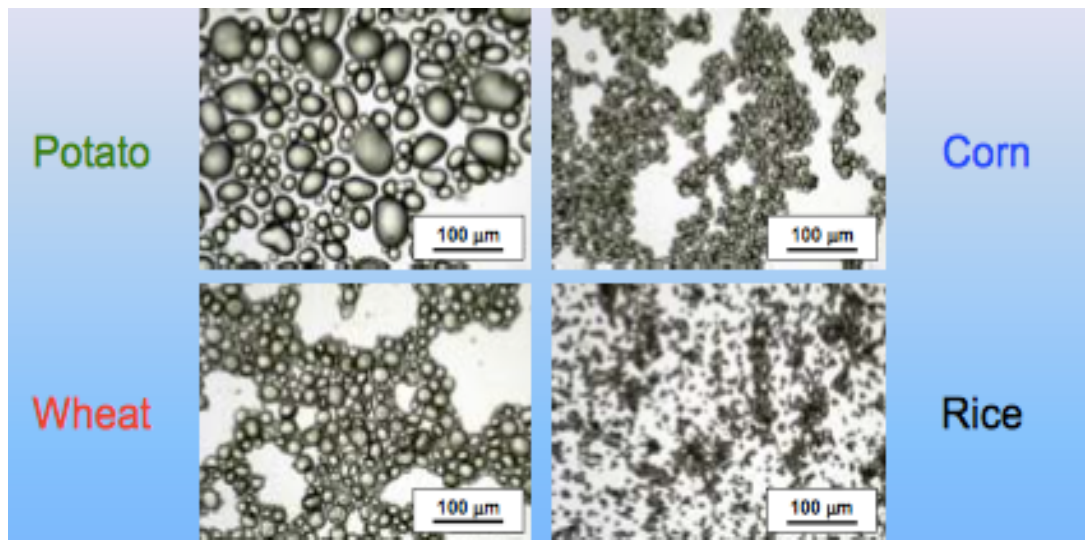
In jelly gum confectionery industry, starches are used mainly in two ways, as moulding medium and gelling agent (Minifie, 1970). In this study, starch was involved both as moulding medium (Figure 1.10) and as gelling agent.



**Figure 1.10** Moulding Starch

Powdered corn starch is effective in absorbing moisture from jelly gums. Regular powdered starch is too dry to hold the jelly gums. However, treating it with a small amount (about 0.5%) of mineral or vegetable oil increases the firmness of the side wall. The functions of moulding starch in jelly gum production are giving the jelly gum its shape, retaining the deposited jelly gum during drying, cooling and setting, and absorbing moisture from the jelly gum during conditioning process (Alikonis, 1979).

The main natural starch used in confectionery, as gelling agent, is corn, however, rice, potato and tapioca are sometimes used. Figure 1.11 shows the appearance and relative size of the starches mentioned.



**Figure 1.11** Granule size- Rice 3-5  $\mu\text{m}$ , Corn 10-25  $\mu\text{m}$ , Potato 15- 100  $\mu\text{m}$ , Wheat 10- 50  $\mu\text{m}$  (Pabst and Gregorova, 2007)

Starches are not soluble in cold water, however, when they are suspended as a slurry and heated, they start to absorb water at 60°C. This causes the cells swell in a great manner and eventually burst resulting into typical starch dispersions. Amount of water, cooking conditions and pH impact the nature of mentioned dispersion. The temperature of initial absorption depends on the starch type. To that effect, cereal starches cook at a slower rate in general. The suspension in water goes through changes on heating with natural starches. When the granules are swollen and before broken, the liquid has a high viscosity and is similar to a smooth paste. Yet, this state is not stable, it is transient and much thinning occurs with increased boiling, lowering of pH or rapid mixing. These starch solutions have a tendency to thicken and form gels during cooling. Increase in viscosity during cooling showed that various constituents present in hot paste tend to reassociate or retrograde as the temperature of the paste decreases. Viscosity change of the gelatinized starch suspension may be due to the frictional dissipation of energy in the movement of the swollen starch granules relative to one another (Miller et al., 1973; Minifie, 1970). Due to secondary bonds between the hydrodynamic units, cooked starch behaved as non-Newtonian fluids, directly or through intermediate water molecules (Schutz, 1971).

When natural or native starch is employed in confectionery syrups, the presence of sugars in solution greatly retards starch cells rupture. This needs to be cooked for a considerable time in water or dilute syrup prior to the concentration of the batch; otherwise uncooked starch appears in the final product, which is not desired. In these conditions, when the starch suspension is very viscous, films form on the surface of the cooking pan and postpone boiling (Minifie, 1970).

Starch modification is performed to decrease retrogradation and syneresis of gel, to enhance clarity, texture, film formation and adhesion and to stabilize granules of starch during processing and make starch convenient for industrial applications. Starch modification can be performed via physical, chemical, enzymatic and biotechnological means (Ashogbon and Akintayo, 2014).

Natural and modified starches are crucial raw materials of many food products (Abd Karim et al., 2000). They are frequently included to these products to promote to their structure and hence improve water and fat holding characteristics (Hermansson and Svegmärk, 1996). Excluding moulding purpose, the role of starch in jellies is to give base to the gel structure and to give contribution to the textural properties. Thin boiled starches are generally involved in confectionery jellies (Cakebread, 1975; Belitz and Grosch, 1999). If we exempt moulding starch, acid hydrolysed thin boiled starches, whose properties are hot water solubility, medium/ low viscosity, rapid setting and high gel power, are generally used in jelly gum production (Mohos, 2010).

In starch gels, the system does not remain static after gelation and the structure of gel starts to change in a process, namely retrogradation, during storage at room temperature (Blanshard, 1979). Kohyama and Nishinari (1991) and Ohtsuka et al. (1994) observed the increase of gel strength due to the retrogradation since it involves the increase in density of gel. It was reported that retrogradation is inhibited by the presence of sugar (Kohyama and Nishinari, 1991), low water content and involvement of other gelling agents like gelatine in the system (Kerr, 1950). Hence, combination of starch and gelatine was used in this study as similar to the several



researchers, who involved starch in their combinations of gelling agents. Siegwein (2010) investigated the effects of soy protein isolate on the physicochemical properties of starch jellies, and he used acid- thinned wheat starch at 3.7%, 5.5%, 7.3% and 11% levels. Marfil et al. (2012) studied texture and microstructure of jellies, where they prepared the gummies with gelatine and acid modified corn starch at varying concentrations from 0% to 10% for both gelling agents. Nicholas (2009) studied conditioning of gelatine and starch confectionery products, which were wine gums and sports mixture. Delgado and Banon (2015) investigated conditioning of jellies prepared with 8.02% acid thinned corn starch and 3.39% type A gelatine in terms of mechanical properties.

#### **1.2.1.4 Other Gelling Agents**

Gelling agents, which confer food products texture by gel formation, are used to thicken and stabilize products and they are used as food additives in the formulations (Banerjee and Bharracharya, 2012). Gelling agents other than gelatine, pectin and starch used in jellies are given in this part of the study.

Agar is used as vegetarian gelatine in jellies according to Armisen et al. (2000) and Stanley (2006). Gellan is also proposed as a gelling agent in confectionery, and gellan gels are said to be stable over a broad pH interval and gel power increases as sugar or ion concentration increases (Jackson, 1995; Gibson and Sanderson, 1997; Edwards, 2000). Whey proteins, soya proteins and egg proteins are reported as gelling agents in confectionery products in several studies (Aguilera and Rademacher, 2004; Bharracharya and Jena, 2007; Siegwein, 2010). Philips and Williams (2000) mentioned carrageenan and Roopa and Bharracharya (2008) mentioned alginate as gelation agents. Curdlan is also a proposed as a useful gelling agent used in applications in confectionery jelly, and gels of different powers are formed in way that are linked to temperature, curdlan amount and heat treatment time (Hino et al., 2003). Costell et al. (1995) studied texture of orange gels by free-choice profiling analysis. They used kappa carrageenan, locust bean, alginate, gellan,

xanthan gums as gelling agents and found differences in sensory were corresponded with mechanical differences except carrageenan and gellan gum gels.

### **1.2.2 Sucrose and Glucose Syrups**

Sugars are the major components of confectionery, which can be in the form of refined crystalline cane or beet sugars, brown sugar, corn syrups, dextrose, lactose, honey, dry or liquid sugar blends, sorbitol and non-nutritive sweeteners. Sugars are mainly used in candy manufacturing for the benefits of their following properties like sweetening or flavouring, bulking, solubility, viscosity, density, crystallization, particle size, hygroscopicity, colour, molecular weight, fermentability, preserving, osmotic pressure and freezing point depression (Alikonis, 1979).

Sucrose, which is one of the mostly found natural sweeteners, is refined from beet and cane. The difference between cane and beet sugar is very significant in terms of flavour and smell. Cane sugar has been reported as pleasant flavour and smell when it is raw; on the other hand it is the opposite for raw beet sugar (Minifie, 1970; Alikonis, 1979).

Sucrose has the solution property of the highest rate, in a way that smaller the crystal size of sucrose, the faster the sugar goes into the solution. Medium granulated sucrose is generally used in the jelly production. Sucrose, a disaccharide sugar, is composed of two sugars (glucose and fructose) chemically bound together. When sucrose is treated with heat and acid, sucrose hydrolyzes into invert sugar, which is a mixture of glucose and fructose and this process is known as inversion (Alikonis, 1979; Hull, 2010).

Sucrose, frequently used in combination with glucose syrups, contributes to the texture and sensory attributes of jelly gums. It is mainly used as sweetener in jelly gums and sucrose addition to gelatine gels helps to decrease haziness, to improve thermal stability and to promote gel structure (Kasapis et al., 2003; Holm et al.,

2009). Sucrose can be used to provide mouthfeel or body to the jelly and to increase product mass and non-crystalline form of sucrose is involved in the production of jelly confectionery (Cakebread, 1975; Richards, 1986). Glucose syrups can improve the solubility of sucrose and retard sucrose crystallization in confectionery; hence sucrose is used together with glucose syrups (Belitz and Grosch, 1999; Edwards, 2000).

Glucose syrups, which are used in hard-boiled candy, toffee, caramel, fudge, jellies, have the following functional characteristics; they control sucrose crystallization and graining in the food system, they reduce cold flow, which occurs when the shape of product changes over a certain period of time, they reduce moisture pick up of product, they enhance processing, they modify the sweetness of the product, and they modify the texture of the product. Glucose syrups are described by its dextrose equivalent, carbohydrate composition, dry solids (Lees, 1980). Dextrose equivalent is the measure of total reducing sugars in the glucose syrup and measured using Lane and Eynon's titration. Both 42 dextrose equivalent and 63 dextrose equivalent glucose syrups are used in jelly manufacture. Glucose syrup with 42 dextrose equivalent makes the jelly product chewier, whereas glucose syrup of 63 dextrose equivalent has a softer bite (Hull, 2010). Generally, glucose syrup of 42 dextrose equivalent is more preferred in candy in order to protect the product from sucrose crystallization and to prevent any changes in physical attributes during storage time (Lees, 1980; Jackson, 1995). Using glucose syrup with higher dextrose equivalent value causes to slow sucrose migration and prevent graining (Lees, 1980; Minifie, 1989). Besides preventing sucrose crystallization in jellies, glucose syrups inhibit microbial growth by decreasing water activity of the jelly gum (Burey et al., 2009). Hence, there is no need to use preservative in the formulation jelly gums as mentioned in the study of Porayanee et al. (2015), where the researchers found that the texture of gelatine jellies was influenced by glucose syrup to sucrose ratios and gelatine levels.

### **1.2.3 Water**

Water is a major component in many foods (Slade and Levine, 1991). Water is needed in order to process ingredients into final products in confectionery manufacture and it impacts texture and shelf life. One of the main functions of water in jelly gum formulations is dissolving the raw materials and water is involved to dissolve and prepare sucrose and glucose syrup mixture. Water is needed for dissolving sugars at an amount between 20% to 35% (Ergun et al., 2010).

Water as a jelly gum ingredient is also important for the quality of the final product. pH of water added to jellies is very critical and should be controlled. Acidic water may cause an increase in reducing sugars, because inversion is favourable at high temperature and acid addition during manufacturing process of jelly gums (Ergun et al., 2010).

Water is also crucial in terms of texture of the confectionery products, where it has role on the hardness or softness of candy (Jackson, 1995). Moisture content can change from 30% in sugar syrup candies to 1-2% in hard boiled candy, and when compared to most of the foods confectionery products have relatively low moisture contents. Water content of jellies was reported as 8-22% with 0.50- 0.75 water activity (Bussiere and Serpelloni, 1985) and jelly is in an amorphous rubber or an amorphous glass, based on the extent and rate of moisture removal (Roos and Karel, 1991b).

### **1.2.4 Acids**

Citric, tartaric, malic, lactic, fumaric, apidic and phosphoric acids are the mostly used organic acids in candy formulations. In addition, potassium acid tartrate, which is known as cream of tartar, is also used in confectionery. Acids have some roles in products like; impacting sweetness of the candy products via inversion, giving tartness, enhancing many flavourings, helping gel formation in pectin jellies,

replacing organic acids in starch jellies with modified thin boiled starch, controlling sugar crystallization in products containing sucrose, lowering viscosity in gelatine and causing coacervation of gelatine in mixed gels and providing shelf stability of confectionery via changing pH (Alikonis, 1979; Lees, 1980; Minifie, 1989; Burey et al., 2009).

The choice of acid in a formulation depends on the desired sharpness level and the effects between the ingredients and the acid (Burey et al. 2009). Johnston- Banks (1990) mentions lactic acid usage in addition of to fermented milk.

Citric acid is the most widely used acid in jellies because it has lowest degradation in other ingredients involved in the food system. Moreover, other types of acids added to products to obtain a different taste profile when compared to citric acid (Burey et al., 2009). In jelly gums manufacture, citric acid is frequently added to the slurry as 50% solution just before depositing into moulds in order to eliminate harsh combination of high temperature and acid, which may impact the other ingredients in a negative manner (Minnifie, 1989; Rix, 1990). As similar with this study, DeMars and Ziegler (2001) and Delgado and Banon (2015) used 50% citric acid solution in their jellies, containing gelatine and pectin and gelatine and starch, respectively.

### **1.2.5 Colours**

Main aim to add colours to jelly gums is to make the products more attractive to the consumers since visual appearance of a jelly gum is one of the most noticed parameters by consumers, who relate visual to both quality and taste. Colours added to confectionery have very little impact on the texture, however, they impact the visual properties of products and they may influence flavour (Edwards, 2000).

Food colours are categorized as artificial colours and natural colours. There is a trend towards natural colours in food products due to the consumer insights and demands. In addition, a study has been done which linked artificial colours to hyperactivity

behaviour of children. Sunset yellow, quinoline yellow, allura red, tartrazine, carmoisine and ponceau 4R are the six artificial colours linked to this study (McCann et al. 2007).

In jelly gums, water-soluble colours in very small amounts less than 1% are used commonly. Selection of colouring agent for the formulation, depend on acid stability, protein presence and other raw materials, which may influence the intensity of colour (Lees, 1980). Besides these factors, consumer demands and regulatory limitations are also taken into account.

Siegwein (2010) used anthocyanin as colour pigment in gummi confections prepared with starch and soy protein isolate. He found monomeric anthocyanin recovery was improved in a significant manner in soy formulation, which showed soy protein a potential as a process aid improving colour stability. Subramaniam (2011) mentioned natural colours to be less stable than artificial ones and studied stability of anthocyanin, lycopene, copper chlorophyllin and chlorophyll in juice-based drinks, jellies and hard boiled candy products.

### **1.2.6 Flavourings**

Jelly gums are confectionery products, whose texture is obtained with gelling agents like gelatine and starch and whose most crucial raw materials are sugars (sucrose, glucose and corn syrups) in terms of quantity, however, flavourings are mostly used to provide authentic sensory properties to these candies (Lubbers and Guichard, 2003; Piccone et al., 2011). Flavourings are the ingredients providing a food product with its final overall differentiated sensory properties in terms of taste and smell. Sugars and acids are the other ingredients that also have contribution to the taste of a food product (Shachman, 2005).

Similar to colours, flavourings are also grouped into two, namely natural and synthetic and can be in intense or diluted form (Edwards, 2000). Synthetic

flavourings are made by mixing legally approved chemical ingredients. Natural flavourings are essential oils coming from fruits or spices (Lees, 1980).

Cooking temperature and time also have a crucial role in final taste and texture in confectionery production, since they have a significant effect on both flavouring and overall taste development during manufacturing (Lawrence and Ashwood, 1999). Hence, flavourings are added at the latest stages of production because they are volatile and can be influenced by high cooking temperatures (Lees, 1980).

Delgado and Banon (2015) used strawberry, raspberry, orange and peach flavourings in gummies prepared with gelatine and starch and added flavourings with acid and colour solutions at the latest step due to heat sensitivity of these ingredients. DeMars and Ziegler (2001) found jellies containing more pectin described as more fruity, more sweet and more tart in their sensory study. Jaime et al. (1993) mentioned about inverse relationship between overall flavour and hardness in gels.

Piccone et al. (2011) studied gelatine, starch and pectin candies, in which same concentration of strawberry flavouring was used, and they found no significant differences in the strawberry flavouring intensity at nose and under saliva dissolution except pectin candies. In the study, pectin candy found to have a significantly higher flavouring intensity and it was mentioned to be more inclined to break and enhance flavouring release and sensation.

### **1.3 Shelf life testing**

Shelf life of a product is the time that a food product retains a quality at an acceptable level (Ergun et al., 2010). Shelf life is determined by holding the food product under typical storage conditions that the product exposed to, and measuring the chemical, microbiological and physical changes taking place during a specified time interval till the food product changes to a state, which is unacceptable to consumers (Kilcast and Subramaniam, 2000).

In confectionery, water influences both physical and chemical properties involving glass transition, crystallization, enzymatic or non-enzymatic reactions, which determine shelf life. Table 1.9 shows shelf life of some confectionery products (Jackson, 1990). Despite the fact that moisture migration in confectionery is not the only factor in product failure, it is stated to be the major factor in the end of shelf life for candy products. Therefore, physicochemical changes related to moisture changes are frequently assessed in shelf life testing of confectionery, which can also involve sensory or analytical methods, or may combine multiple tests. Sensory testing is mostly included to the shelf life testing of confectionery products (Ergun et. al, 2010).

Accelerated shelf life study is carried out by holding the product under controlled conditions, which enhance degradation rates occurring in the product during storage (Subramaniam, 2009). High temperatures and high humidities are generally used to favour product degradation in accelerated shelf life testing of confectionery products. Empirical relationships are often built up to correlate accelerated conditions with normal storage conditions. Yet, these correlations are generally specific to product and careful considerations should be done when extrapolating from one recipe to another (Ergun et al., 2010).

**Table 1. 9** Shelf life of some sugar confectionery products (Jackson, 1990).

<b>Product</b>	<b>Normal shelf life (months)</b>
High boilings (Moisture content less than 2.5%)	12
Gums and jellies	6-12 (Depends on recipe and moisture content of final product)
Liquorice	Up to 12
Toffees	9
Jelly beans	9



In accelerated shelf life studies, high temperatures are generally involved to alter the reaction rates since a rise of 10°C induces doubling of the rates of degradation in general (Taub and Singh, 1998). But, not all degradation reactions obey this doubling rule. In addition to water migration, physicochemical changes generally occur due to the temperature changes. Cycling of temperatures enhances faster degradations in the products when compared to storing them at constant temperatures because average rate at cycling temperatures is a little higher than the rate at the equivalent average of cycle temperatures. This is due to the fact that many of the degradations and physical changes increase with temperature in an exponential way. Also, altering of storage relative humidity can change water migration rate within the product. Holding of hard boiled confectionery products at high humidities causes water migration through packaging material, on the other hand, holding of marshmallow products at low humidities causes drying; both case results in short shelf life.

Accelerated shelf life tests are helpful not only for product stability investigations but also for determination of product safety under abused conditions, for troubleshooting and for packaging performance of a food commodity. Predictions of stability obtained from accelerated tests may not always be dependable for complex food products and accelerated tests must be seen as supplementary to real time shelf life (Hough et al., 2006; Subramaniam, 2009). Accelerated shelf life study is done via holding of products at controlled conditions of temperature, relative humidity and light in storage cabinets, which accelerates degradation rates similar to the degradation rates occurring at normal storage conditions (Subramaniam, 2007). Degradation rate can be measured against that taking place under ambient conditions and for shelf life prediction relationships can be used to construct models (Subramaniam, 2011).

Shelf life testing needs a good comprehension of all of the quality parameters changing through storage and main quality drivers limiting shelf life of the food product are required to be determined from these parameters. Moreover, critical levels of change regarding these different parameters should be found for product

failure. Due to the high levels of sugars in the formulations and low moisture contents, confectioneries are microbiologically safe in general (Richardson, 1980). Hence, shelf life of confectionery products is mainly based on the loss of sensory attributes during storage. It is a hard task to determine the end points for a sensory parameter since a change level that a consumer may recognise as acceptable may be found as unacceptable by another consumer. Scientific measurements help to find shelf life of products, however, the most crucial quality attribute regarding consumer acceptance is sensory quality during shelf life of a product. Purpose of involving instrumental, physical and chemical measurements as part of a shelf life test is transforming consumer acceptance of quality to parameters that can be measured. Critical limits in quality parameters are frequently set depending on compromise on the views of taste panels or experienced employees or marketing personnel with the support of top management of the company (Thursby, 1974; Kilcast and Subramaniam, 2000; Subramaniam, 2009).

Shelf life tests are performed during product development. These tests are also carried out in order to evaluate modified formulations (for cost saving purpose for example) and to evaluate shelf life extension of existing products; in which a comparison with old formulation having a known shelf life with the new modified formulation at the same storage conditions is done to check whether there is a change in the shelf life. In the tests, product samples held in the storage cabinets are taken out in an order at certain time periods and changes in quality parameters with the help of sensory, physical and chemical tests are evaluated till the time when the product becomes unacceptable (Subramaniam, 2009; Barnett, 1980).

Choosing of test conditions and understanding of the storage, handling and climates of the markets where the product meets with the consumers are crucial regarding the accuracy of shelf life studies. Distribution cycle of the food product should be considered while deciding test conditions. Most widely used shelf life testing conditions are 38-40°C and 80-90% relative humidity for tropical climates and 20-25°C and 50-70% relative humidity for market conditions. Sample of food products,

that are expected to have twelve to eighteen months shelf life, is analysed at one to two month intervals for shelf life investigation (Subramaniam, 2009).

Baiano and Del Nobile (2005) studied shelf life prolonging of almond paste pastries. In the study, it was found that moisture loss and hence hardening of samples is the main limiting parameter influencing quality and shelf life of products and sensory panel test determined the maximum acceptable hardness of paste pastries and correlated to a maximum force of 25 N by using texture instrument. As a result, acceptable shelf life was found as the time when the maximum compression remained under 25 N. Hardness determination can also be involved whether the confectionery product soften or lose crispiness during shelf life studies (Ergun et al., 2010). Koster and Westerbeek (1991) studied prolonging the shelf life of aerated foods and they described the mechanisms of destabilization of foams in accordance to shelf life improvement.

#### **1.4 Sensory Evaluation**

Sensory evaluation is one of the quality indices used commonly in shelf life studies of foods besides chemical, microbiological and physical instrumental measurements since shelf life of a product means the time till a product is unacceptable to consumers at a known storage condition (Labuza, 2000). It is composed of methodologies to find out the reaction of sensory attributes, like taste, texture, colour and flavour of food products, with senses of consumers and it enables consumer tests, quality control, correlating among physical and chemical measurements and product development. Sensory analysis is carried out by counting in function of the reactions to the senses, which come from physiologic responses and this induces estimation of intrinsic characteristics of food products (Pertuzatti, 2015).

Sensory changes are not due to the microbiological spoilage in most of the confectionery products, hence changes in sensory properties of the product is associated with product acceptability. Different aspects of product shelf life should

be evaluated with proper sensory tests (Subramaniam, 2009). Changes in sensory attributes are followed in the shelf life measurements of jellies, where a trained sensory panel can be involved to define changes regarding product degradation. Table 1.10 illustrates some of the sensory properties that can be evaluated during storage of fruit jellies (Subramaniam, 2000).

**Table 1.10** Some of the sensory properties evaluated during fruit jelly storage (Subramaniam, 2000).

Sensory property	Definition
Gloss	Amount of shine on product surface
Hardness on 1 <sup>st</sup> bite	Resistance for biting as evaluated on front teeth
Stickiness on 1 <sup>st</sup> bite	Degree to which the sample sticks to front teeth
Gelatinous	Texture of raw jelly
Stickiness	Degree to which the sample sticks to the teeth and surfaces of mouth during chewing
Cohesive	Degree to which sample holds together as a mass
Chewiness	Effort needed to break down

Analytical tests like descriptive and difference tests and hedonic tests are involved in product stability studies. Analytic tests are beneficial to describe and track variations in a food product. Difference tests are conducted to compare the sensory properties of two products. Paired comparison, duo- trio and triangle tests are examples of difference tests and they are capable of giving only limited information regarding the sensory history of food product during storage despite they are regarded as sensitive.

On the other hand, descriptive tests can measure changes in product properties such as texture or colour. Findings of these tests can be used in correlations with instrumental analyses, which measure the same or a similar property, and this allows quantification of the sensory variations of a food product. Trained sensory panelists are involved in the quantitative profile methods in order to measure intensity level for each product property, and due to this these methods provide more information with respect to difference tests (Subramaniam, 2009).

According to Kilcast (2000), quantitative descriptive analysis is the most widely used analysis, in which the information obtained can be evaluated statistically and shown visually. Preference and acceptability tests are hedonic tests ensuring to measure consumer liking level and quality perception level. Mentioned tests are used to find out the end points of product shelf life, when consumer acceptability decreases in a significant manner. In addition, quantitative descriptive analysis on the same product samples could be utilized for determining the level of change for a specific property to define the critical variations making the food product unacceptable to the consumers. A correlation of these two kinds of tests is frequently needed to conclude on the product shelf life (Kilcast and Subramaniam, 2000). Consumers are involved in hedonic tests to perform assessments, in which product samples are scored. These tests enable to measure consumer acceptance in a direct way, however, their costs are high since they are conducted with many consumers to show the market that the product to be sold (Subramaniam, 2009).

Kim et al. (2014) studied the effect of medicinal plant extracts on the sensory properties of gelatine jellies and they found that colour and taste scores were highest in the gelatine jelly samples with *Cudrania tricuspidata* Bureau with respect to other jelly samples. On the other hand, no significant differences were seen in flavour, texture, gel intensity and acceptability. Piccone et al. (2011) studied the effect of gelling agents (gelatine, starch and pectin) and sugars on the sensory perception of flavouring compounds at the same strawberry flavouring level and it was found that only candies prepared with pectin had higher strawberry flavour intensity after

chewing of candies and under saliva dissolution. In this study, pectin was found to help the release of flavouring and perception in candy systems. Siegwein (2010) carried out sensory analysis of gummies containing starch and soy protein isolate as gelling agents with hedonic scale of acceptability. Addition of soy protein isolate increased acceptance scores for flavour and texture of gummy confections.

## **1.5 Primary parameters measured by instrumental analysis for confectionery**

Chemical and physical analyses are conducted to measure primary factors causing deterioration of a confectionery during normal storage conditions. The data of these instrumental tests can be correlated with sensory data in order to determine critical limits for the end of shelf life.

### **1.5.1 Water activity**

Water activity term describes the equilibrium amount of water available for hydration of foods. Unity shows pure water, zero shows the total absence of free water molecules. Water activity ( $a_w$ ) is the equilibrium vapour pressure that water in a food exerts ( $p_w$ ) divided by pure water's vapour pressure ( $p_0$ ) given in Equation 1.1 (Mohos, 2010).

$$a_w = \frac{p_w}{p_0} \quad (1.1)$$

Water activity is also used to predict equilibrium relative humidity (ERH%), where candy neither gains nor loses moisture from air and given in Equation 1.2 (Nielsen, 2003).

$$\text{ERH (\%)} = a_w (100) \quad (1.2)$$

Water activity is mainly affected by the presence of dissolved sugars and their concentrations, other sweeteners like polyols, salts like caramel and humectants in

confectionery products. Main types of deterioration dependent on water activity in foods. At  $a_w$  between 0.2 and 0.4, lipid oxidation is relatively slow, at lower and higher values stimulated. At about  $a_w$  of 0.2, non-enzymatic browning starts and attains its maximum at about  $a_w$  of 0.8. At about  $a_w$  of 0.3, enzymatic activity starts, stimulated when  $a_w$  increases. At about  $a_w$  of 0.75, mold growth starts, stimulated when  $a_w$  increases. At  $a_w$  of 0.8, yeast growth starts, stimulated when  $a_w$  increases. At  $a_w$  of 0.9, bacterial growth starts, stimulated when  $a_w$  increases (Mohos, 2010).

As mentioned above, microbial growth is directly related to water activity, with certain types of microorganisms unable to grow if water activity is below some critical value (Table 1.11). Fortunately; water activity in gummies and jellies is in the interval of 0.50-0.75, generally below the critical values for microbial growth. Hence, microbial growth in jelly gums is generally not an issue. On the other hand, end of shelf life of a food product because of moisture loss or gain, besides subsequent changes in texture and other properties, is the main problem in most of the confectionery products in the market. Due to this, water activity is crucial for control of stability and shelf life of confections (Bussiere and Serpelloni, 1985; Ergun et al., 2010). In addition, Scott (1957), who introduced water activity as measurement of water availability, mentioned that water availability determines food stability, not water content only.

**Table 1.11** Microorganism growth in confectionery (Beuchat, 1981; Fontana, 2006; Minifie, 1999; Ergun et al., 2010)

<b>aw</b>	<b>Microorganism which can grow</b>	<b>Product</b>
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.

### 1.5.2 Glass transition temperature

Processed foods, generally low moisture content and sugar-rich products, are characterized by structural properties, flavour and colour. They may go through physical structural changes and these changes are strongly related to Tg. Processing types which need an understanding of the glass transition and amorphous state of products are the ones taking place at low moisture content, like cereal systems, frozen foods, confectionery and candies, dehydrated foods, extruded foods. Carbohydrates are the main glass forming compounds in foods and proteins are to a lesser extent. On the other hand, fats do not show glass transition (Haque and Roos, 2004; Jouppila and Roos, 1997).

The importance of glass transition temperature to confectionery products has been regarded for many years. Confectionery products can be grouped as crystalline or amorphous based on their molecular mobility. Crystalline form is the most stable molecular organization. Amorphous states can be categorized as rubbery or glassy.



Glassy state is more rigid and has less internal mobility, however, rubbery state is viscous and has fluid like properties. Transition between glassy to rubbery takes place at the glass transition temperature (Ergun et al., 2010; Kauzmann, 1948).

As mentioned earlier, glass transition temperature is a physicochemical parameter. This parameter is a good indicator of food properties, stability and safety according to Levine and Slade (1992) since  $T-T_g$  (temperature difference between glass transition temperature and storage temperature) was found to control the rates of physical, chemical and biological changes (Champion et al., 2000). Diffusion based deteriorations are severely retarded if the product is kept at a temperature below  $T_g$ . Molecular mobility increases and viscosity is reduced above  $T_g$ , this causes time dependent structural transformations. Some examples are stickiness and collapse during processing and storage of food products (Roos, 1995). A minimum degree of mobility should be ensured for reactant molecules to collide with, position toward and react with one another, taking place at reduced rates if the food product is held at temperatures below  $T_g$  (Serwin et al., 2002; Sherwin and Labuza, 2006; Ergun et al., 2010).

Glass transition temperature depends on a range of factors; important ones are reviewed shortly below.

- **Molecular weight:** Chain end concentration in straight chain polymers is reduced when molecular weight increases. It has been seen that by changing the end groups, molecular weight dependence of glass transition temperature can also be altered (Roth et al., 2006). According to the previous research findings, glass transition temperature of homologous and amorphous polymers decreases when the average molecular weight decreases or the amount of plasticizer increases (White and Cakebread, 1966; Orford et al., 1990; Roos and Karel, 1991; Buera et al., 1992).
- **Molecular structure:** Due to reducing mobility, presence of inflexible side

groups (or chains) increases  $T_g$ . As side groups' length increases, polymer chains have the tendency of being further apart; this increases free volume hence result of lower  $T_g$  is seen. Polymers with double bonds in the backbone structure have less bond rotation resulting into lower  $T_g$ s. In addition, cross-linking of chains increases  $T_g$  value as it decreases mobility (Champion et al., 2004)

- **Plasticizer effect:** Presence of plasticizers decreases  $T_g$  of the system since plasticizers increase the free volume by increasing the space between polymer chains hence these can slide past each other in a more easy manner. For example; water acts as a plasticizer because of the formation of hydrogen bonds with polymeric chains, thus increasing the distance between polymeric chains, as a result decreasing the  $T_g$  (Ljungberg et al., 2003).
- **Cooling rate:** Cooling rate is directly proportional to glass transition temperature, as cooling rate increases  $T_g$  also increases (Hsu et al., 2003).
- **Entropy effect:** If entropy value increases,  $T_g$  value also increases since the entropy of an amorphous material is higher than the entropy of a crystalline material (Debenedetti and Stillinger, 2001).

### 1.5.3 pH

Sørensen (1909) defined pH as below;

$$\text{pH} = -\lg (C_{\text{H}}/C^{\circ}) \quad (1.3)$$

where  $C_{\text{H}}$  is the hydrogen ion concentration ( $\text{mol m}^{-3}$ ) and  $C^{\circ} = 1 \text{ mol dm}^{-3}$ , which is the standard amount concentration.

Afterwards in 1924, Sørensen and Linderstrøm-Lang defined the term in terms of the relative activity of hydrogen ions in solution as;

$$\text{pH} = -\lg a_{\text{H}} = -\lg (m_{\text{H}}\gamma_{\text{H}} / m^{\circ}) \quad (1.4)$$

where,  $a_{\text{H}}$  is the relative activity,  $\gamma_{\text{H}}$  is the molal activity coefficient of the hydrogen ion at the molality  $m_{\text{H}}$ , and  $m^{\circ}$  is the standard molality. Hence, pH is measure of the activity of hydrogen ions in the system. Buck et al. (2002) mentioned that Equation 1.4 above could be only a notional definition since pH is defined in terms of a quantity that can not be measured with a thermodynamically valid method in IUPAC Recommendations 2002.

Changes on the pH can have an influence on some of the components of the food, for instance proteins can undergo conformational changes. In addition, it may cause changes in texture, colour and nutritional value of the final product (Heldman and Hartel, 1997; Enrione, 2005). Confectionery products produced with gelling agents like starch, gelatine or pectin are affected by pH, thus it is crucial to maintain pH control and appropriate acidity levels in gelled products in order to provide stability. In pectin jellies, lack of pH control may cause changes in the gel setting of pectin, such as setting may be too slow or too fast. In gelatine jellies, pH and acidity influence the strength of the gelatine setting in a way that it may degrade it. The gelling power of gelatine decreases at lower pH values independent of acid type. If the acidity is too high, gelatine gel strength is affected and firmness of the product decreases. A possible solution to this is to add buffer salts or buffered acids in the recipes to assist pH control. It is important to know the effect of pH on gelatine gel strength when employing acid to acidify, flavor or fortify the confectionery products (Schrieber, 1976; Jarrett, 2012).

#### **1.5.4 Texture**

Analysis of the effect of recipe change or time-dependent variations on textural properties can be realized with texture profile analysis (Siegwein et al., 2011). The instrumental texture profile analysis is a technique commonly used for evaluation of food textural behaviour since it can provide an indication of sensory eating properties of the food (Olkku and Rha, 1975; Bourne, 1986; Hough et al., 1996; Antoniou et al., 2000; Burey et al., 2009). Texture profile analysis imitates first two bites of food mastication process with an instrument and relates the data obtained to textural descriptors and developed at General Foods and it is a common and robust method to analyze foods in a way meant to compare to mastication (Daubert and Foegeding, 2003). The test involves a two-cycle penetration test into the food and the force developed is observed during testing time. Output of texture profile analysis is a two-peak force curve, from which calculations are performed to determine textural parameters (Friedman et al., 1963; Borwankar, 1992; Antoniou et al., 2000). It is important to be consistent with sample size because magnitudes of the textural parameters depend on testing geometries (Burey et al., 2009).

#### **1.5.5 Sugar Profile**

Sugars such as sucrose, glucose or corn syrups are widely used as key ingredients due to their versatility and cost effectiveness by both food and pharmaceutical industries due to the following reasons:

- ❖ They manage the sweetness of food products for taste and indulgence, for instance in fat-phase confectionery products in addition to contributing sweetness they enhance mouthfeel and fatty flavour
- ❖ They preserve the product and improve and increase shelf life, for example sugars act as humectants both for preservation and extended shelf life in bakery products like jams and jellies.
- ❖ They give easily metabolized energy

- ❖ They help the body absorb water
- ❖ They regulate the texture of product, such as they act as binders in order to increase crispiness, spread and surface porosity in breakfast cereals.
- ❖ They have role in a product's overall flavor character
- ❖ They have impact on appearance of finished product
- ❖ They provide a cost-effective source of bulking solids and fermentable carbohydrates
- ❖ They are solidified into crystalline products
- ❖ They have influence on freezing and boiling point, as such sugars have role in lowering the freezing point in the production of ice-cream and frozen desserts.
- ❖ They have the capability to mask the bitter taste of vitamins, minerals and medicines
- ❖ They can be used as a processing aid.

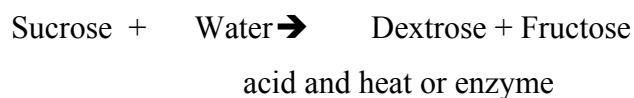
(Lindley, 1988; Jeffery, 1993; Mathlouthi, 1995; Helstad, 2006; Lee, 2010).

Sugars in a product can be found dissolved in the water, dispersed as a crystalline phase, immobilized in the glassy or amorphous state or various combinations of these (Hartel et al., 2011). In syrup- phase confectioneries, sugars provide different quality features with respect to the product type. For fondants and fudges recrystallization of the syrup phase is needed, however, for jam and jellies crystallization of sugars is not desirable; for hard-boiled candy and caramel products formation of a stable syrup phase is needed (Lee, 2010). According to the categorization of sugar-based confectionery based on the state of sugar done by Ergun and Hartel (2009), jellies are in noncrystalline liquid category with solid-like structure due to hydrocolloids such as gelatine, starch and pectin.

During processing (mostly thermal processing) of confectionery products, sugars in the formulation go through one phase transition or more. Changes in phase behaviour may also happen during storage, which may affect shelf life of the product in a negative way. Sugars show two main phase/state transitions, namely crystallization

and glass transition (Hartel et al., 2011). Glass transition topic was mentioned earlier in this study. Crystallization occurs if solubility limit of sucrose has been exceeded and a supersaturation medium has been created either by decreasing the temperature or by increasing the sucrose level in the formulation, or both. In jelly gums, glucose syrups and invert sugar are used together with sucrose in order to eliminate crystallization of sucrose in a way that they decrease the solubility concentration of sucrose with less sucrose is required for saturation at higher addition levels and they act as insulation around the individual sucrose crystals, besides ingredients such as proteins, texturisers and stabilizers also affect the crystallization in the food system. Undesirable crystallization of sugars in jellies may affect the appearance of the product in a manner that they have grainy visual and a greyish colour and may also affect texture in a way that products become coarse and gritty. In addition, water activity of jellies may increase since moisture is squeezed out when sugar solids are concentrated in crystals, this may impact the product shelf life negatively (Hull, 2010; Hartel et al., 2011).

In addition to glass and crystallization transitions in the production and storage of jelly gums, sucrose inversion is also very crucial to be considered as jelly gums are produced at high temperatures and acid addition is involved in processing. When sucrose is cooked in the presence of acid, chemical bond between dextrose and fructose within sucrose is broken to produce invert. Invert is a mixture of dextrose and fructose and conversion of sucrose into dextrose and fructose is called inversion (Figure 1.12). It can be understood from below figure, reaction starts with 342 (molecular weight of sucrose) parts of sucrose and end up with 360 (dextrose and fructose has a molecular weight of 180) parts of dextrose and fructose, due to the inclusion of a water molecule it is called hydrolysis gain.



**Figure 1.12** Sucrose inversion (Hull, 2010)

Ingredient list on the pack of a commercial food product states that sucrose is used in the recipe, however, there might not be any sucrose or there might be very little sucrose found in the end product. Hence, in addition to quality effects caused by inversion, changes due to sucrose inversion into dextrose and fructose should be tracked during the shelf life of a food product for legal declarations as well.

Quality parameters of jelly gums such as texture, shelf life and microstructure also depend on processing (high temperature, acid addition...etc.) and the types and amounts of sugars used due to the possibility of inversion. Therefore, it is important to determine the sugar profiles before launching a jelly gum product, which is a very high sugar containing product. Sucrose and glucose syrup are used together in the production of jellies since the syrup can improve solubility of sucrose and retard sucrose crystallization during storage of the products (Belitz and Grosch, 1999; Edwards, 2000; Burey et al., 2009).

## 1.6 Aim of the Study

Food products are active, complex and diverse systems, where microbiological, enzymatic and physicochemical reactions occur at the same time, which makes the study of shelf life of food systems difficult. The approaches and methods that are used to determine shelf life depend on the product type tested and food industry is continuously searching better and faster analytical and testing methods for improved shelf life determinations by taking account quality, safety, regulatory and market pillars. To achieve superior quality with extended shelf life of confectionery products

is an up to date improvement and innovation topic as there are lots of types in confectionery market, one of which is jelly gum confectionery products. Hence, the main aim of this study was to correlate sensorial changes with instrumental techniques during the shelf life of jelly gums to identify specific markers for product failure and shelf life prediction.

Jelly gums on the market have a lot of types in terms of ingredients and processing. The studies regarding shelf life of jelly gums are limited in the literature and it is believed that there are sensitive interactions between gelling agents and ratio of sugars in mogul process, which is not known very precisely. Thus, it is crucial to investigate the effects of gelling agent type, gelling agent ratio and sugars ratio on quality of gums in shelf life testing. In addition, when the commercial products in different geographic regions were analysed, gelatine jellies with only 6-10% gelatine, and 4-6% gelatine in combination with 6-12% starch are preferred. Hence, another purpose in this study was to find an optimized formulation that could be compatible to different geographic regions in a cost effective way.

