GIDA
THE JOURNAL OF FOOD
E-ISSN 1309-6273, ISSN 1300-3070

Research / Araştırma
GIDA (2021) 46(2) 279-295
doi: 10.15237/gida.GD20093

DETERMINATION OF THE BEST DRYING CONDITIONS FOR GELATIN BASED CANDIES

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Received / Geliş: 06.08.2020; *Accepted* / Kabul: 08.10.2020; *Published online* / Online baskı: 08.02.2021

Pocan, P., Kaya, D., Mert, B., Oztop, M.H. (2021). Determination of the best drying conditions for gelatin based candies. GIDA (2021) 46(2) 279-295 doi: 10.15237/gida.GD20093.

Pocan, P., Kaya, D., Mert, B., Oztop, M.H. (2021). Jelatin bazlı yumuşak şekerlerin kurutma koşullarının optimizasyonu. GIDA (2021) 46(2) 279-295 doi: 10.15237/gida.GD20093.

ABSTRACT
In this study, objective was to determine the best drying and conditioning times for achieving the desired quality for jelly candies of different weights. Drying time (also known as *Stoving time*) (12, 16, 20, 24 h) in oven, conditioning time (WIP time) (0, 24, 48, 72 h), unit weight of the candies (2.5 g, 3 g, 6 g) were the variable parameters investigated. TPA, moisture content, water activity, TSSC, TD-NMR Relaxometry experiments were performed. Optimum drying time was found as 20 h for 2.5 g and 24 h for both 3 g and 6 g. Following drying, the best conditioning time with respect to unit weight was determined as 72 h for 2.5 g and 48 h for 3 g and 6 g candies. The results of the study suggested that, to achieve and keep the desired quality parameters during shelf life, each unit weight candy should be processed separately.
Keywords: Gelatin based soft candy; texture; moisture; drying; conditioning; time domain NMR relaxometry

JELATİN BAZLI YUMUŞAK ŞEKERLERİN KURUTMA KOŞULLARININ OPTİMİZASYONU

ÖZ
Bu çalışmada amaç, farklı ağırlıklardaki jelatin bazlı şekerlerde istenen kaliteyi elde etmek için optimum kurutma ve tavlama sürelerini belirlemektir. Fırında kuruma süresi (*Fırınlama süresi*) (12, 16, 20, 24 saat), tavlama süresi (WIP zamanı) (0, 24, 48, 72 saat), şekerlerin birim ağırlığı (2.5 g, 3 g, 6 g) incelenen değişken parametrelerdir. Şekerlerin karakterizasyonu için Tekstür Profil Analizi, nem içeriği, su aktivitesi, toplam çözünür katı içerik ve NMR Relaksometre deneyleri yapılmıştır. En iyi kuruma süresi 2.5 g için 20 saat, 3 g ve 6 g için 24 saat olarak bulunmuştur. Kurutmadan sonra, birim ağırlığa göre en iyi tavlama süresi, 2.5 g için 72 saat, 3 g ve 6 g şekerlemeler için ise 48 saat olarak belirlenmiştir. NMR relaksasyon süreleri olarak tanımlanan T_1 ve T_2 fiziksel parametrelerle ilişkili bulunmuştur. Çalışmanın sonuçları, raf ömrü boyunca istenen kalite parametrelerini elde etmek ve korumak için, farklı birim ağırlıkta olan şekerlemelerin ayrı olarak işlenmesi gerektiğini önermiştir.
Anahtar kelimeler: Jelatin bazlı yumuşak şeker, tekstür, nem, kurutma, tavlama, zamansal alanlı NMR relaksometre

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INTRODUCTION

Jellies, pastilles and gums belong to the same classification in confectionary products and have a moisture content around 20% (Lees and Jackson 2012). Jelly candies are composed of gelling agents such as gelatin, pectin, starch or gum arabic. Sucrose and glucose syrup are used as the main components and as the additional ingredients; jelly candies also include food acids, flavouring and colouring agents (Burey et al. 2009).

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

During drying in the oven, moisture migration, occurs between starch - candy and air. Firstly, moisture migration occurs from candy into the starch bed. After that, moisture migrates from the candy into the air. Finally, moisture migration occurs from starch bed into the air depending on the % RH of the air (Troutman et al. 2001).

Dehydration is a crucial parameter in texture profile of the candy. The highest dehydration rate of candy usually occurs at the beginning of the drying in starch mold. As stated above, water inside a candy is transferred to the outside and consequently increases the solid content (Delgado and Bañón 2015). Mostly, jelly candies' water activity changes between 0.5 and 0.7 and the final product should achieve at least 75% total soluble solids to prevent mold growth (Ergun et al. 2010a).