Accelerated shelf life testing is the most often used method by food industry; however, it is commonly abused in design and interpretation of results. In this study, important parameters of jellies were determined and measured before, during and after storage at different conditions and accelerated test conditions were suggested for shelf life prediction of jelly gums accompanied with sensory testing. As confectionery companies export products to both cold and hot climates, 10°C and 30°C temperature studies were involved in the study.

Glass transition theory and water content changes are believed to understand the physical and textural changes of jelly gums, so other goals in this study were to obtain sorption isotherms and to measure glass transition temperatures of the products. In addition, diffusion coefficient of jellies were determined in order to have information related to conditioning in starch.



## **CHAPTER II**

### **MATERIALS AND METHODS**

#### **2.1. Materials**

Commercially available beef gelatine having 250 bloom was provided by Halavet Gıda (Istanbul, Turkey). Acid thinned corn starch (Cleargum MB70, Roquette, Valencia, Spain) with 0.5% protein, 12% moisture and at 5.5-7.5 pH was used in the experiments. Sucrose was bought from Balküpü (Kocaeli, Turkey) and 42DE glucose syrup was obtained from Cargill (Bursa, Turkey). The following ingredients were also used in the formulations of jelly gums: food grade citric acid monohydrate (Jungbunzlauer, Basel, Switzerland), natural red liquid colour (Black carrot anthocyanin, Endemix, Kocaeli, Turkey), and natural identical strawberry flavouring (Aromsa, Kocaeli, Turkey).

#### **2.2 Methods**

##### **2.2.1 Production of Jelly Gums**

Jelly gums were made according to the recipes given in Table 2.1.

**Table 2.1** Recipes used for preparation of samples (1000 g batches)

<b>Ingredient</b>	<b>Mass (g)</b>	<b>Mass (g)</b>
	(Glucose syrup:sucrose=1.1)	(Glucose syrup:sucrose=1.5)
Water	104	104
42 DE Glucose Syrup	364	418.8
Sucrose	334	279.2
Gelling Solution <sup>a</sup>	290.4	290.4
Citric acid solution (50% w/v) <sup>b</sup>	Variable	Variable
Flavouring and Colour	2	2

a: Composition of gelling solution depends on final gelatine and starch concentrations. The quantities were adjusted in order to achieve the desired concentrations of gelling agent.

b: Citric acid solution (50% w/v) was added so that the final pH of the jelly gum was  $3.4 \pm 0.1$  and varied from 9.6 g to 25.2 g depending on gelling agent concentration.

In the experiments, gelatine concentrations used were 3.0%, 4.5% and 6.0% and starch concentrations were 0%, 1.0% and 1.5% in both glucose syrup to sucrose ratios, namely 1.1 and 1.5. Summary of the formulations was given in Table 2.2. Two replicate batches of each formulation were prepared.

**Table 2.2** Different formulations used in the experiment

Formula #	Glucose/ Sucrose Ratio	Starch (%)	Gelatine (%)
1	1.1	0	3
2	1.1	0	4.5
3	1.1	0	6
4	1.1	1	3
5	1.1	1	4.5
6	1.1	1	6
7	1.1	1.5	3
8	1.1	1.5	4.5
9	1.1	1.5	6
10	1.5	0	3
11	1.5	0	4.5
12	1.5	0	6
13	1.5	1	3
14	1.5	1	4.5
15	1.5	1	6
16	1.5	1.5	3
17	1.5	1.5	4.5
18	1.5	1.5	6

The gelatine and starch powders were mixed together and were soaked in warm water at 55°C before use. Then, the slurry was mixed at low speed by using a kitchen blender (Moulinex, DDF4 Optipro, Cedex, France) for 30 seconds and deaerated in glass beakers at 90°C in a water bath (Nuve, BM 15/30, Ankara, Turkey).

Jelly gums were prepared using an open saucepan with electric heating (Kumtel KH/LX 7010/7011, Kayseri, Turkey). The sucrose, glucose syrup and water were heated to boiling. Heating continued slightly until a target weight calculated to achieve 78% dry solids. The solution was then allowed to cool down to 90°C. The

prepared gelling slurry was added to the solution and mixed to ensure adequate mixing in Hobart Mixer N50 (Troy, USA). Citric acid, colour and flavouring were added and the final desired mass was 1000 g. Starch trays were prepared using pre-dried warm moulding starch (03703 Cargill, Bergen op Zoom, Holland) containing 1.2% oil warmed to 50°C prior to using. The shapes were made to yield gently tapering moulds achieving dimensions of approximately 15 mm×15 mm×15 mm. The cooked mass was poured into a metal funnel (inner stem diameter= 1/ 8 in) and deposited into the starch moulds. A layer of warm moulding starch was poured over the filled trays to a depth of a few mm above the gum level. Filled trays were stored at 20°C and jelly gums were removed from starch until 83% dry solids were achieved in the conditioning. Removed products were brushed to remove the excess starch on the samples. Sufficient number of samples (15 pieces) was taken randomly from the cleaned jelly gums and instrumental tests were performed.

### **2.2.2 Bloom Measurement of Gelatine**

Bloom measurements were done according to Official Procedure of the Gelatine Manufacturers Institute of America (AOAC, 1986). Three of  $7.5 \pm 0.01$  g of dry gelatine samples were prepared and  $105 \text{ ml} \pm 0.2 \text{ ml}$  distilled water was added to each sample with a magnetic stirrer. The concentration of gelatine solution should be 6.67%. The samples were left at the room temperature for 1-4 h (min 1h). The samples were taken and put into water bath at 65°C and kept for  $20 \pm 5$  minutes until the temperature of the samples increased to 60°C. The samples were mixed after removal from water bath. Then, gelatine solutions were cooled by leaving them at room temperature for 15-20 minutes. Afterwards, the samples were left  $17 \text{ h} \pm 1 \text{ h}$  at 10°C. The bloom characteristics of the samples were measured with texture analyser (CT3 Texture Analyser, Brookfield, Middleboro, USA) in terms of hardness in grams. The bloom measurement was done three times and bloom of the gelatine used in the experiments was reported as the average of the three measurements and given in Table 2.3.

**Table 2.3** Bloom measurement data of gelatine

<b>Sample #</b>	<b>Bloom Measurement</b>
1	250.2
2	251.8
3	249.8
Average	250.6

### **2.2.3 Instrumental Analysis**

During the experiments jelly gums were stored at laboratory conditions at 15-22°C and 30-40% RH for 52 weeks except Tg experiments, in Tg part storage was done for 53 weeks. Temperature storage experiments were done at 10°C, 20°C and 30°C by using incubator (Nuve EN 300, Istanbul, Turkey) for 12 weeks.

#### **2.2.3.1 Moisture Content Measurement**

Moisture content of the jelly gums was measured by Karl Fischer titration using Titrino 702 SM with a double Pt-wire electrode (Methrom Schweiz, Zofingen, Switzerland). Minced gum sample of 0.1 g was dissolved in a medium of 20 ml Hidranal dry methanol and 20 ml formamide (Sigma- Aldrich, St. Louis, Missouri, USA). The sample solution was heated at 50°C and then titrated to dryness with Hidranal Composite (Sigma-Aldrich). All the samples were analyzed in two replicates every week.

#### **2.2.3.2 Water Activity Measurement**

Water activity were measured with water activity meter (Aqualab 4TE, Pullman, USA) at 25°C. Slices from the surface of the samples were cut and measurements were reported at constant readings. Also, portions from the center of jelly gums were measured. All of the samples were analyzed in two replicates every week at all conditions mentioned.

### **2.2.3.3 Total Soluble Solid Content Measurement**

Weight of (g) of total soluble solids in 100 g of jelly gum sample or °Brix were determined using a refractometer (Atago PAL 3, Tokyo, Japan) with a measurement range of 58-90 °Brix. Samples were sliced in 2 mm thickness and put into the equipment for the measurements. Analyses were conducted in two replicates every week at all of the conditions studied.

### **2.2.3.4 pH Measurement**

The pH of the jelly gum samples were measured using a pHmeter (Hanna HI2211, Woonsocket, USA) after the samples were cut in thin slices and mix with hot water. One part of sample was dissolved in three parts of hot water. The solution was maintained at 25°C before the measurements of jelly gums. All of the measurements were performed in two replicates weekly.

### **2.2.3.5 Texture Measurement**

A Texture Profile Analysis (TPA) test was performed using a texture analyser (CT3 Brookfield, Middleboro, USA) in two replicates weekly. Jelly gums were compressed two times with a 3 mm diameter cylindrical probe, allowing the sample to be deformed without penetration. Texture settings for measurements were done as two consecutive cycles of 50% compression; cross-head moved at a constant speed of 30 mm/min and a trigger point of 0.05 N at 24°C (Delgado and Banon, 2015). TPA force and time diagram was calculated according to the studies in literature (Bourne, 1968; Mochizuki, 2001). Table 2.4 shows the texture parameters investigated in the study by TPA test for the jelly gums.

**Table 2.4** TPA parameters for jelly gums (Delgado and Banon, 2015)

Texture variable (unit)	Description
Hardness (N)	Force required to compress the material by a given amount
Cohesiveness (no units)	Strength of the internal bonds in the sample
Springiness (mm)	Elastic recovery that occurs when the compressive force is removed
Gumminess (N)	The energy required to break down a semi-solid food ready for swallowing

#### **2.2.3.6 Colour Measurement**

Colour of the jelly gums were evaluated by measuring CIE L\*, a\*, b\* parameters by using Color Flex Ez Spectrophotometer (HunterLab, Reston, USA).

L\*, a\*, b\* parameters show whiteness/ darkness, greenness/ redness, and blueness/ yellowness values, respectively. Two replicates of colour data were recorded every week.

Hue (H\*) value gives information regarding the shade of colour and chroma (C\*) value gives information on the intensity or saturation of the colour in a product. Equations combining L\*, a\* and b\* values provide parameters that can be used for quantifying the overall colour change for the products according to Bonerz et al. (2007) and Ibarz et al. (1999). Change in hue ( $\Delta H^*$ ) is a useful parameter for colour analysis of the products because it shows the magnitude of colour change taking into account total colour difference, change in L\* value and change in chroma; hence it was used as the measure of colour change in this study. They were calculated from equations 2.1, 2.2 and 2.3.

$$\Delta H^* \text{ (Change in hue)} = [(\Delta E^*)^2 - (\Delta L^*)^2 - (\Delta C^*)^2]^{1/2} \quad (2.1)$$

$$\Delta E^* \text{ (Total colour difference)} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2.2)$$

$$\Delta C^* \text{ (Change in Chroma, saturation)} = [(\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2.3)$$

Jelly gums were also prepared without the addition of colour in order to use as control samples.

### 2.2.3.7 Sugar Profile Analysis

Determination of sucrose, glucose and fructose percentages of jelly samples were performed every 4 weeks of storage by a high performance liquid chromatography (HPLC) (Agilent 1100, San Diego, USA) as two replicates. HPLC equipment parts were; G1311A pump, G1379A vacuum degasser, G1313A ALS auto-sampler/auto-injector, G1316A Colcom column compartment and RID10A refractive index detector at 68°C.

Glucose, fructose and sucrose were separated on a 300 × 7.8 mm column (Bio-Rad Aminex HPX-87K). A column (A Bio-Rad Guard) with the same stationary phase as the separation column was placed in front of the separation column. Mobile phase was ultra pure water and flow rate used was 0.6 mL/min. 20-μL of sample volume was injected. Sugar identification was done according to the comparison of their retention times with pure standards' (Sigma-Aldrich) retention times. Quantification was done with standard calibration curves (1 mg/100 mL -5 mg/100 mL). Software of HPLC instrument (Agilent Chem Station software) was used to for data analysis.

### 2.2.3.8 Glass Transition Temperature Measurement

Differential scanning calorimetry (DSC) (PerkinElmer, Wellesly, USA) was used for measurement of glass transition temperature ( $T_g$ ) of jelly gum samples twice, once in the beginning of storage and once in the end of storage as three replications.



Samples were weighed as 10 mg and loaded into stainless steel pans. For the experiments, Q100 DSC with RCS (TA Instruments, New Castle, USA) and purging gas nitrogen with 50 mL/min flow rate was used. Indium was used for calibration. As a reference, an empty pan was involved in the measurements. Analysis was conducted with Universal Analysis software (TA Instruments, New Castle, USA).

### **2.3 Sensory Analysis**

The panel was composed of 10 trained technical staff of Kervan Gıda Sanayi ve Tic. A. Ş. All of the panelists had experience in sensory analysis.

Appearance, taste and texture descriptors of the jelly gums were scored using hedonic scale of 9 points every 4 weeks by the panelists. Samples were scored as the following scale; 1- dislike extremely, 2- dislike very much, 3- dislike moderately, 4- dislike slightly, 5- neither like nor dislike, 6- like slightly, 7- like moderately, 8- like very much and 9- like extremely. Overall acceptability score was found by taking the average of appearance, taste and texture scores.

Each batch was evaluated twice by the panelists. At least three samples were placed in plastic cups with coding of 3-digit random numbers. Three samples were evaluated at every sensory session. At each session panelists were given individual score sheet, reference sample (fresh), experimental jelly gum samples, crackers and water. Crackers and water were given to the panelists for neutralizing the mouth before tasting of each sample.

### **2.4 Preparation of Sorption Isotherms**

Desiccator method was used for preparation of sorption isotherms of selected jelly gums. Firstly, samples were put into the aluminium pans and weighed. Then, samples were equilibrated over saturated salt solutions given in Table 2.5 inside glass jars at 20°C until equilibrium was attained. Equilibrium was reached when the

difference between two consecutive weight measurements was equal or less than 1 mg/ g solids. Moisture content was determined by oven drying for 2 hours at 115°C. Three replications were conducted for each measurement. Equilibrium moisture contents of jelly gums were plotted as a function of water activity in order to obtain sorption isotherms.

**Table 2.5** Water activity of saturated salt solutions at 20°C (Sahin and Sumnu, 2006)

<b>Saturated salt solution</b>	<b>Water activity</b>
Lithium chloride	0.113
Magnesium chloride	0.331
Magnesium nitrate	0.544
Sodium chloride	0.755
Potassium chloride	0.851

## 2.5 Data Analysis

IBM SPSS Statistics 24 and StatPlus®:mac (2015 AnalystSoft Inc.) software were used to perform the statistical analysis and the regressions of experimental data. ANOVA (Analysis of Variance) was used to determine if there were significant differences between treatments ( $p \leq 0.05$ ). Duncan's Multiple Comparison Test was done for comparisons ( $p \leq 0.05$ ) when significant differences were found. Multiple regressions were performed in order to explain the relationships between independent and dependent variables.

Partial least scores (PLS) correlation is a useful tool which can be used to relate a set of y-variables versus a set of x- variables. Correlation between sensory and instrumental data was done using PLS modelling with Smart PLS 3 software (<http://www.smartpls.com>).

## CHAPTER III

### RESULTS AND DISCUSSION

#### 3.1 Moisture Content of Jelly Gums

Moisture content, brix, water activity and pH are measured in confectionery plants in order to control rheological properties and hydration degree during processing (Delgado and Banon, 2015). Water in confectionery products is one of the main factors affecting shelf life and texture (Ergun et al., 2010; Jackson, 1990). In addition to texture, which has huge impact on eating quality of products, moisture content influences the formulation percentages and thus the nutrition information per 100 g finished product. Since it is essential to provide the accurate nutritional content on the packaging and significant moisture increase or decrease affects this accuracy, moisture content is an important quality parameter (Nicholas, 2009).

Changes in moisture content in all of the formulations were measured weekly for long time storage analysis for 52 weeks. Second order multiple regression equations showing the effects of different ingredients on moisture content of fresh jelly gums are shown in Table 3.1 and Table A.1. Coefficient of determination ( $R^2$ ) value of the model was 1.000. Glucose:sucrose ratio ( $p \leq 0.001$ ) affected moisture content significantly. According to the regression; when glucose:sucrose ratio increased at the same gelatine and starch level, moisture content increased. This was an expected result since the dry solids of glucose syrup used in the experiments was 81.5% and dry solids of sucrose used in the study was 99.6%. In addition, the interactions between starch and gelatine was found to be significant. Although they did not affect moisture content as single ingredients, they had impact on moisture content together ( $p \leq 0.05$ ).

**Table 3.1** Model constants for instrumental results of jelly gums determined by multiple regression.

Parameter	Equation	R <sup>2</sup>
Moisture content (%)	$Y_1 = 22.778 \text{ G:S}^{***} - 1.375 \text{ starch}^{NS} - 0.046 \text{ gelatine}^{NS} - 7.198 \text{ G:S} \times \text{G:S}^{***} + 0.098 \text{ starch} \times \text{starch}^{NS} - 0.014 \text{ gelatine} \times \text{gelatine}^{NS} + 0.790 \text{ G:S} \times \text{starch}^{NS} + 0.063 \text{ G:S} \times \text{gelatine}^{NS} + 0.607 \text{ starch} \times \text{gelatine}^* - 0.449 \text{ G:S} \times \text{starch} \times \text{gelatine}^*$	1.000
Water activity	$Y_2 = 1.194 \text{ G:S}^{***} - 0.006 \text{ starch}^{NS} - 0.006 \text{ gelatine}^{NS} - 0.479 \text{ G:S} \times \text{G:S}^{***} - 0.015 \text{ starch} \times \text{starch}^{***} - 3.704\text{E-}5 \text{ gelatine} \times \text{gelatine}^{NS} + 0.002 \text{ G:S} \times \text{starch}^{NS} + 0.002 \text{ G:S} \times \text{gelatine}^{NS} + 0.004 \text{ starch} \times \text{gelatine}^{NS} - 0.004 \text{ G:S} \times \text{starch} \times \text{gelatine}^{NS}$	1.000
Total soluble solids (%)	$Y_3 = 134.801 \text{ G:S}^{***} - 1.342 \text{ starch}^{NS} - 0.045 \text{ gelatine}^{NS} - 53.419 \text{ G:S} \times \text{G:S}^{***} - 0.101 \text{ starch} \times \text{starch}^{NS} - 0.013 \text{ gelatine} \times \text{gelatine}^{NS} - 0.762 \text{ G:S} \times \text{starch}^{NS} - 0.058 \text{ G:S} \times \text{gelatine}^{NS} - 0.600 \text{ starch} \times \text{gelatine}^* + 0.444 \text{ G:S} \times \text{starch} \times \text{gelatine}^*$	1.000
pH	$Y_4 = 4.960 \text{ G:S}^{***} - 0.125 \text{ starch}^{NS} + 0.006 \text{ gelatine}^{NS} - 1.755 \text{ G:S} \times \text{G:S}^{***} + 0.006 \text{ starch} \times \text{starch}^{NS} - 0.001 \text{ gelatine} \times \text{gelatine}^{NS} + 0.070 \text{ G:S} \times \text{starch}^{NS} - 0.001 \text{ G:S} \times \text{gelatine}^{NS} + 0.052 \text{ starch} \times \text{gelatine}^* - 0.038 \text{ G:S} \times \text{starch} \times \text{gelatine}^*$	1.000
Hardness	$Y_5 = 1.339 \text{ G:S}^{***} - 0.037 \text{ starch}^{**} - 0.010 \text{ gelatine}^* - 0.548 \text{ G:S} \times \text{G:S}^{***} + 0.001 \text{ starch} \times \text{starch}^{NS} + 4.023\text{E-}15 \text{ gelatine} \times \text{gelatine}^{NS} + 0.032 \text{ G:S} \times \text{starch}^{**} + 0.010 \text{ G:S} \times \text{gelatine}^{***} + 0.005 \text{ starch} \times \text{gelatine}^{NS} - 0.005 \text{ G:S} \times \text{starch} \times \text{gelatine}^*$	1.000

\* means term is significant at  $p \leq 0.05$ , \*\* means term is significant at  $p \leq 0.01$ , \*\*\* means term is significant at  $p \leq 0.001$  and NS means term is not significant.

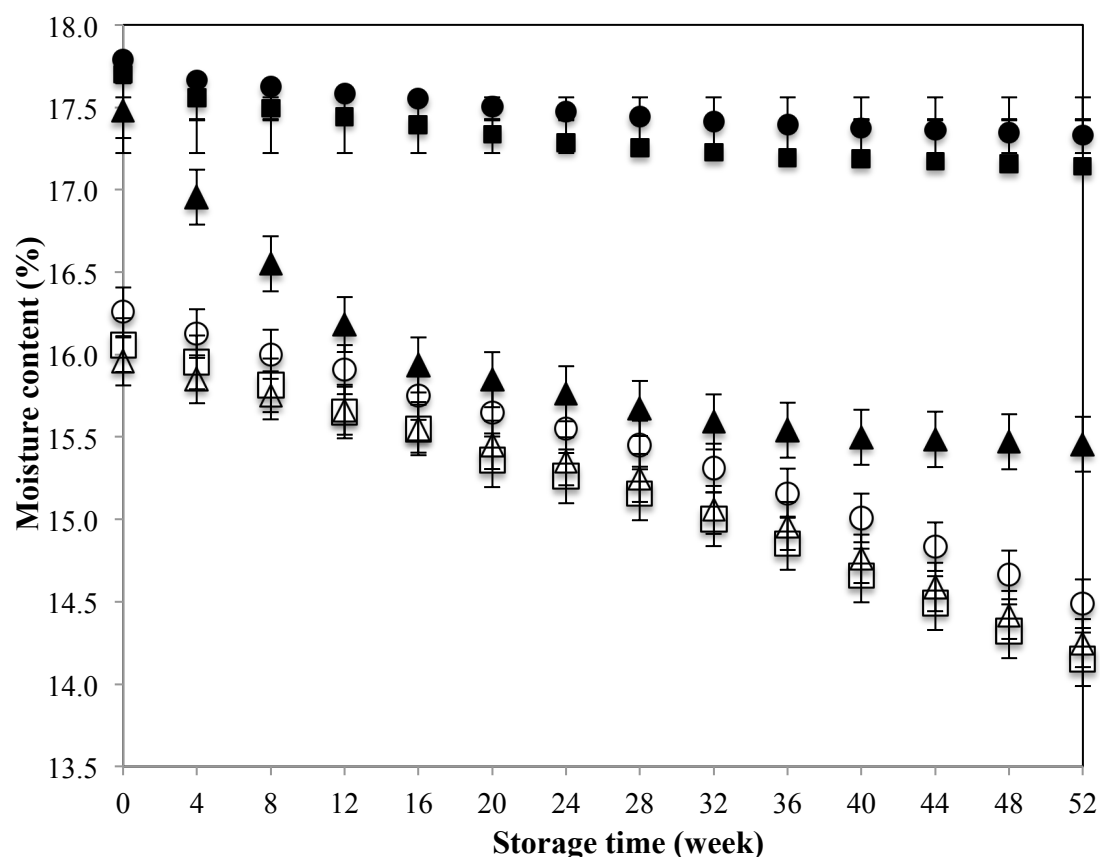
**Table 3.1** (Continued)

Parameter	Equation	R <sup>2</sup>
Cohesiveness	$Y_6 = 0.884 \text{ G:S}^{***} + 0.028 \text{ starch}^* + 0.005 \text{ gelatine}^{\text{NS}} - 0.348 \text{ G:S} \times \text{G:S}^{***} - 0.002 \text{ starch} \times \text{starch}^{\text{NS}} + 0.000 \text{ gelatine} \times \text{gelatine}^{\text{NS}} - 0.017 \text{ G:S} \times \text{starch}^* - 0.001 \text{ G:S} \times \text{gelatine}^{\text{NS}} - 0.007 \text{ starch} \times \text{gelatine}^{**} + 0.005 \text{ G:S} \times \text{starch} \times \text{gelatine}^*$	1.000
Springiness	$Y_7 = 4.752 \text{ G:S}^{***} - 0.047 \text{ starch}^{\text{NS}} + 0.006 \text{ gelatine}^{\text{NS}} - 1.673 \text{ G:S} \times \text{G:S}^{***} + 0.002 \text{ starch} \times \text{starch}^{\text{NS}} + 0.000 \text{ gelatine} \times \text{gelatine}^{\text{NS}} + 0.018 \text{ G:S} \times \text{starch}^{\text{NS}} - 0.008 \text{ G:S} \times \text{gelatine}^{\text{NS}} + 0.037 \text{ starch} \times \text{gelatine}^* - 0.027 \text{ G:S} \times \text{starch} \times \text{gelatine}^*$	1.000
Gumminess	$Y_8 = 0.764 \text{ G:S}^{***} - 0.016 \text{ starch}^{\text{NS}} - 0.005 \text{ gelatine}^{\text{NS}} - 0.317 \text{ G:S} \times \text{G:S}^{***} + 0.003 \text{ starch} \times \text{starch}^* + 0.000 \text{ gelatine} \times \text{gelatine}^{\text{NS}} + 0.10 \text{ G:S} \times \text{starch}^{\text{NS}} + 0.002 \text{ G:S} \times \text{gelatine}^{\text{NS}} - 0.001 \text{ starch} \times \text{gelatine}^{\text{NS}} + 0.001 \text{ G:S} \times \text{starch} \times \text{gelatine}^{\text{NS}}$	1.000

\* means term is significant at  $p \leq 0.05$ , \*\* means term is significant at  $p \leq 0.01$ , \*\*\* means term is significant at  $p \leq 0.001$  and NS means term is not significant.

When the variation of moisture content with storage time was analysed; it was observed that samples with glucose syrup:sucrose ratio of 1.5 and gelatine levels of 3% and 4.5% had tendency to be stabilized after 16 weeks while in 6% gelatine level, the same behaviour was observed after 28 weeks (Figure 3.1). However, moisture content of samples having glucose syrup:sucrose ratio of 1.1 decreased linearly during storage. This meant that formulations having higher glucose syrup:sucrose ratio were more stable in terms of moisture content. This result was expected since more glucose syrup was available in the formulations with glucose syrup:sucrose ratio of 1.5. Glucose syrup is good at retention of moisture and used

for the humectancy purpose in confectionery industry in order to prevent the product from drying out during shelf life (Hull, 2010).



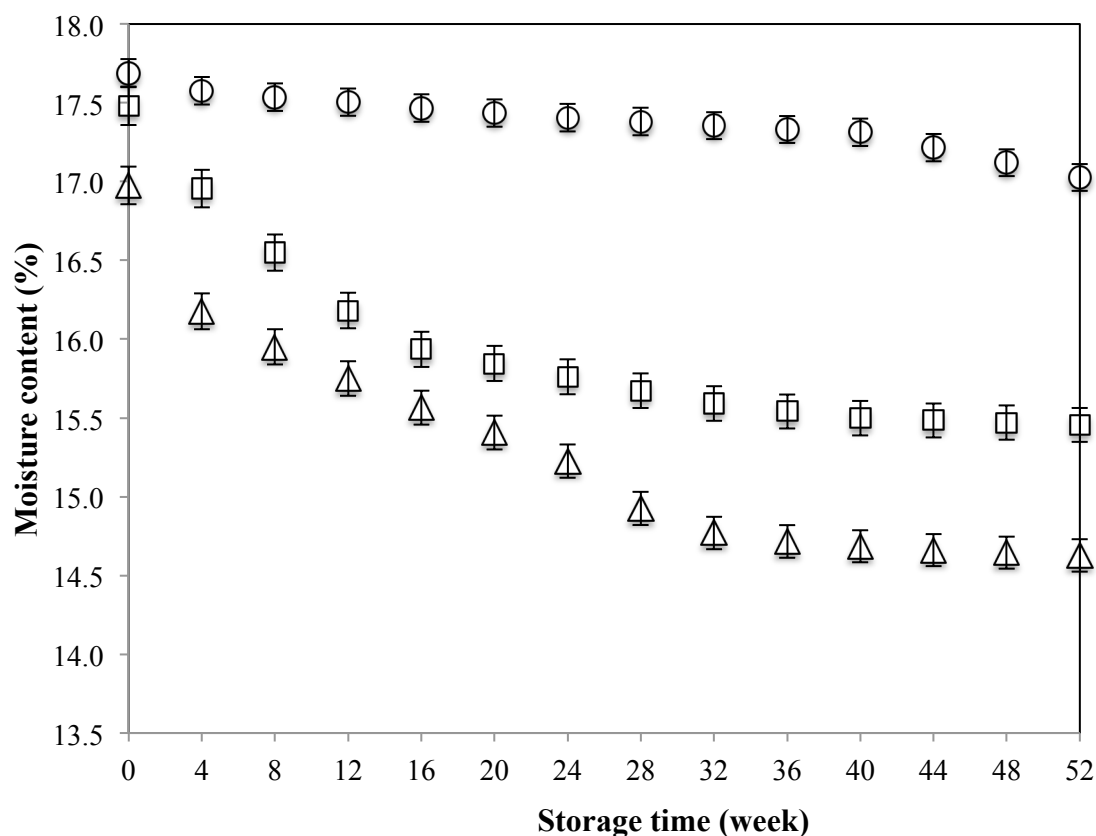
**Figure 3.1** Change in moisture content with storage time at 15-22°C with different glucose syrup:sucrose ratios and gelatine amounts at starch level of 1%

○ G:S=1.1, Gelatine=3%; □ G:S=1.1, Gelatine=4.5%; △ G:S=1.1, Gelatine=6%; ● G:S=1.5, Gelatine=3%; ■ G:S=1.5, Gelatine=4.5%; ▲ G:S=1.5, Gelatine=6%

Moisture loss during storage is an indicator for quality deterioration for confectionery products since it affects sugar crystallization and glass formation (Jackson, 1995). According to Figure 3.1, it was seen that moisture decrease was higher for the formulations having lower glucose syrup:sucrose ratio. This might have been a sign of sucrose crystallization as sucrose molecules in the jellies might have expelled moisture during sugar crystallization (Hartel, 1993; Edmond, 2000). In

addition, it was seen that as the gelatine level increased moisture loss increased. This result was similar to the results given in a previous study (Tan and Lim, 2008), where marshmallows having 2.2% gelatine had faster moisture loss when compared to the marshmallows with 2.0% gelatine during storage of 25 weeks at 25°C. This might be due to the increase of gel networking.

Figure 3.2 shows the effect of starch concentration on moisture change at glucose syrup:sucrose ratio of 1.5 and 6% gelatine. It was observed that as the starch concentration increased moisture loss increased during storage. Jelly gums containing the highest starch concentration of 1.5% had the highest moisture loss with 2.35% at the end of 52 weeks of storage at laboratory conditions, whereas moisture loss was 0.66% and 2.02% at starch levels of 0% and 1%, respectively. This might have been due to the fact that increase of starch might have increased the interfacial area that caused rapid moisture loss during storage (Lim et al., 2006).

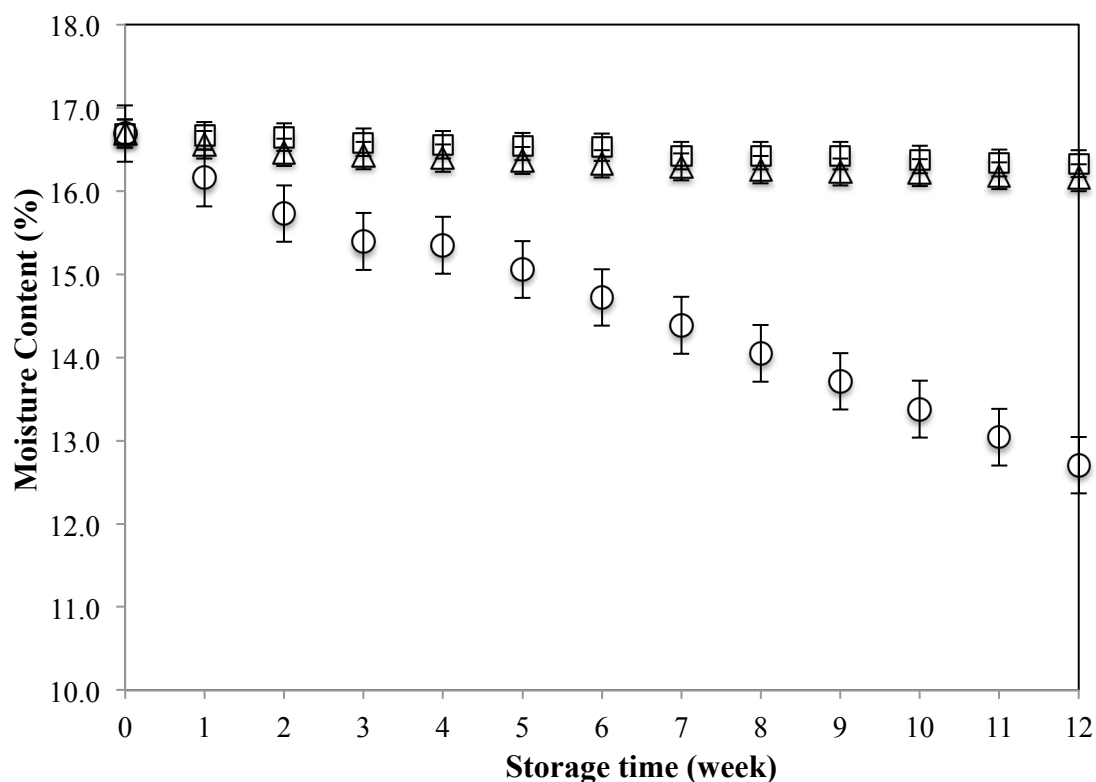


**Figure 3.2** Change in moisture content with storage time at 15-22°C with different starch amounts at glucose syrup:sucrose ratio of 1.5 and 6% gelatine

O no starch; □ starch= 1.0%; Δ starch= 1.5%

Storage experiments in the study were also conducted at different temperatures (10°C, 20°C and 30°C) for 12 weeks (Figure 3.3). These temperatures were selected since the recommended storage conditions of confectionery products were 15-25°C and jelly gum containing gelatine had a melting temperature below 35°C. Moisture losses of the samples stored at 20°C mimicked the ones stored at laboratory conditions at 15-22°C for all formulations. For these formulations, rate of moisture loss at 10°C and 20°C was close to each other but it was higher during storage at 30°C. This was expected as the diffusion coefficient was known to depend on temperature. Similarly, supporting this result, in the study of Nicholas (2009), wine gums containing gelatine and starch stored at 25°C lost moisture at faster rate than those stored at 20°C.





**Figure 3.3** Change in moisture content with storage time for jelly gums having glucose syrup:sucrose ratio of 1.1, Starch= 1.5% and Gelatine= 6% at different temperatures

□ 10°C, Δ 20°C, O 30°C

It was observed that slope of the variation of moisture content with respect to storage time were similar at storage temperature of both 10°C and 30°C for all gelatine levels at the highest glucose syrup: sucrose ratio at the same starch level (Table 3.2). On the contrary, for lower glucose syrup: sucrose ratio, 1.5% starch level was found to be more effective than gelatine concentrations when the slopes given in Table 3.2 were compared. This might be due to the fact that starch might have had different interfacial area properties than gelatine in jelly system (Lim et. al, 2006). According to the study of Ward and Courts (1977), high sucrose was observed to increase the gel strength depending on the age and the temperature of the gel. Hence, in the light

of the mentioned study with glucose syrup:sucrose ratio of 1.5, gel strength was thought to be lower, which might decrease the stability at low temperature of 10°C. The properties of jelly gums containing both gelatine and starch as gelling agents were affected by the presence of other ingredients in the recipe (Nicholas, 2009). This makes these systems difficult to be predicted adequately due to the complexity of the ingredient interactions.

In the jelly gum samples containing no starch, the gelling agent role only belonged to gelatine in the formulations. When compared with starch, gelatine has different setting properties. Gelatine is very temperature dependent ingredient and it sets the product to a rubbery one (US Patent 5626896). In addition; in US 6403140 B1 patent, it was also mentioned that desired gelling during shelf life was preferably at about 5-20%, calculated as percentages from the jellying product. Thus, gelatine amounts more than 5% might have been more stable at low temperatures (Table 3.2). In addition, variation of moisture content was similar at all temperatures independent of gelatine concentration.

Volume loss was observed when the temperature was increased over the region of 18°C to 24°C, and slight weakening of the elastic forces of jelly system was reported from 0°C to 18°C (Ling, 2003). Therefore, moisture loss was easier and faster in less volume with weak elastic forces during increase of temperature. Similarly, in our study it was observed that when the storage temperature increased, rate of moisture loss increased when the rates of the predictive equations given in Table 3.2 were compared. In other words, higher slope of the line showed faster moisture loss during storage of jelly gums. This might be due to the loss of volume and weakening of the elastic forces as the temperature increased. In addition, the most moisture stable formulations were jelly gums with 1% starch and 6% gelatine, 1.5% starch and 3% gelatine, 1.5% starch and 6% gelatine and 0% starch and 3% gelatine with glucose syrup:sucrose ratio of 1.1 at 10°C storage since these formulations had the lowest slope of moisture content variation at mentioned temperature (Table 3.2).

As can be seen in Table 3.2; at glucose syrup:sucrose ratio of 1.1 the samples were more stable at 1.5% starch level as compared to 1% starch level at 10°C. Modified starches were stated to improve the mechanical properties like acid, heat and moisture stability at low temperatures (Ellis et al., 1998; Kunaik and Marchessault, 1972; Seker and Hanna, 2006; Singh et al., 2007). On the other hand, it was seen that when glucose syrup:sucrose ratio increased, rate of moisture loss was decreased at 3% gelatine level at 30°C at all starch levels (Table 3.2). This result might be related to the glucose syrup concentration since it could regulate moisture stability of jellies (Hull, 2010).

When moisture content values of the jelly gums stored at laboratory conditions at 15-22°C for 52 weeks and moisture content values of samples stored at different temperatures for 12 weeks were compared; for all of the recipes, it was seen that the moisture content changes in one week at 30°C were similar to the changes in eight weeks at laboratory conditions at 15-22°C (Figure 3.1 and Table 3.2). According to this finding, 30°C was found to be a good accelerated temperature condition for the shelf life study of jelly gums containing gelatine and starch in terms of moisture content.

**Table 3.2** Predictive equations obtained for moisture content (m.c.) with respect to time (t) at different temperatures for selected jelly gums.

G:S ratio	Starch (%)	Gelatine (%)	Temperature (°C)	Predictive equation	R <sup>2</sup>
1.1	0	3	10	$m.c. (\%) = -0.2305(t)+16.0420$	0.985
1.1	0	3	30	$m.c. (\%) = -0.3783(t)+16.2270$	0.998
1.1	0	6	10	$m.c. (\%) = -0.0428(t)+15.9880$	0.963
1.1	0	6	30	$m.c. (\%) = -0.2395(t)+15.9020$	0.993
1.5	0	3	10	$m.c. (\%) = -0.1673(t)+17.9630$	0.984
1.5	0	3	30	$m.c. (\%) = -0.2652(t)+17.9480$	1.000
1.5	0	6	10	$m.c. (\%) = -0.1239(t)+17.6890$	0.999
1.5	0	6	30	$m.c. (\%) = -0.1624(t)+17.6730$	0.984
1.1	1	3	10	$m.c. (\%) = -0.0367(t)+16.2680$	0.962
1.1	1	3	30	$m.c. (\%) = -0.2224(t)+16.1390$	0.988
1.1	1	6	10	$m.c. (\%) = -0.0302(t)+15.9600$	0.978
1.1	1	6	30	$m.c. (\%) = -0.1988(t)+15.9190$	0.999
1.5	1	3	10	$m.c. (\%) = -0.0401(t)+17.7250$	0.977
1.5	1	3	30	$m.c. (\%) = -0.1193(t)+17.7750$	0.999
1.1	1.5	3	10	$m.c. (\%) = -0.0307(t)+16.2590$	0.978
1.1	1.5	3	30	$m.c. (\%) = -0.3971(t)+15.9900$	0.991
1.1	1.5	6	10	$m.c. (\%) = -0.0316(t)+16.6910$	0.978
1.1	1.5	6	30	$m.c. (\%) = -0.3142(t)+16.5280$	0.993
1.5	1.5	3	10	$m.c. (\%) = -0.1144(t)+17.5690$	0.999
1.5	1.5	3	30	$m.c. (\%) = -0.2820(t)+17.2730$	0.973
1.5	1.5	6	10	$m.c. (\%) = -0.1067(t)+16.8920$	0.991
1.5	1.5	6	30	$m.c. (\%) = -0.2819(t)+16.7630$	0.966

### 3.2 Apparent Diffusion Coefficient of Moisture in Jelly Gums

Conditioning in starch moulds is one of the most important stages in the production of jelly gums (Figure 1.2). Conditioning decreases moisture and increases total soluble content, and also modifies textural properties of jellies (Vieira et al., 2008). There are three main goals of conditioning of jellies, which are to strengthen the gummies in a way that they can resist mechanical handling without deformation, to decrease stickiness and to protect the jellies from sweating after packaging (Nicholas, 2010). Since most of the water in jellies is removed during conditioning step, diffusion coefficient information may help to prevent texture defects and to ensure optimum conditions in terms of product quality and time and energy consumption (Ziegler et al., 2003; Sudharsan et al. 2004; Burey et al., 2009).

Change in moisture concentration of jelly gums can be shown by Fick's second law of diffusion (Crank, 1975).

$$\frac{\delta M}{\delta t} = D_a \frac{\delta^2 M}{\delta x^2} \quad (3.1)$$

where  $M$  is the moisture concentration,  $t$  is the time,  $Da$  is the diffusion coefficient and  $x$  is the distance (m). Assuming the jelly gum is a slab of infinite extent in starch conditioning, Newman's solution was given in Equation 3.2 below (Newman, 1931).

$$\frac{M_t}{M_i} = \frac{8}{\pi^2} \left[ \exp\left(\frac{-1\pi^2 D_a t}{4 h^2}\right) + \frac{1}{9} \exp\left(\frac{-9\pi^2 D_a t}{4 h^2}\right) + \frac{1}{25} \exp\left(\frac{-1\pi^2 D_a t}{4 h^2}\right) + \dots \right] \quad (3.2)$$

Higher order terms were neglected in Equation 3.2 by assuming long time solution and Equation 3.3 was obtained.

$$\frac{M_t}{M_i} = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_a t}{4 h^2}\right) \quad (3.3)$$

Equation 3.3 was arranged to Equation 3.4.

$$-\ln\left(\frac{\pi^2 Mt}{8 Mi}\right) = \frac{\pi^2 Da t}{4 h^2} \quad (3.4)$$

where  $Da$  is the diffusion coefficient ( $\text{m}^2/\text{s}$ ), and  $h$  is the thickness of jelly gums ( $\text{m}$ ) during conditioning in starch, which was 15 mm in our study.