Time domain NMR Relaxometry is a valuable tool to get information about the dehydration behaviours of soft candies. Nuclear Magnetic Resonance (NMR) is a non-destructive characterization technique and can be used to determine food quality (Kirtil et al. 2017). Troutman et al. (2011) used NMR relaxation times to observe the effect of environmental factors on drying and moisture migration. Since foods are good examples to the chemically and structurally heterogeneous systems, various contributions to the NMR signal is possible due to the changes in molecular mobility (Kirtil et al. 2017). These changes in NMR signal could be explained with two main variables: longitudinal (T_1) and transverse (T_2) relaxation times (Kirtil et al. 2017). These time constants are used to characterize the mobility of protons and variations of conformations within the biopolymer (Ozel et al. 2017a).

From the previous studies, it was known that T_1 (spin-lattice relaxation time) highly relied on the mobility of protons which came from the water component of the gel matrices (Pocan et al. 2019a). In that regard, T_1 relaxation time could be considered as a valuable tool to detect the moisture distribution on food samples (Pocan et

al. 2019a). Longitudinal relaxation time (T_1) was utilized in many studies for the analysis of food systems such as effect of microwave heating on starch-water interactions (Ozel et al. 2017b), impact of pectin methyl esterase and $CaCl_2$ infusion on mangoes (Kirtil et al. 2014), effect of D-Allulose (formerly known as D-Psicose) addition on gelatin based soft candies (Pocan et al. 2019a) and moisture migration in soft-panned confections during aging (Troutman et al. 2001).

T_2 (spin-lattice) relaxation time is also an important parameter to deduce water content, interaction of water with surrounding molecules and physical properties of water (Kirtil et al. 2014). Both multi-exponential and mono-exponential approaches could be utilized to interpret the transverse (T_2) relaxation times (Pocan et al. 2019a). T_2 relaxation time of food products having a multi-compartmental nature such as gluten free cakes (Yildiz et al. 2018), thawed and frozen mangoes (Kirtil et al. 2014) and gelatin based soft candies (Pocan et al. 2019a) were analysed with the help of multi-exponential approach while emulsion stabilization properties of some gums like gum tragacanth (Pocan et al. 2019b) and characterization of capsaicin emulsions (Akbas et al. 2016) were explored with the help of T_2 relaxation times that were expressed as mono-exponential.
In this study, the objective is to determine the best stoving and conditioning times (WIP) for achieving the desired quality for soft candies of different weights. Moisture content, water activity, total soluble solid content, hardness and NMR Relaxometry experiments were performed for samples stored at different stoving and conditioning times.

MATERIALS AND METHODS

Materials

In soft candy production; sucrose (Kayseri Şeker, Kayseri, Turkey), glucose syrup (Cargill, Bursa, Turkey) (DE: 42), gelatin (Halavet Gıda, İstanbul, Turkey) (Blooming index: 240), coloring and flavoring agents (International Flavors & Fragrances (IFF), Kocaeli, Turkey), citric acid monohydrate (Yılmaz Kimya, İstanbul, Turkey) were used as the ingredients.

Methods

Gelatin Based Soft Candy Production

Firstly, gelatin (240 bloom) solution was prepared around 65 °C in the gelatin preparation tank. In weighing tank, gelatin solution, glucose syrup and sucrose were mixed while heating to dissolve sugar crystals. After, slurry (mixed solution) was cooked at approximately 100 °C, it was fed into vacuum chamber to adjust the solid content, remove air bubbles and eliminate the excess water. Solid content was around 75 to 80 %. Flavor, acid and coloring agent were added into the slurry immediately prior to molding to minimize time & temperature effect on volatile flavor components and to avoid inversion of the sucrose. Then, the slurry was molded into starch trays. The filled trays were taken to an oven at approximately 30 ºC and 45 % RH to condition and gain the desired texture. Time that products were kept in oven is called *stoving time*. When the desired total soluble solid content and texture was attained, drying was completed. Following drying, the trays were inverted to remove the jellies from the molds and were brushed gently to eliminate starch powder. Finally, the jellies were coated with oil using a drum to avoid a sticky surface. After coating, products were transferred to cases and kept on conditioning area before packaging. Conditioning room temperature was around 20 ºC and <65 % RH. In jelly candy technology, *conditioning* is termed as WIP *(Work in Process)*. In the following sections, *WIP* term is used instead of conditioning. Production flow chart is also given in Fig 1.

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

Samples molded into starch molds and put into oven for drying process. Effect of four levels of stoving time (12, 16, 20, 24 h) were studied. Moisture content, water activity, total soluble solid content, texture analysis and NMR experiments were performed. After stoving cycle

was completed, samples were transferred to the coating section. In this study, samples that were hold 12 h and 16 h on oven were not coated with oil, only 20 h and 24 h samples were coated. Products that were hold 12 h and 16 h on oven were not found to be proper even from the beginning due to the textural properties of these candies not being appropriate for the consumer (too sticky and too soft). So, 12 and 16 h products were not coated. In order to analyse the effect of stoving time on the moisture content, total solid content and water activity, non-coated samples were used.

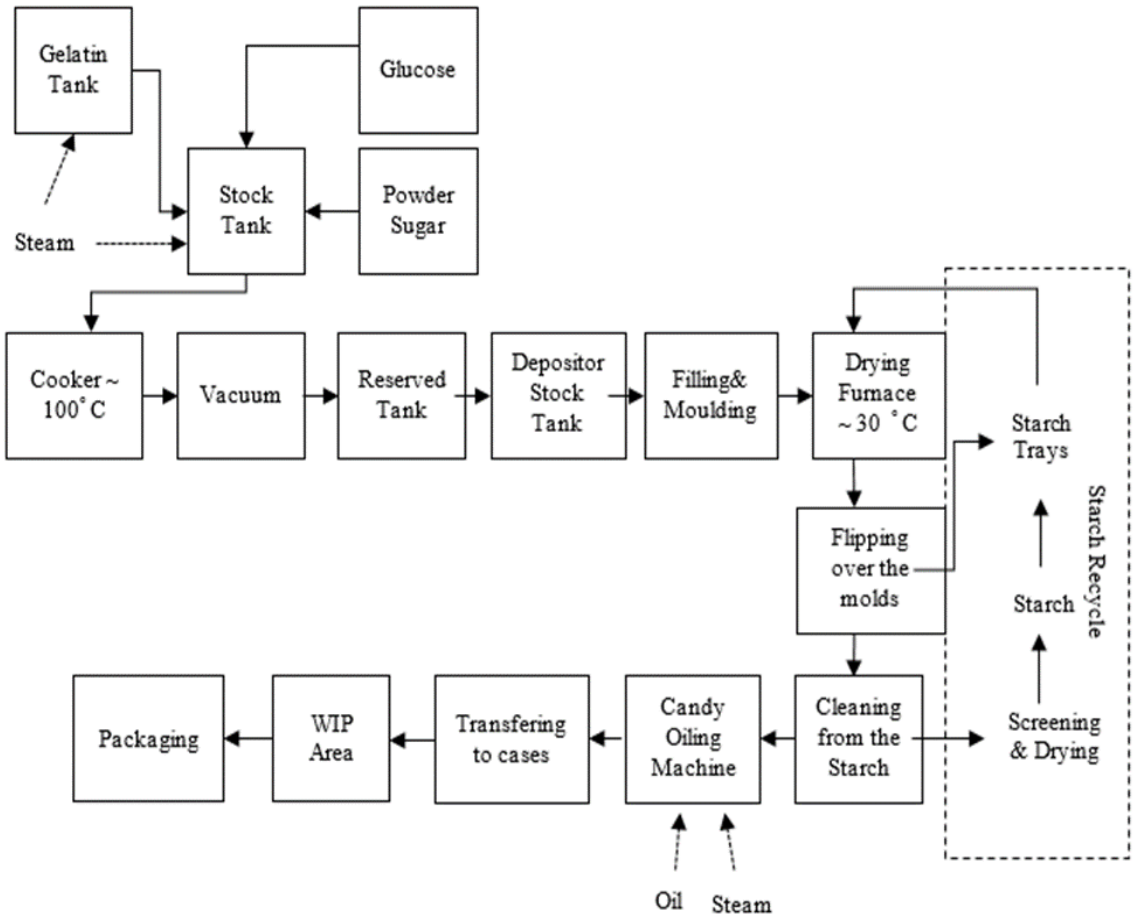

Figure 1. Flow chart of jelly candy production

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

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

Moisture Analysis – Vacuum Oven Method
Moisture content was determined by the vacuum oven method. 20 g sea sand and glass pieces were put on aluminium dish and dishes were put in oven at 105 ºC for 1 hour. Then, the dish was held in a desiccator at room temperature to cool down. One piece of sample was added to the dish and 1-2 ml hot water was then added and mixed into sea sand and glass pieces. This mixture was placed on drying oven at 60 º C for half an hour to evaporate excess water. After that, dishes were put into vacuum oven (DAIHAN, Germany) at 70 ˚and at a pressure of > 950 mbar and kept for 18 hours. After 18 hours, dishes were cooled in the desiccator and weighed.