Diffusion coefficient values of selected jelly gums after conditioning for 24 hours in starch were determined by using Equation 3.4. Selected formulations were the ones which were scored the highest in terms overall acceptability in sensory part of this study (Table 3.9). Diffusion coefficient values of formulation 9 (Table 2.2) with glucose syrup:sucrose ratio of 1.1, 1.5% starch and 6% gelatine and formulation 12 (Table 2.2) with glucose syrup:sucrose ratio of 1.5, no starch and 6% gelatine were found as  $5.93 \times 10^{-10} \text{ m}^2/\text{s}$  and  $5.46 \times 10^{-10} \text{ m}^2/\text{s}$ , respectively. Fourier number was checked for the mentioned formulations and it was found higher than 0.2. Diffusion coefficient was slightly higher when starch was included to the formulation, which might be due to higher interfacial area that allowed faster moisture loss during conditioning of jellies (Lim et. al., 2006).

Nicholas (2010) reported diffusion coefficient of wine gummies, containing high amount of starch, as  $3.14 \times 10^{-10} \text{ m}^2/\text{s}$  by using dynamic vapour sorption machine. In another study, Sudharsan et al. (2004) found moisture diffusion coefficient of starch moulded confectionery as  $1.73 \times 10^{-10} \text{ m}^2/\text{s}$ . The reasons why the results were different from mentioned studies might be due to the formulation differences of the model systems tested as our formulations contain very little starch and due to the differences in processing and test conditions because jelly gums are complex food systems with interactions of sugars and gelling agents.

### 3.3 Water Activity of Jelly Gums

Water is one of the most important components of confectionery products and understanding of the behaviour of water in confectionery has grown over years, proceeding from the moisture content to water activity and recently to water mobility. Water activity is used to describe moisture migration, texture and microbial stability during storage of products (Ergun et al., 2010). According to Lees (1995), water activity provides insight into physical and chemical effects seen during and after manufacture.

Multiple regression result of water activity was given in Table 3.1 and Table A.2. Ingredient parameters of glucose:sucrose ratio and starch level were significantly important for water activity of jellies ( $p \leq 0.001$ ). This finding was in accordance with the study of Cakebread (1969), who mentioned that each of the ingredients used in confectionery recipes had its own affect on water activity, dependent on the nature and concentration in the final product.

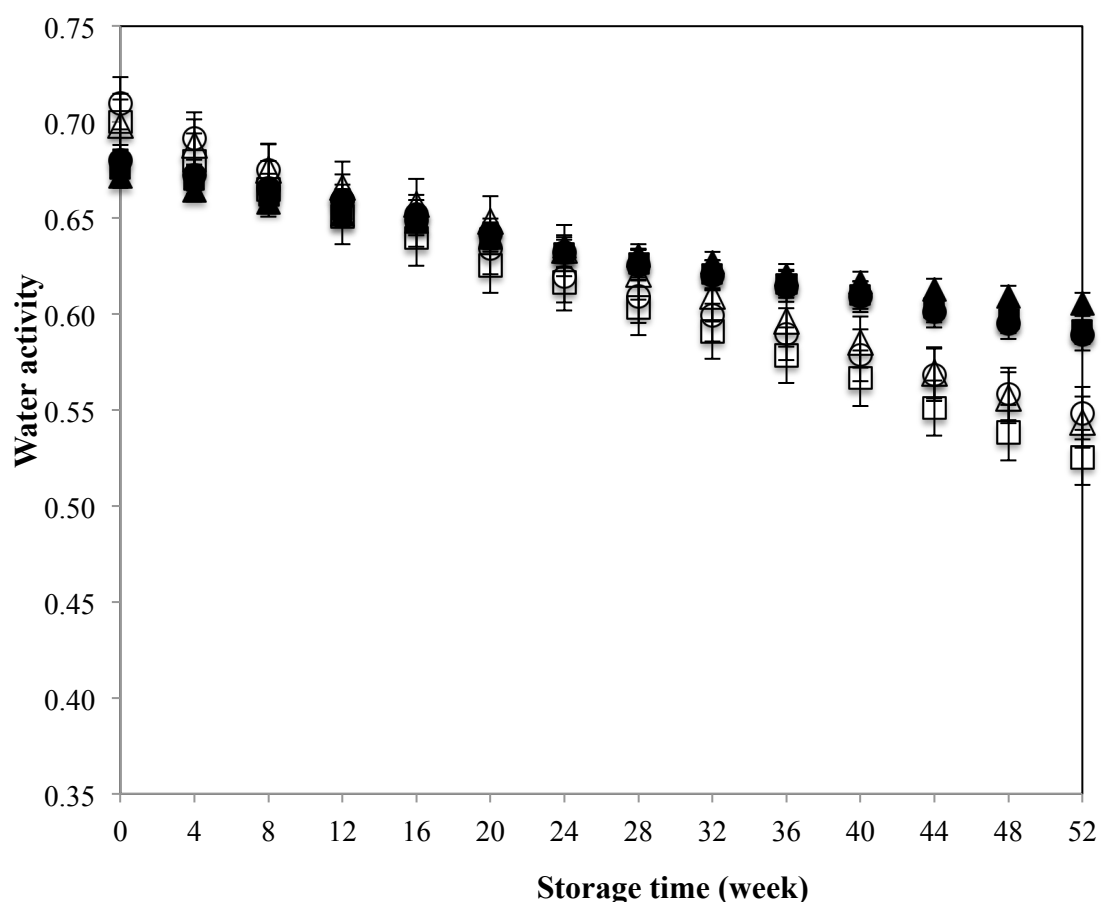
According to the regression equation shown in Table 3.1, it was seen that as glucose syrup to sucrose ratio increased (either by increasing glucose syrup or by decreasing sucrose) in the formulation, water activity of fresh jelly gums decreased. This can be explained by the humectancy property of the glucose syrup, which reduces water activity (Ergun et al., 2010; Hull, 2010). Similarly, in the study of Enrione (2005); at moisture contents higher than 15% (similar to the moisture content values of fresh jelly gums), adding glycerol contributed to a decrease in water activity which was related to humectant property of glycerol.

As mentioned earlier, it was seen that starch level was an important ingredient for water activity. According to Enrione (2005), who studied waxy corn, rice and wheat starch; starch could aid to control moisture dependent processes in foods. Starch affecting water availability, might have been due to the fact that water had a significant plasticizing effect in the case of food polymers like starch and gelatine (Healey et al. 1974; Attenburrow et al. 1989; Peleg, 1993; Haris and Peleg, 1996;

Laohakunjit and Noomhorm, 2004; Enrione, 2005), which were used in confectionery as gelling agents. Regression coefficient of starch level was significant, whereas that of gelatine level was not (Table 3.1). This situation might be explained by high molecular weight soluble ingredients such as proteins (gelatine) and hydrocolloids having little influence on reducing water activity (Ergun et al., 2010).

It was seen that water activity for all jellies decreased linearly with respect to time (Figure 3.4). Predictive equations were shown in Table 3.3. Coefficient of determination values ( $R^2$ ) ranged from 0.944 to 0.999, meaning a good correlation between actual and predictive values.





**Figure 3.4** Change in water activity with storage time at 15-22°C with different glucose syrup:sucrose ratios and gelatine amounts at starch level of 1%

O G:S=1.1, Gelatine=3%; □ G:S=1.1, Gelatine=4.5%; △ G:S=1.1, Gelatine=6%; ● G:S=1.5, Gelatine=3%; ■ G:S=1.5, Gelatine=4.5%; ▲ G:S=1.5, Gelatine=6%

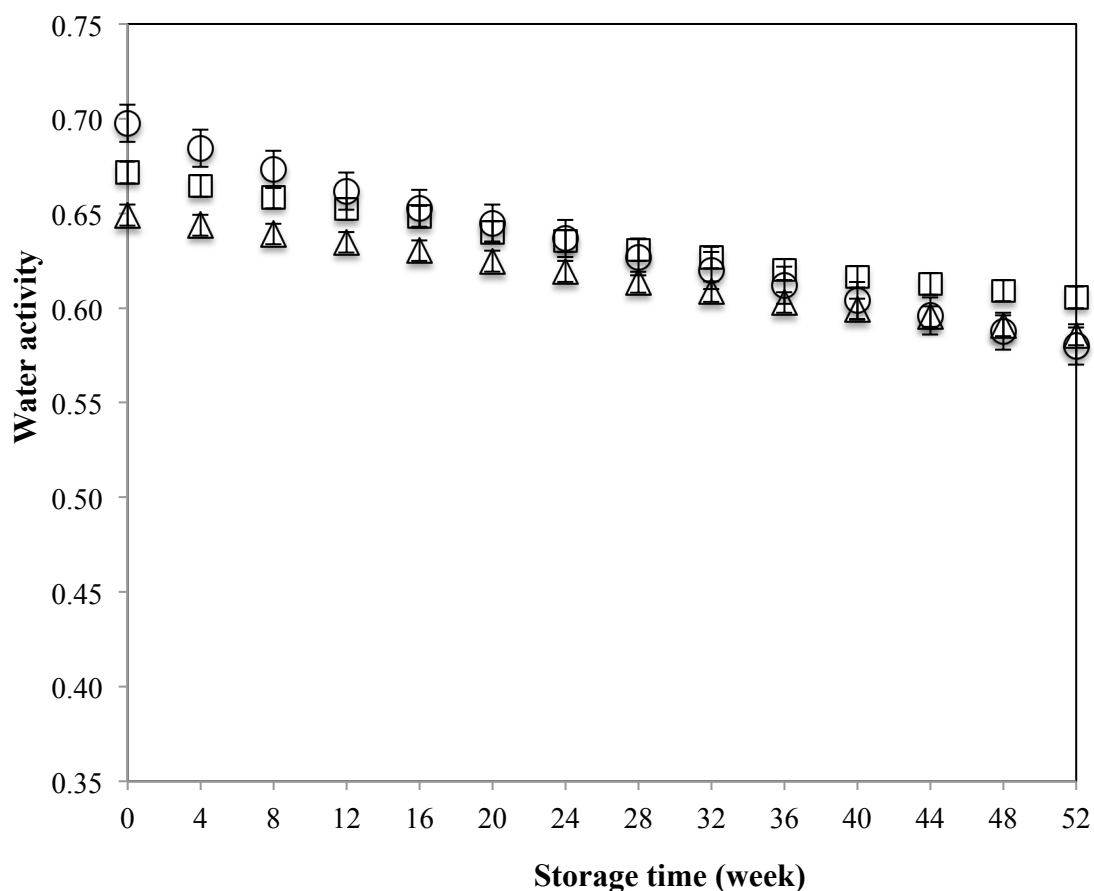
Water activity was measured during storage of jelly gums. The relative rate of water activity change of samples with glucose syrup:sucrose ratio of 1.5 was slower than that of the samples with glucose syrup:sucrose ratio of 1.1 (Table 3.3). The result of formulation containing more glucose syrup, namely glucose syrup:sucrose ratio of 1.5, were more stable and showed slower change of water activity during storage. This could be explained with the humectant property of glucose syrup as an ingredient since it prevented jelly gums from drying out by regulating water activity (Hull, 2010).

**Table 3.3** Predictive equations obtained for water activity ( $a_w$ ) with respect to time (t) for all jelly gum formulations for storage at 15-22°C and 30-40% RH

<b>G:S ratio</b>	<b>Starch (%)</b>	<b>Gelatine (%)</b>	<b>Predictive equation</b>	<b>R<sup>2</sup></b>
1.1	0	3	$a_w = -0.0079(t) + 0.7099$	0.982
1.1	0	4.5	$a_w = -0.0050(t) + 0.7198$	0.999
1.1	0	6	$a_w = -0.0039(t) + 0.7040$	0.998
1.1	1	3	$a_w = -0.0030(t) + 0.6993$	0.990
1.1	1	4.5	$a_w = -0.0032(t) + 0.6932$	0.998
1.1	1	6	$a_w = -0.0030(t) + 0.7022$	0.997
1.1	1.5	3	$a_w = -0.0025(t) + 0.6807$	0.999
1.1	1.5	4.5	$a_w = -0.0022(t) + 0.6763$	0.999
1.1	1.5	6	$a_w = -0.0026(t) + 0.6731$	0.999
1.5	0	3	$a_w = -0.0017(t) + 0.6947$	0.944
1.5	0	4.5	$a_w = -0.0019(t) + 0.6990$	0.997
1.5	0	6	$a_w = -0.0022(t) + 0.6910$	0.995
1.5	1	3	$a_w = -0.0018(t) + 0.6788$	0.995
1.5	1	4.5	$a_w = -0.0016(t) + 0.6739$	0.996
1.5	1	6	$a_w = -0.0013(t) + 0.6681$	0.991
1.5	1.5	3	$a_w = -0.0012(t) + 0.6612$	0.994
1.5	1.5	4.5	$a_w = -0.0012(t) + 0.6595$	0.998
1.5	1.5	6	$a_w = -0.0012(t) + 0.6489$	0.999

The effect of starch amount on water activity of jellies when glucose syrup:sucrose ratio was 1.5 and 6% gelatine was used can be seen in Figure 3.5. It was seen that the effect of starch amount on water activity was not as sharp as it was observed on moisture content (Figure 3.2). Samples containing starch had less water activity decrease of 0.0660 and 0.0632 at starch levels 1% and 1.5%, respectively, after 52 weeks storage at laboratory conditions. However, the decrease in water activity of no starch sample was 0.1177 at week 52. This difference between no starch and with

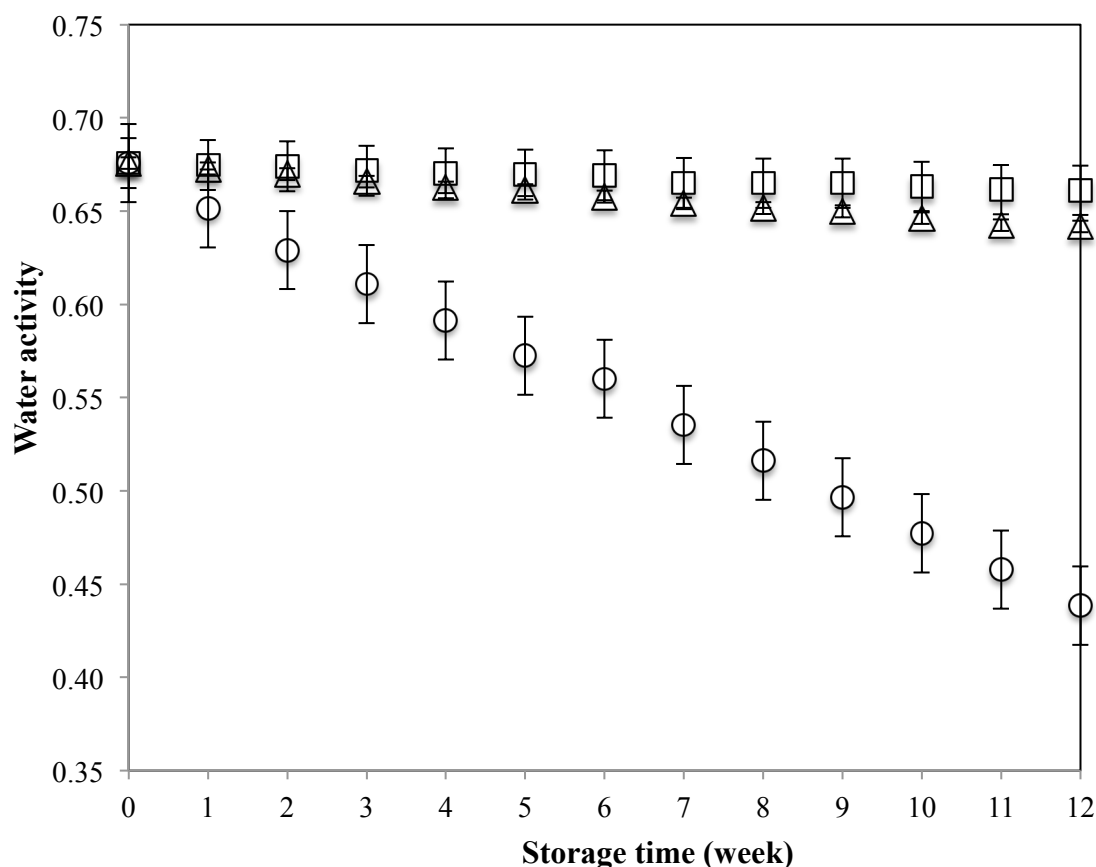
starch formulations might be in accordance with the water activity regression equation given in Table 3.1. Because coefficient of starch was not significant, however, that of starch $\times$ starch was significant and can be seen in the mentioned table.



**Figure 3.5** Change in water activity with storage time at 15-22°C with different starch amounts at glucose syrup:sucrose ratio of 1.5 and 6% gelatine

O no starch; □ starch= 1.0%; Δ starch= 1.5%

Knowledge of water activity of food products as a function of temperature is important during processing, handling, packaging and storage to prevent deleterious results such as collapse, caking and stickiness (Chuy and Labuza, 1994). Thus, temperature studies of jelly gums on water activity change were conducted at 10°C, 20°C and 30°C for 12 weeks. It was observed that water activity decreased linearly for all formulations for studied temperatures as shown in Figure 3.6.



**Figure 3.6** Change in water activity with storage time for jelly gums having glucose syrup:sucrose ratio of 1.1, Starch= 1.5% and Gelatine= 6% at different temperatures  
□ 10°C, Δ 20°C, O 30°C

When jelly gums were stored at high temperature of 30°C, the relative rates of water activity change were higher as compared to the samples stored at 20°C (Figure 3.6). Hence, 30°C storage could be a choice of a condition in the accelerated shelf life studies of jelly gums containing gelatine and starch for water activity parameter since rate of acceleration in terms of water activity was higher at the stated temperature. This might be the result of loss of volume and weakening of the elastic forces by the jelly gums when the temperature was elevated (Ling, 2003). Similarly, according to Ergun et al. (2010), elevated temperatures and humidities are used to enhance degradation in accelerated shelf life tests of confections. Taub and Singh (1998) mentioned that increasing temperature was often used to change reaction rates in

accelerated storage studies and an increase of 10°C could generally double reaction rates. Yet, in our study higher rates that were more than double were observed in terms of water activity loss. This might be due to the fact that jelly gums were complex food systems and the rates were product specific (Hough et al. 2006; Ergun et al., 2010).

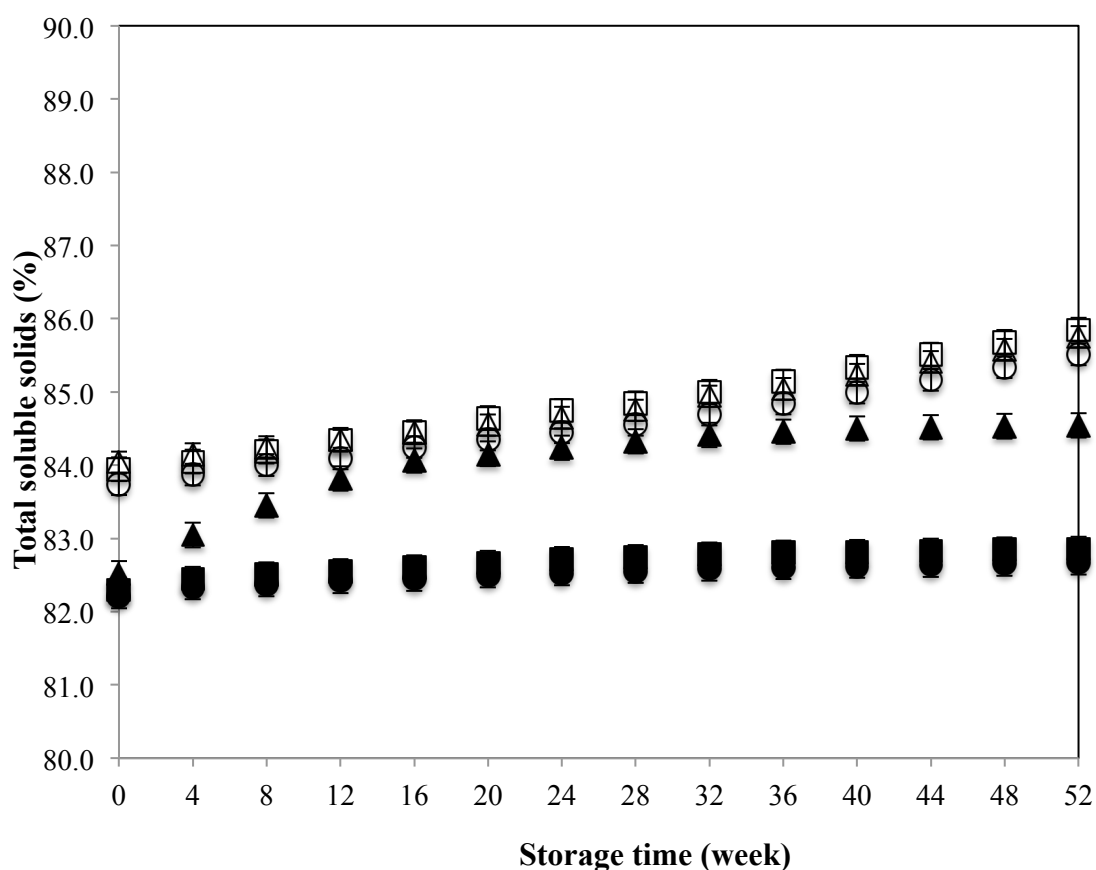
### **3.4 Total Soluble Solids of Jelly Gums**

Total soluble solids (%) is a physical parameter that is measured in an easy and fast way in plants for controlling the degree of hydration and rheology of products at different manufacturing steps of jelly gums, especially at depositing stage before conditioning and destarching stage after conditioning (Delgado and Banon, 2015). In addition, in a private conversation with Quality Assurance Manager of Kervan Gıda (Istanbul, Turkey), total soluble solids (%) is stated to be one of the first parameters that is investigated first and in a fast manner in a jelly gum.

According to multiple regression results of total soluble solids of fresh samples given in Table 3.1 and Table A.3, glucose syrup:sucrose ratio was found to be an important factor affecting total soluble solids ( $p \leq 0.001$ ). It was seen that significant terms affecting total soluble solids (%) was identical to those affecting moisture content (%) although rapid refractometer measurement is known to be less sensitive than Karl Fischer titration. As mentioned before, moisture content measurements were done by Karl Fischer titration. This method is useful for the analyses of dried fruits, candies and fats (Pomeranz and Meloan, 1994) and is widely used for confectionery applications (Ergun et al., 2010). In Karl Fischer method, the reagent reacts only with water and this eliminates the error caused from the detection of the volatile components (Beard, 2001). Refractometer was used to measure total soluble solids (%) values of jelly gums in this study, in which refractive index value was recorded. According to Pancoast and Junk (1980) and Beard (2001), refractive index can be used to determine the percentage of dry substance and hence the moisture content of the sample. On the other hand, readings of a refractometer used for confectionery

products give slightly erroneous results as most refractometers read in a scale of sucrose weight percent (Ergun et. al, 2010). However, jelly gums contain not only sucrose but also high amount of glucose syrups in the formulations. Similar to moisture content regression results, there might have been some possible interactions between starch and gelatine since they did not affect total soluble solids of jelly gums as single ingredients, whereas, they affected total soluble solids together ( $p \leq 0.05$ ) (Table 3.1).

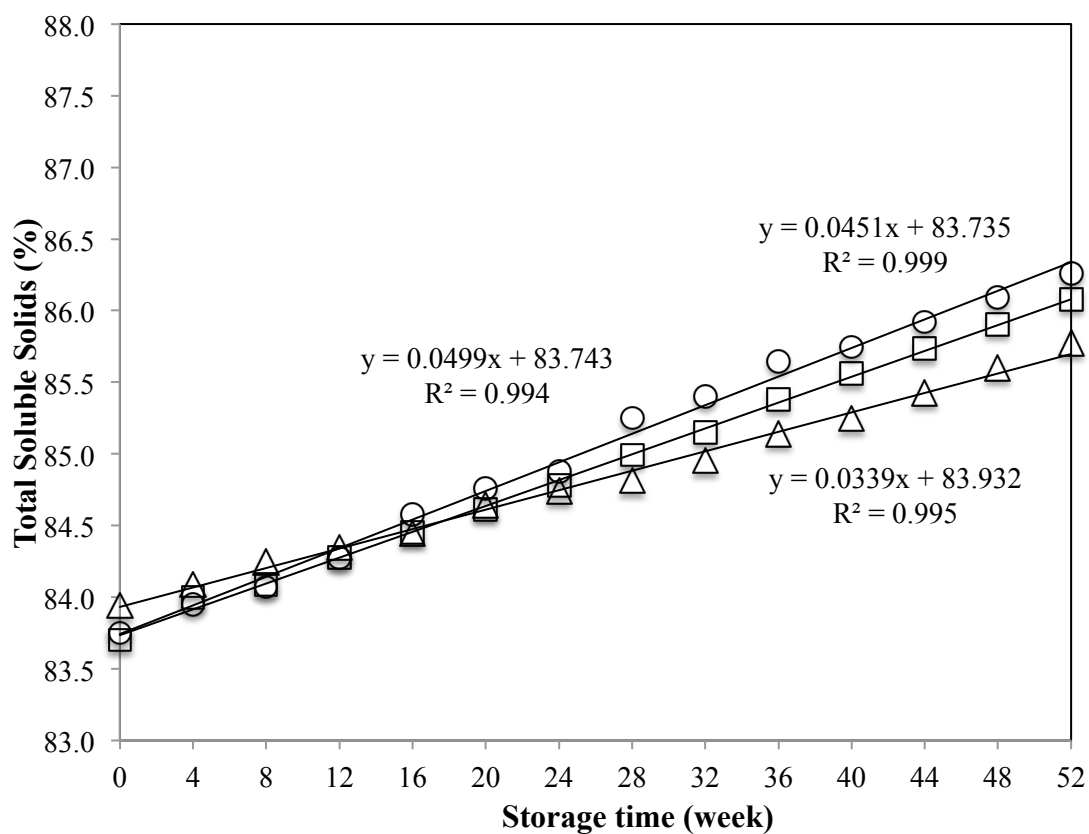
Figure 3.7 shows variation of total soluble solids (%) with respect to storage time at laboratory conditions for all of the jelly gum formulations. It was seen that total soluble solids of jellies with glucose syrup:sucrose ratio of 1.5 became constant after 16 weeks, but total soluble solids (%) of samples with glucose syrup:sucrose ratio of 1.1 increased linearly during storage. This result, which was similar to the one discussed in the moisture content part of this study, showed that jelly gums containing more glucose syrup were more stable than the ones having less glucose syrup in terms of total soluble solids parameter as well. This finding was due to the stabilizing property of glucose syrup by retaining moisture in the jelly gum during storage (Jackson, 1995; Hull 2010). In addition, Fontana (2016) mentioned that combinations of sucrose with invert or glucose syrups, or both, increased total soluble solids and thus they were often used in jams and preserves.



**Figure 3.7** Change in total soluble solids (%) with storage time at 15-22°C with different glucose syrup:sucrose ratios and gelatine amounts at starch level of 1%

O G:S=1.1, Gelatine=3%; □ G:S=1.1, Gelatine=4.5%; △ G:S=1.1, Gelatine=6%; ● G:S=1.5, Gelatine=3%; ■ G:S=1.5, Gelatine=4.5%; ▲ G:S=1.5, Gelatine=6%

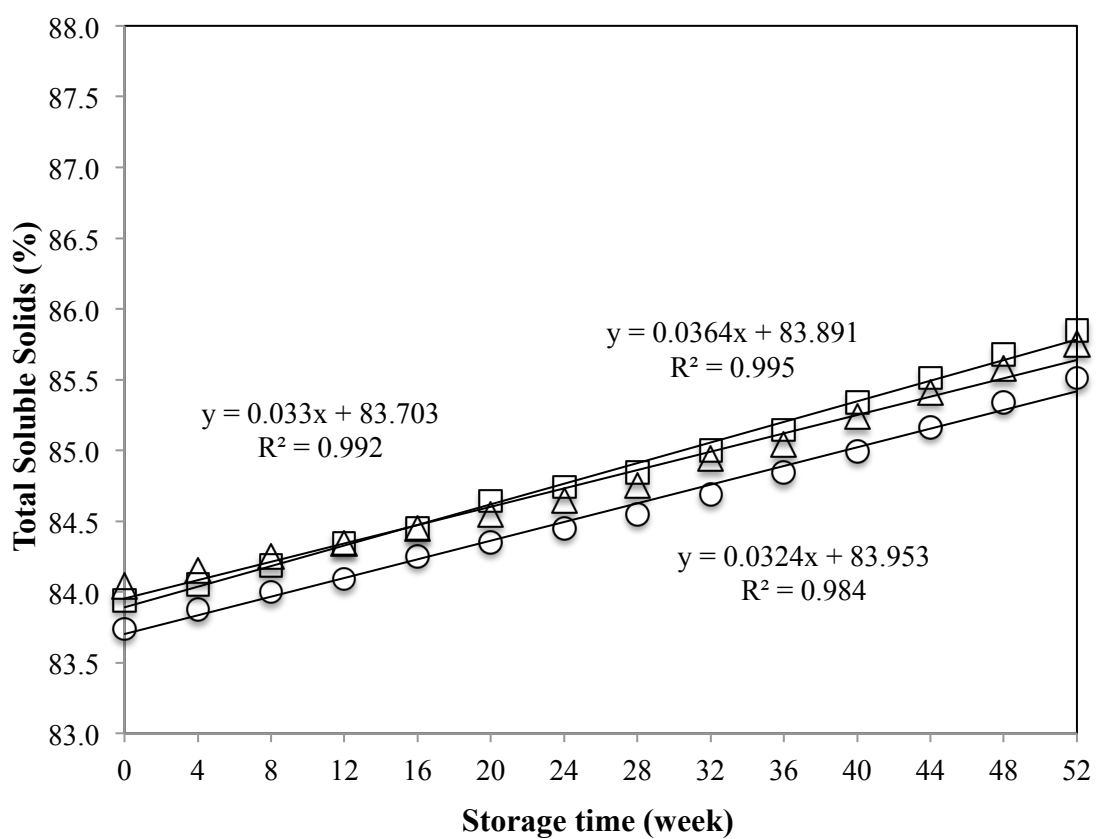
As mentioned before total soluble solids of jelly gums having glucose syrup:sucrose ratio of 1.1 increased linearly and linear lines of best fit for samples were shown in Figures 3.8-3.10 with high coefficient of determination values between 0.984-0.999. Predictive equations are tabulated in Table 3.4. It was seen that gelatine level was not effective as can be seen from the rates, which were very identical.



**Figure 3.8** Variation of total soluble solids of jelly gums with formulations of glucose syrup:sucrose ratio of 1.1 and no starch during storage at laboratory conditions at 15-22°C and 30-40% RH

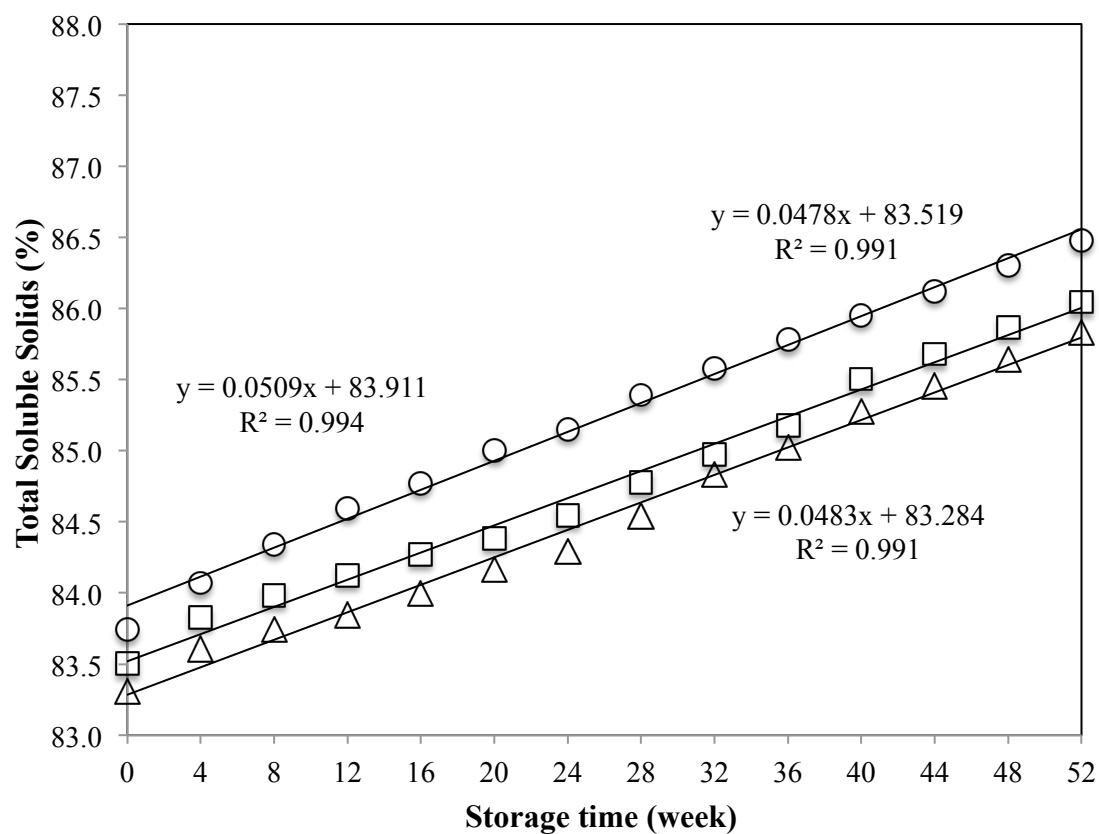
O Gelatine=3%, □ Gelatine=4.5%, Δ Gelatine=6%





**Figure 3.9** Variation of total soluble solids of jelly gums with formulations of glucose syrup:sucrose ratio of 1.1 and 1% starch during storage at laboratory conditions at 15-22°C and 30-40% RH

O Gelatine=3%, □ Gelatine=4.5%, Δ Gelatine=6%



**Figure 3.10** Variation of total soluble solids of jelly gums with formulations of glucose syrup:sucrose ratio of 1.1 and 1.5% starch during storage at laboratory conditions at 15-22°C and 30-40% RH

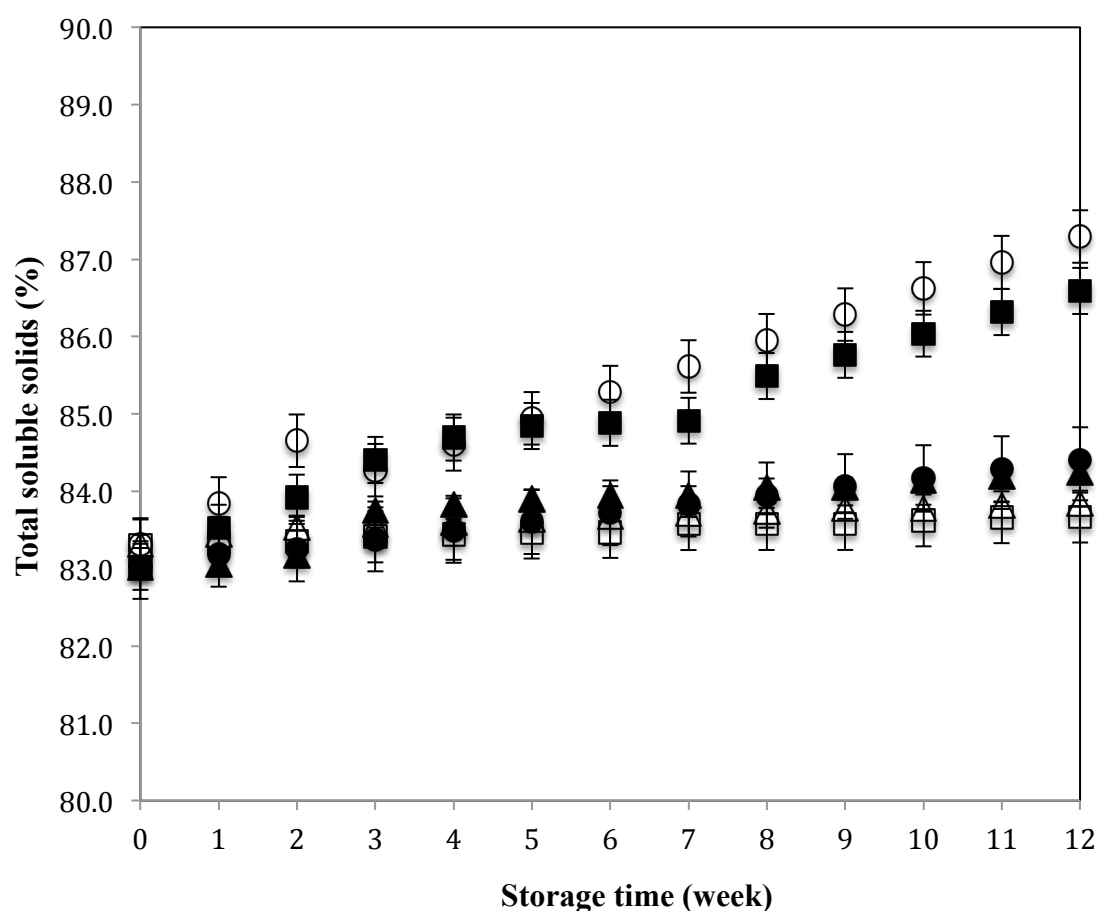
O Gelatine=3%, □ Gelatine=4.5%, Δ Gelatine=6%

**Table 3.4** Predictive equations obtained for total soluble solids (TSS) with respect to time (t) for the formulations with glucose syrup:sucrose ratio of 1.1 for storage at 15-22°C and 30-40% RH

Starch (%)	Gelatine (%)	Predictive equation	R <sup>2</sup>
0	3	$TSS(\%) = 0.0499 (t) + 83.743$	0.994
0	4.5	$TSS(\%) = 0.0450 (t) + 83.735$	0.999
0	6	$TSS(\%) = 0.0339 (t) + 83.932$	0.994
1	3	$TSS(\%) = 0.0330 (t) + 83.703$	0.991
1	4.5	$TSS(\%) = 0.0364 (t) + 83.891$	0.993
1	6	$TSS(\%) = 0.0324 (t) + 83.953$	0.982
1.5	3	$TSS(\%) = 0.0509 (t) + 83.911$	0.993
1.5	4.5	$TSS(\%) = 0.0478 (t) + 83.519$	0.999
1.5	6	$TSS(\%) = 0.0483 (t) + 83.284$	0.989

Temperature experiments on total soluble solids were performed at 10°C, 20°C and 30°C for 12 weeks. It was seen that total soluble solids parameter increased in a linear manner (Figure 3.11). Relative rate of total soluble solids was higher at glucose syrup:sucrose ratio 1.1 when compared with the rate at glucose syrup:sucrose ratio of 1.5 for 30°C storage. When storage results were analysed at 10°C, it was seen that relative rates were smaller.

According to the evaluation of these relative rate values in temperature storage studies, a specific trend could not be found. This was rather expected since total soluble solids results were obtained via refractometer readings of sucrose weight percent as discussed previously. Hence, it has been thought that total soluble solids is not a very sensitive parameter to be involved at temperature storage studies of jelly gums containing high amount of corn syrups although it is very easy and fast method.



**Figure 3.11** Change in total soluble solids (%) with storage time for jelly gums having Starch= 1.5% and Gelatine= 6% at different temperatures and glucose syrup:sucrose ratios

□ G:S=1.1 at 10°C, Δ G:S=1.1 at 20°C, O G:S=1.1 at 30°C, ■ G:S=1.5 at 10°C, ▲ G:S=1.5 at 20°C, ● G:S=1.5 at 30°C

### 3.5 Glass Transition Temperature of Jelly Gums

Stability of food products is affected by reactions, namely chemical, physical and enzymatic reactions and it depends on the physical state of the system. In addition, stability is related to both thermodynamic and kinetic aspects and can be described by water activity and glass transition temperature ( $T_g$ ) (Eskin and Robinson, 2000).

T<sub>g</sub> is defined as the temperature at which liquid to glass transformation or glass to liquid transformation takes place and it was recognized that this transition generally occurs over a temperature range (Roos, 1995; Ergun et al., 2010).

In the study, glass transition temperature of the jelly gum formulations were measured twice, namely freshly and at the end of week 53<sup>th</sup>. In the experiments, it was seen that measured T<sub>g</sub> values given in Table 3.5 were much lower than the T<sub>g</sub> values of the main ingredients used in the jelly gums given in Table 3.6. This can be due to the fact that inversion of sucrose into glucose and fructose often occurs during cooking and acid addition in the production of jelly gums. The mixture containing glucose and fructose had a much lower T<sub>g</sub> when compared to the glass transition temperature value of sucrose given in Table 3.6. Hence, inversion of confectionery syrup during cooking and acid addition could lead to a decrease in T<sub>g</sub> (Bhandari and Hartel, 2005; Ergun et al., 2010).

It was seen that samples with glucose syrup:sucrose ratio of 1.1 had higher and positive T<sub>g</sub> values, whereas jelly gums having glucose syrup:sucrose ratio of 1.5 had lower and negative T<sub>g</sub> values both in fresh samples and aged samples (Table 3.5). As discussed before, high glucose syrup amount had the humectancy effect for the formulations with glucose syrup:sucrose ratio of 1.5. A humectant is known to ensure water retention and help to keep a confection moist. Thus, high glucose syrup in the formulations might have increased plasticizing effect of water and lowered T<sub>g</sub> values.

**Table 3.5** Glass transition temperatures (°C) of jelly gums stored at laboratory conditions at 15-22°C and 30-40% RH.

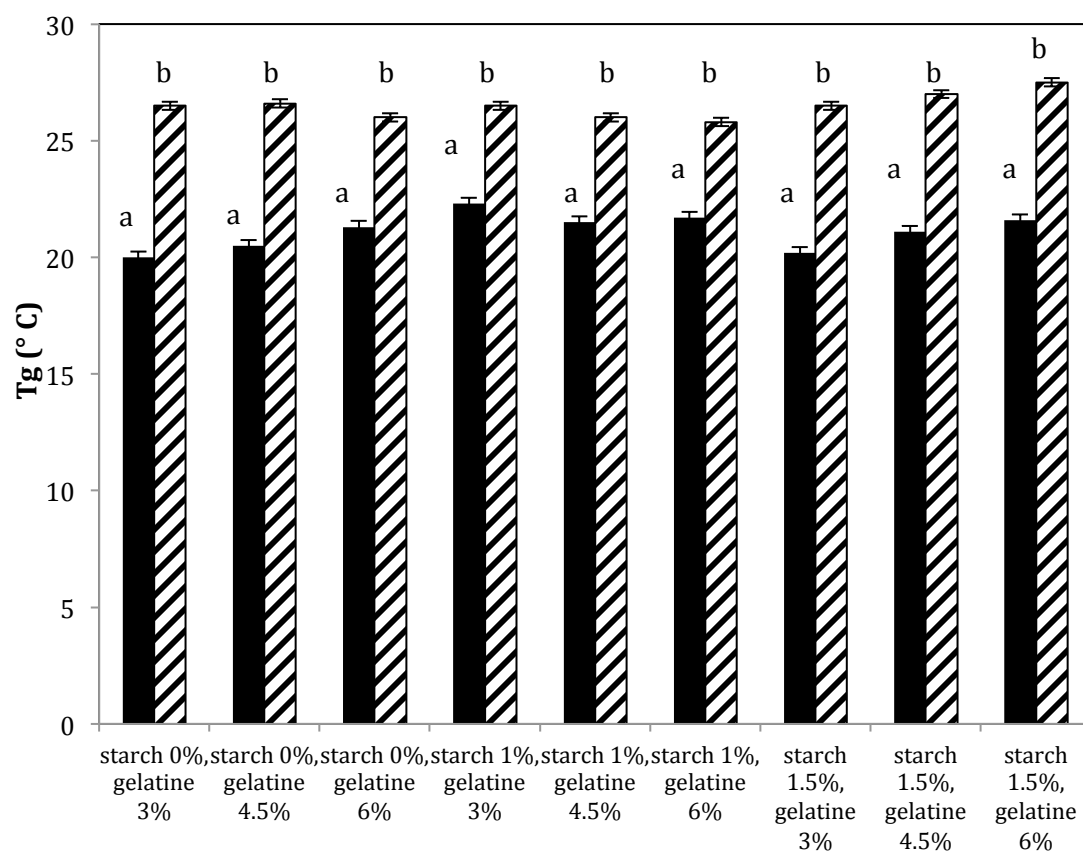
<b>G:S ratio</b>	<b>Formulation</b>		<b>Tg (°C)</b>	
	<b>Starch (%)</b>	<b>Gelatine (%)</b>	<b>Fresh (t= 0 weeks)</b>	<b>Aged (t= 53 weeks)</b>
1.1	0	3	20.0 ± 1.40	26.5 ± 1.30
1.1	0	4.5	20.5 ± 1.20	26.6 ± 1.20
1.1	0	6	21.3 ± 1.30	26.0 ± 1.20
1.1	1	3	22.3 ± 0.50	26.5 ± 1.00
1.1	1	4.5	21.5 ± 0.40	26.0 ± 0.70
1.1	1	6	21.7 ± 0.70	25.8 ± 0.60
1.1	1.5	3	20.2 ± 2.00	26.5 ± 1.70
1.1	1.5	4.5	21.1 ± 1.60	27.0 ± 1.50
1.1	1.5	6	21.6 ± 1.90	27.5 ± 1.40
1.5	0	3	-32.0 ± 1.00	-20.5 ± 0.90
1.5	0	4.5	-30.0 ± 1.10	-25.4 ± 1.20
1.5	0	6	-29.5 ± 1.00	-23.3 ± 1.10
1.5	1	3	-30.1 ± 0.50	-29.3 ± 0.60
1.5	1	4.5	-29.3 ± 0.60	-28.5 ± 0.70
1.5	1	6	-27.4 ± 0.60	-24.3 ± 0.70
1.5	1.5	3	-28.3 ± 0.40	-25.1 ± 0.60
1.5	1.5	4.5	-25.5 ± 0.50	-22.1 ± 0.50
1.5	1.5	6	-23.1 ± 0.60	-20.1 ± 0.40

**Table 3.6** Tg values of some confectionery components (Orford et al., 1989; Forssell et al., 1997; Mousia et al., 2000; Parker and Ring, 2001; Ergun et al., 2010)

Component	Tg (° C)
Sucrose	67
Glucose	31
Fructose	5
Citric acid	6
Starch	226.9
Water	-139.2
Glycerol	-83.2
Sorbitol	-9

In the light of the finding of this study, glass transition temperature results were analysed in two groups with respect to glucose to sucrose ratio. It was observed that glass transition temperatures of samples having glucose syrup:sucrose ratio of 1.1 increased significantly after storage of 53 weeks (Figure 3.12 and Table B.1 given in Appendix). This was an expected result since the moisture content decreased significantly after 53 weeks of storage. In other words, the decrease in concentration of plasticizer in the samples increased Tg value. This result was in accordance with the study of Nowakowski (2000), which showed the significant effect of increasing water content on decreasing Tg of a confection. Similarly, Lim et al. (2006) studied Tg of marshmallows during storage. It was shown that Tg was below -40 °C when the marshmallow samples were fresh with higher moisture content and increased to just above 0°C after 20 weeks when the moisture content decreased to 7.9 %.

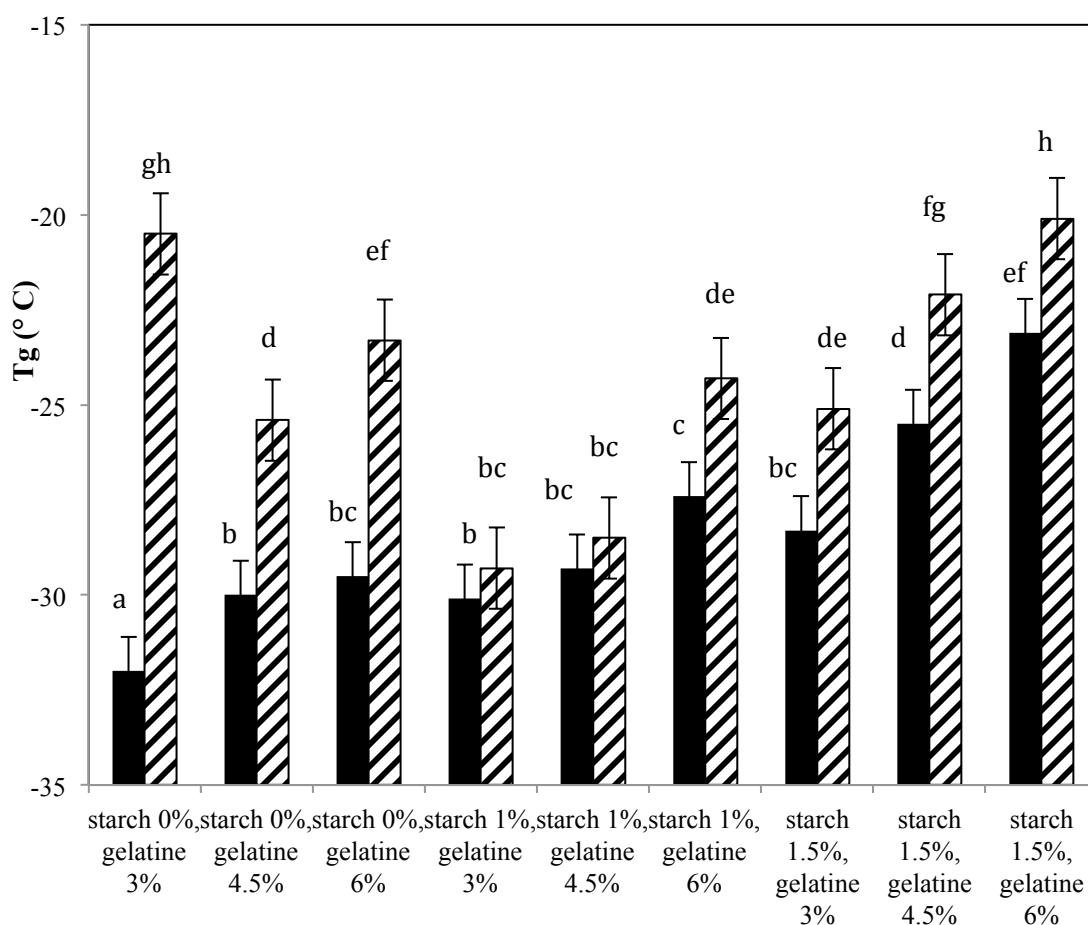
When glass transition temperatures of the samples having glucose syrup:sucrose ratio of 1.5 formulations were analysed, it was seen that Tg values increased in a significant manner after storage of 53 weeks (Figure 3.13 and Table B.2). This observation was expected as the moisture content decreased significantly after 53 weeks due to the plasticizing effect of water mentioned earlier. This result was in accordance with the observation at glucose syrup:sucrose ratio of 1.1.



**Figure 3.12** Change in glass transition temperature during storage of 53 weeks for the jelly gums with glucose syrup:sucrose ratio of 1.1

■  $t=0$  weeks, ▨  $t=53$  weeks





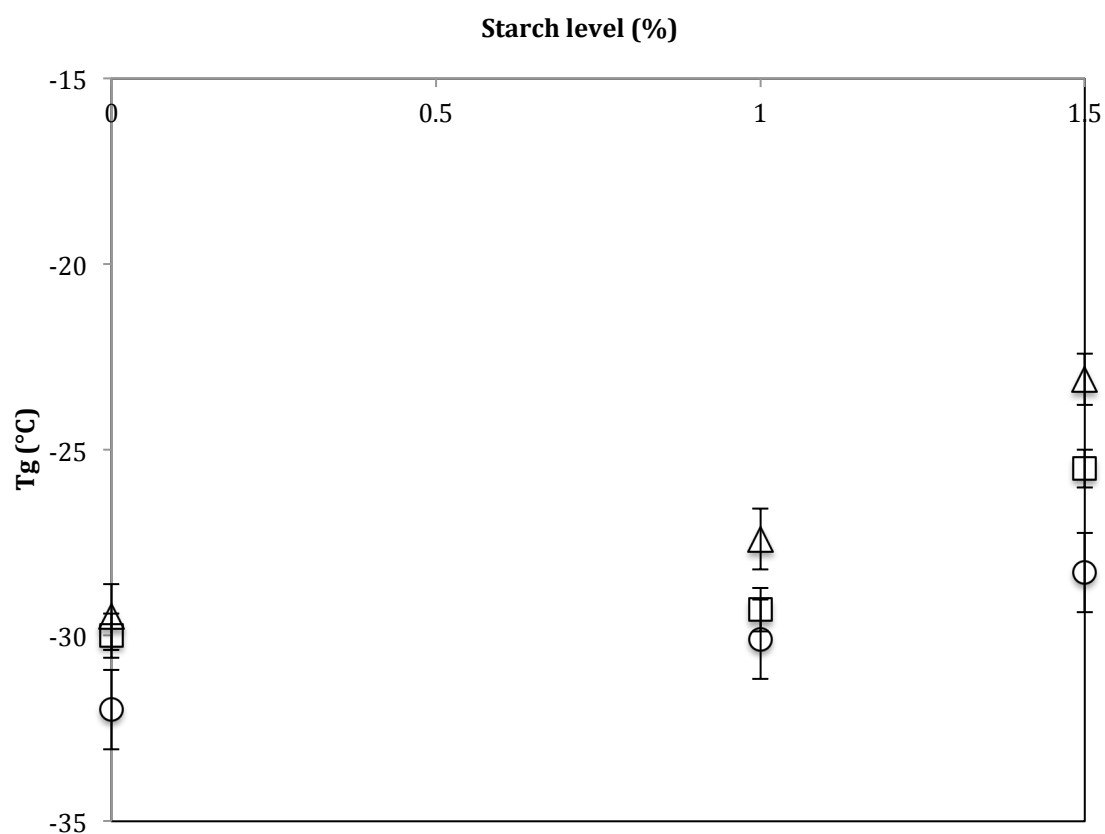
**Figure 3.13** Change in glass transition temperature during storage of 53 weeks for the jelly gums with glucose syrup:sucrose ratio of 1.5

■ t= 0 weeks, ▨ t= 53 weeks

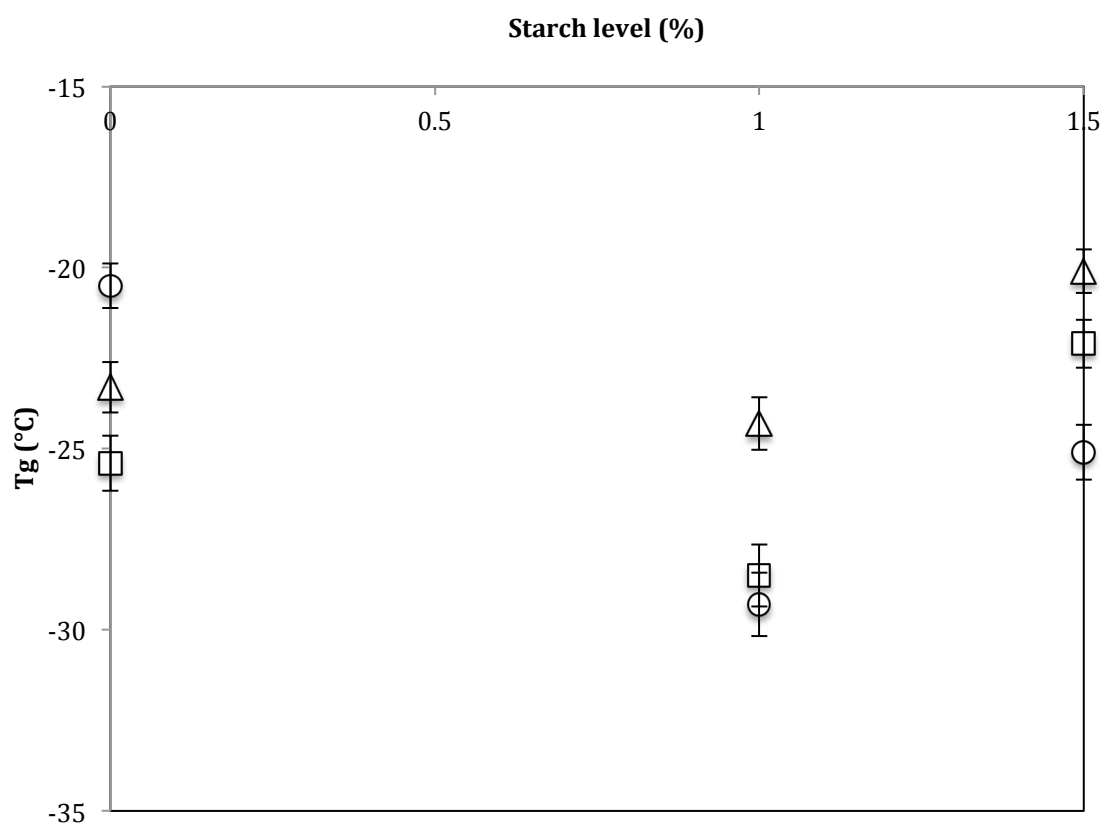
Unlike jelly gums with glucose to sucrose ratio of 1.1, jelly gums having glucose to sucrose ratio of 1.5 had significant differences in Tg values with respect to different starch and gelatine concentrations (Figure 3.13). Hence, starch and gelatine effects on Tg were studied for the samples having glucose to sucrose ratio 1.5 during aging of 53 weeks (Figure 3.14- 3.17). Labuza (1998) summarized some of the ingredient effects as following; sugars lower Tg curve, starches increase Tg lines and proteins have lower Tg values as compared to starches. These ingredient effects were observed when the samples were fresh. At constant starch levels Tg increased as

gelatine concentration increased, and similarly at constant gelatine levels  $T_g$  increased as starch level increased (Figure 3.14 and Figure 3.16). This might be explained with  $T_g$  being an increasing function of the molecular weight of a material as gelatine and starch had higher molecular weight when compared to sugars such as glucose, fructose and sucrose (Roos, 1995; Meste et al., 2006). Supporting to this, Roos (1995) found a glass transition temperature of  $-54^{\circ}\text{C}$  for a 80% sugar solution and Kasapis and Sablani (2005) found higher glass transition temperature between  $-44^{\circ}\text{C}$  and  $-45^{\circ}\text{C}$  for 5% gelatine in the presence of 50% glucose syrup and 25% sucrose. Similarly, Liu et al. (2007) reported starch syrup with higher  $T_g$  in a model system containing sucrose and starch syrup solids.

As can be seen from Figures 3.14-3.17; when  $T_g$  values of aged samples were considered, the trends with respect to starch and gelatine levels were different than those of fresh samples, especially at the lowest levels (gelatine = 3% and starch = 0%). This might be due to the fact that in a complex food system, like jelly gums, decrease of moisture content can affect glass transition temperature in different manners, based on structure, microstructure, composition and geometric factors (Peleg, 1998). Hence, an increase in protein or starch mobility caused by water might not have the same effect on different products or processes (Li, 2010). Siegwins (2010) studied the effect of soy protein isolate in starch confectionery, and observed no significant differences in glass transition temperatures between the samples containing no soy protein isolate ( $T_g = 18^{\circ}\text{C}$ ) and samples containing 50% soy protein isolate ( $T_g = 17^{\circ}\text{C}$ ) after 20 days storage.

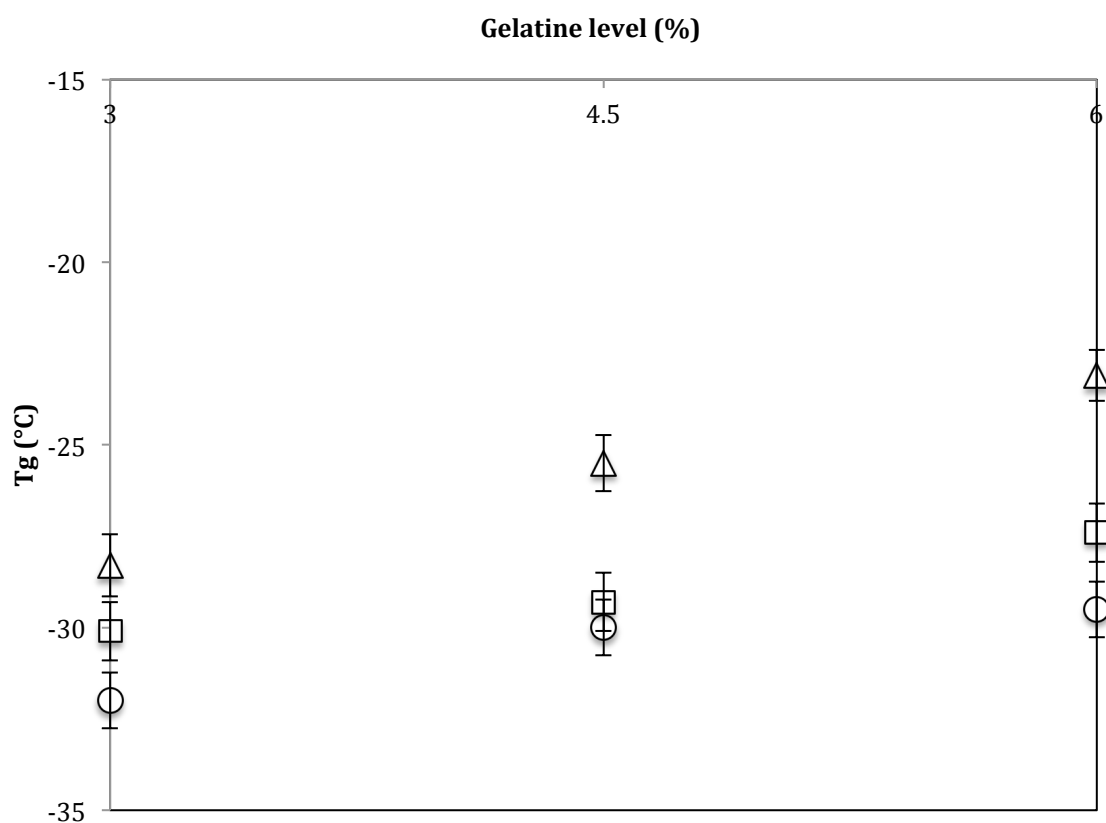


**Figure 3.14** Glass transition temperature of fresh jelly gums having glucose syrup:sucrose ratio of 1.5 at different gelatine levels  
O Gelatine=3%, □ Gelatine=4.5%, Δ Gelatine=6%



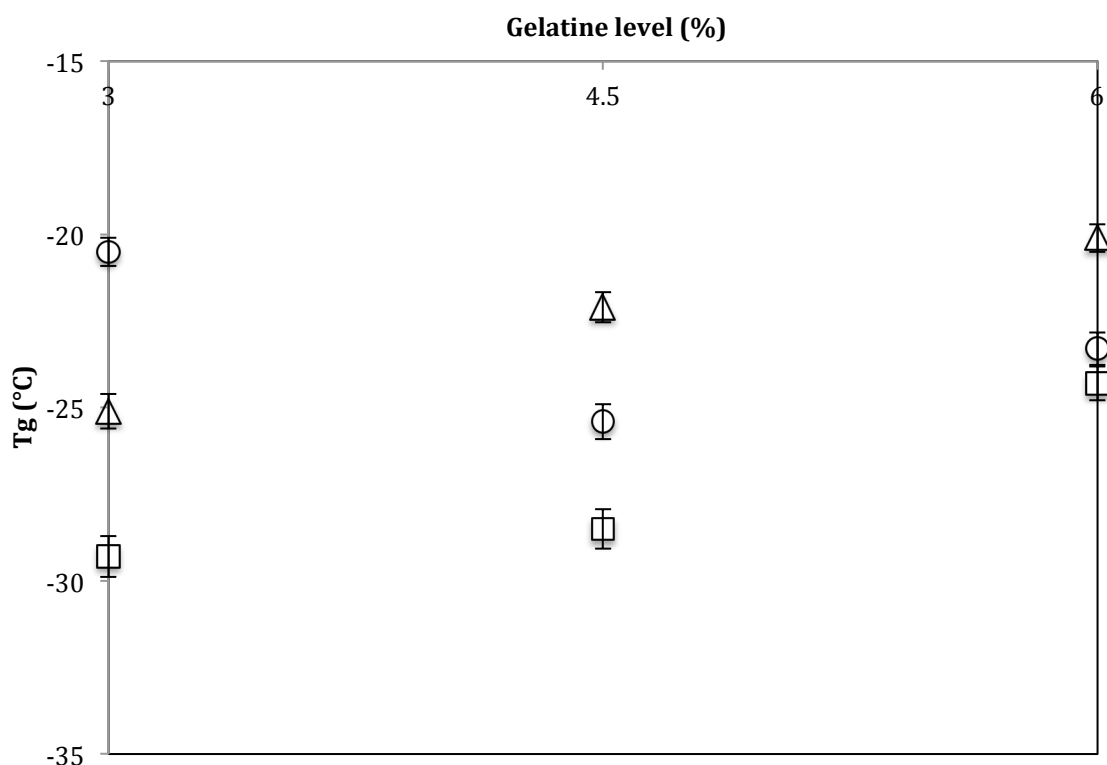
**Figure 3.15** Glass transition temperature of aged jelly gums having glucose syrup:sucrose ratio of 1.5 at different gelatine levels after storage of 53 weeks

O Gelatine=3%, □ Gelatine=4.5%, Δ Gelatine=6%



**Figure 3.16** Glass transition temperature of fresh jelly gums having glucose syrup:sucrose ratio of 1.5 at different starch levels

O No Starch, □ Starch=1.0%, Δ Starch=1.5%



**Figure 3.17** Glass transition temperature of aged jelly gums having glucose syrup:sucrose ratio of 1.5 at different starch levels after storage of 53 weeks

O No Starch, □ Starch=1.0%, Δ Starch=1.5%

Glass transition studies were conducted in sugar-based candies (Gabarra and Hartel 1998; Nowakowski and Hartel 2002; Labuza and Labuza, 2004; Lim et al., 2006) like hard boiled candy, marshmallow, cotton candy. In all of these studies, it was seen that  $T_g$  increased as moisture content decreased. However, the slopes and trends were all different because of the differences in sugar composition (Hartel et al., 2011), which was similar to our study as we studied two different sugar compositions.

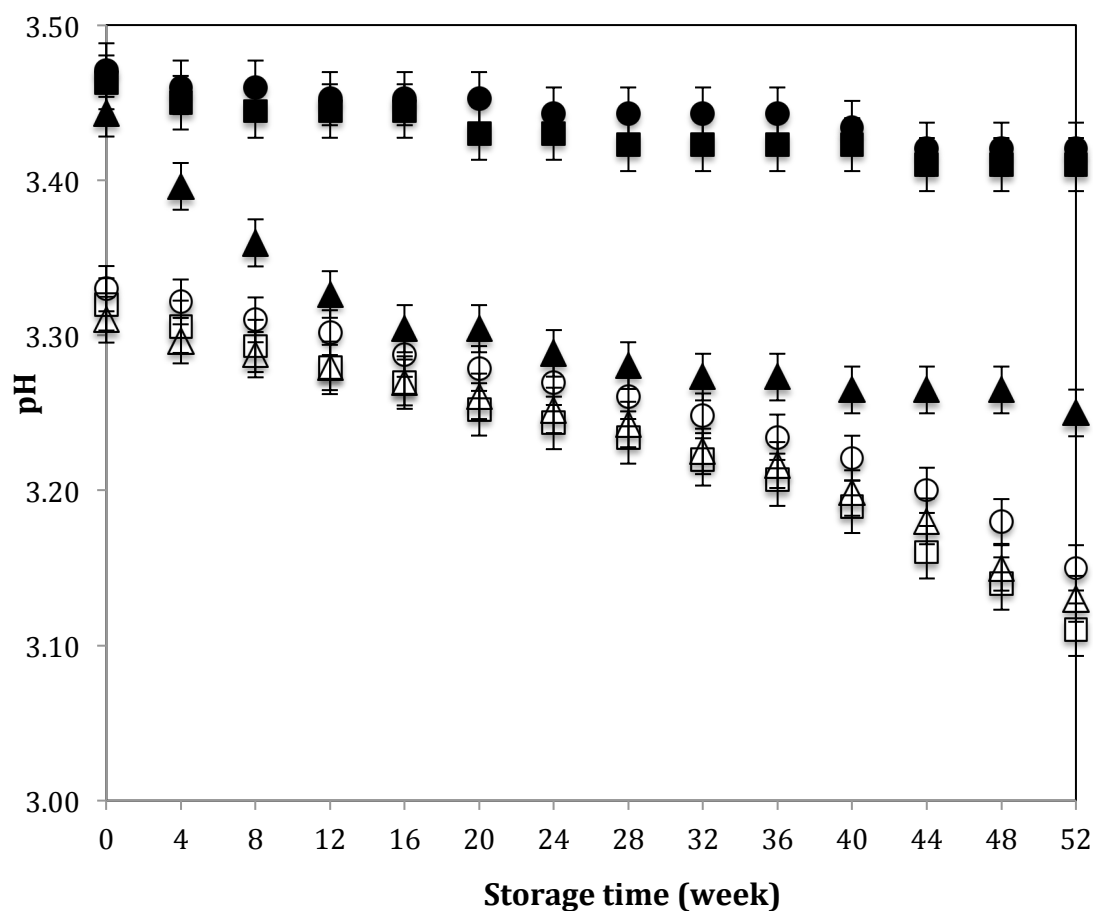
As a conclusion, measurement of  $T_g$  is useful for commercial products as composition of sugars in the final product may be quite different from the ingredients used in the product, especially for jelly gums, whose process and individual ingredients are favorable for inversion, making the food system even more complex and unique.

### 3.6 pH of Jelly Gums

In food products like jams and jellies, not only low water activity due to high sugar concentration, but also low pH of such products contribute to prolonged shelf life in terms of microbial stability (Minifie, 1999; Nordic Sugar, 2016). In jelly gums, pH is also important in terms of gelling as mentioned earlier. Thus, pH is required to be measured during manufacturing stages in confectionery plants.

It was seen that glucose:sucrose ratio was an important parameter affecting the pH of jelly gums ( $p \leq 0.001$ ) (Table 3.1 and Table A.4). As the ratio of glucose syrup to sucrose ratio increased, pH value increased (Figure 3.18). This might be due to the differences in the end products of sucrose inversion that could occur at different glucose syrup and sucrose concentrations in jellies during production of jellies where acid and heat treatment were involved at the same time. There are fundamental differences between glucose syrups and sucrose. Glucose syrups are made up of reducing sugars. Sucrose, on the other hand, is a disaccharide composed of glucose and fructose. In addition, some confectionery companies depend on the acidity of the glucose syrup to reduce the risk of inversion and crystallization. When the pH is very critical, pH of the glucose syrup can be adjusted using citric acid. According to Hull (2010), sugar spectrum of a glucose syrup does not change during processing or during storage, however, sucrose can invert in the presence of acid and heat, meaning that sucrose changes into a mixture of dextrose and fructose. Hence, there might have been less inversion during cooking and conditioning of jellies with glucose syrup to sucrose ratio of 1.5 resulting to higher pH as compared to the jellies with glucose syrup to sucrose ratio of 1.1, despite the fact that the same amount of citric acid solution was added to all of the formulations. In addition, interaction of sugar and starch might be responsible regarding to the significant effect of starch on pH. According to Chang et al. (2004) certain sugars can affect protein-polysaccharide interactions. In addition, Burey et al. (2009) mentioned that the level of this interaction depends on many factors including polymer properties such as charge, size and morphology in protein-polysaccharide systems like confectionery gels.

Morphology should be noted because the glucose syrup used in the experiments was corn origin and was produced via treatment with enzyme. In a similar manner, the corn starch used in the study was acid thinned modified corn starch, which might have affected the acidity level of jellies.



**Figure 3.18** Change in pH with storage time at 15-22°C with different glucose syrup:sucrose ratios and gelatine amounts at starch level of 1%

O G:S=1.1, Gelatine=3%; □ G:S=1.1, Gelatine=4.5%; △ G:S=1.1, Gelatine=6%; ● G:S=1.5, Gelatine=3%; ■ G:S=1.5, Gelatine=4.5%; ▲ G:S=1.5, Gelatine=6%

Like moisture content and total soluble solids regression findings, there might have been some possible ingredient interactions between gelatine and starch. It was seen



that these ingredients did not affect pH of jelly gums when used alone, but, they influenced pH together with glucose syrup:sucrose ratio ( $p \leq 0.05$ ) (Table 3.1).

Temperature storage studies of pH were done at 10°C, 20°C and 30°C for 12 weeks. pH values decreased linearly for almost all of the recipes (Table 3.7). Relative rates of pH change at 10°C and 30°C were compared to 20°C. As can be seen from Table 3.7, recipes with high glucose syrup to sucrose ratio were more stable having lower relative rates than the ones with low glucose syrup to sucrose ratio in terms of pH. This can be explained by sucrose inversion during storage in the presence of acid. As the glucose syrup in the recipe increased (or sucrose in the recipe decreased) inversion decreased and this might have protected the formulation from acidity denaturation during storage (Hull, 2010). It was observed that the most stable formulations having lowest rates were the jellies with 1% starch + 6% gelatine, 1.5% starch + 3% gelatine and 1.5% starch + 6% gelatine having glucose syrup to sucrose ratio of 1.5 (Table 3.7).

Acids are added to jellies primarily to give a tangy and sour taste to the consumer (Burey et al. 2009), and pH is a good measure to the confectionery manufacturer. Hence, it is beneficial to follow the changes in pH at different storage temperatures since it was learnt in a private conversation with Quality Assurance Manager of Kervan Gıda (Istanbul, Turkey) that consumers mostly complain about the decrease in sourness of their gummies.

**Table 3.7** Predictive equations obtained for pH with respect to time (t) at different temperatures for selected jelly gums.

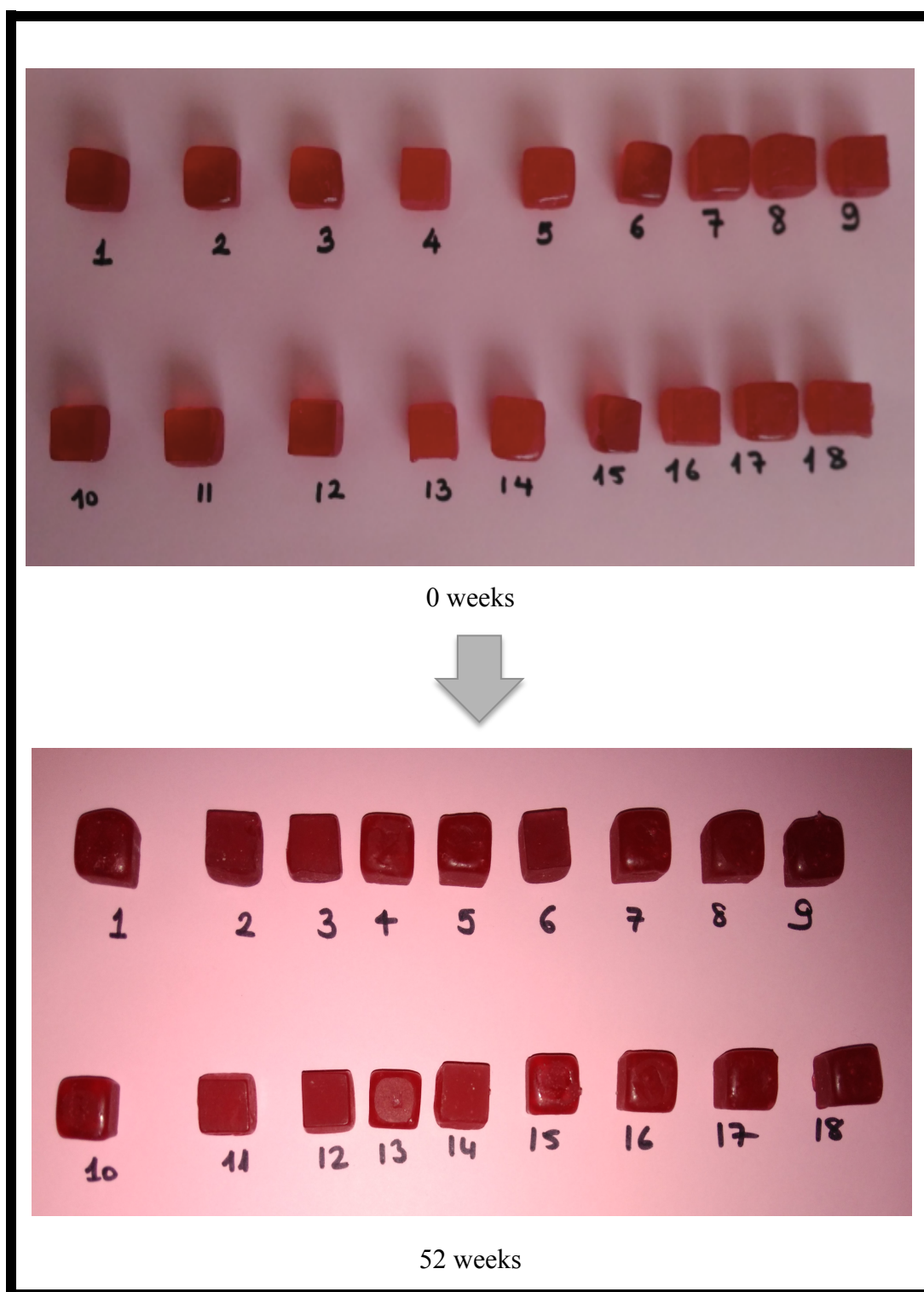
<b>G:S ratio</b>	<b>Starch (%)</b>	<b>Gelatine (%)</b>	<b>Temperature (°C)</b>	<b>Predictive equation</b>	<b>R<sup>2</sup></b>
1.1	0	3	10	$pH = -0.0346(t)+3.3267$	1.000
1.1	0	3	30	$pH = -0.0375(t)+3.3350$	1.000
1.1	0	6	10	$pH = -0.0342(t)+3.3140$	1.000
1.1	0	6	30	$pH = -0.0271(t)+3.3213$	0.999
1.5	0	3	10	$pH = -0.0092(t)+3.4700$	0.915
1.5	0	3	30	$pH = -0.0161(t)+3.4797$	0.995
1.5	0	6	10	$pH = -0.0076(t)+3.4468$	0.957
1.5	0	6	30	$pH = -0.0063(t)+3.4577$	0.991
1.1	1	3	10	$pH = -0.0343(t)+3.3240$	1.000
1.1	1	3	30	$pH = -0.0239(t)+3.3360$	0.997
1.1	1	6	10	$pH = -0.0344(t)+3.3067$	1.000
1.1	1	6	30	$pH = -0.0254(t)+3.3176$	0.996
1.5	1	3	10	$pH = -0.0078(t)+3.4586$	0.977
1.5	1	3	30	$pH = -0.0080(t)+3.4684$	0.996
1.1	1.5	3	10	$pH = -0.0343(t)+3.3240$	1.000
1.1	1.5	3	30	$pH = -0.0379(t)+3.3223$	0.999
1.1	1.5	6	10	$pH = -0.0347(t)+3.3639$	1.000
1.1	1.5	6	30	$pH = -0.0346(t)+3.3724$	0.999
1.5	1.5	3	10	$pH = -0.0074(t)+3.4522$	0.989
1.5	1.5	3	30	$pH = -0.0165(t)+3.3684$	0.793
1.5	1.5	6	10	$pH = -0.0070(t)+3.3991$	0.971
1.5	1.5	6	30	$pH = -0.0205(t)+3.3215$	0.871

### 3.7 Colour of Jelly Gums

Visual appearance of a product is one of the first sensory parameters that consumers notice. Due to the fact that colour of a food predetermines consumer's expectation of taste and quality, it is the main property influencing consumer acceptance (Subramaniam, 2011). Hence, food industry aims to optimize the colour of products in a way that it attracts consumers until the best before date. For confectionery products, especially jelly gums; this involves adding of food colours to the formulations.

As there is a recent trend towards the usage of natural ingredients in confectionery products black carrot anthocyanin, being a natural colour, was used in this study. Pigments of anthocyanin are antocyanidins glycosides providing purple and red shades in foods (Matsufuji et al., 2007). Colour shade of antocyanins depends on the pH of the product and source of anthocyanin used in the formulation (Subramaniam, 2011). In the experiments, anthocyanin of black carrot gave red shade to all of the jelly gum formulations, which can be seen in Figure 3.19. This was expected, since anthocyanin gave red shade to the product typically at a pH of 3 (Subramaniam, 2011), which was the pH value of the jelly gums prepared in the experiments. In addition, formulations were also prepared without the addition of colour for use as control samples.

Colour results given in this part of the study are based on the changes of jelly gums over a period of storage up to 52 weeks at 15-22°C and 30-40% RH and up to 12 weeks at 10°C, 20°C and 30°C. Results were given as change in hue ( $\Delta H$ ), which is a useful parameter for colour analysis of the products since it shows the magnitude of change in colour over time by taking into account the total colour difference ( $\Delta E$ ), change in lightness ( $\Delta L$ ) and change in chroma ( $\Delta C$ ).

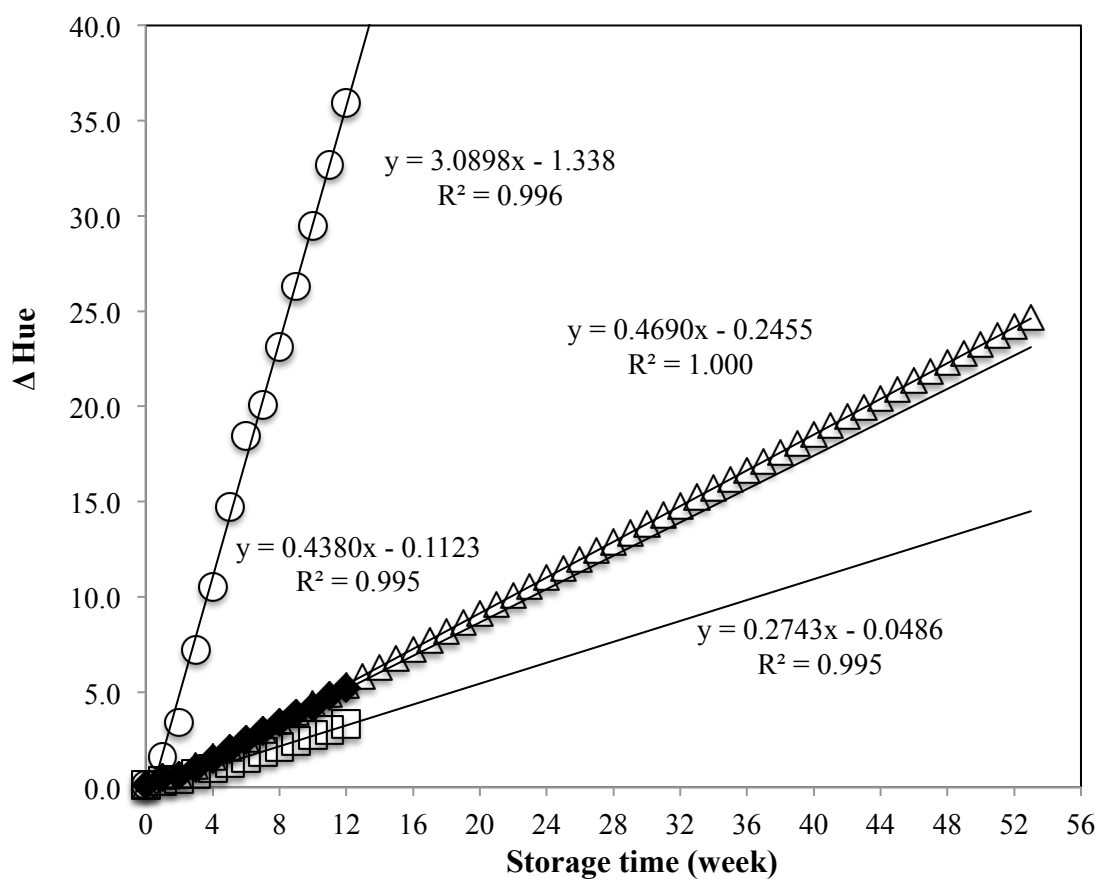


**Figure 3.19** Appearance of jelly gums after storage at 15-22°C and 30-40% RH (Formulation numbers are described in Table 2.2)

Figure 3.19 shows the appearance of jelly gums over a period of storage up to 52 weeks at laboratory conditions, i.e. 15-22°C and 30-40% RH. As can be seen from the mentioned figure, the redness coming from anthocyanin had a very slight fading when the samples were observed visually. Subramaniam (2011) studied natural colour stability in model food systems involving hard boiled candy, gelatine gellies and juice based drinks. A similar result was observed for gelatine jellies containing anthocyanin in visual observation after storage of 20 weeks at 20°C.