Total Soluble Solids Content
Total soluble solids (expressed as g of total soluble solids contained in 100 g or °Brix) are measured using a refractometer (Atago Co. Ltd. RX- 5000) with an accuracy of 0.01 °Brix. Firstly, the device was adjusted to 30º C and calibrated three times with water.

Texture Analysis
Penetration test measurements were made using Stable Micro Systems (SMS) Texture Analyzer Plus with a 5 kg load cell. The penetration test used for this analysis was performed with a 3mm cylindrical probe which penetrated to 80% of the full depth of a standard unit, and then retracted. Hardness and stickiness values were recorded. Analysis was made at room temperature (25 ºC) with minimum 10 replicates.

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

Time Domain NMR Relaxometry (TD-NMR) Experiments
NMR relaxometry experiments were performed by using the parameters that were mentioned in the study Pocan et al. (2019) with some modifications. TD NMR relaxometry experiments were carried out using 0.5 T (20.34 MHz) system (Spin Track, Resonance Systems GmbH, Kirchheim/Teck, Germany). For T_1 measurements, saturation-recovery sequence was used with a 300 ms relaxation period (TR) and 400 ms observation time. For T_2 measurements, the Carr–Purcell–Meiboom–Gill (CPMG) sequence was utilized with parameters of 40 us echo time, 500 echoes, and four scans. T_1 and T_2 measurements were performed for all samples.

The T_1 and T_2 data were analysed as indicated in the study of Pocan et al. (2019). First, mono-exponential fitting was conducted on the relaxation curves using MATLAB. Discrete component analysis was performed using XPFit (Softonics Inc., Israel) to investigate the proton pools and components in CPMG decay curve as mentioned in the study of Efe et al. (2019). For the analysis of T_2 relaxation times, both mono-exponential and bi-exponential approach were utilized. For T_1 relaxation times, only mono-exponential approach was used.

Statistical Analysis
All measurements except texture measurements were conducted with three replicates. Texture measurements were carried out with at least 10 replicates. Data were analysed using Minitab 17 (Minitab Inc., Penn State, USA) at %5 significance level. One-way ANOVA was conducted for the analysis of stoving time on non- coated samples. In order to determine the difference between samples, Tukey's comparison tests were used at %95 confidence interval.

RESULTS AND DISCUSSION

Total Soluble Solid Content
In these experiments, refractometer was used to investigate % total soluble solid content (TSSC) content. ºBrix value is a fast and adequate parameter that is used to understand TSSC in confectionery products (Lees and Jackson, 2012). In gelatin-based candies, total soluble solid content should be between % 75-82 to prevent graining (Lees and Jackson, 2012).

As mentioned before, in this study gelatin-based soft candies of different weights (2.5 g, 3 g and 6 g) were investigated for different stoving and WIP

times. As it is shown in Table 1., 2.5 g non-coated samples that were kept for 24 h in the oven had the highest value (P <0.05). As expected, for these samples with 2.5 g unit weight, TSSC (%) increased significantly as the stoving time increased. On the contrary, for the other jelly candies with 3 and 6 g unit weights, significant deviations in TSSC (%) were not observed and remained nearly constant as the stoving time increased from 12 to 24 hours (P >0.05).

Table 1. Effect of stoving time on total solid content (%), moisture content (%) and water activity (a_w) of non-coated jelly candies

Stoving Time (h)	Type (g)	Total Solid Content (%)	Moisture Content (%)	Water activity (a_w)
12 h	2.5	$78.60^{c}\pm0.18$	$18.76^{a}\pm0.13$	$0.45^{b}\pm0.03$
16 h	2.5	$78.83^{c}\pm0.02$	$18.37^{ab}\pm0.38$	$0.53^{a}\pm0.01$
20 h	2.5	$79.49^{b}\pm0.00$	$17.72^{bc}\pm0.15$	$0.53^{a}\pm0.01$
24 h	2.5	$82.26^{a}\pm0.00$	$17.55^{c}\pm0.11$	$0.56^{a}\pm0.01$
12 h	3	$78.92^{a}\pm0.36$	$19.17^{a}\pm0.05$	$0.41^{c}\pm0.01$
16 h	3	$79.05^{a}\pm0.25$	$19.04^{a}\pm0.06$	$0.40^{c}\pm0.01$
20 h	3	$79.09^{a}\pm0.12$	$18.96^{a}\pm0.02$	$0.48^{b}\pm0.01$
24 h	3	$79.45^{a}\pm0.15$	$18.64^{b}\pm0.14$	$0.53^{a}\pm0.01$
12 h	6	$78.98^{a}\pm0.01$	$19.79^{a}\pm0.09$	$0.44^{c}\pm0.02$
16 h	6	$79.10^{a}\pm0.01$	$19.49^{a}\pm0.11$	$0.49^{b}\pm0.01$
20 h	6	$78.85^{a}\pm0.00$	$19.34^{a}\pm0.22$	$0.54^{a}\pm0.01$
24 h	6	$79.70^{a}\pm0.31$	$18.46^{b}\pm0.07$	$0.44^{c}\pm0.01$