Colour changes increased linearly during storage for all formulations with high coefficient of determination values between 0.989 to 1.000 (Table 3.8). It was seen that relative rate of colour change of jelly gums increased with increase in storage temperature (Figure 3.20). This might be due to the effect of gelatine, which is known to have colour change when held at high temperatures, by developing browning at high temperature (Farahnaky et al., 2003) and increasing  $\Delta H$  values in a fast manner. In addition, in accordance with our results, Subramaniam (2011) mentioned that the temperature increase gave significant rates of acceleration of colour change for anthocyanin containing boiled confectionery.

Figure 3.21 shows the effect of glucose syrup:sucrose ratio on colour change of jelly gums for different gelatine levels at starch level of 1.5. It was observed that when the jellies were fresh,  $\Delta H$  values were similar at all gelatine concentrations and glucose syrup:sucrose ratios. After 12 weeks storage at 30°C, it was seen that change in colour increased significantly at both glucose syrup:sucrose ratios and at all gelatine levels. In addition,  $\Delta H$  values of jelly gums were higher at glucose syrup:sucrose ratio of 1.1 after 12 weeks storage at mentioned temperature. This meant samples with glucose syrup:sucrose ratio of 1.5 were more stable in terms of colour. Hull (2010) stated that use of glucose syrups improve the appearance of confectionery products because they overcome sucrose crystallization causing opaque and dull appearance.



**Figure 3.20** Variation of hue value of jelly gums with glucose syrup:sucrose ratio of 1.1, starch=1.5% and gelatine=6% during storage at laboratory conditions for 52 weeks, at 10°C for 12 weeks, at 20°C for 12 weeks and at 30°C for 12 weeks

Δ Laboratory conditions (15-22°C), □ 10°C, ◆ 20°C, O 30°C, — Linear line of best fit

**Table 3.8** Predictive equations obtained for change in hue ( $\Delta H$ ) with respect to time (t) for all jelly gum formulations at different storage conditions

G:S ratio	Starch (%)	Gelatine (%)	Storage Temperature(°C)	Predictive equation	R <sup>2</sup>
1.1	0	3.0	15-22	$\Delta H = 0.1593(t)-0.0834$	1.000
			10	$\Delta H = 0.1007(t)-0.0187$	0.995
			20	$\Delta H = 0.1527(t)-0.0319$	0.995
			30	$\Delta H = 1.1784(t)-0.7341$	0.996
1.1	0	4.5	15-22	$\Delta H = 0.1770(t)-0.0926$	1.000
			10	$\Delta H = 0.1048(t)-0.0187$	0.995
			20	$\Delta H = 0.1671(t)-0.0346$	0.995
			30	$\Delta H = 1.3492(t)-0.9233$	0.995
1.1	0	6.0	15-22	$\Delta H = 0.2212(t)-0.1158$	0.999
			10	$\Delta H = 0.1294(t)-0.0229$	0.995
			20	$\Delta H = 0.2117(t)-0.0500$	0.995
			30	$\Delta H = 1.6752(t)-1.1522$	0.994
1.1	1.0	3.0	15-22	$\Delta H = 0.3009(t)-0.1575$	1.000
			10	$\Delta H = 0.1782(t)-0.0318$	0.994
			20	$\Delta H = 0.2850(t)-0.0752$	0.992
			30	$\Delta H = 2.2023(t)-1.3236$	0.996
1.1	1.0	4.5	15-22	$\Delta H = 0.3097(t)-0.1621$	0.999
			10	$\Delta H = 0.1835(t)-0.0328$	0.995
			20	$\Delta H = 0.2912(t)-0.0703$	0.995
			30	$\Delta H = 2.2619(t)-1.3946$	0.996
1.1	1.0	6.0	15-22	$\Delta H = 0.3362(t)-0.1760$	1.000
			10	$\Delta H = 0.1992(t)-0.0356$	0.994
			20	$\Delta H = 0.3147(t)-0.0693$	0.995
			30	$\Delta H = 2.4848(t)-1.6168$	0.996
1.1	1.5	3.0	15-22	$\Delta H = 0.4247(t)-0.2224$	0.999
			10	$\Delta H = 0.2484(t)-0.0440$	0.995
			20	$\Delta H = 0.3934(t)-0.1476$	0.989
			30	$\Delta H = 3.0865(t)-1.8831$	0.996

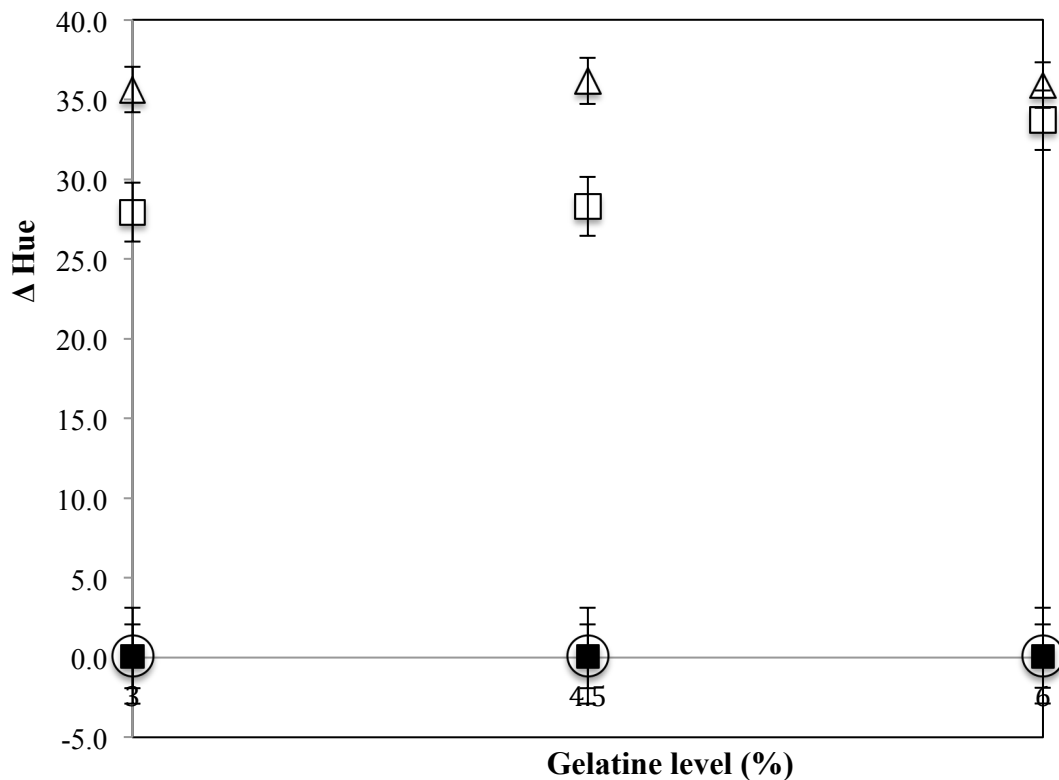
**Table 3.8** (Continued)

<b>G:S ratio</b>	<b>Starch (%)</b>	<b>Gelatine (%)</b>	<b>Storage Temperature (°C)</b>	<b>Predictive equation</b>	<b>R<sup>2</sup></b>
1.1	1.5	4.5	15-22	$\Delta H = 0.4424(t) - 0.2316$	0.999
			10	$\Delta H = 0.2588(t) - 0.0461$	0.994
			20	$\Delta H = 0.4190(t) - 0.1345$	0.993
			30	$\Delta H = 3.1371(t) - 1.7909$	0.996
1.1	1.5	6.0	15-22	$\Delta H = 0.4690(t) - 0.2455$	1.000
			10	$\Delta H = 0.2743(t) - 0.0486$	0.995
			20	$\Delta H = 0.4380(t) - 0.1123$	0.995
			30	$\Delta H = 3.0898(t) - 1.3380$	0.996
1.5	0	3.0	15-22	$\Delta H = 0.1168(t) - 0.0751$	0.999
			10	$\Delta H = 0.0788(t) - 0.0224$	0.994
			20	$\Delta H = 0.1065(t) - 0.0497$	0.991
			30	$\Delta H = 0.8445(t) - 0.5189$	0.996
1.5	0	4.5	15-22	$\Delta H = 0.1362(t) - 0.0876$	1.000
			10	$\Delta H = 0.0992(t) - 0.0302$	0.994
			20	$\Delta H = 0.1257(t) - 0.0520$	0.991
			30	$\Delta H = 1.0293(t) - 0.7526$	0.994
1.5	0	6.0	15-22	$\Delta H = 0.1752(t) - 0.1126$	0.999
			10	$\Delta H = 0.1275(t) - 0.0384$	0.994
			20	$\Delta H = 0.1637(t) - 0.0770$	0.992
			30	$\Delta H = 1.3165(t) - 0.9446$	0.995
1.5	1.0	3.0	15-22	$\Delta H = 0.2433(t) - 0.1564$	0.999
			10	$\Delta H = 0.1770(t) - 0.0522$	0.993
			20	$\Delta H = 0.2289(t) - 0.0980$	0.993
			30	$\Delta H = 1.6937(t) - 0.9038$	0.995
1.5	1.0	4.5	15-22	$\Delta H = 0.2530(t) - 0.1627$	1.000
			10	$\Delta H = 0.1842(t) - 0.0549$	0.994
			20	$\Delta H = 0.2374(t) - 0.0976$	0.993
			30	$\Delta H = 1.8005(t) - 1.0679$	0.994



**Table 3.8** (Continued)

<b>G:S ratio</b>	<b>Starch (%)</b>	<b>Gelatine (%)</b>	<b>Storage Temperature (°C)</b>	<b>Predictive equation</b>	<b>R<sup>2</sup></b>
1.5	1.0	6.0	15-22	$\Delta H = 0.2725(t) - 0.1752$	1.000
			10	$\Delta H = 0.1985(t) - 0.0604$	0.994
			20	$\Delta H = 0.2557(t) - 0.1051$	0.994
			30	$\Delta H = 2.0484(t) - 1.4429$	0.994
1.5	1.5	3.0	15-22	$\Delta H = 0.3406(t) - 0.2190$	0.999
			10	$\Delta H = 0.2387(t) - 0.0706$	0.994
			20	$\Delta H = 0.3219(t) - 0.1400$	0.993
			30	$\Delta H = 2.4275(t) - 1.4307$	0.995
1.5	1.5	4.5	15-22	$\Delta H = 0.3504(t) - 0.2253$	0.999
			10	$\Delta H = 0.2455(t) - 0.0732$	0.993
			20	$\Delta H = 0.3310(t) - 0.1436$	0.992
			30	$\Delta H = 2.4408(t) - 1.1667$	0.995
1.5	1.5	6.0	15-22	$\Delta H = 0.4430(t) - 0.2903$	0.999
			10	$\Delta H = 0.3120(t) - 0.1072$	0.995
			20	$\Delta H = 0.4212(t) - 0.1800$	0.995
			30	$\Delta H = 2.8811(t) - 0.8868$	0.993



**Figure 3.21** Effect of glucose syrup:sucrose ratio on colour change with gelatine concentration at starch level of 1.5%

○ Glucose Syrup:Sucrose=1.5 fresh, ■ Glucose Syrup:Sucrose=1.1 fresh, □ Glucose Syrup:Sucrose=1.5 after 12 weeks storage at 30°C, △ Glucose Syrup:Sucrose=1.1 after 12 weeks storage at 30°C

### 3.8 Texture of Jelly Gums

Texture encompasses all structural and rheological properties of a food product, which are perceptible by means of mechanical, tactile and when appropriate, visual and auditory receptors according to the definition of International Organization for Standardization (Borwankar, 1992). Similarly, Edwards (2000), mentioned texture as an indication of gel eating quality. Hence, texture is one of the important primary determinants of quality in confectionery products (DeMars and Ziegler, 2001). Texture study may be used as a quantitative supplement to sensory panels in texture evaluation. Texture analysis could be correlated with sensory analysis since final

judge of confectionery texture is the consumer. In the case of confectionery gels, hardness, chewiness and gumminess are examples of sensory descriptors (Szczeniak, 1986; Borwankar, 1992). Jowitt (1982) stated that jellies can be termed as chewy and this behaviour is a consequence of their ductile nature.

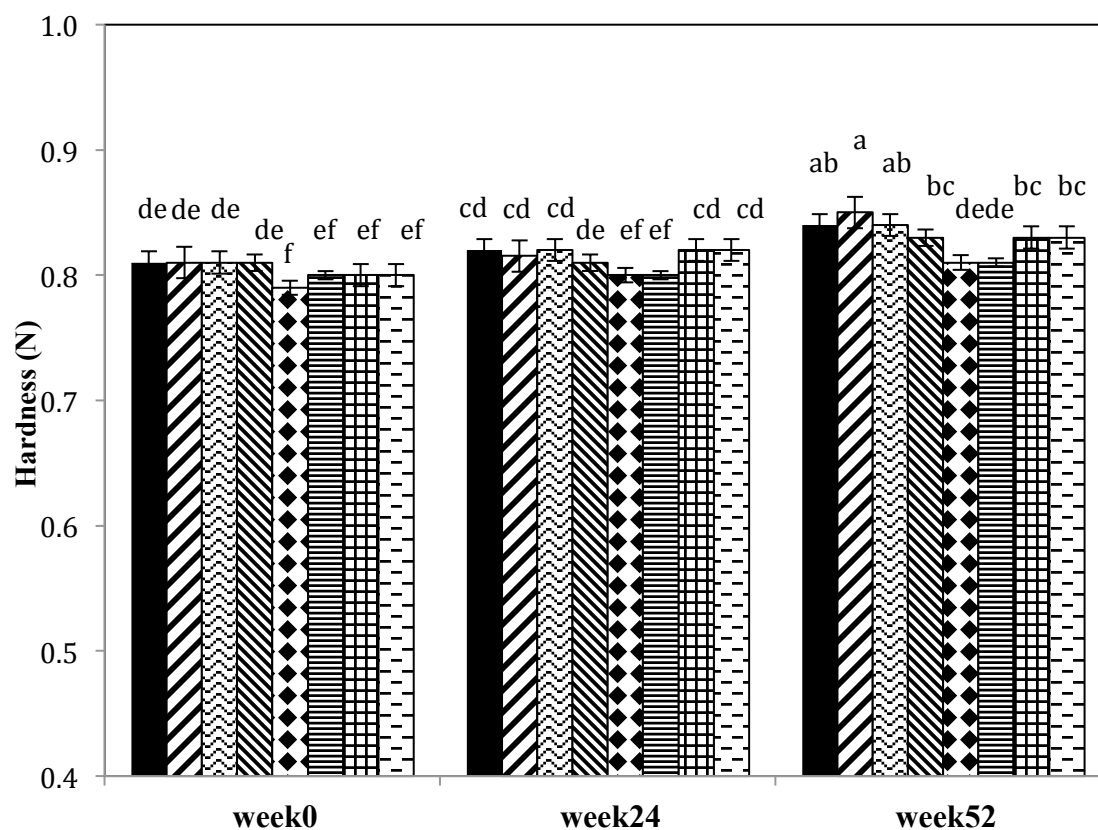
Hardness is resistance to deformation and expressed as the maximum force applied during the first compression (Siegwein, 2010). It is directly proportional to the maximum force needed for the jelly gums to be deformed in the first bite (Delgado and Banon, 2015). Multiple regression results between ingredient parameters and hardness of fresh jelly gums are shown in Table 3.1 and Table A.5. It was observed that hardness was affected by ingredients, namely glucose syrup to sucrose ratio ( $p \leq 0.001$ ), starch level ( $p \leq 0.01$ ) and gelatine level ( $p \leq 0.05$ ). The increase in glucose syrup to sucrose ratio decreased hardness. However, the increase in starch and gelatine levels increased hardness of jelly gums. From the point of gelatine, gelatine concentration might have resulted in higher gel strength since higher gelatine amounts gave more intermolecular contacts, and hence stronger protein interactions (Zayas, 1997). In the previous study of Lau et al. (2000), gel strength was found to be dependent on gelatine concentration in gellan and gelatine mixed gel. Similarly, it has been reported that gel force increased with increasing gelatine concentration in gelatine gels (Munoz et al., 1986). More recently, Porayanee et al. (2015) found that hardness significantly increased with increasing gelatine concentration by using gelatine levels of 7.75%, 8.00% and 8.25%, which was similar to the results in this study. Similar to the gelatine effect, hardness also increased with starch level. This might be due to the increase of interconnected network as starch amount increased (Prokopowich and Biliaderis, 1995). Glucose syrup to sucrose ratio can increase either by increasing the amount of glucose syrup or decreasing the amount of sucrose in the recipe of jelly gums. When the glucose syrup increased or sucrose decreased in the recipe, hardness of the jelly gums decreased. This result was in agreement with the previous study of Porayanee et al. (2015). They reported that addition of sucrose significantly increased the hardness compared to the samples having no sucrose. Sugars were known to stabilize protein by increasing the rigidity and by favoring protein-to-protein interaction in protein gels like jelly gums (Semenova et al., 2002).

Sucrose aids gelatine dissolution and combination of sucrose and glucose syrup makes a continuous phase in gelatine, strengthening the gel, hence increasing the hardness of the food system (Burey et al., 2009). Similarly, addition of sugar has been stated to increase the gel strength in gelatine gel, corn starch gel and gellan gum gel model systems (Kasapis et al., 2003; Holm et al., 2009; Sun et al., 2014), which is in accordance with the hardness results of this study. Furthermore, the decrease in hardness may have resulted from plasticizing effect of higher glucose syrup and higher moisture content in the jelly gums having glucose syrup to sucrose ratio of 1.5 when compared to the ones having glucose syrup to sucrose ratio of 1.1 (Figure 3.1). Martinez and Chiralt (1995) also showed that hardness of xixona turrón (a typical Spanish confectionery product) decreased as moisture content increased due to the plasticizing effect. Additionally, Vieira et al. (2008) noted that hardness increased as the solid content increased in gummy candies and found that hardness values were dependent on the total soluble solids content. It was observed that jelly gums with 1.1 glucose syrup to sucrose ratio had higher total soluble solids content and higher hardness values when compared with the samples with 1.5 glucose syrup to sucrose ratio (Figure 3.7). Likewise, it has been reported that total solids content were correlated well with hardness of gummy confections with glucose syrup to sucrose ratio of 1.1 (Delgado and Banon, 2015).

Hardness is the strength of gelling agent's (such as gelatine, starch) gel structure under compression (Porayanee et al., 2015). Hardness results of selected jelly gums held at laboratory conditions with respect to storage time are presented in Figure 3.22. According to this figure, as gelling agent amount increased in the recipe, hardness of jelly gums were found to be similar when fresh values (week 0) were analysed. This was in accordance with regression results given for starch-gelatine interaction term being insignificant (Table 3.1).

It was seen that hardness increased after one-year storage (Figure 3.22, Table C.1), which was an expected result due to the simultaneous actions of gelatine and starch system containing sugars, water and other minor ingredients resulting in firm and chewy structure of gelled confectionery (Warnecke, 1991; Burey et. al, 2009).

Supporting to this result, Marfil et al. (2012) reported that gelatine gives elasticity, while addition of starch increased gel strength and hardness of gelatine based gummies. In the study of Delgado and Banon (2015), it was found that hardness increased from 0.79 N (12<sup>th</sup> hour at conditioning) to 0.88 N (16<sup>th</sup> hour at conditioning) and then became constant until the end of conditioning step taking 24 hours in total. Formulation with glucose syrup to sugar ratio of 1.5, 0% starch and 6% gelatine was the most stable formulation in terms of hardness with the smallest increase of 0.01 N after 52 weeks storage. On the other hand, the highest increase in hardness was observed for jelly gums formulated with glucose syrup to sugar ratio of 1.1, no starch and 6% gelatine with 0.04 N increase after one year of storage at laboratory conditions. This finding suggested that glucose syrup to sugar ratio in the formulation might have had stronger effect on hardness than gelling agent levels in the recipe, which was also in accordance with the multiple regression results between the ingredients (Table 3.1). This result was similar to the study of Porayanee et al. (2015). They also mentioned that sugars had stronger influence on textural properties as compared to gelatine concentration.



**Figure 3.22** Change in hardness with storage time for jelly gums at 15-22°C and 30-40% RH

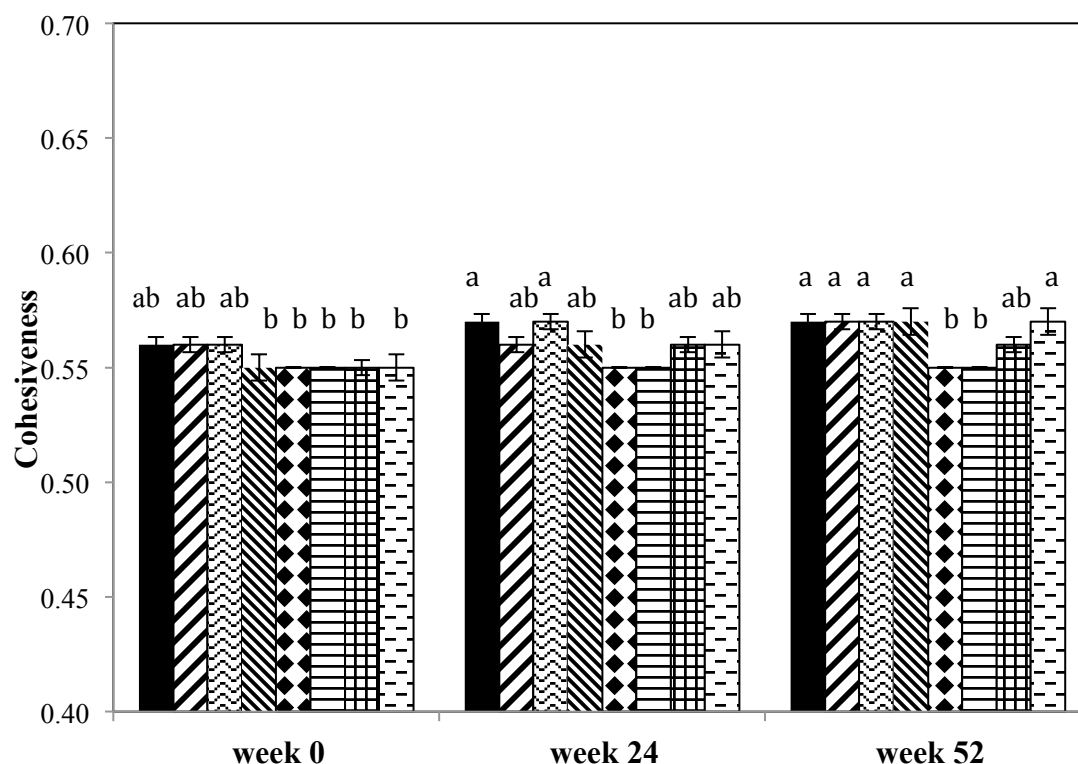
■ G:S=1.1, Starch=0%, Gelatine=3%; ▨ G:S=1.1, Starch=0%, Gelatine=6%; ▩ G:S=1.1, Starch=1.5%, Gelatine=3%; ▪ G:S=1.1, Starch=1.5%, Gelatine=6%;  
 ◆ G:S=1.5, Starch=0%, Gelatine=3%; ≡ G:S=1.5, Starch=0%, Gelatine=6%; + G:S=1.5, Starch=1.5%, Gelatine=3%; - G:S=1.5, Starch=1.5%, Gelatine=6%

Cohesiveness is the strength of internal bonds within a product and shows how well the product resists a second deformation relative to the first deformation. It also demonstrates behaviour of the delivery system under deformation before rupturing (Pons and Fiszman, 1996; Abd Karim et al., 2000; Sandhu and Singh, 2007; Besbes et al., 2009). Multiple regression results were carried out to understand the effect of ingredients on cohesiveness of fresh jelly gums (Table 3.1 and Table A.6). It was seen that cohesiveness was affected by glucose syrup to sucrose ratio ( $p \leq 0.001$ ) and starch level ( $p \leq 0.05$ ), but not by gelatine level ( $p > 0.05$ ). As can be seen from

Table 3.1, cohesiveness of jellies reduces as glucose syrup increases or sucrose decreases in the formulation. This finding was in accordance with the previous study of Porayanee et al. (2015), in which they observed a small difference in value of cohesiveness at all levels of sugars ratio. Siegwein (2010) studied effects of soy protein isolate addition on the properties of starch confections, where significant decrease was seen when 50% soy protein for starch replacement was used. However, unlike our study Munoz et al. (1986) found small differences in cohesiveness values of gelatine gels at different gelatine amounts. This difference might be explained with ingredient interactions between starch and sugars ratio and gelatine as shown in Table 3.1.

In the light of our findings and results of previous studies in literature, it can be concluded that gelling agents like acid thinned starch and gelatine can have significant impacts on hardness in fresh jelly gums with protecting cohesiveness. Cohesiveness is a measure of attractive forces between similar molecules and hence structural integrity (Siegwein, 2010).

Cohesiveness results of selected jelly gums held at laboratory conditions are presented in Figure 3.23 and Table C.2 for two 6-month storage intervals. Selected jellies were the ones having both maximum and minimum gelatine and starch levels for each of the sugars ratio. There was no change in cohesiveness over 6 months for all of the selected jelly gums, namely formulation numbers 1, 3, 7, 9, 10, 12, 16 and 18 (Table 2.2). Exceptions for one-year storage were samples with maximum starch level and maximum gelatine level for both sugars ratio (formulation numbers 9 and 18). As for these two recipes, that are 9 and 18, cohesiveness value increased slightly from 0.55 to 0.57 after 52 weeks storage, which might have been due to the high gelling agent concentration which was transformed into interconnected network during one-year storage (Prokopowich and Biliaderis, 1995; Siegwein, 2010). Regarding samples with formulation numbers 1, 3, 7, 10, 12 and 16, there were no significant changes in cohesiveness after one-year, meaning that internal networks in these jellies remained relatively static, in accordance with several studies found in literature (Siegwein, 2010; Fisher's, 2011; Delgado and Banon, 2015).



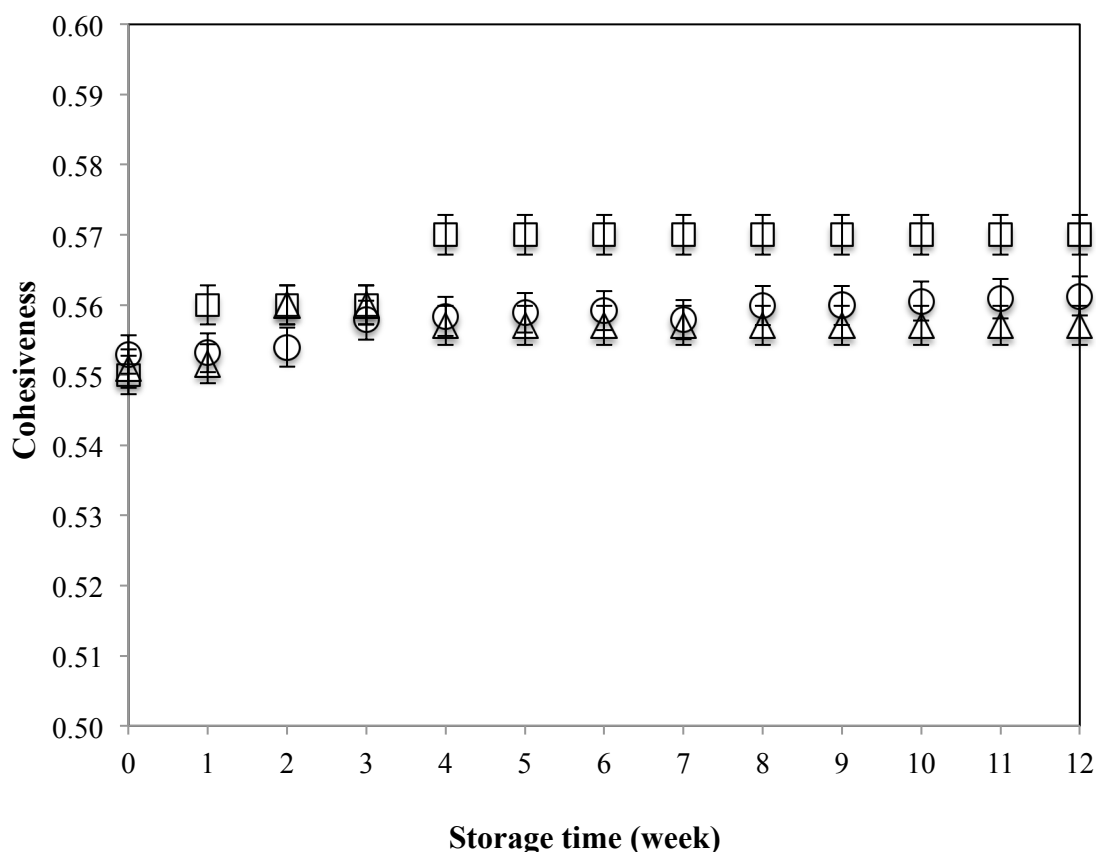
**Figure 3.23** Change in cohesiveness with storage time for jelly gums at 15-22°C and 30-40% RH

■ G:S=1.1, Starch=0%, Gelatine=3%; ▨ G:S=1.1, Starch=0%, Gelatine=6%; ▩ G:S=1.1, Starch=1.5%, Gelatine=3%; ▪ G:S=1.1, Starch=1.5%, Gelatine=6%;  
 ◆ G:S=1.5, Starch=0%, Gelatine=3%; ≡ G:S=1.5, Starch=0%, Gelatine=6%; ⚡ G:S=1.5, Starch=1.5%, Gelatine=3%; - G:S=1.5, Starch=1.5%, Gelatine=6%

Cohesiveness was also measured during storage at 10°C, 20°C and 30°C for 12 weeks. It was noticed that cohesiveness value did not change during 12 weeks at all temperatures, which remained constant around 0.56 except formulation 9, 17 and 18. Cohesiveness change with respect to time and temperature for formulation 18 can be seen in Figure 3.24 and it was seen that cohesiveness increased at high temperature. In addition, similar trend was observed for formulation 9 and 17. Under the light of these observations, it can be concluded that temperature change had very little effect on cohesiveness of jellies at temperatures between 10°C and 30°C as it changed internal network in a very slight manner (only 3 formulations out of 18



formulations). Thus, it can be stated that cohesiveness is not a critical parameter to be measured during temperature studies of jellies having similar formulations given in our study. Yet, it should be a parameter that should be considered and measured in fresh product since when cohesiveness is low (about 0.25), the product could be more prone to moisten on the surface during storage (Masmoudi et al., 2010).

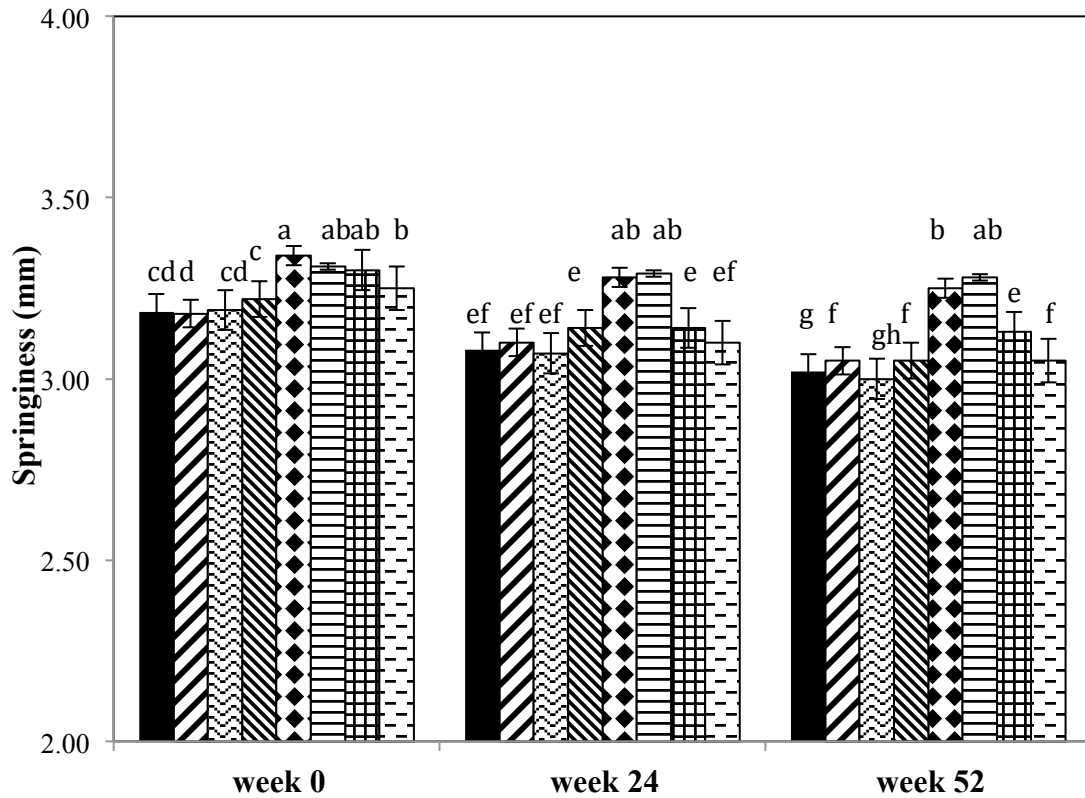


**Figure 3.24** Change in cohesiveness with storage time for jelly gums having glucose syrup:sucrose ratio of 1.5, Starch=1.5% and Gelatine=6% stored at different temperatures

Δ 10°C, O 20°C, □ 30°C

Springiness is defined as the extent to which a compressed food returns to its original size when the load is removed, and can indicate the durability of the confection in the oral cavity during eating when deformed by palate or tongue (Szczesniak, 1962; Pons and Fiszman, 1996; Rosenthal, 1999; Abd Karim et al., 2000; Sandhu and Singh, 2007). In the multiple regression, it was seen that springiness of fresh jellies

was affected by only glucose syrup to sucrose ratio significantly ( $p \leq 0.001$ ) (Table 3.1 and Table A.7). When glucose syrup to sucrose ratio in the recipe increased, springiness of the jelly gums increased. When springiness values of fresh samples were analysed, it was seen that springiness increased from around 3.19 mm to around 3.30 mm as glucose syrup to sucrose increased from 1.1 to 1.5, which supported the regression equations and can also be seen from the results of week 0 given in Figure 3.25. Sun et al. (2014) found closer springiness values at different sugar levels in corn starch gel systems and Porayane et al. (2015) reported differences in springiness of gels at all gelatine concentrations and sugars ratio. Finding of differences in springiness among all recipes might be due to the dependence of springiness on mechanical behaviour during the second bite and jellies remained deformed after the first bite in a way that they had similar springiness measurements. Besides this, interactions between starch and other ingredients might have been the other reason (Table 3.1).



**Figure 3.25** Change in springiness with storage time for jelly gums at 15-22°C and 30-40% RH

■ G:S=1.1, Starch=0%, Gelatine=3%; ▨ G:S=1.1, Starch=0%, Gelatine=6%; ▩ G:S=1.1, Starch=1.5%, Gelatine=3%; ▪ G:S=1.1, Starch=1.5%, Gelatine=6%;  
 ◆ G:S=1.5, Starch=0%, Gelatine=3%; ≡ G:S=1.5, Starch=0%, Gelatine=6%; ⋈ G:S=1.5, Starch=1.5%, Gelatine=3%; ⚡ G:S=1.5, Starch=1.5%, Gelatine=6%

Figure 3.25 shows the springiness results of the selected jellies stored at laboratory conditions for 12 months (Table C.3). As discussed before in previous texture parameters, selected samples were the ones having the lowest and highest gelatine (3% and 6%) and starch (0% and 1.5%) concentrations for each of the sugar ratio. After the first 6-month time interval, it was seen that springiness decreased for all of the formulations chosen except formulations 10 and 12. When the changes between 6 months and 12 months were viewed, it was observed that springiness of selected jellies remained similar except formulations 1, 7 and 9. Decreases during storage

might be due to the fact that it was quite likely that air pockets formed within the jelly gum matrix changed very little during storage and were in charge of the springiness values measured during compression cycles (Fisher, 2011). It was noticed that springiness value of formulations 10 and 12 did not show any significant differences during storage at laboratory conditions for 52 weeks, meaning that air pockets formed within the jelly matrix were maintained throughout storage. This was a very important observation because springiness was related to the chewing energy in mouth (Rahman and Al-Mahrouqi, 2009) and in sensory testing formulation 12 was found to be one of the most acceptable formulations by the panelists (Table 3.9). Studies about time dependence of springiness are very limited regarding confectionery products like jelly gums in the literature. Delgado and Banon (2015) found no changes in springiness during 24 hours of conditioning time of jellies formulated with 1.1 sugars ratio with 8.02% starch and 3.39% gelatine. They observed springiness around 3.94 mm in their study.

To conclude, springiness is an important parameter in jelly product development and should be taken into account in the earlier stages of formula development as it gives clue about chewing energy in mouth. It affects sensory experience of the consumer since the panelists gave highest scores to formulation 12, which was one of the most stable formulations when springiness was concerned (Table 3.9).

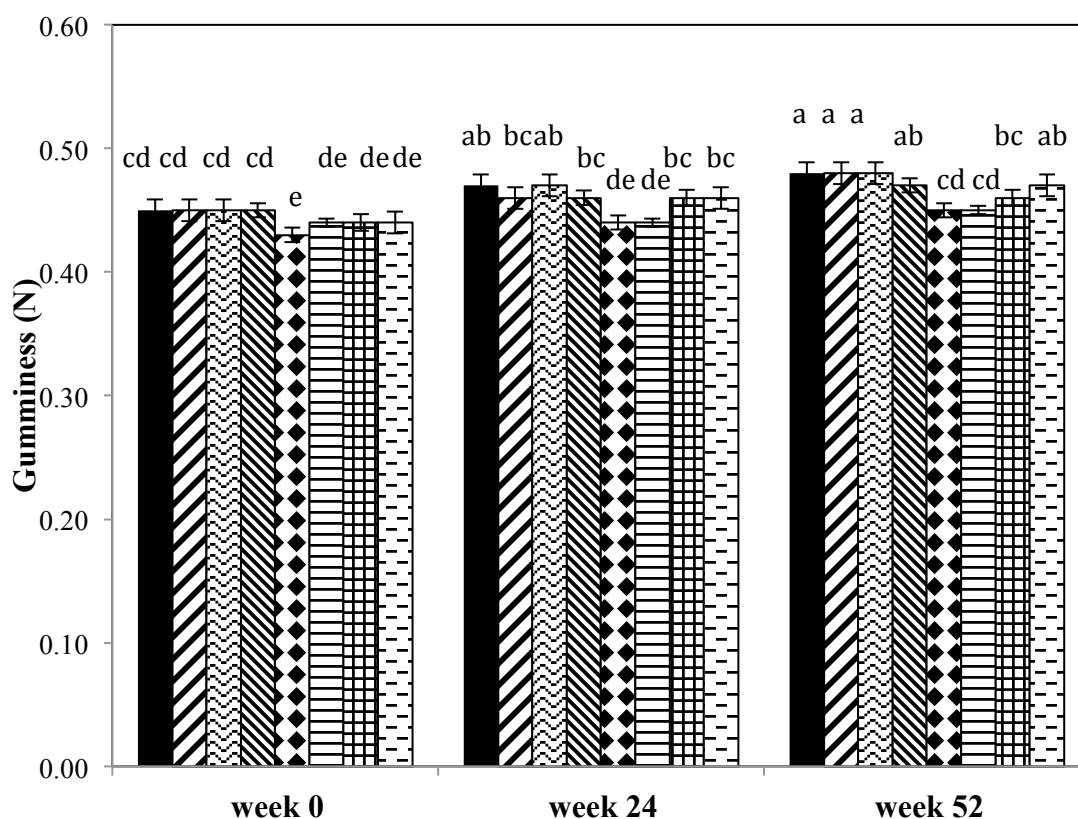
Gumminess is the result of multiplying hardness and cohesiveness and it was used as a comparison attribute between samples (Delgado and Banon, 2015). Gumminess is defined as the energy needed to disintegrate a semisolid food during mastication in mouth to a point where the food can be swallowed (Bourne, 2002). Table 3.1 and Table A.8 show the results of multiple regression. It was found that gumminess of fresh samples was affected by glucose syrup:sucrose ratio ( $p \leq 0.001$ ), but not by gelatine and starch level ( $p > 0.05$ ).

Gumminess results of fresh jellies of selected formulations can be seen from Figure 3.26 as week 0, from this figure it was noticed that gumminess values were similar.

Different from this study, Porayanee et al. (2015) reported significant increases in gumminess in a way that when sucrose amount increased (or glucose amount decreased) gumminess also increased. In addition, they have observed the highest gumminess value at maximum gelatine level, which was 8.25%, at the same sugars ratio (70:30; glucose syrup: sucrose ratio). However, this finding was not in accordance with the results of this study regarding gelling agents concentrations since gelling agent concentration did not affect gumminess values when the jellies were fresh in this study (Figure 3.26 week 0 and Table C.4). This might be due to the fact that gelling agent concentrations used in the experiments were not as high as in the previous research (Porayanee et al., 2015) as gumminess means the energy required to break down product into a ready to swallow state. Porayanee et al. (2015) used gelatine levels of 7.75%, 8.00% and 8.25%, whereas gelatine levels of 3%, 4.5% and 6% combined with starch levels of 0%, 1% and 1.5% were used in this study, which gave similar results at the same sugars ratio.

Gumminess results of selected jelly gums, having minimum and maximum gelatine (3% and 6%) and starch (0% and 1.5%) levels for each of the sugars ratio, are presented in Figure 3.26 (Table C.4). After 24 weeks storage at laboratory conditions, an increase of gumminess of 0.02 N was seen only for the formulations 1 and 7 with 1.1 sugars ratio and formulations 16 and 18 with 1.5 sugars ratio. On the other hand, the only formulation whose gumminess increased by 0.02 N between week 24 and week 52 was formulation 3 with 1.1 sugars ratio, 0% starch and 6% gelatine. Little information on gumminess during long storage of jellies is available in the literature. Delgado and Banon (2015) reported increase of gumminess from 0.50 N (12 hours) to 0.56 N (16 hours) during conditioning. Siegwein (2010) compared the gumminess of sample with 11% acid-thinned wheat starch and the one with 5.5% acid-thinned wheat starch and 5.5% soy protein isolate during 20 days storage. It was observed that gumminess value of sample with 11% acid-thinned wheat starch increased significantly and peaked at day 10, on the other hand, samples with 5.5% acid-thinned wheat starch and 5.5% soy protein isolate did not increase significantly until day 20. Confections, prepared with 10.1% modified corn starch,

stored for 14 days were significantly gummier than the ones at day 1 (Fisher, 2011). Under the light of these studies and comparison of the findings of the current study, changes in gumminess value may be more pronounced when the gelling agent was starch and the concentration was high. It was also realized one more time that formulation 12 encountered the lowest increase (0.01 N) in gumminess during 52 weeks storage at laboratory conditions. This was expected due to the results of hardness and cohesiveness since gumminess is the multiplication of these two attributes. The lowest increase in gumminess value meant that the mentioned formulation would require a stable mastication to dissolve during storage (Siegwein, 2010).



**Figure 3.26** Change in gumminess with storage time for jelly gums at 15-22°C and 30-40% RH

■ G:S=1.1, Starch=0%, Gelatine=3%; ▨ G:S=1.1, Starch=0%, Gelatine=6%; ▩ G:S=1.1, Starch=1.5%, Gelatine=3%; ▪ G:S=1.1, Starch=1.5%, Gelatine=6%;  
 ◆ G:S=1.5, Starch=0%, Gelatine=3%; ≡ G:S=1.5, Starch=0%, Gelatine=6%; ⦶ G:S=1.5, Starch=1.5%, Gelatine=3%; ⦶ G:S=1.5, Starch=1.5%, Gelatine=6%

### 3.9 Sugar Profiles of Jelly Gums

Sugar spectrum of a glucose syrup do not change during storage or during processing, however, sucrose can invert into a mixture of dextrose and fructose via heat and acid treatment. Hence, it is important to have knowledge about the sugar profiles of jelly gums during storage at different temperature conditions, which can give idea about inversion and crystallization behaviour of the product, to predict and

to control texture and shelf life. Thus, sucrose (%), glucose (%) and fructose (%) analysis of each jelly gum formulation were carried out at different storage temperatures, namely 10°C, 20°C and 30°C during 52 weeks.

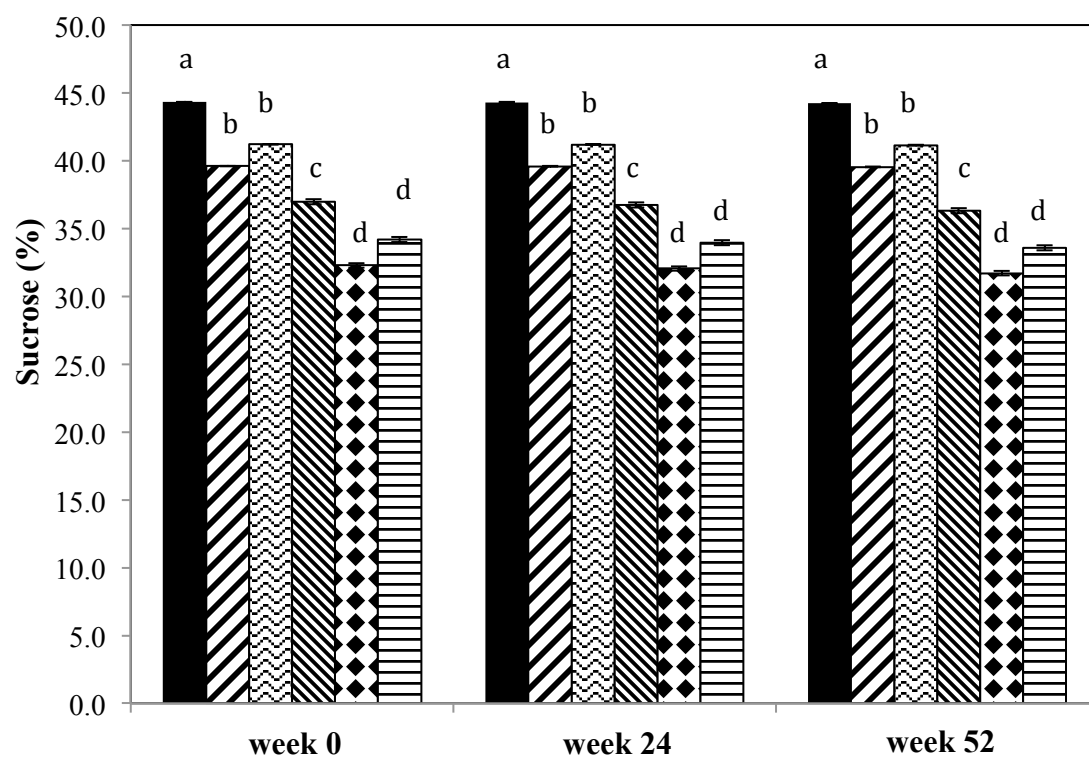
In this part of the study, formulations were selected in order to investigate the impacts of most important ingredient parameters, sugars ratio and starch concentration, that were thought to have interactions in jellies and could cause changes in sugar profiles during processing and storage. Starch was preferred as a parameter since all sugars could increase the gelatinization temperature. In jellies high levels of sugars are used with respect to starch, which might increase gelatinization temperature in a significant way. This might lead to quality problems. Type of the sugar used in the recipe affects the gelatinization temperature in a different way (Helstad, 2006). Sucrose ranks the first in the order of influence in increasing gelatinization temperature, glucose ranks the second, fructose ranks the third and xylose ranks the fourth (Slade and Levine, 1987). In addition, as sugar concentration increases, gelatinization temperature of starch also increases (Helstad, 2006). Gelatine level was not chosen as a parameter because gelatine, which was a protein, had different interactions with sugars in jelly matrix. Glucose syrups can react with proteins as they are made up of reducing sugars. On the other hand, sucrose not being a reducing sugar does not go into Maillard reaction with gelatine (Hull, 2010).

Figure 3.27, 3.28 and 3.29 showed the effect of storage time on sucrose content of selected jelly formulations at different temperatures, 10°C, 20°C and 30°C, respectively. Three-way Anova tables on sucrose content are given in Table D.1.1, D.1.2 and D.1.3 for 10°C, 20°C and 30°C, respectively. It was observed that 10°C and 20°C storage had very similar effects on sucrose content of jellies. It was noticed that as glucose syrup to sucrose ratio increased from 1.1 to 1.5, sucrose content of samples decreased. This result was expected because glucose syrup to sucrose ratio of 1.1 in the recipe contained more sucrose when compared with sugars ratio of 1.5. When storage time was concerned, it was seen that sucrose content did not change



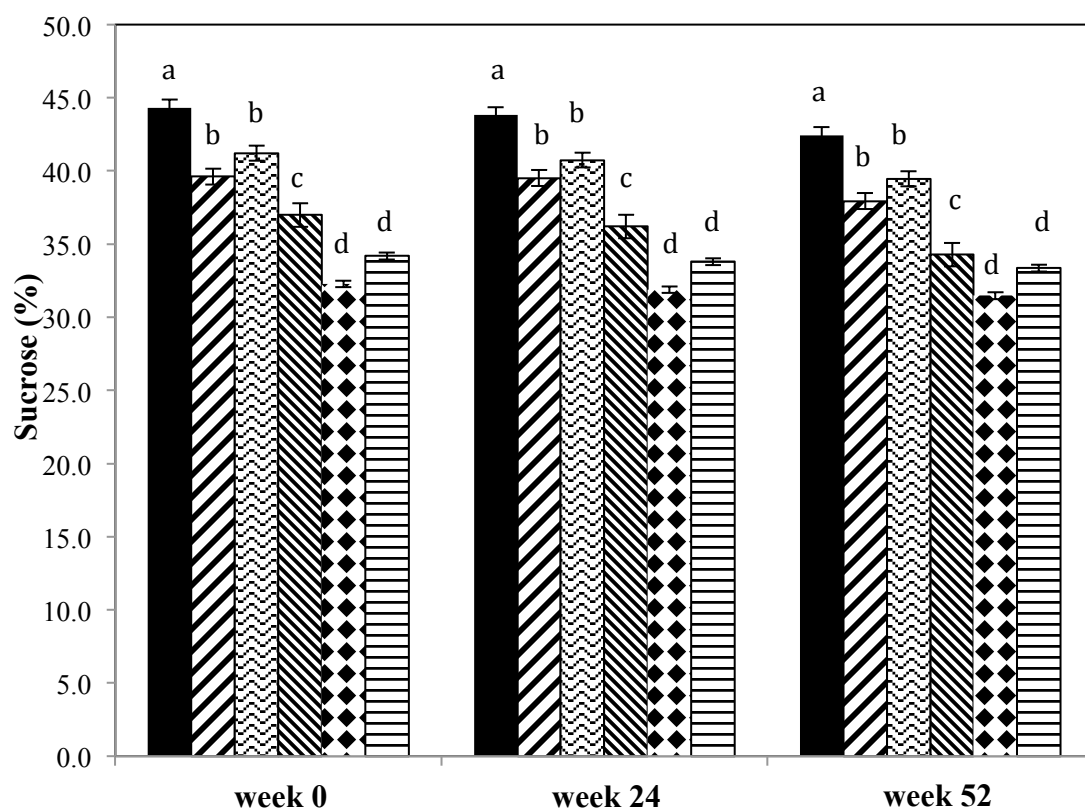
significantly for each of the formulation during 52 weeks at 10°C and 20°C, meaning that mentioned formulations remained stable in terms of sucrose content. This showed that sucrose inversion might have occurred insignificantly since these temperatures was not sufficient to favor sucrose inversion (Hull, 2010).

Influence of starch concentration on sucrose content was also very similar for 10°C and 20°C. According to 3-way Anova results given in Table D.1.1 and D.1.2, significant differences were determined between starch concentrations. As starch concentration increased from 0% to 1.5%, sucrose content decreased in a time independent way. This might be due to the interaction of starch with sugars in the recipe in a way to bind moisture. Sugars have strong affinity than starch regarding competition on water. When glucose syrup:sugar ratio was fixed and starch level increased, there would be less water in the food matrix for starch. Sucrose in the recipe might have used the available water for inversion reaction described as hydrolysis gain previously. Rate of inversion was known to be a function of moisture availability (Helstad, 2006).



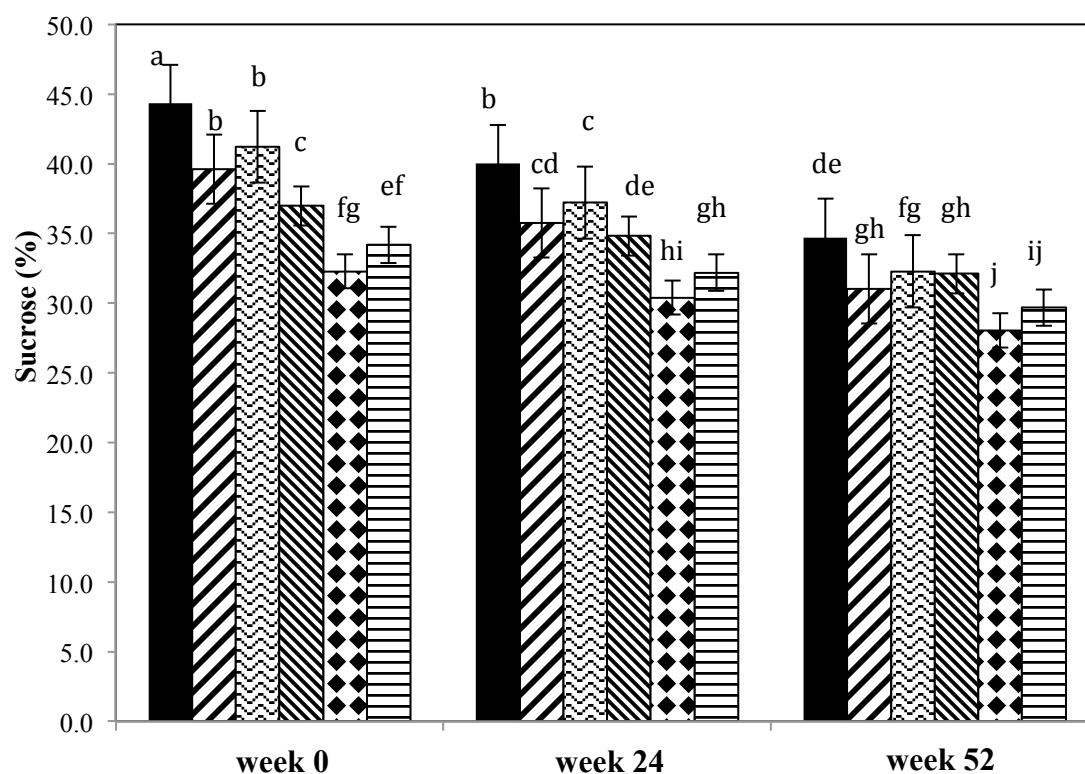
**Figure 3.27** Sucrose content of jelly gums with 6% gelatine as a function of storage time at 10°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪  
 G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ≡ G:S=1.5, Starch=1.5%



**Figure 3.28** Sucrose content of jelly gums with 6% gelatine as a function of storage time at 20°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪ G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ≡ G:S=1.5, Starch=1.5%



**Figure 3.29** Sucrose content of jelly gums with 6% gelatine as a function of storage time at 30°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪ G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ▤ G:S=1.5, Starch=1.5%

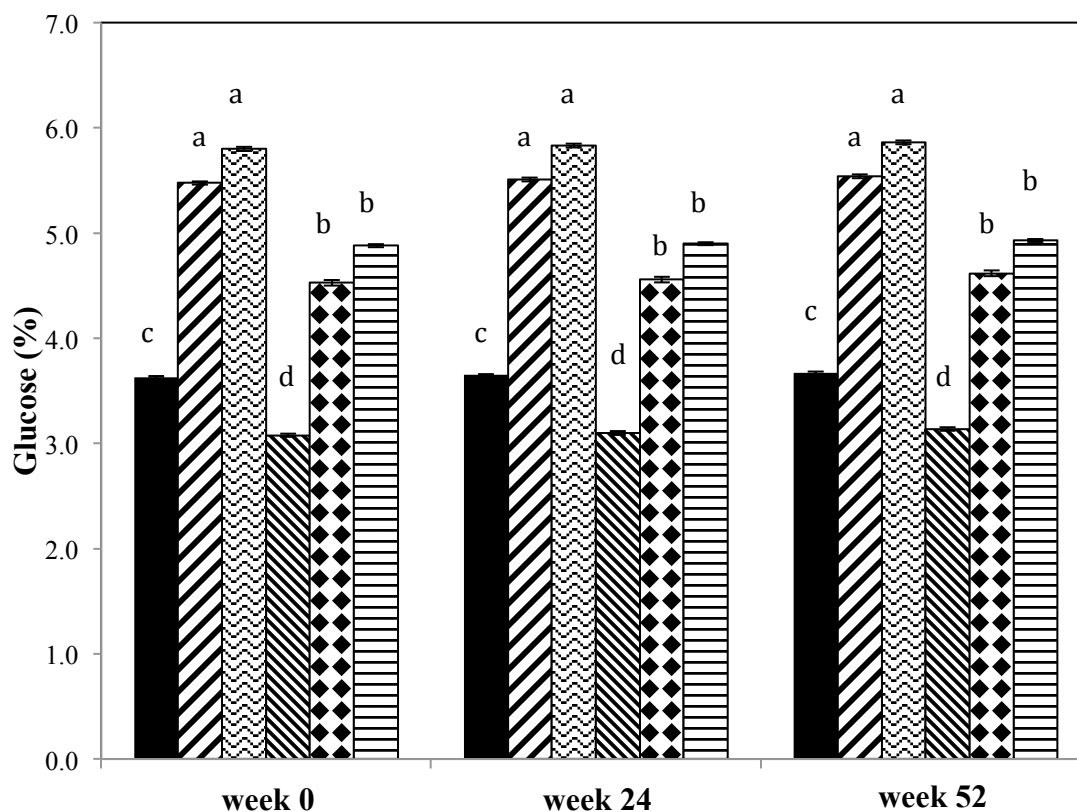
Figure 3.29 represents sucrose content of jelly gums with respect to storage time at 30°C. Unlike 10°C and 20°C storage, sucrose content decreased with storage time for all of the jellies (Table D.1.3). This might be due to inversion reaction of sucrose during storage at 30°C since rate of inversion was a function of temperature. The higher the temperature was faster the rate of inversion (Helstad, 2006). Similar to 10°C and 20°C storage, at 30°C significant changes in sucrose content of jellies were realized between different starch levels. Hence, when the storage temperature increased, sucrose content decreased in a faster manner. The impact of sugars ratio on sucrose content at 30°C was very similar with 10°C and 20°C due to the same reason discussed previously.

Effect of storage time on glucose content of selected jellies are given in Figure 3.30, 3.31 and 3.32 at 10°C, 20°C and 30°C, respectively. Tables D.2.1, D.2.2 and D.2.3 showed 3-way Anova results on glucose content at these temperatures. According to these results, it was found that as the glucose syrup to sucrose ratio in jellies increased from 1.1 to 1.5, glucose content decreased independently from storage time at all temperatures. This was an expected result, because when the glucose syrup in the formulation increased, sucrose in the formulation decreased, which meant less sucrose inversion occurred in the jelly matrix due to cooking at high temperature and acid addition. Hence, there was less glucose, which was one of the end products of sucrose inversion, in the system (Helstad, 2006; Hull, 2010).

On the other hand, it was observed in the opposite way when starch concentration increased in the recipe. In other words, when starch increased from 0% to 1% or from 0% to 1.5%, glucose content of jelly increased. This might be clarified via competition for water as mentioned previously in sucrose content part. As starch concentration increased in the formulation, there would be less available water for starch. As sucrose had higher attraction for water when compared to corn starch, sucrose inversion rate increased and thus there were more glucose formed as an end product (Helstad, 2006). In addition, less starch conversion to glucose might have happened during preparation of jelly gums in a way to contribute to the increase of glucose content of jelly gums (Johnson and Padmaja, 2013). This contribution was thought to be low due to the fact that there were no significant differences between 1% and 1.5% starch levels in affecting glucose content at all temperatures. The increment of 0.5% starch was not sufficient to cause an interaction. In addition, when changes in glucose content were investigated, no statistically significant interactions were found between sugars ratio, starch level and storage time. There were only single effects found which were sugars ratio and starch (%) for all of the storage temperatures (Tables D.2.1, D.2.2 and D.2.3).

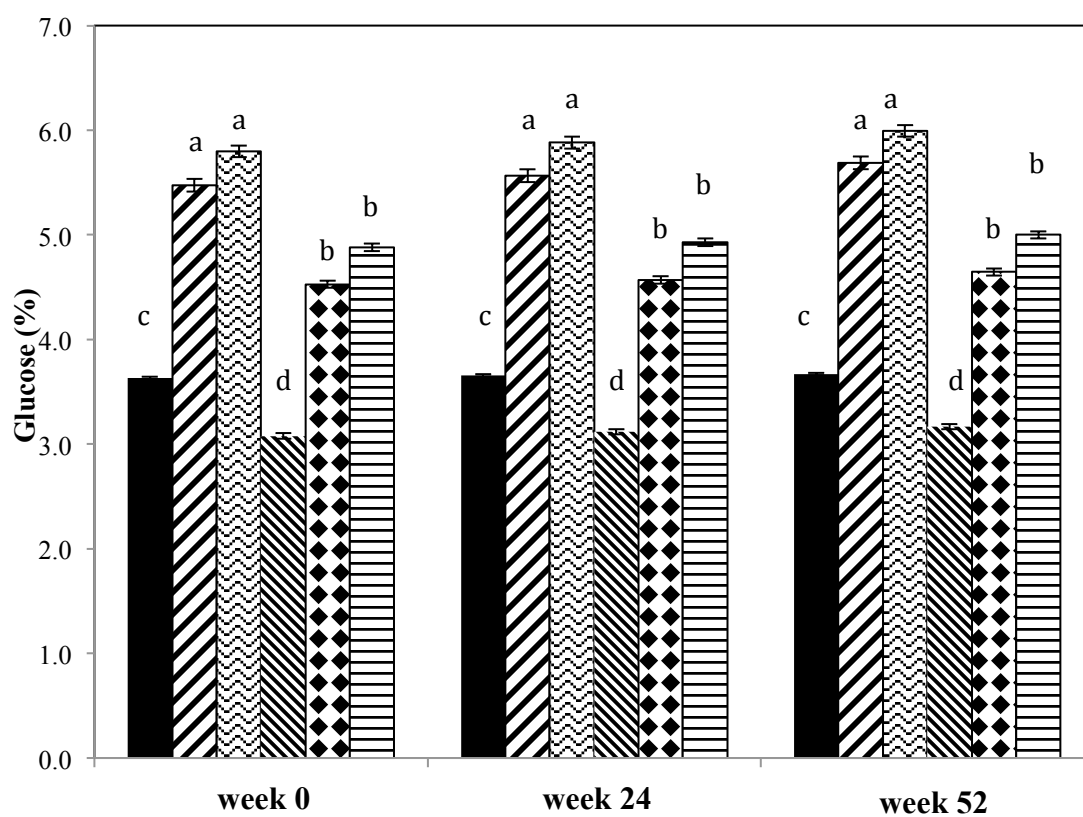
Despite the case that there were no significant changes in glucose content of each jelly formulation during storage time at all temperatures studied, it was seen that when storage temperature increased glucose yield increased accordingly. In other

words, 0.06% glucose increase at 10°C, 0.13% glucose increase at 20°C and 0.29% glucose increase at 30°C were seen. This described that every 10°C increase nearly doubled the glucose content of jellies. This finding was in accordance with the study of Helstad (2006), who noted sucrose inversion was driven by temperature. It was stated that as the temperature increased, rate of sucrose inversion into glucose and fructose increased.



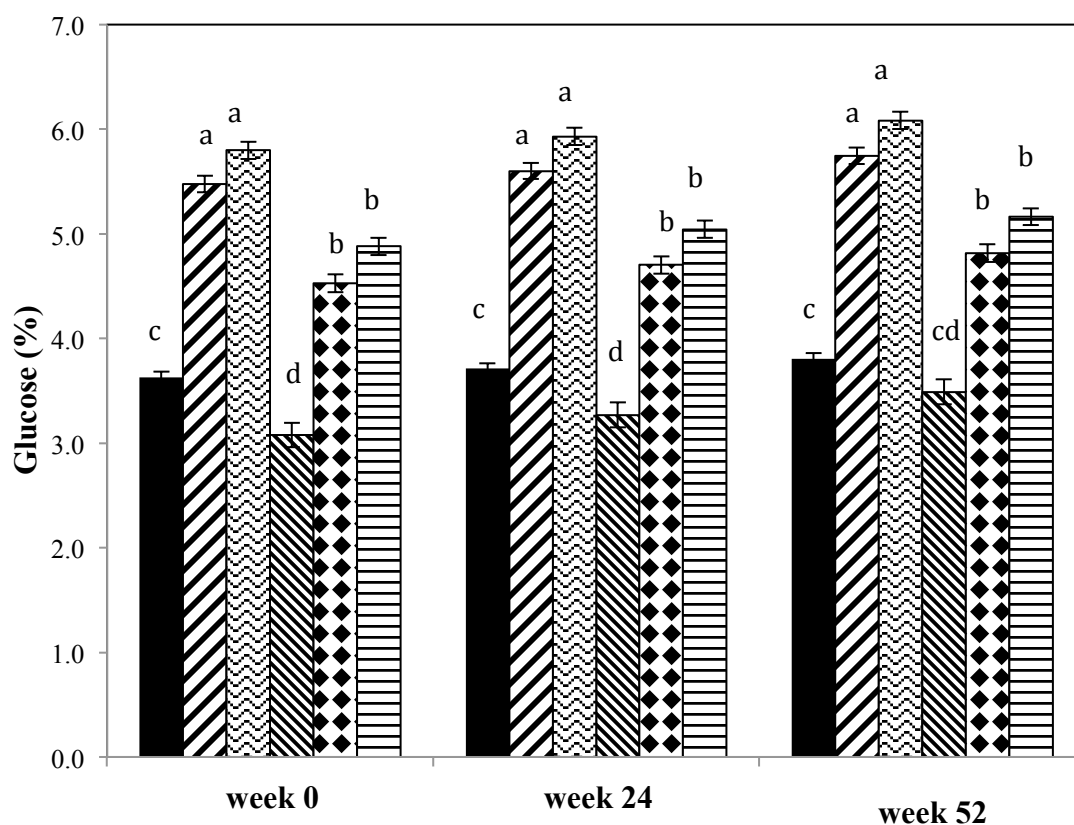
**Figure 3.30** Glucose content of jelly gums with 6% gelatine as a function of storage time at 10°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪ G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ▤ G:S=1.5, Starch=1.5%



**Figure 3.31** Glucose content of jelly gums with 6% gelatine as a function of storage time at 20°C

■ G:S=1.1, Starch=0%; 
 ▨ G:S=1.1, Starch=1%; 
 ▩ G:S=1.1, Starch=1.5%; 
 ▪ G:S=1.5, Starch=0%; 
 ◆ G:S=1.5, Starch=1%; 
 ≡ G:S=1.5, Starch=1.5%



**Figure 3.32** Glucose content of jelly gums with 6% gelatine as a function of storage time at 30°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪ G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ≡ G:S=1.5, Starch=1.5%

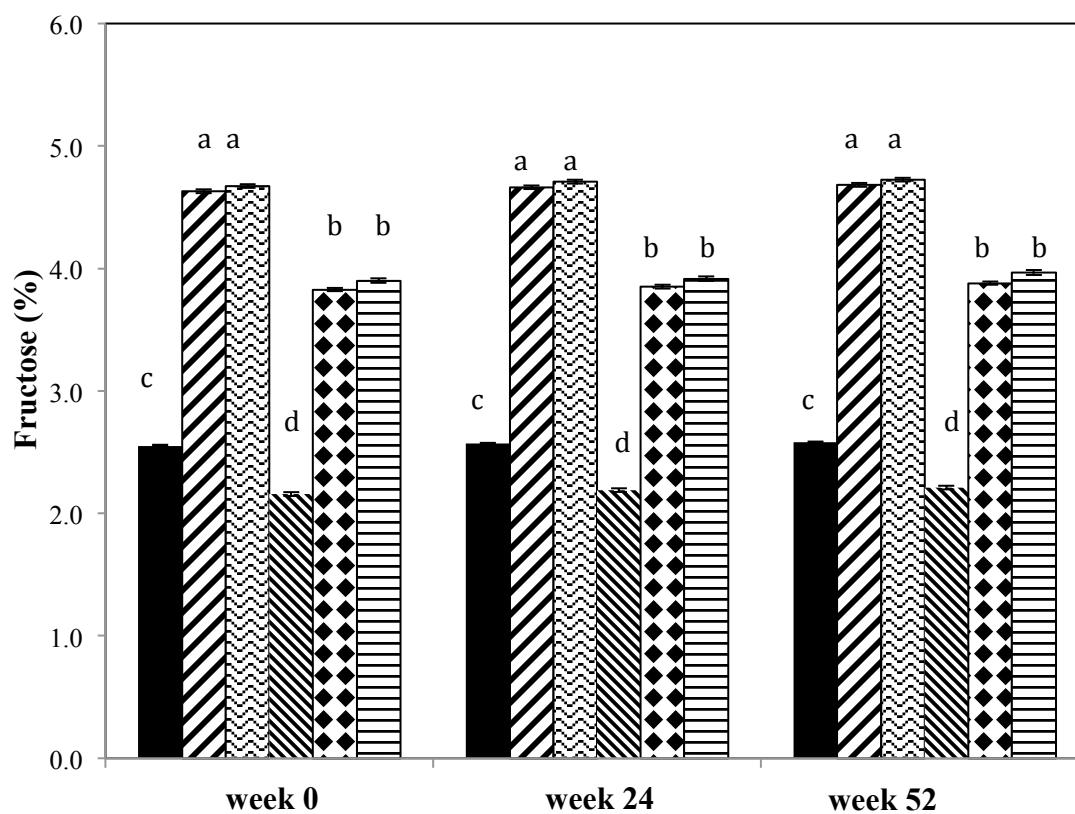
When the changes in fructose content of jellies were analysed, it was seen that the results were very identical to the results of glucose content. This was an expected result since both were the end products of sucrose inversion and their molecular weights were the same. Figures 3.33, 3.34 and 3.35 represented the effect of storage time on fructose content. Tables D.3.1- D.3.3 designated 3-way Anova findings on fructose content at 10°C, 20°C and 30°C storage temperatures. Similar with glucose content findings, when the glucose syrup to sucrose ratio in the formulation increased from 1.1 to 1.5, fructose content declined at all temperatures in a storage time independent manner. This was because of less sucrose inversion occurred in the



jelly matrix as there were less sucrose in the system and there were more glucose syrup, which had stable sugar spectrum during processing or storage unlike sucrose (Helstad, 2006; Hull, 2010).

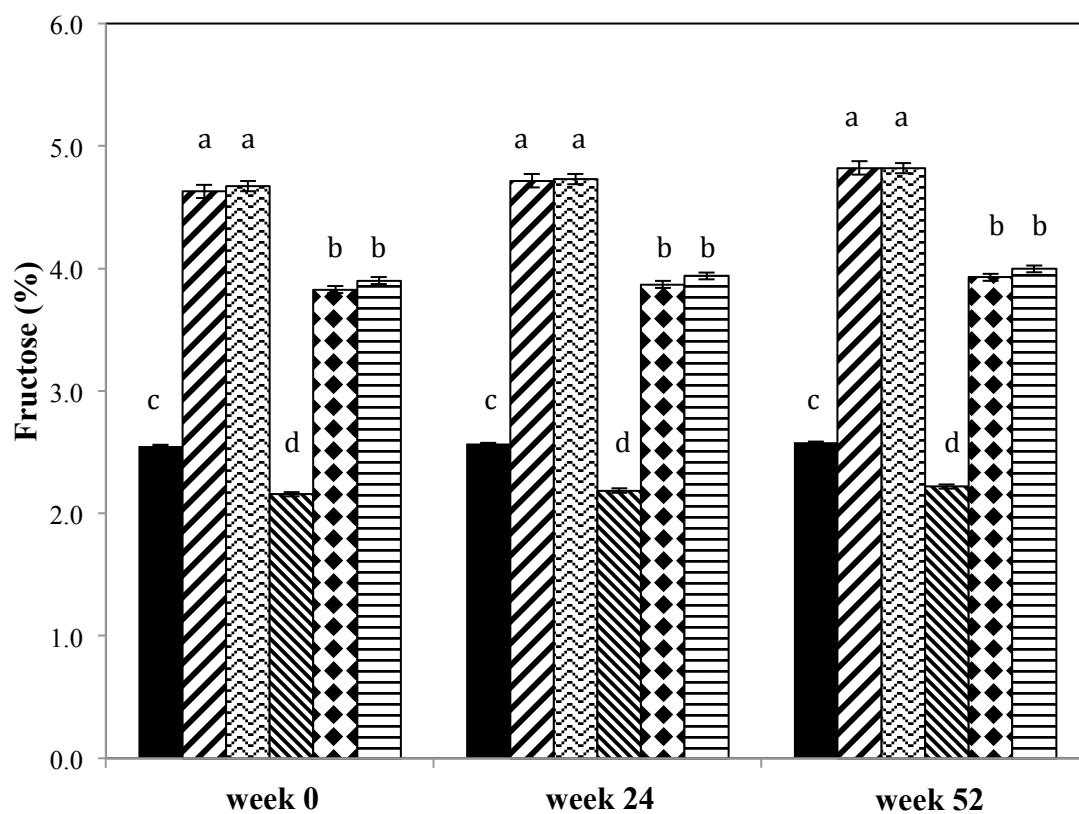
Aligned with glucose content results, it was seen that fructose contents of jellies increased when starch level changed from 0% to 1% or 1.5%. Due the mentioned reasons explained previously, increase in starch level might have enhanced sucrose inversion such that more water was available to be utilized by sucrose in inversion reaction during processing mostly. Hence, the effect of storage time was insignificant in terms of fructose content of samples (Table D.3.1- D.3.3). Furthermore, no significant changes were found in fructose content between 1% and 1.5% starch concentrations as 0.5% of starch change was not enough to increase and fasten the interactions, which was an identical observation seen in glucose content measurements.

When the fructose content results for different storage temperatures were compared, it was seen that as temperature of the environment, that jellies were held, increased fructose gain increased. Average of fructose increase was 0.05%, 0.10% and 0.19% at 10°C, 20°C and 30°C, respectively. Like the case for glucose content, every 10°C temperature increase caused increase in fructose levels almost twice due to the temperature driven characteristic of sucrose inversion (Helstad, 2006; Hull, 2010). When these values were compared with glucose results, fructose results were slightly lower than glucose. This difference was thought to be due to the small contribution of starch conversion to glucose (Johnson and Padmaja, 2013) in jellies. Moreover, hydrolysis of complex sugars mainly maltose to glucose might have had a role in glucose content being slightly higher than fructose content.



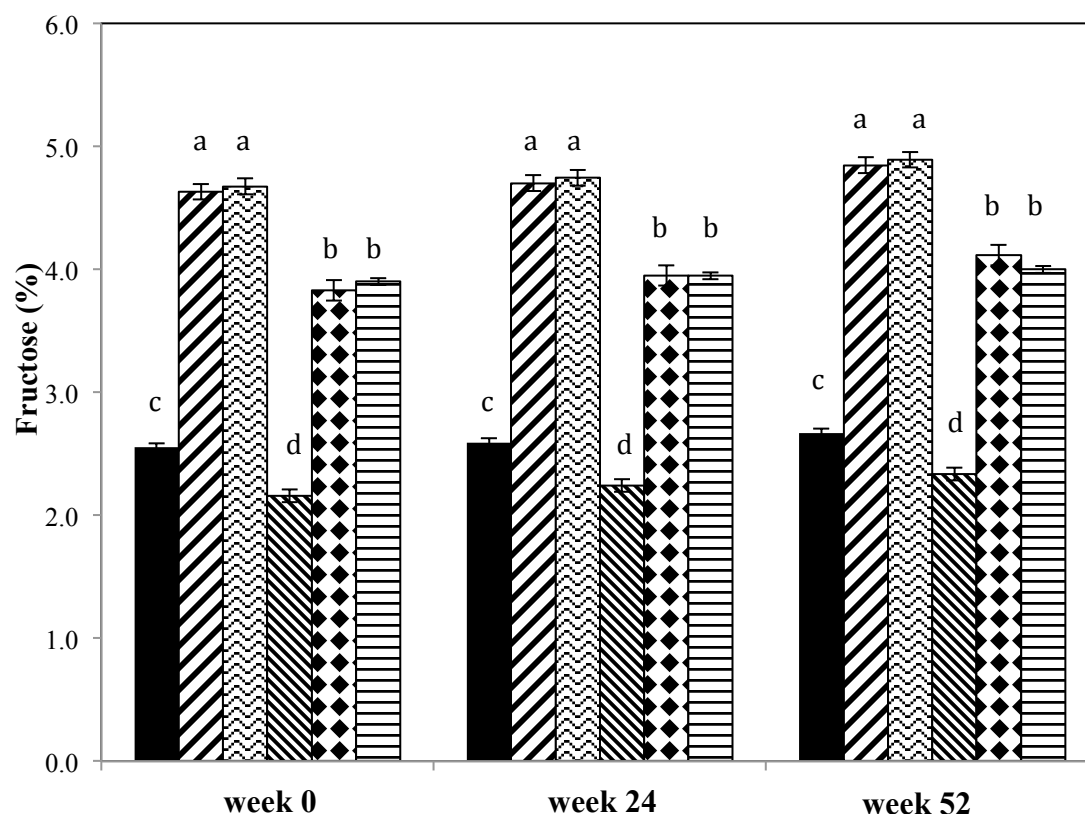
**Figure 3.33** Fructose content of jelly gums with 6% gelatine as a function of storage time at 10°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪  
 G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ≡ G:S=1.5, Starch=1.5%



**Figure 3.34** Fructose content of jelly gums with 6% gelatine as a function of storage time at 20°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪  
 G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ≡ G:S=1.5, Starch=1.5%



**Figure 3.35** Fructose content of jelly gums with 6% gelatine as a function of storage time at 30°C

■ G:S=1.1, Starch=0%; ▨ G:S=1.1, Starch=1%; ▩ G:S=1.1, Starch=1.5%; ▪ G:S=1.5, Starch=0%; ◆ G:S=1.5, Starch=1%; ▬ G:S=1.5, Starch=1.5%

To conclude, it is important to investigate sucrose inversion phenomena while designing a jelly formulation as it may cause many consequences. First of all, it causes chemical gain up to 5% (342 parts of sucrose end up with up to  $180 \times 2 = 360$  parts of glucose and fructose by gaining 18 parts water). This mass gain can affect the mouth-feel of consumers. In our study, our panelists did not give feedback about this, supporting negligible inversion during storage, as sugars ratio of 1.1 and 1.5 were found to be stable. Also, sweetness of product increases during sucrose inversion, which may affect the flavor of the product. In addition, conversion from nonreducing to reducing sugars may have some future impacts during shelf life of confectionery. In order to track sucrose inversions within a product, sugar profile

analysis can be used, in the light of the findings of this study to concentrate on sucrose content may be a time saving action since it showed differences during storage at high temperature.

### **3.10 Sensory Evaluation of Jelly Gums**

#### **3.10.1 Overall Acceptability of Jelly Gums**

Generally in sensory testing; when the product is scored as minimum 6.5 out of 9, the product is said to be acceptable as it means it is liked moderately by the panelists as described in sensory analysis part in Chapter II previously. Table 3.9 and Table E.1-E.4 show overall acceptability scores of jelly gums that were stored at different conditions. It was noticed that jelly gums containing highest gelatine level, i.e. 6%, had acceptable scores when the samples were fresh.

When the samples were held at laboratory conditions (15-22°C) for 52 weeks, at the end of the storage only two formulations had acceptable results, namely, formulation with 1.5% starch, 6% gelatine and glucose syrup to sucrose ratio of 1.1 and formulation with no starch, 6% gelatine and glucose syrup to sucrose ratio of 1.5 (Table 3.9).

Sensory studies were performed at 10°C and at 30°C for 12 weeks. It was seen that samples with 6% gelatine had scores more than 6.5 after 12-week storage at 10°C which was similar to the scores of fresh samples. However, when the samples were aged at 30°C for 12 weeks, none of the jellies scored more than 6.5 points by the panelists (Table 3.9). This showed that end points of shelf life in terms of sensory analysis were already reached during storage at high temperature. Thus, 30°C was a suitable temperature for shelf life studies of jelly gums with gelatine and starch in terms of sensory testing.

In addition, it was found that formulation with 1.5% starch, 6% gelatine and glucose syrup to sucrose ratio of 1.1 and formulation with no starch, 6% gelatine and glucose

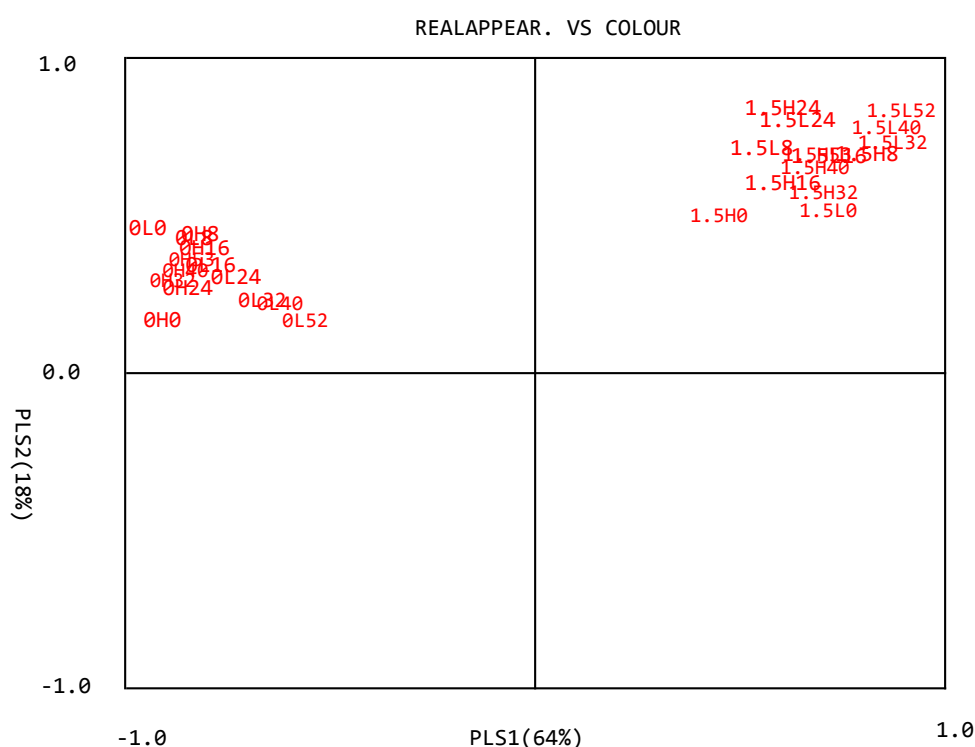
syrup to sucrose ratio of 1.5 had the highest scores among all formulations before and after storage at all conditions (Table 3.9). According to the gumminess part of this study, formulation with no starch, 6% gelatine and glucose syrup to sucrose ratio of 1.5 had the lowest increase in gumminess, which meant formulation had a stable mastication to dissolve product in the mouth (Figure 3.26). This might be one of the reasons why mentioned formulation was evaluated to be one of the most acceptable recipes by the panelists as given in Table 3.9.

**Table 3.9** Overall acceptability scores of jelly gums stored at different conditions

Formulation			Overall acceptability			
G:S ratio	Starch (%)	Gelatine (%)	Fresh (0 weeks)	Aged (52 weeks) 15-22°C	Aged (12 weeks) 10°C	Aged (12 weeks) 30°C
1.1	0	3	4.1 ± 0.26	3.5 ± 0.30	3.9 ± 0.36	1.8 ± 0.30
1.1	0	4.5	4.9 ± 0.28	4.4 ± 0.62	4.8 ± 0.44	2.2 ± 0.42
1.1	0	6	7.2 ± 0.34	6.0 ± 0.32	7.0 ± 0.84	3.2 ± 0.22
1.1	1	3	4.2 ± 0.50	3.6 ± 0.56	4.0 ± 0.38	2.0 ± 0.78
1.1	1	4.5	4.8 ± 0.40	4.4 ± 0.70	4.7 ± 0.35	2.3 ± 0.83
1.1	1	6	7.1 ± 0.70	6.1 ± 0.62	6.9 ± 0.72	3.2 ± 0.45
1.1	1.5	3	5.2 ± 0.41	4.5 ± 0.74	5.0 ± 0.22	2.5 ± 0.54
1.1	1.5	4.5	6.1 ± 0.60	5.5 ± 0.53	5.9 ± 0.54	2.7 ± 0.69
1.1	1.5	6	8.6 ± 0.90	6.6 ± 0.40	8.4 ± 0.76	4.1 ± 0.67
1.5	0	3	4.2 ± 0.25	3.5 ± 0.90	4.0 ± 0.48	1.2 ± 0.25
1.5	0	4.5	6.2 ± 0.85	5.4 ± 0.45	6.0 ± 0.74	2.8 ± 0.39
1.5	0	6	8.5 ± 0.27	6.5 ± 0.92	8.3 ± 0.56	4.1 ± 0.18
1.5	1	3	3.9 ± 0.50	3.3 ± 0.62	3.6 ± 0.42	1.4 ± 0.28
1.5	1	4.5	4.3 ± 0.60	3.5 ± 0.76	4.1 ± 0.88	1.6 ± 0.64
1.5	1	6	6.8 ± 0.62	5.8 ± 0.77	6.6 ± 0.38	3.3 ± 0.62
1.5	1.5	3	4.0 ± 0.40	3.6 ± 0.63	3.9 ± 0.55	1.2 ± 0.71
1.5	1.5	4.5	4.2 ± 0.50	3.8 ± 0.57	4.0 ± 0.49	1.9 ± 0.76
1.5	1.5	6	6.9 ± 0.60	5.9 ± 0.37	6.7 ± 0.35	3.4 ± 0.44

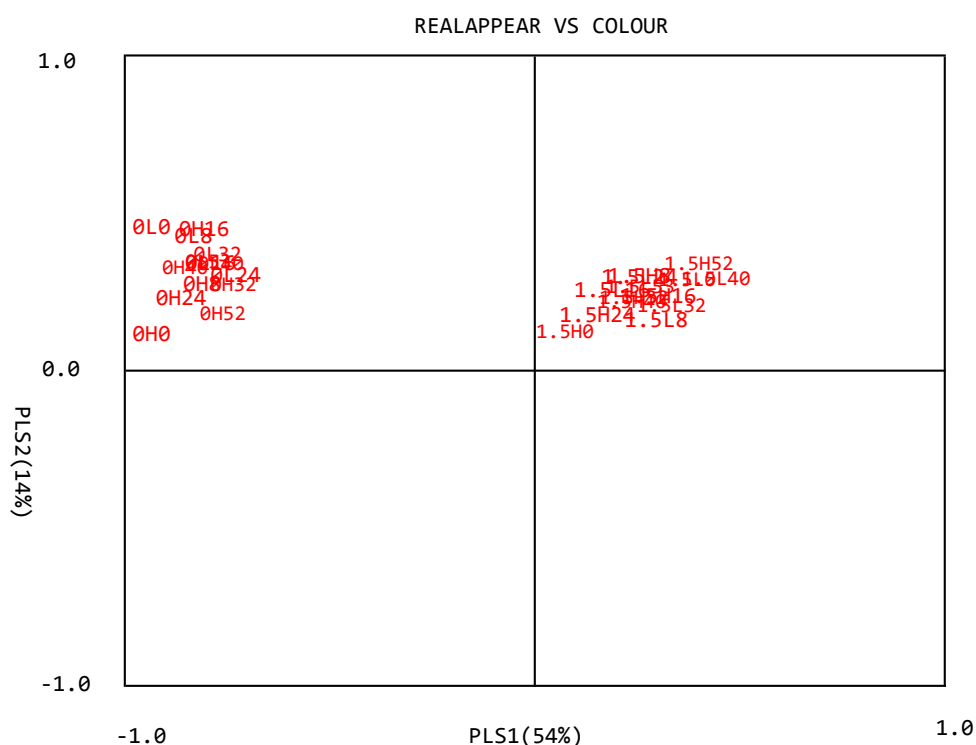
### 3.10.2 Correlation of Sensory Appearance Liking and Colour of Jelly Gums

Figure 3.36 investigates the effect of starch level and the effect of gelatine level on appearance liking for the jelly gums with glucose syrup to sucrose ratio are 1.1 stored at laboratory conditions at 15-22°C and 30-40% RH with exposure to light for 52 weeks. The results of recipes with starch level of 1% and with gelatine level of 4.5% were not involved as they had similar sensory scores with 1.5% starch level and 3% gelatine level. According to the results given in Figure 3.36, starch level was more dominant with respect gelatine level on appearance liking in a way that starch affected the sensory appearance liking of jellies negatively. This might be due to the opaque appearance of starch confectionery where the way starch molecules interact to form a gel making confectionery translucent to opaque (Hartel and Hartel, 2014).



**Figure 3.36** Appearance liking sensory versus colour change ( $\Delta$ Hue) for jelly gums with glucose syrup:sucrose of 1.1. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 8<sup>th</sup> week, 16<sup>th</sup> week, 24<sup>th</sup> week, 32<sup>nd</sup> week, 40<sup>th</sup> week, 52<sup>nd</sup> week)

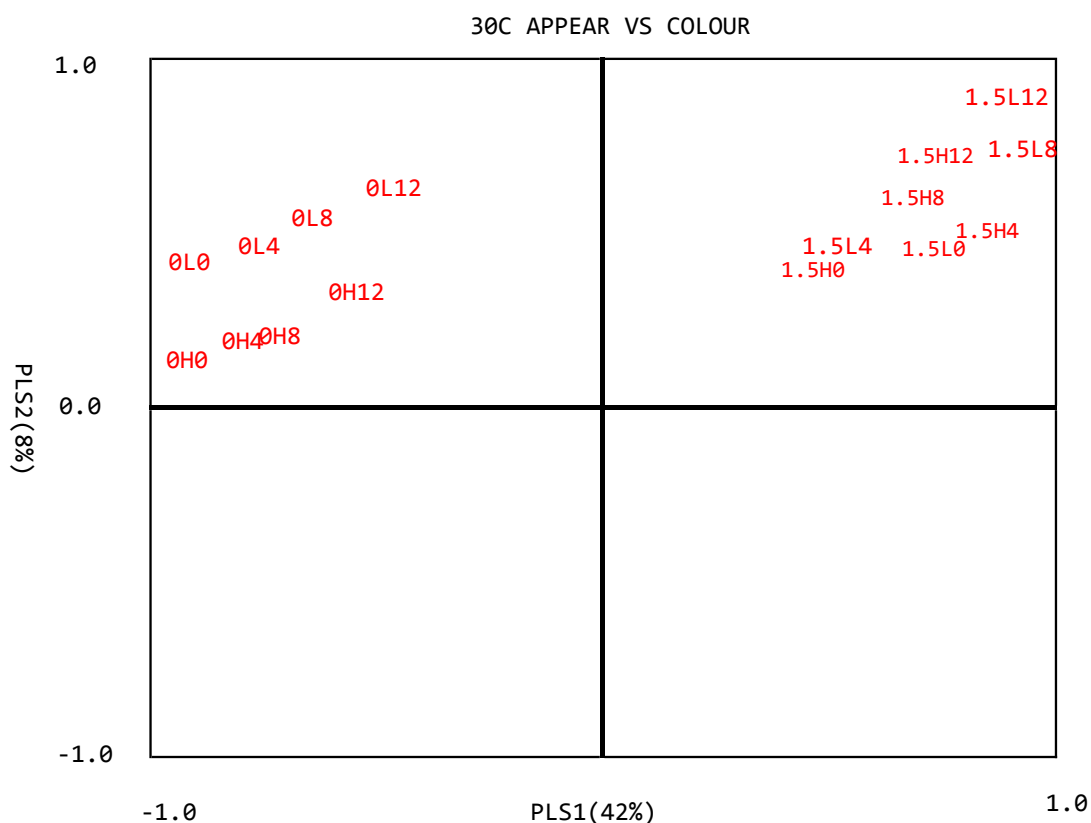
Similarly, when the glucose syrup to sucrose ratio was 1.5, it was seen that starch amount in the jelly gum recipe was effective on appearance liking, whereas gelatine amount was not (Figure 3.37). This result was also related to the opaque appearance of jellies caused by the modified corn starch in the formulation (Hartel and Hartel, 2014).



**Figure 3.37** Appearance liking sensory versus colour change ( $\Delta$ Hue) for jelly gums with glucose syrup:sucrose of 1.5. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 8<sup>th</sup> week, 16<sup>th</sup> week, 24<sup>th</sup> week, 32<sup>nd</sup> week, 40<sup>th</sup> week, 52<sup>nd</sup> week)

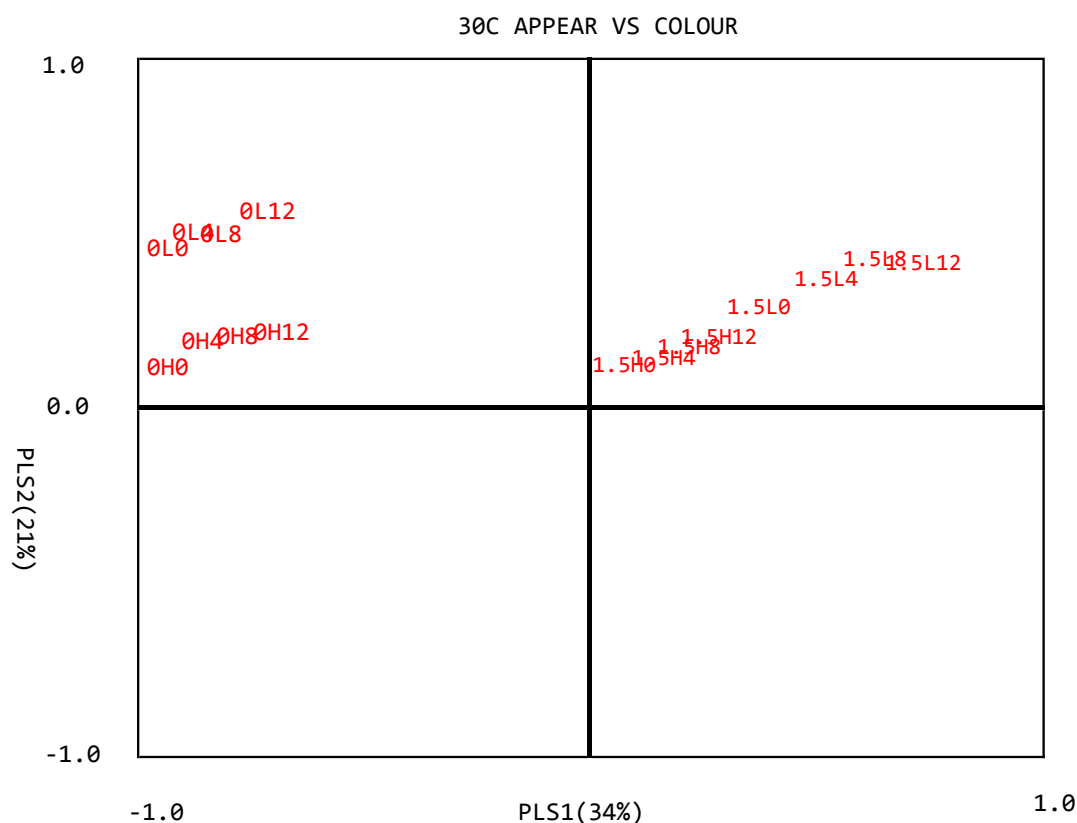
Figure 3.38 and 3.39 show the effect of gelling agents on appearance liking stored at 30°C for the samples with glucose syrup:sucrose ratio of 1.1 and 1.5, respectively. It was seen that starch level was effective on appearance liking of panelists, however, gelatine level was not. This was similar to the long time storage results at laboratory conditions. Appearance liking of jellies decreased as the amount of corn starch was elevated.





**Figure 3.38** Appearance liking sensory versus colour change ( $\Delta$ Hue) for jelly gums with glucose syrup:sucrose of 1.1 stored at 30°C. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 4<sup>th</sup> week, 8<sup>th</sup> week, 12<sup>th</sup> week)

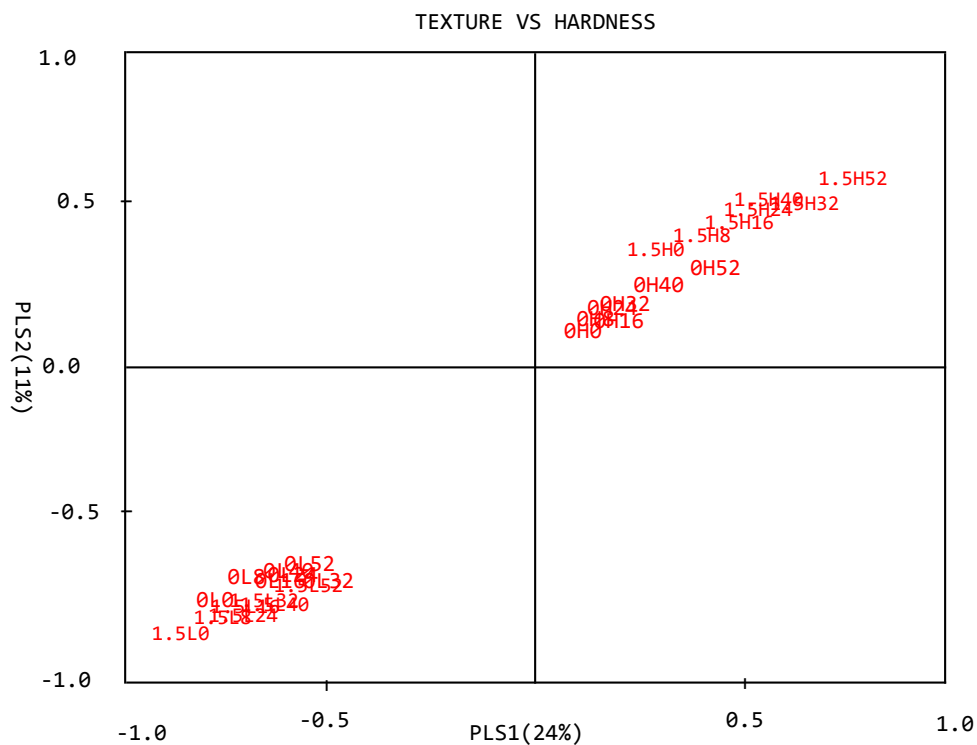
Although magnitudes of colour sensory changes over storage time were small for the long time storage study conducted at laboratory conditions (Figure 3.36 and 3.37), colour sensory measurements showed clear changes with storage time at 30°C, especially for the jellies with glucose syrup to sucrose ratio of 1.1 (Figure 3.38 and 3.39). Therefore, 30°C was found to be an adequate temperature for shelf life studies of jellies containing gelatine and starch.



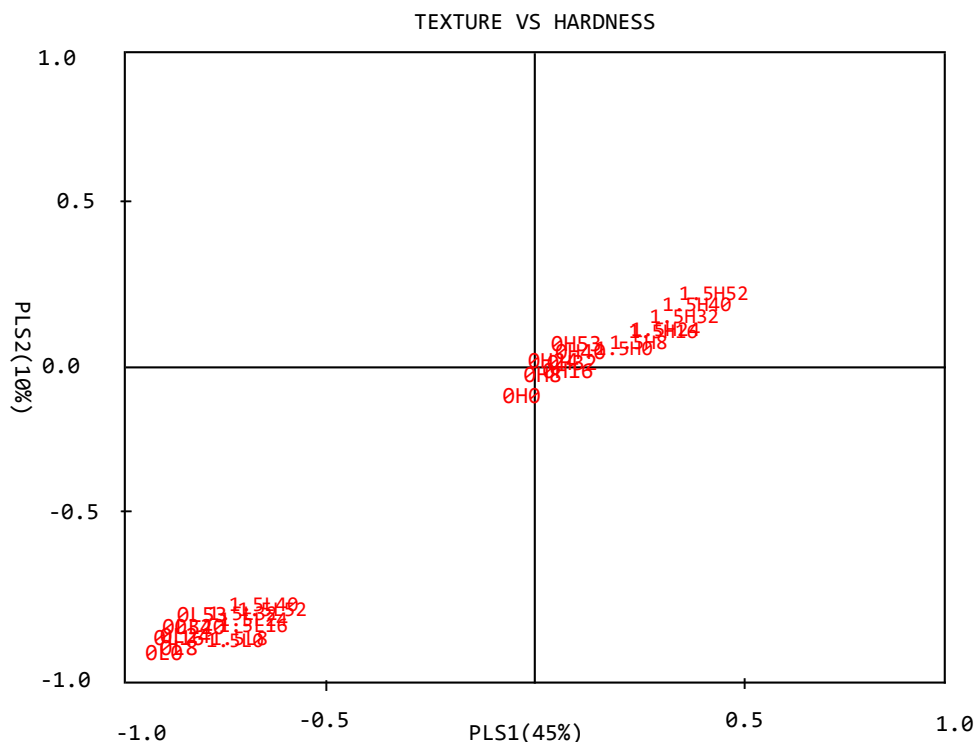
**Figure 3.39** Appearance liking sensory versus colour change ( $\Delta$ Hue) for jelly gums with glucose syrup:sucrose of 1.5 stored at 30°C. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 4<sup>th</sup> week, 8<sup>th</sup> week, 12<sup>th</sup> week)

### 3.10.3 Correlation of Sensory Texture Liking and Hardness of Jelly Gums

Figure 3.40 and 3.41 show the effect of starch level and the effect of gelatine level on texture liking for the jellies with glucose syrup to sucrose ratio of 1.1 and 1.5 stored at 15-22°C for 52 weeks. It was seen that gelatine level was more effective than starch level on texture liking at both sugars ratio of 1.1 and 1.5. Gelatine level positively affected the sensory texture liking of jellies. Similar, in one of the former studies, Siegewein (2010) observed significant improvements in texture scores on incorporation of soy protein at 50% starch replacement.



**Figure 3.40** Texture liking sensory versus hardness change for jelly gums with glucose syrup:sucrose of 1.1. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 8<sup>th</sup> week, 16<sup>th</sup> week, 24<sup>th</sup> week, 32<sup>nd</sup> week, 40<sup>th</sup> week, 52<sup>nd</sup> week)

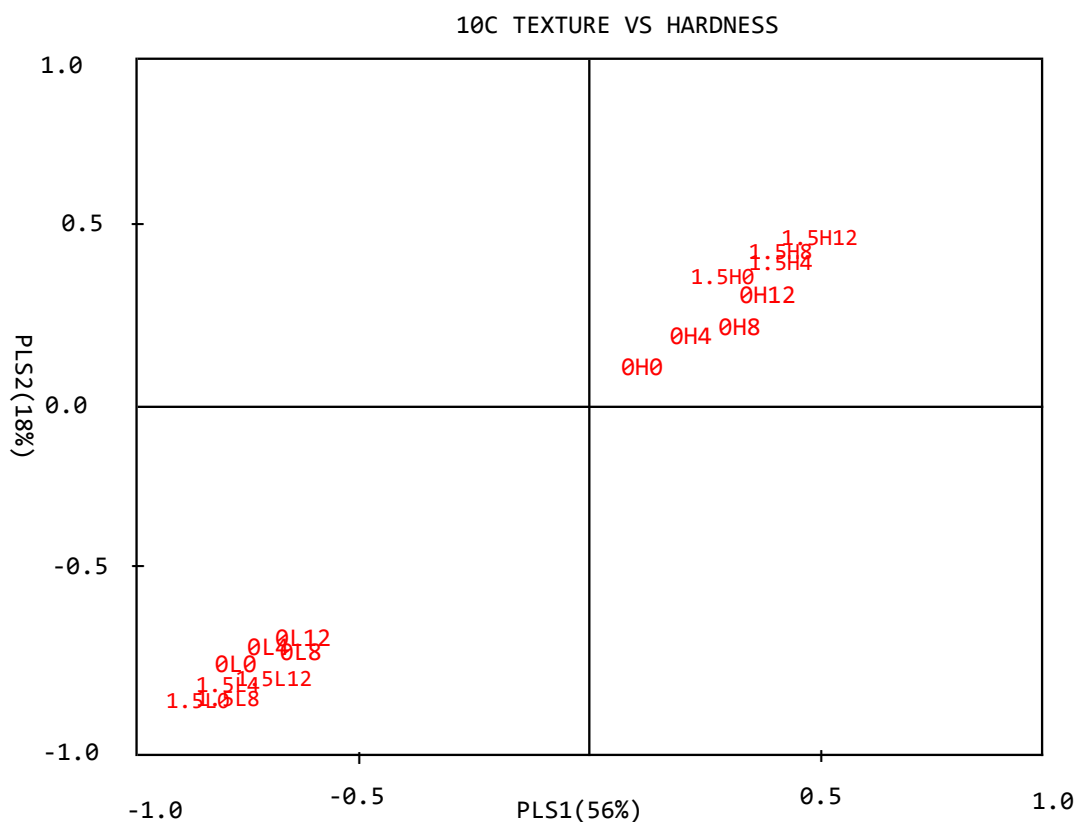


**Figure 3.41** Texture liking sensory versus hardness change for jelly gums with glucose syrup:sucrose of 1.5. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 8<sup>th</sup> week, 16<sup>th</sup> week, 24<sup>th</sup> week, 32<sup>nd</sup> week, 40<sup>th</sup> week, 52<sup>nd</sup> week)

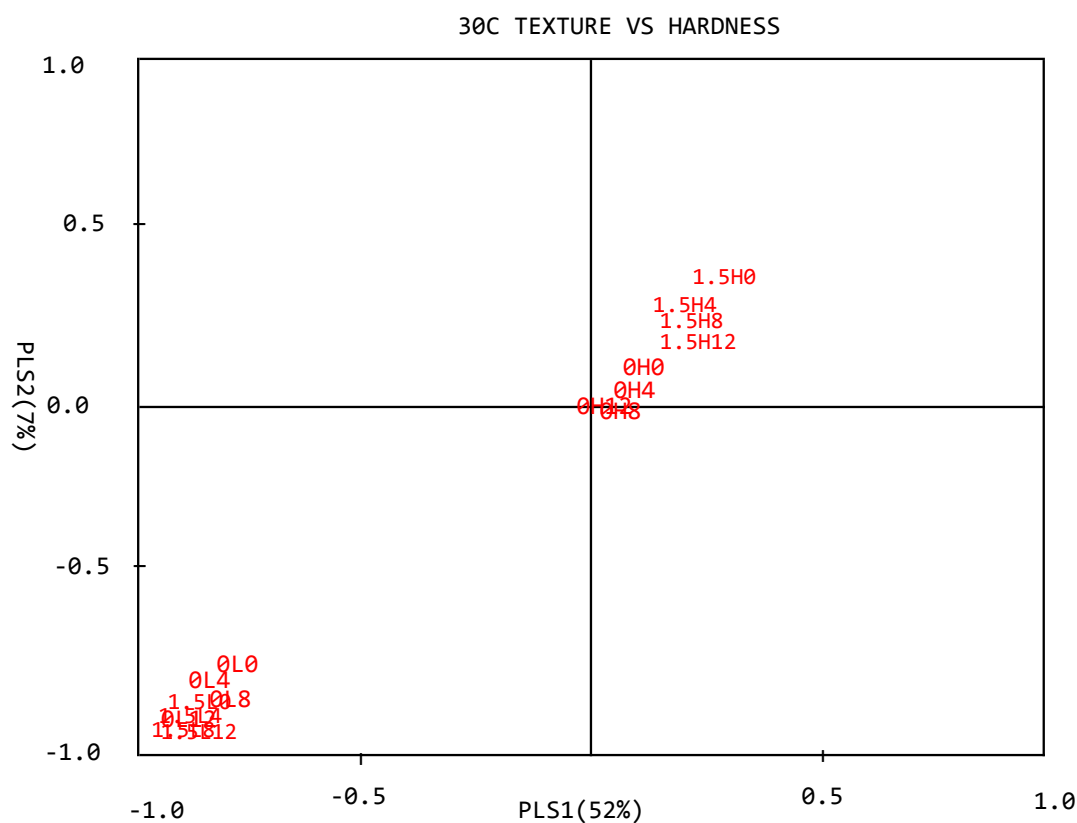
Impact of gelling agents on texture liking of jellies with glucose syrup to sucrose ratio of 1.1 were observed at different temperatures (Figure 3.42 and 3.43). In accordance with the long time storage results performed at 15-22°C, it was observed that gelatine level of samples were more effective than starch level on texture liking at both 10°C and 30°C. In addition, at these mentioned temperatures as the amount of gelatine increased texture liking increased.

Texture sensory values showed changes with time at both 10°C and 30°C for the samples with glucose syrup to sucrose ratio of 1.1 (Figure 3.42 and 3.43). Low temperature positively affected the sensory texture liking scores during storage time similar to the long time storage at laboratory conditions, on the other hand, high

temperature affected as the opposite. Therefore, 10°C and 30°C can be used as test temperatures for shelf life study of jellies having gelatine and starch as the gelling agents in terms of hardness.



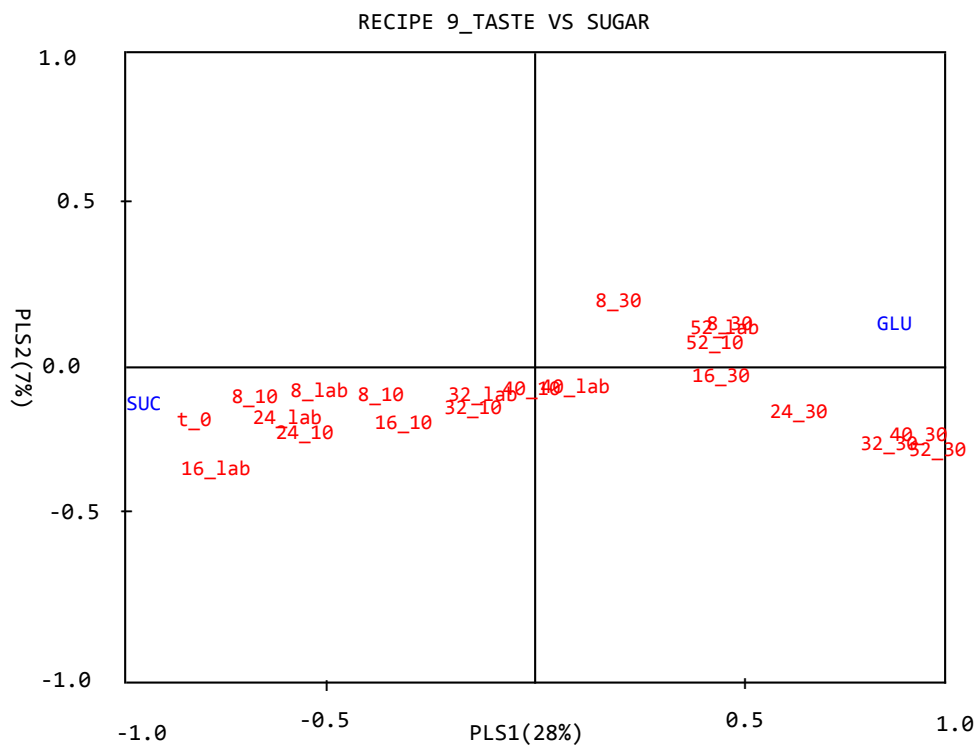
**Figure 3.42** Texture liking sensory versus hardness change for jelly gums with glucose syrup:sucrose of 1:1 stored at 10°C. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 4<sup>th</sup> week, 8<sup>th</sup> week, 12<sup>th</sup> week)



**Figure 3.43** Texture liking sensory versus hardness change for jelly gums with glucose syrup:sucrose of 1.1 stored at 30°C. First letter: Starch level (0 or 1.5), Second letter: Gelatine level (Low or High; Low: 3%, High: 6%), Last digit: storage time in weeks (0<sup>th</sup> week, 4<sup>th</sup> week, 8<sup>th</sup> week, 12<sup>th</sup> week)

### 3.10.4 Correlation of Sensory Taste Liking and Sugar Profile of Jelly Gums

Figure 3.44 demonstrates the impact of storage time and storage condition on taste liking for the jelly gum with 1.5% starch, 6% gelatine and glucose syrup to sucrose ratio of 1.1, which was one of the most acceptable formulations (Table 3.9). It was seen that storage of about 8 weeks at 30°C was equivalent to storage of 52 weeks at 15-22°C when sensory taste liking was correlated with sugar profile.



**Figure 3.44** Taste liking sensory versus glucose and sucrose change for jelly gum with 1.5% starch, 6% gelatine and glucose syrup:sucrose ratio of 1.1. First letter: Storage time in weeks, Second letter: Storage conditions, t\_0: fresh value

As can be seen from Figure 3.44, sugar profile of jellies showed clear changes with temperature and storage time. Hence, especially 30°C could be involved as shelf life test temperatures in terms of taste liking versus sugar profile in confectionery products containing gelatine and corn starch.

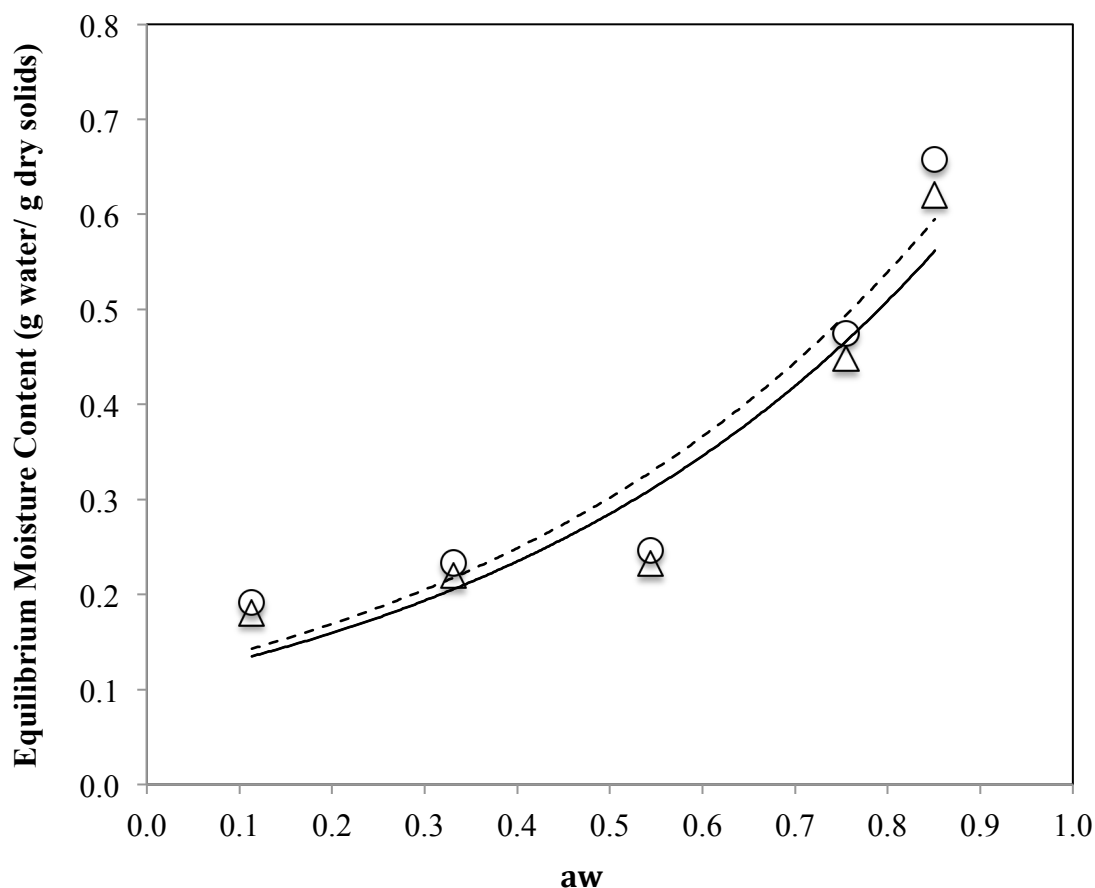
### 3.11 Sorption Isotherms of Jelly Gums

Moisture sorption isotherm is a graphical representation form of the relationship between moisture content and corresponding water activity of a food over a wide range at constant temperature (Karel et al., 1975; Labuza, 1984). It is one of the most fundamental elements in order to understand a drying process based on removal of moisture content in products (Dauthy, 1995).

In this part of the study, moisture sorption isotherm study was carried out for the jelly gum formulations which were scored the highest in terms of overall acceptability in sensory analysis part, namely formulation number 9 with glucose syrup to sucrose ratio of 1.1, 1.5% starch and 6% gelatine and formulation number 12 with glucose syrup to sucrose ratio of 1.5, no starch and 6% gelatine as jellies go through conditioning in the manufacturing, which is also a drying process and very critical in jelly plants.

Figure 3.45 represents the sorption data of jellies with formulation numbers 9 and 12 (Table 2.2). It took 18-20 days to reach equilibrium for samples with formulation number 9 and 23-25 days for samples with formulation number 12.





**Figure 3.45** Moisture sorption isotherms of selected jelly gum formulations at 20°C  
 (Δ): Glucose Syrup:Sucrose= 1.1, Starch= 1.5%, Gelatine= 6%, (O): Glucose Syrup:Sucrose= 1.5, Starch= 0%, Gelatine= 6%. Markers represent data points, lines represent models.

Several models have been used in order to describe sorption behaviour of a product by various researchers (Rizvi, 1995). Halsey equation is one of the widely used equations in modeling experiments for sorption data of both high sugar and starch containing foods (Crapiste and Rotstein, 1982; Levine and Slade 1991; Houssein, 2007). Halsey equation is given in Equation 3.5 as below.

$$M = X_m(-\ln(a_w))^n \quad (3.5)$$

where,  $M$  is equilibrium moisture content (g water / g dry solids),  $X_m$  is monolayer moisture content and  $a_w$  is the water activity.

It was seen that data fitted Halsey equation well with a coefficient of determination value of 0.965. The values of the parameters are represented in Table 3.10, Table F.1 and Table F.2. It was seen that Halsey equation parameters were very similar for both of the recipes; only there was a slight difference in monolayer moisture content, which was important for drying of foods in terms of stability (Labuza, 1972). Between two recipes no starch formulation had slightly higher value of monolayer water content. Similar equation parameters might show that there was no difference in water sorption capacity of both jelly gums.

**Table 3.10** Halsey equation parameters for sorption isotherms of selected formulations at 20°C

Sample	$X_m$	$n$	Standard Error	$R^2$
Formulation 9	0.2174	-0.5658	0.0410	0.965
Glucose Syrup:Sucrose= 1.1				
Starch= 1.5%				
Gelatine=6%				
Formulation 12	0.2303	-0.5658	0.0434	0.965
Glucose Syrup:Sucrose= 1.5				
Starch= 0%				
Gelatine=6%				

## **CHAPTER 4**

### **CONCLUSIONS AND RECOMMENDATIONS**

Multiple regression analysis was performed to understand the impact of ingredients on quality parameters of fresh jelly gums. glucose syrup:sucrose ratio, starch and gelatine level in the formulation affected hardness of jelly gums. Moisture content, water activity, total soluble solids, pH, springiness and gumminess of jellies were influenced by only glucose syrup to sucrose ratio. Glucose syrup:sucrose ratio and starch amount had an impact on cohesiveness.

It was found that jellies having glucose syrup:sucrose ratio of 1.5 were more stable in terms of moisture content, water activity, pH and colour. pH decreased linearly for almost all of the recipes at all storage temperatures. It was seen that relative rate of colour change increased with increase in temperature.  $\Delta$ Hue values of jellies were higher at glucose syrup:sucrose ratio of 1.1 at 30°C, meaning that colour of the samples with glucose syrup to sucrose ratio of 1.1 were less stable.

Jelly gum with glucose syrup:sucrose ratio of 1.5, 1% starch and 6% gelatine was the most stable one in terms of total soluble solid content and pH. Formulation with glucose syrup:sucrose ratio of 1.5, no starch and 6% gelatine was the most stable formulation in terms of hardness and gumminess, which meant that mentioned formulation would require a stable mastication during storage. The highest increase in hardness was observed for jellies with glucose syrup:sucrose ratio of 1.1, no starch and 6% gelatine. Temperature change had very little effect on cohesiveness of jellies at studied temperatures.

Jelly gums with glucose syrup:sucrose ratio of 1.1 had higher glass transition temperatures. It was observed that T<sub>g</sub> increased after storage of 53 weeks significantly. T<sub>g</sub> increased with gelatine and starch concentration. Trends of T<sub>g</sub> of aged samples with respect to starch and gelatine levels were different than those of fresh samples. Glass transition information is valuable in terms of quality and shelf life for confectionery as the composition of sugars in the finished product may be rather different than the individual ingredients due to process and ingredient interactions. Every 10°C temperature increase caused nearly twice increase in glucose and fructose levels. The higher the temperature, faster was the rate of sucrose inversion. It is important to investigate sucrose inversion phenomena while designing a jelly gum formulation since it may affect mouth-feel of consumers.

It was found that formulations with 1.5% starch, 6% gelatine and glucose syrup:sucrose ratio of 1.1 and with no starch, 6% gelatine and glucose syrup:sucrose ratio of 1.5 had the highest scores before and after storage at all conditions. According to the results of correlation of sensory appearance liking and colour, starch level was more effective on appearance scores at long time and 30°C storage. In the correlation of sensory texture liking and hardness, it was seen that gelatine level was more effective than starch level on texture liking at all glucose syrup:sucrose ratios at 10°C and 30°C storage temperature. 10°C storage positively affected the sensory texture liking scores during storage, which was similar to the long time storage at 15-22°C, however, high temperature affected as the opposite.

Diffusion coefficients of jellies having the highest scores for overall acceptability were determined. Samples with glucose syrup:sucrose ratio of 1.1, 1.5% starch and 6% gelatine and with glucose:sucrose ratio of 1.5, no starch and 6% gelatine were found as  $5.93 \times 10^{-10} \text{m}^2/\text{s}$  and  $5.46 \times 10^{-10} \text{m}^2/\text{s}$ , respectively. Moisture sorption isotherm study was also done for these formulations and it was seen that data fitted Halsey equation.

According to the findings of this study, it was concluded that 30°C was found as a

suitable temperature for accelerated shelf life studies of jelly gums containing gelatine and starch. Sensorial changes were correlated with the instrumental results.

Shelf life studies of jelly gums are very limited in the literature. For the future studies, it can be recommended to investigate different gelling agents such as pectins, carrageenan gum and starches with different origins and different sugar syrups like rice syrup and fructose syrups and sweeteners like maltitol syrup on quality and sensorial properties of jelly gums. The studies with these ingredients can be performed under different conditioning and storage temperatures. Besides temperature effect, the effect of relative humidity on shelf life of jellies can be studied. Moreover, microstructure of the jelly gums can be tracked during storage. In addition to these, conditioning can be monitored and investigated at different conditions.



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## APPENDIX A

### MULTIPLE REGRESSIONS

**Table A.1** Multiple regression results for moisture content of jelly gums

**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	Change Statistics			
					F Change	df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.165	1.00	38016.962	10	26	0

- a. Predictors: Gsratio\_starch\_gelatine, GSratio\_square, gelatine\_square, starch\_square, starch, starch\_gelatine, gelatine, GSratio\_gelatine, GSratio\_starch, GSratio
- b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	10326.778	10	1032.678	38016.962	0.000 <sup>c</sup>
	Residual	0.706	26	0.027		
	Total	10327.485 <sup>d</sup>	36			

- a. Dependent Variable: moisture
- b. Linear Regression through the Origin
- c. Predictors: Gsratio\_starch\_gelatine, GSratio\_square, gelatine\_square, starch\_square, starch, starch\_gelatine, gelatine, GSratio\_gelatine, GSratio\_starch, GSratio
- d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Table A.1** (Continued)**Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients	t	Sig.
	B	Std. Error	Beta		
GSratio	22.778	1.224	1.769	18.603	0.000
starch	-1.375	1.116	-0.084	-1.232	0.229
gelatine	-0.046	0.339	-0.013	-0.136	0.893
GSratio_square	-7.198	0.756	-0.768	-9.527	0.000
starch_square	0.098	0.119	0.008	0.823	0.418
gelatine_square	-0.014	0.026	-0.020	-0.534	0.598
GSratio_starch	0.790	0.839	0.064	0.942	0.355
GSratio_gelatine	0.063	0.187	0.023	0.335	0.740
starch_gelatine	0.607	0.237	0.174	2.568	0.016
Gsratio_starch_gelatine	-0.449	0.180	-0.169	-2.497	0.019

a. Dependent Variable: moisture

b. Linear Regression through the Origin

**Table A.2** Multiple regression results for water activity of jelly gums**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	Change Statistics			
					F Change	df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.003	1.00	266502.024	10	26	0

a. Predictors: Gsratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**Table A.2 (Continued)****ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	17.095	10	1.709	266502.024	0.000 <sup>c</sup>
	Residual	0.000	26	0.000		
	Total	17.095 <sup>d</sup>	36			

a. Dependent Variable: aw

b. Linear Regression through the Origin

c. Predictors: Gsratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, Gsratio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients	t	Sig.
	B	Std. Error	Beta		
GSratio	1.194	0.019	2.279	63.445	0.000
starch	-0.006	0.017	-0.010	-0.376	0.710
gelatine	-0.006	0.005	-0.039	-1.096	0.283
Gsratiosq	-0.479	0.012	-1.256	-41.280	0.000
Gsratio_starch	0.002	0.013	0.004	0.156	0.878
starchsq	-0.015	0.002	-0.032	-8.417	0.000
Gsratio_gelatine	0.002	0.003	0.020	0.774	0.446
gelatinesq	-3.704E-5	0.000	-0.001	-0.093	0.927
starch_gelatine	0.004	0.004	0.032	1.232	0.229
Gsratio_starch_gelatine	-0.004	0.003	-0.033	-1.292	0.208

a. Dependent Variable: aw

b. Linear Regression through the Origin

**Table A.3** Multiple regression results for total soluble solids (%) of jelly gums

**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	F Change	Change Statistics		
						df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.166	1.00	899366.400	10	26	0.000

a. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	248471.055	10	24847.106	899366.400	0.000 <sup>c</sup>
	Residual	0.718	26	0.028		
	Total	248471.774 <sup>d</sup>	36			

a. Dependent Variable: TSSs

b. Linear Regression through the Origin

c. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Table A.3 (Continued)**Coefficients<sup>a,b</sup>

Model	Unstandardized Coefficients		Standardized Coefficients	t	Sig.
	B	Std. Error	Beta		
GRatio	134.801	1.235	2.134	109.167	0.000
starch	1.342	1.126	0.017	1.192	0.244
gelatine	0.045	0.342	0.003	0.132	0.896
Gsratosq	-53.419	0.762	-1.162	-70.106	0.000
Gsratio_starch	-0.762	0.846	-0.013	-0.900	0.376
starchsq	-0.101	0.120	-0.002	-0.847	0.405
Gsratio_gelatine	-0.058	0.189	-0.004	-0.306	0.762
gelatinesq	0.013	0.026	0.004	0.500	0.621
starch_gelatine	-0.600	0.239	-0.035	-2.514	0.018
GRatio_starch_gelatine	0.444	0.181	0.034	2.446	0.022

a. Dependent Variable: TSSs

b. Linear Regression through the Origin

**Table A.4** Multiple regression results for pH of jelly gums**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	Change Statistics			
					F Change	df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.014	1.00	224626.188	10	26	0.000

a. Predictors: GRatio\_starch\_gelatine, Gsratosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GRatio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**Table A.4** (Continued)**ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	414.288	10	41.429	224626.188	0.000 <sup>c</sup>
	Residual	0.005	26	0.000		
	Total	414.293 <sup>d</sup>	36			

a. Dependent Variable: pH

b. Linear Regression through the Origin

c. Predictors: GSRatio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSRatio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients		Sig.
	B	Std. Error	Beta	t	
GSRatio	4.960	0.101	1.923	49.160	0.000
starch	-0.125	0.092	-0.038	-1.357	0.187
gelatine	0.006	0.028	0.009	0.224	0.824
Gsratiosq	-1.755	0.062	-0.934	-28.183	0.000
Gsratio_starch	0.070	0.069	0.028	1.016	0.319
starchsq	0.006	0.010	0.002	0.590	0.560
Gsratio_gelatine	-0.001	0.015	-0.003	-0.093	0.926
gelatinesq	-0.001	0.002	-0.010	-0.640	0.528
starch_gelatine	0.052	0.019	0.074	2.663	0.013
GSRatio_starch_gelatine	-0.038	0.015	-0.071	-2.542	0.017

a. Dependent Variable: pH

b. Linear Regression through the Origin

**Table A.5** Multiple regression results for hardness of jelly gums

**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	Change Statistics			
					F Change	df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.002	1.00	618825.810	10	26	0.000

a. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq,

starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	23.234	10	2.323	618825.810	0.000 <sup>c</sup>
	Residual	0.000	26	0.000		
	Total	23.234 <sup>d</sup>	36			

a. Dependent Variable: hardness

b. Linear Regression through the Origin

c. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Table A.5** (Continued)**Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients		Sig.
	B	Std. Error	Beta	t	
GSratio	1.339	0.014	2.192	93.016	0.000
starch	-0.037	0.013	-0.048	-2.816	0.009
gelatine	-0.010	0.004	-0.061	-2.628	0.014
Gsratiosq	-0.548	0.009	-1.232	-61.662	0.000
Gsratio_starch	0.032	0.010	0.055	3.260	0.003
starchsq	0.001	0.001	0.002	0.796	0.433
Gsratio_gelatine	0.010	0.002	0.073	4.328	0.000
gelatinesq	4.023E-15	0.000	0.000	0.000	1.000
starch_gelatine	0.005	0.003	0.032	1.884	0.071
GSratio_starch_gelatine	-0.005	0.002	-0.038	-2.252	0.033

a. Dependent Variable: hardness

b. Linear Regression through the Origin

**Table A.6** Multiple regression results for cohesiveness of jelly gums**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	Change Statistics			
					F Change	df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.002	1.00	436834.239	10	26	0.000

a. Predictors: Gsratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, Gsratio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.



**Table A.6 (Continued)****ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	11.068	10	1.107	436834.239	0.000 <sup>c</sup>
	Residual	0.000	26	0.000		
	Total	11.068 <sup>d</sup>	36			

a. Dependent Variable: cohesiveness

b. Linear Regression through the Origin

c. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients		Sig.
	B	Std. Error	Beta	t	
GSratio	0.884	0.012	2.096	74.736	0.000
starch	0.028	0.011	0.053	2.614	0.015
gelatine	0.005	0.003	0.043	1.563	0.130
Gsratiosq	-0.348	0.007	-1.134	-47.681	0.000
Gsratio_starch	-0.017	0.008	-0.041	-2.058	0.050
starchsq	-0.002	0.001	-0.006	-1.939	0.063
Gsratio_gelatine	-0.001	0.002	-0.013	-0.659	0.516
gelatinesq	0.000	0.000	-0.016	-1.481	0.151
starch_gelatine	-0.007	0.002	-0.063	-3.127	0.004
GSratio_starch_gelatine	0.005	0.002	0.055	2.742	0.011

a. Dependent Variable: cohesiveness

b. Linear Regression through the Origin

**Table A.7** Multiple regression results for springiness of jelly gums

**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	F Change	Change Statistics		
						df1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.011	1.00	300564.222	10	26	0.000

a. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq,

starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	379.467	10	37.947	300564.222	0.000 <sup>c</sup>
	Residual	0.003	26	0.000		
	Total	379.470 <sup>d</sup>	36			

a. Dependent Variable: springiness

b. Linear Regression through the Origin

c. Predictors: GSratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GSratio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Table A.7 (Continued)****Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients		Sig.
	B	Std. Error	Beta	t	
GSratio	4.752	0.083	1.925	56.925	0.000
starch	-0.047	0.076	-0.015	-0.618	0.542
gelatine	0.006	0.023	0.008	0.255	0.801
Gsratiosq	-1.673	0.052	-0.931	-32.474	0.000
Gsratio_starch	0.018	0.057	0.008	0.323	0.749
starchsq	0.002	0.008	0.001	0.275	0.786
Gsratio_gelatine	-0.008	0.013	-0.015	-0.606	0.549
gelatinesq	0.000	0.002	-0.003	-0.210	0.835
starch_gelatine	0.037	0.016	0.056	2.311	0.029
GSratio_starch_gelatine	-0.027	0.012	-0.054	-2.233	0.034

a. Dependent Variable: springiness

b. Linear Regression through the Origin

**Table A.8** Multiple regression results for gumminess of jelly gums**Model Summary**

R	R <sup>2b</sup>	Adjusted R <sup>2</sup>	Std. Error of the Estimate	R <sup>2</sup> Change	Change Statistics			
					F Change	df 1	df 2	Sig.F Change
1.00 <sup>a</sup>	1.00	1.00	0.002	1.00	161341.545	10	26	0.000

a. Predictors: Gsratio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, Gsratio

b. For regression through the origin (the no-intercept model), R Square measures the proportion of the variability in the dependent variable about the origin explained by regression. This CANNOT be compared to R Square for models which include an intercept.

**Table A.8** (Continued)**ANOVA<sup>a,b</sup>**

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	7.043	10	0.704	161341.545	0.000 <sup>c</sup>
	Residual	0.000	26	0.000		
	Total	7.043 <sup>d</sup>	36			

a. Dependent Variable: gumminess

b. Linear Regression through the Origin

c. Predictors: GRatio\_starch\_gelatine, Gsratiosq, gelatinesq, starchsq, starch, starch\_gelatine, gelatine, Gsratio\_gelatine, Gsratio\_starch, GRatio

d. This total sum of squares is not corrected for the constant because the constant is zero for regression through the origin.

**Coefficients<sup>a,b</sup>**

Model	Unstandardized Coefficients		Standardized Coefficients		Sig.
	B	Std. Error	Beta	t	
GRatio	0.764	0.016	2.273	49.252	0.000
starch	-0.016	0.014	-0.037	-1.123	0.272
gelatine	-0.005	0.004	-0.056	-1.233	0.229
Gsratiosq	-0.317	0.010	-1.295	-33.101	0.000
Gsratio_starch	0.010	0.011	0.031	0.952	0.350
starchsq	0.003	0.002	0.011	2.216	0.036
Gsratio_gelatine	0.002	0.002	0.025	0.753	0.458
gelatinesq	0.000	0.000	0.020	1.128	0.270
starch_gelatine	-0.001	0.003	-0.014	-0.437	0.666
GRatio_starch_gelatine	0.001	0.002	0.017	0.522	0.606

a. Dependent Variable: gumminess

b. Linear Regression through the Origin

## APPENDIX B

### ANOVA AND DUNCAN TABLES FOR GLASS TRANSITION TEMPERATURE

**Table B.1** ANOVA and Duncan's Multiple Range Test for Tg of jelly gums with for Glucose/ Sucrose Ratio= 1.1 measured at week 0 (fresh) and at week 53 (aged).

Class	Level	Values
Formulation	18	1, 2, 3, 4, 5, 6, 7, 8, 9, 153, 253, 353, 453, 553, 653, 753, 853, 953
Replication	3	1, 2, 3
Number of observations in data set= 54		

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	407.813	17	23.989	14.549	.000
Within Groups	59.360	36	1.649		
Total	467.173	53			

**Table B.1** (Continued)**Duncan Results**

Means with the same letter are not significantly different.

<b>Formulation</b>	<b>N</b>	<b>alpha = 0.05</b>	
		<b>Mean</b>	<b>Grouping</b>
1 (G/S=1.1, starch 0%, gelatine 3% @week 0)	3	20.000	A
7 (G/S=1.1, starch 1.5%, gelatine 3% @week 0)	3	20.200	A
2 (G/S=1.1, starch 0%, gelatine 4.5% @week 0)	3	20.500	A
8 (G/S=1.1, starch 1.5%, gelatine 4.5% @week 0)	3	21.100	A
3 (G/S=1.1, starch 0%, gelatine 6% @week 0)	3	21.300	A
5 (G/S=1.1, starch 1%, gelatine 4.5% @week 0)	3	21.500	A
9 (G/S=1.1, starch 1.5%, gelatine 6% @week 0)	3	21.600	A
6 (G/S=1.1, starch 1%, gelatine 6% @week 0)	3	21.700	A
4 (G/S=1.1, starch 1%, gelatine 3% @week 0)	3	22.300	A
653 (G/S=1.1, starch 1%, gelatine 6% @week 53)	3	25.800	B
353 (G/S=1.1, starch 0%, gelatine 6% @week 53)	3	26.000	B
553 (G/S=1.1, starch 1%, gelatine 4.5% @week 53)	3	26.000	B
153 (G/S=1.1, starch 0%, gelatine 3% @week 53)	3	26.500	B
453 (G/S=1.1, starch 1%, gelatine 3% @week 53)	3	26.500	B
753 (G/S=1.1, starch 1.5%, gelatine 3% @week 53)	3	26.500	B
253 (G/S=1.1, starch 0%, gelatine 4.5% @week 53)	3	26.600	B
853 (G/S=1.1, starch 1.5%, gelatine 4.5% @week 53)	3	27.000	B
953 (G/S=1.1, starch 1.5%, gelatine 6% @week 53)	3	27.500	B

**Table B.2** ANOVA and Duncan's Multiple Range Test Table for Tg of jelly gums with for Glucose Syrup:Sucrose Ratio= 1.5 measured at week 0 (fresh) and at week 53 (aged).

Class	Level	Values
Formulation	18	10, 11, 12, 13, 14, 15, 16, 17, 18, 1053, 1153, 1253, 1353, 1453, 1553, 1653, 1753, 1853
Replication	3	1, 2, 3

Number of observations in data set= 54

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	644.253	17	37.897	64.843	.000
Within Groups	21.040	36	.584		
Total	665.293	53			

### Duncan Results

Means with the same letter are not significantly different

Formulation	N	Mean	Grouping
10 (G/S=1.5, starch 0%, gelatine 3% @week 0)	3	-32.000	A
13 (G/S=1.5, starch 1%, gelatine 3% @week 0)	3	-30.100	B
11 (G/S=1.5, starch 0%, gelatine 4.5% @week 0)	3	-30.000	B
12 (G/S=1.5, starch 0%, gelatine 6% @week 0)	3	-29.500	BC
14 (G/S=1.5, starch 1%, gelatine 4.5% @week 0)	3	-29.300	BC
1353 (G/S=1.5, starch 1%, gelatine 3% @week 53)	3	-29.300	BC
1453 (G/S=1.5, starch 1%, gelatine 4.5% @week 53)	3	-28.500	BC
16 (G/S=1.5, starch 1.5%, gelatine 3% @week 0)	3	-28.300	BC
15 (G/S=1.5, starch 1%, gelatine 6% @week 0)	3	-27.400	C
17 (G/S=1.5, starch 1.5%, gelatine 4.5% @week 0)	3	-25.500	D
1153 (G/S=1.5, starch 0%, gelatine 4.5% @week 53)	3	-25.400	D
1653 (G/S=1.5, starch 1.5%, gelatine 3% @week 53)	3	-25.100	DE
1553 (G/S=1.5, starch 1%, gelatine 6% @week 53)	3	-24.300	DE
1253 (G/S=1.5, starch 0%, gelatine 6% @week 53)	3	-23.300	EF
18 (G/S=1.5, starch 1.5%, gelatine 6% @week 0)	3	-23.100	EF
1753 (G/S=1.5, starch 1.5%, gelatine 4.5% @week 53)	3	-22.100	FG
1053 (G/S=1.5, starch 0%, gelatine 3% @week 53)	3	-20.500	GH
1853 (G/S=1.5, starch 1.5%, gelatine 6% @week 53)	3	-20.100	H





## APPENDIX C

### ANOVA AND DUNCAN TEST TABLES FOR TEXTURE

**Table C.1** ANOVA and Duncan Test Table for hardness of jelly gum formulations 1, 3, 7, 9, 10, 12, 16 and 18 (Table 2.2) at week 0, 24 and 52.

Class	Level	Values
Formulation	24	1_0, 3_0, 7_0, 9_0, 10_0, 12_0, 16_0, 18_0, 1_24, 3_24, 7_24, 9_24, 10_24, 12_24, 16_24, 18_24, 1_52, 3_52, 7_52, 9_52, 10_52, 12_52, 16_52, 18_52

Replication                      3                      1, 2, 3

Number of observations in data set= 72

	Sum of Squares	df	Mean Square	F	p-level	Fcrit
Between Groups	0.01639	23	0.00071	57	0	1.75676
Within Groups	0.00060	48	0.00001			
Total	0.01699	71				

#### Duncan Results

Means with the same letter are not significantly different.

Formulation	N	alpha = 0.05	
		Mean	Grouping
10_0	3	0.79	F
12_0	3	0.80	EF
16_0	3	0.80	EF
18_0	3	0.80	EF
10_24	3	0.80	EF
12_24	3	0.80	EF
1_0	3	0.81	DE
3_0	3	0.81	DE
7_0	3	0.81	DE
9_0	3	0.81	DE
9_24	3	0.81	DE

**Table C.1 (Continued)****(Continued) Duncan Results**

Means with the same letter are not significantly different.

Formulation	N	alpha = 0.05	
		Mean	Grouping
10_52	3	0.81	DE
12_52	3	0.81	DE
1_24	3	0.82	CD
3_24	3	0.82	CD
7_24	3	0.82	CD
16_24	3	0.82	CD
18_24	3	0.82	CD
9_52	3	0.83	BC
16_52	3	0.83	BC
18_52	3	0.83	BC
1_52	3	0.84	AB
7_52	3	0.84	AB
3_52	3	0.85	A

**Table C.2** ANOVA and Duncan Test Table for cohesiveness of jelly gum formulations 1, 3, 7, 9, 10, 12, 16 and 18 (Table 2.2) at week 0, 24 and 52.

Class	Level	Values
Formulation	24	1_0, 3_0, 7_0, 9_0, 10_0, 12_0, 16_0, 18_0, 1_24, 3_24, 7_24, 9_24, 10_24, 12_24, 16_24, 18_24, 1_52, 3_52, 7_52, 9_52, 10_52, 12_52, 16_52, 18_52
Replication	3	1, 2, 3

Number of observations in data set= 72

**Table C.2 (Continued)**

	Sum of					
	Squares	df	Mean Square	F	p-level	Fcrit
Between Groups	0.00375	23	0.00016	9.78261	2.58672E-11	1.75676
Within Groups	0.00080	48	0.00002			
Total	0.00455	71				

**Duncan Results**

Means with the same letter are not significantly different.

<b>alpha = 0.05</b>			
<b>Formulation</b>	<b>N</b>	<b>Mean</b>	<b>Grouping</b>
9_0	3	0.55	B
10_0	3	0.55	B
12_0	3	0.55	B
16_0	3	0.55	B
18_0	3	0.55	B
10_24	3	0.55	B
12_24	3	0.55	B
10_52	3	0.55	B
12_52	3	0.55	B
1_0	3	0.56	AB
3_0	3	0.56	AB
7_0	3	0.56	AB
3_24	3	0.56	AB
9_24	3	0.56	AB
16_24	3	0.56	AB
18_24	3	0.56	AB
16_52	3	0.56	AB
1_24	3	0.57	A
7_24	3	0.57	A
1_52	3	0.57	A
3_52	3	0.57	A
7_52	3	0.57	A
9_52	3	0.57	A
18_52	3	0.57	A

**Table C.3** ANOVA and Duncan Test Table for springiness of jelly gum formulations 1, 3, 7, 9, 10, 12, 16 and 18 (Table 2.2) at week 0, 24 and 52.

Class	Level	Values
Formulation	24	1_0, 3_0, 7_0, 9_0, 10_0, 12_0, 16_0, 18_0, 1_24, 3_24, 7_24, 9_24, 10_24, 12_24, 16_24, 18_24, 1_52, 3_52, 7_52, 9_52, 10_52, 12_52, 16_52, 18_52
Replication	3	1, 2, 3
Number of observations in data set= 72		

	Sum of Squares	df	Mean Square	F	p-level	Fcrit
Between Groups	0.73061	23	0.03177	2028.99631	0	1.75676
Within Groups	0.00075	48	0.00002			
Total	0.73136	71				

### Duncan Results

Means with the same letter are not significantly different.

Formulation	N	Mean	alpha = 0.05
			Grouping
7_52	3	3.00	GH
1_52	3	3.02	G
3_52	3	3.05	F
9_52	3	3.05	F
18_52	3	3.05	F
7_24	3	3.07	EF
1_24	3	3.08	EF
3_24	3	3.10	EF
18_24	3	3.10	EF
16_52	3	3.13	E
9_24	3	3.14	E
16_24	3	3.14	E
3_0	3	3.18	D
1_0	3	3.19	CD
7_0	3	3.19	CD
9_0	3	3.22	C

**Table C.3 (Continued)****(Continued) Duncan Results**

Means with the same letter are not significantly different.

Formulation	N	Mean	alpha = 0.05
			Grouping
18_0	3	3.25	B
10_52	3	3.25	B
10_24	3	3.28	AB
12_52	3	3.28	AB
12_24	3	3.29	AB
16_0	3	3.30	AB
12_0	3	3.31	AB
10_0	3	3.34	A

**Table C.4** ANOVA and Duncan Test Table for gumminess of jelly gum formulations 1, 3, 7, 9, 10, 12, 16 and 18 (Table 2.2) at week 0, 24 and 52.

Class	Level	Values
Formulation	24	1_0, 3_0, 7_0, 9_0, 10_0, 12_0, 16_0, 18_0, 1_24, 3_24, 7_24, 9_24, 10_24, 12_24, 16_24, 18_24, 1_52, 3_52, 7_52, 9_52, 10_52, 12_52, 16_52, 18_52
Replication	3	1, 2, 3
Number of observations in data set= 72		

	Sum of Squares	df	Mean Square	F	p-level	Fcrit
Between Groups	0.01475	23	0.00064	153.91304	0	1.75676
Within Groups	0.00020	48	4.16667E-6			
Total	0.01495	71				

**Table C.4 (Continued)****Duncan Results**

Means with the same letter are not significantly different.

Formulation	N	alpha = 0.05	
		Mean	Grouping
10_0	3	0,43	E
10_24	3	0,44	DE
12_0	3	0,44	DE
12_24	3	0,44	DE
16_0	3	0,44	DE
18_0	3	0,44	DE
10_52	3	0,45	CD
12_52	3	0,45	CD
3_0	3	0,45	CD
7_0	3	0,45	CD
9_0	3	0,45	CD
1_0	3	0,45	CD
3_24	3	0,46	BC
9_24	3	0,46	BC
16_24	3	0,46	BC
16_52	3	0,46	BC
18_24	3	0,46	BC
1_24	3	0,47	AB
7_24	3	0,47	AB
9_52	3	0,47	AB
18_52	3	0,47	AB
1_52	3	0,48	A
3_52	3	0,48	A
7_52	3	0,48	A

## APPENDIX D

### 3-WAY ANOVA TABLES FOR SUGAR CONTENTS

#### D.1 Sucrose (%)

**Table D.1.1** ANOVA Table for sucrose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 10°C storage.

##### Summary

Response	sucrose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

##### ANOVA

Source of Variation	SS	d.f.	MS	F	p-level	Fcrit
Factor #1 (age)	0.72544	2	0.36272	0.08123	0.92232	3.55456
Factor #2 (GSratio)	506.37136	1	506.37136	113.39636	3.36575E-9	4.41387
Factor #3 (starch)	134.73719	2	67.3686	15.08646	0.00014	4.41387
Factor #1 + #2 (age x GSRatio)	0.42417	2	0.21209	0.04749	0.95373	3.55456
Factor #1 + #3 (age x starch)	0.01581	4	0.00395	0.00089	1.00000	2.92774
Factor #2 + #3 (GSratio x starch)	0.11825	2	0.05913	0.01324	0.98686	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.01495	4	0.00374	0.00084	1.00000	0.00084
Within Groups	80.37899	18	4.4655			
Total	722.78616	35	20.65103			
Omega squared for combined effect	0.77895					

**Table D.1.1** (Continued)

Source of Variation	Omega Sqr.
Factor #1 (age)	0.00000
Factor #2 (GSratio)	0.69014
Factor #3 (starch)	0.17299
Factor #1 + #2 (age x GSRatio)	0.00000
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

## Comparisons among groups (Factor 3 - starch)

## Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	4.68365	7.67786	0.00014	Yes
0 vs 1.5	2.9661	4.86229	0.00789	Yes
1 vs 1.5	-1.71755	2.81556	0.14322	No

**Table D.1.2** ANOVA Table for sucrose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 20°C storage.**Summary**

Response	sucrose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed



**Table D.1.2 (Continued)****ANOVA**

Source of Variation	SS	d. f.	MS	F	p-level	Fcrit
Factor #1 (age)	16.21618	2	8.10809	1.80615	0.19282	3.55456
Factor #2 (GSratio)	458.86421	1	458.86421	102.21590	7.54294E-9	4.41387
Factor #3 (starch)	111.42206	2	55.71103	12.4101	0.00041	4.41387
Factor #1 + #2 (age x GStratio)	0.23652	2	0.11826	0.02634	0.97404	3.55456
Factor #1 + #3 (age x starch)	1.56447	4	0.39112	0.08712	0.98533	2.92774
Factor #2 + #3 (GSratio x starch)	1.58489	2	0.79245	0.17652	0.83961	3.55456
Factor #1 + #2 + #3 (age x GStratio x starch)	1.10988	4	0.27747	0.06181	0.99232	0.06181
Within Groups	80.80500	18	4.48917			
Total	671.80321	35	19.19438			
Omega squared for combined effect	0.76104					

Source of Variation	Omega Sqr.
Factor #1 (age)	0.01070
Factor #2 (GSratio)	0.67186
Factor #3 (starch)	0.15148
Factor #1 + #2 (age x GStratio)	0.00000
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GStratio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

**Comparisons among groups (Factor 3 - starch)****Tukey HSD**

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	4.28474	7.00538	0.00031	Yes
0 vs 1.5	2.54052	4.15366	0.02288	Yes
1 vs 1.5	-1.74422	2.85173	0.13694	No

**Table D.1.3** ANOVA Table for sucrose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 30°C storage.

### Summary

Response	sucrose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

### ANOVA

Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	277.94335	2	138.97167	30.95712	1.49223E-6	3.55456
Factor #2 (GSratio)	229.76904	1	229.76904	51.18300	1.15828E-6	4.41387
Factor #3 (starch)	113.80199	2	56.90099	12.67518	0.00037	4.41387
Factor #1 + #2 (age x GSRatio)	30.56056	2	15.28028	3.40381	0.05574	3.55456
Factor #1 + #3 (age x starch)	0.69149	4	0.17287	0.03851	0.99690	2.92774
Factor #2 + #3 (GSratio x starch)	0.17213	2	0.08606	0.01917	0.98103	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.04413	4	0.01103	0.00246	0.99999	0.00246
Within Groups	80.80500	18	4.48917			
Total	733.78767	35	20.96536			
Omega squared for combined effect	0.78110					

Source of Variation	Omega Sqr.
Factor #1 (age)	0.36431
Factor #2 (GSratio)	0.30514
Factor #3 (starch)	0.14198
Factor #1 + #2 (age x GSRatio)	0.02923
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

### Comparisons among groups (Factor 1 - age)

#### Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
0 vs 24	3.03542	4.96279	0.00677	Yes
0 vs 53	6.79337	11.10690	0.00005	Yes
24 vs 53	3.75795	6.14411	0.00109	Yes

**Table D.1.3** (Continued)

Comparisons among groups (Factor 3 - starch)

Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	4.30953	7.04592	0.00029	Yes
0 vs 1.5	2.69901	4.41278	0.01558	Yes
1 vs 1.5	-1.61052	2.63315	0.17845	No

## D.2 Glucose (%)

**Table D.2.1** ANOVA Table for glucose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 10°C storage.

### Summary

Response	glucose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

### ANOVA

Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	0.01453	2	0.00726	0.01105	0.98901	3.55456
Factor #2 (GSratio)	5.80948	1	5.80948	8.83968	0.00815	4.41387
Factor #3 (starch)	27.36502	2	13.68251	20.81924	0.00002	4.41387
Factor #1 + #2 (age x GSRatio)	0.00030	2	0.00015	0.00023	0.99977	3.55456
Factor #1 + #3 (age x starch)	0.00067	4	0.00017	0.00025	1.00000	2.92774
Factor #2 + #3 (GSratio x starch)	0.29984	2	0.14992	0.22812	0.79830	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00066	4	0.00016	0.00025	1.00000	0.00025
Within Groups	11.82969	18	0.65720			
Total	45.32018	35	1.29486			
Omega squared for combined effect	0.48541					

**Table D.2.1** (Continued)

Source of Variation	Omega Sqr.			
Factor #1 (age)	0.00000			
Factor #2 (GSratio)	0.11206			
Factor #3 (starch)	0.56660			
Factor #1 + #2 (age x GSratio)	0.00000			
Factor #1 + #3 (age x starch)	0.00000			
Factor #2 + #3 (GSratio x starch)	0.00000			
Factor #1 + #2 + #3 (age x GSratio x starch)	0.00000			
Within Groups				
Total				
Omega squared for combined effect				
Comparisons among groups (Factor 3 - starch)				
Tukey HSD				
Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	-1.66093	7.09729	0.00027	Yes
0 vs 1.5	-1.99305	8.51645	0.00007	Yes
1 vs 1.5	-0.33212	1.41915	0.58416	No

**Table D.2.2** ANOVA Table for glucose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 20°C storage.**Summary**

Response	glucose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

**ANOVA**

Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	0.08441	2	0.04221	0.06445	0.93779	3.55456
Factor #2 (GSratio)	6.17753	1	6.17753	9.43383	0.00658	4.41387
Factor #3 (starch)	28.37859	2	14.18929	21.66876	0.00002	4.41387
Factor #1 + #2 (age x GSRatio)	0.00225	2	0.00112	0.00172	0.99829	3.55456
Factor #1 + #3 (age x starch)	0.01237	4	0.00309	0.00472	0.99995	2.92774
Factor #2 + #3 (GSratio x starch)	0.40068	2	0.20034	0.30595	0.74018	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00599	4	0.0015	0.00229	0.99999	0.00229
Within Groups	11.78689	18	0.65483			
Total	46.84870	35	1.33853			
Omega squared for combined effect	0.50375					

**Table D.2.2** (Continued)

Source of Variation	Omega Sqr.
Factor #1 (age)	0.00000
Factor #2 (GSratio)	0.11626
Factor #3 (starch)	0.56983
Factor #1 + #2 (age x GRatio)	0.00000
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GRatio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

Comparisons among groups (Factor 3 - starch)

Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	-1.69374	7.25062	0.00023	Yes
0 vs 1.5	-2.02829	8.68275	0.00007	Yes
1 vs 1.5	-0.33454	1.43213	0.57860	No

**Table D.2.3** ANOVA Table for glucose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 30°C storage.**Summary**

Response	glucose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

**Table D.2.3 (Continued)****ANOVA**

Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	0.44988	2	0.22494	0.34351	0.71383	3.55456
Factor #2 (GSratio)	5.17810	1	5.17810	7.90759	0.01154	4.41387
Factor #3 (starch)	27.11383	2	13.55691	20.70304	0.00002	4.41387
Factor #1 + #2 (age x GSRatio)	0.01200	2	0.00600	0.00916	0.99089	3.55456
Factor #1 + #3 (age x starch)	0.00095	4	0.00024	0.00036	1.00000	2.92774
Factor #2 + #3 (GSratio x starch)	0.46088	2	0.23044	0.35191	0.70808	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.01721	4	0.00430	0.00657	0.99991	0.00657
Within Groups	11.78689	18	0.65483			
Total	45.01974	35	1.28628			
Omega squared for combined effect	0.48388					

Source of Variation	Omega Sqr.
Factor #1 (age)	0.00000
Factor #2 (GSratio)	0.09903
Factor #3 (starch)	0.56496
Factor #1 + #2 (age x GSRatio)	0.00000
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

**Comparisons among groups (Factor 3 - starch)****Tukey HSD**

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	-1.64856	7.05721	0.00029	Yes
0 vs 1.5	-1.98657	8.50414	0.00007	Yes
1 vs 1.5	-0.33800	1.44692	0.57228	No

### D.3 Fructose (%)

**Table D.3.1** ANOVA Table for fructose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 10°C storage.

<b>Summary</b>						
Response	fructose(%)					
Factor #1	age				Fixed	
Factor #2	GSratio				Fixed	
Factor #3	starch				Fixed	
<b>ANOVA</b>						
Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	0.00858	2	0.00429	0.00651	0.99351	3.55456
Factor #2 (GSratio)	3.82736	1	3.82736	5.81196	0.02683	4.41387
Factor #3 (starch)	29.21431	2	14.60715	22.18142	0.00001	4.41387
Factor #1 + #2 (age x GStratio)	0.00040	2	0.00020	0.00030	0.99970	3.55456
Factor #1 + #3 (age x starch)	0.00037	4	0.00009	0.00014	1.00000	2.92774
Factor #2 + #3 (GSratio x starch)	0.33997	2	0.16998	0.25813	0.77531	3.55456
Factor #1 + #2 + #3 (age x GStratio x starch)	0.00010	4	0.00003	0.00004	1.00000	0.00004
Within Groups	11.85356	18	0.65853			
Total	45.24464	35	1.29270			
Omega squared for combined effect	0.48354					
Source of Variation				Omega Sqr.		
Factor #1 (age)				0.00000		
Factor #2 (GSratio)				0.06903		
Factor #3 (starch)				0.60774		
Factor #1 + #2 (age x GStratio)				0.00000		
Factor #1 + #3 (age x starch)				0.00000		
Factor #2 + #3 (GSratio x starch)				0.00000		
Factor #1 + #2 + #3 (age x GStratio x starch)				0.00000		
Within Groups				0.00000		
Total				0.06903		
Omega squared for combined effect						

**Table D.3.1** (Continued)

Comparisons among groups (Factor 3 - starch)

Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	-1.87907	8.02132	0.00010	Yes
0 vs 1.5	-1.94134	8.28711	0.00008	Yes
1 vs 1.5	-0.06226	0.26579	0.98082	No

**Table D.3.2** ANOVA Table for fructose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 20°C storage.

### Summary

Response	fructose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

### ANOVA

Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	0.05109	2	0.02555	0.03898	0.96185	3.55456
Factor #2 (GSratio)	4.09688	1	4.09688	6.25125	0.02230	4.41387
Factor #3 (starch)	30.22689	2	15.11345	23.06093	0.00001	4.41387
Factor #1 + #2 (age x GSRatio)	0.00163	2	0.00081	0.00124	0.99876	3.55456
Factor #1 + #3 (age x starch)	0.01096	4	0.00274	0.00418	0.99996	2.92774
Factor #2 + #3 (GSratio x starch)	0.40305	2	0.20152	0.30750	0.73907	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00328	4	0.00082	0.00125	1.00000	0.00125
Within Groups	11.79666	18	0.65537			
Total	46.59044	35	1.33116			
Omega squared for combined effect	0.50063					



**Table D.3.2** (Continued)

Source of Variation	Omega Sqr.
Factor #1 (age)	0.00000
Factor #2 (GSratio)	0.07284
Factor #3 (starch)	0.61204
Factor #1 + #2 (age x GRatio)	0.00000
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GRatio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

Comparisons among groups (Factor 3 - starch)

Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	-1.92119	8.22085	0.00009	Yes
0 vs 1.5	-1.96565	8.41113	0.00008	Yes
1 vs 1.5	-0.04447	0.19028	0.99016	No

**Table D.3.3** ANOVA Table for fructose (%) of jelly gum formulations 3, 6, 9, 12, 15 and 18 (Table 2.2) at week 0, 24 and 52 at 30°C storage.

### Summary

Response	fructose(%)	
Factor #1	age	Fixed
Factor #2	GSratio	Fixed
Factor #3	starch	Fixed

**Table D.3.3 (Continued)****ANOVA**

Source of Variation	SS	d.f.	MS	F	p-level	F crit
Factor #1 (age)	0.18426	2	0.09213	0.14058	0.86980	3.55456
Factor #2 (GSratio)	3.77774	1	3.77774	5.76429	0.02738	4.41387
Factor #3 (starch)	29.73496	2	14.86748	22.68562	0.00001	4.41387
Factor #1 + #2 (age x GSRatio)	0.00128	2	0.00064	0.00097	0.99903	3.55456
Factor #1 + #3 (age x starch)	0.01297	4	0.00324	0.00495	0.99995	2.92774
Factor #2 + #3 (GSratio x starch)	0.38605	2	0.19302	0.29453	0.74841	3.55456
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.01051	4	0.00263	0.00401	0.99996	0.00401
Within Groups	11.79666	18	0.65537			
Total	45.90444	35	1.31156			
Omega squared for combined effect	0.49327					

Source of Variation	Omega Sqr.
Factor #1 (age)	0.00000
Factor #2 (GSratio)	0.06706
Factor #3 (starch)	0.61049
Factor #1 + #2 (age x GSRatio)	0.00000
Factor #1 + #3 (age x starch)	0.00000
Factor #2 + #3 (GSratio x starch)	0.00000
Factor #1 + #2 + #3 (age x GSRatio x starch)	0.00000
Within Groups	
Total	
Omega squared for combined effect	

**Comparisons among groups (Factor 3 - starch)****Tukey HSD**

Groups	Difference	Test Statistics	p-level	Significant
0 vs 1	-1.92086	8.21947	0.00009	Yes
0 vs 1.5	-1.93490	8.27952	0.00008	Yes
1 vs 1.5	-0.01404	0.06006	0.99909	No

## APPENDIX E

### ONE-WAY ANOVA TABLES FOR ACCEPTABILITY SCORES

**Table E.1** ANOVA Table with Tukey test for acceptability scores of fresh jelly gums

#### Descriptive Statistics

Groups	Sample size	Sum	Mean	Variance
formula no.	36	342.0	9.5	27.6857
fresh acceptability	36	202.4	5.6222	2.7219
Total	72		7.5611	18.8019

#### ANOVA

Source of Variation	d.f.	SS	MS	F	p-level	F crit	Omega Sqr.
Between Groups	1	270.6689	270.6689	17.8027	0.00007	3.9778	0.1892
Within Groups	70	1064.2662	15.2038				
Total	71	1334.9351					

#### Comparisons among groups (Factor 1 - Factor #1)

##### Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
formula no. vs fresh acceptability	3.8778	5.9670	0.00014	Yes

**Table E.2** ANOVA Table with Tukey test for acceptability scores of jelly gums stored at 15-22°C storage for 53 weeks.

Groups	Sample size	Sum	Mean	Variance
aged 53 @lab acceptability	36	171.8	4.7722	1.7681
formula no.	36	342.0	9.5	27.6857
Total	72		7.1361	20.1862

#### ANOVA

Source of Variation	d.f.	SS	MS	F	p-level	F crit	Omega Sqr.
Between Groups	1	402.3339	402.3339	27.3196	0.000002	3.9778	0.2677
Within Groups	70	1030.8838	14.7269				
Total	71	1433.2177					

#### Comparisons among groups (Factor 1 - Factor #1)

##### Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
aged 53 @lab acceptability vs formula no.	-4.7278	7.3918	0.00007	Yes

**Table E.3** ANOVA Table with Tukey test for acceptability scores of jelly gums stored at 10°C storage for 12 weeks.

### Descriptive Statistics

Groups	Sample size	Sum	Mean	Variance
aged 12 @10C acceptability	36	195.6	5.4333	2.7339
formula no.	36	342.0	9.5	27.6857
Total	72		7.4667	19.1883

### ANOVA

Source of Variation	d.f.	SS	MS	F	p-level	F crit	Omega Sqr.
Between Groups	1	297.68	297.68	19.5716	0.00003	3.9778	0.2051
Within Groups	70	1064.6872	15.2098				
Total	71	1362.3672					

### Comparisons among groups (Factor 1 - Factor #1)

#### Tukey HSD

Groups	Difference	Test Statistics	p-level	Significant
aged 12 @10C acceptability vs formula no.	-4.0667	6.2565	0.0001	Yes

**Table E.4** ANOVA Table with Tukey test for acceptability scores of jelly gums stored at 30°C storage for 12 weeks.

Groups	Sample size	Sum	Mean	Variance
aged 12 @30C acceptability	36	89.8	2.4944	1.1277
formula no.	36	342.0	9.5	27.6857
Total	72		5.9972	26.6461

**ANOVA**

Source of Variation	d.f.	SS	MS	F	p-level	F crit	Omega Sqr.
Between Groups	1	883.4006	883.4006	61.3186	3.7461E-11	3.9778	0.4559
Within Groups	70	1008.4707	14.4067				
Total	71	1891.8712					

**Comparisons among groups (Factor 1 - Factor #1)**

**Tukey HSD**

Groups	Difference	Test Statistics	p-level	Significant
aged 12 @30C acceptability vs formula no.	-7.0056	11.0742	0.00007	Yes

## APPENDIX F

### HALSEY EQUATION MODEL FITTING

**Table F.1** Halsey equation model fitting results of Formula 9 (Table 2.2) by using CurveExpert Professional 2.5.3

Reading file:C:\Users\Suzan\Desktop\formula9\_halsey\_equation\_101116.cxp

\* read result: halsey\_jelly (ID=1)

\* read 5 rows, 2 columns of data.

\* read 1 results.

\* read 2 graphs.

- Data Plot

- Top Results

\* read 0 images.

\* read 84 characters in notes.

Distributing the calculation over 4 cores...

Final Result [Custom/halsey\_jelly]:

Equation :  $a * (-\ln(x))^{**b}$

a = 2.173593501042235E-01

b = -5.658033627814445E-01

Standard Error : 4.095452111247585E-02

Correlation Coefficient : 9.820902164759632E-01

Run time : 12.1530 seconds

**Table F.2** Halsey equation model fitting results of Formula 12 (Table 2.2) by using CurveExpert Professional 2.5.3

Reading file: C:\Users\Suzan\Desktop\formula12\_halsey\_equation\_101116.exp

\* read result: halsey\_jelly (ID=1)

\* read 5 rows, 2 columns of data.

\* read 1 results.

\* read 2 graphs.

- Data Plot

- Top Results

\* read 0 images.

\* read 84 characters in notes.

Autoscaling graph Top Results...

Final Result [Custom/halsey\_jelly]:

Equation :  $a * (-\ln(x))^{**b}$

a = 2.303466850299935E-01

b = -5.658356785267225E-01

Standard Error : 4.339829649576824E-02

Correlation Coefficient : 9.820959230070234E-01

Run time : 0.0160 seconds



## **CURRICULUM VITAE**

### **PERSONAL INFORMATION**

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### **EDUCATION**

<b>Degree</b>	<b>Institution</b>	<b>Year of Graduation</b>
M.S.	Middle East Technical University	2005
B.S.	Middle East Technical University	2002
High School	Kdz. Ereğli Anatolian High School	1997

### **PROFESSIONAL EXPERIENCE**

<b>Year</b>	<b>Place</b>	<b>Enrollment</b>
2013- current	Kervan Gıda San. Ve Tic. A. S.	R&D Manager
2012- 2013	Yıldız Holding A. S.	R&D Project Manager
2011- 2012	Yıldız Holding A. S.	R&D Assist. Manager
2010- 2011	NWG Human Resources	R&D Engineer
2008- 2010	Yıldız Holding A. S.	Food Research Specialist
2007- 2008	Coca Cola Services	R&D Product Developer
2005- 2007	Eti Group of Companies	R&D Engineer
2002- 2005	Middle East Technical University	Research Assistant

## **PUBLICATIONS**

- 1) Tireki S., Sumnu, G. and Esin, A. Production of bread crumbs by infrared-assisted microwave drying, Article published in European Food Research and Technology [222 (1-2): 8-14 (2006)].
- 2) Tireki S., Sumnu, G. and Esin, A. Effect of Microwave, Infrared and Infrared-assisted Microwave Heating on the Drying Rate of Bread Dough, Article published in American Journal of Food and Technology [1(2): 82-93, 2006)].
- 3) Tireki, S. Technology of Cake Production. In: Food Engineering Aspects of Baking Sweet Goods. Edited by Sumnu, S. G. and Sahin, S. CRC Press, Taylor & Francis, 2008.
- 4) Tireki, S. Technology of Cookie Production. In: Food Engineering Aspects of Baking Sweet Goods. Edited by Sumnu, S. G. and Sahin, S. CRC Press, Taylor & Francis, 2008.