*Different small letters indicate significant difference (P < 0.05) for the same unit weight with different stoving

In addition, if the unit weight was examined as the only factor, it was deduced that, TSSC (%) decreased significantly as the unit weight increased from 2.5 g to 3 g (P <0.05) as seen in Table 2. However, detectable changes were not observed as the unit weight increased to 6 g (P >0.05). A similar drying study was performed by Delgado et al. (2015) and they illustrated that TSSC increased from 76.9 °Brix to 77.7 °Brix when drying time increased from 12h to 20 h and then remained constant. However, they utilized porcine gelatin and starch together as the gelling agent in production of gummy candies. The differences observed in our study might have stemmed from gelling agent that utilized since bovine gelatin was the only gelling agent that used in our study. It is highly probable that; drying behaviour was affected from the type of the gelling agent.

Table 2. Effect of unit weight on Total Solid Content & Moisture Content (%) and Water Activity (aw) results for non-coated jelly candies

Type (g)	Total Solid Content (%)	Moisture Content (%)	Water activity (a_w)
2.5	$79.79^{a}\pm0.84$	$18.10^{c}\pm0.31$	$0.52^{a}\pm0.03$
3	$79.13^{b}\pm0.18$	$19.07^{b}\pm0.12$	$0.45^{c}\pm0.03$
6	$79.16^{b}\pm0.31$	$19.27^{a}\pm0.29$	$0.48^{b}\pm0.02$

*Different small letters indicate significant difference (P < 0.05) considering the unit weight as the only factor

When the results were examined for the oil coated samples, 2.5 g oil coated candy (Stoving time: 24 h, WIP: 72 h) had the highest total soluble solid content (TSSC %) and 6 g oil coated candy (Stoving time: 20 h, WIP time: 0 h) had the lowest TSSC (%) as seen in Table 3. TSSC (%) increased and moisture content reduced with drying. So, as expected, increasing stoving and WIP time increased TSSC (%).

Table 3. Effect of WIP and stoving time on the physical and textural properties of oil coated jelly candies

Stoving time (h)	WIP Time (h)	Type (g)	Total Solid Content (%)	Moisture Content (%)	Water activity (a_w)	Hardness (N)	Stickiness (N)
20	0	2.5	$79.49^{d}\pm0.01$	$18.74^{a}\pm0.08$	$0.53^{a}\pm0.01$	$560.64^{bc}\pm10.59$	$-38.60^{a}\pm3.26$
20	24	2.5	$80.74^{c}0.08$	$18.67^{a}\pm0.13$	$0.60^{a}\pm0.01$	$556.77^{bc}\pm19.62$	$-38.30^{a}\pm2.93$
20	48	2.5	$82.32^{b}\pm0.03$	$18.66^{a}\pm0.13$	$0.57^{a}\pm0.02$	$550.80^{bc}\pm10.18$	$-37.14^{a}\pm1.94$
20	72	2.5	$82.34^{b}\pm0.03$	$17.05^{a}\pm0.10$	$0.60^{a}\pm0.03$	$516.54^{c}\pm15.78$	$-38.45^{a}\pm1.88$
24	0	2.5	$82.26^{b}\pm0.01$	$18.57^{a}\pm0.06$	$0.56^{a}\pm0.01$	$582.67^{ab}\pm7.41$	$-37.18^{a}\pm1.32$
24	24	2.5	$82.29^{b}\pm0.02$	$18.54^{a}\pm0.13$	$0.62^{a}\pm0.01$	$557.88^{bc}\pm17.63$	$-36.89^{a}\pm2.22$
24	48	2.5	$82.38^{b}\pm0.03$	$18.53^{a}\pm0.08$	$0.42^{a}\pm0.25$	$563.56^{ab}\pm14.99$	$-39.65^{a}\pm1.23$
24	72	2.5	$86.97^{a}\pm0.04$	$18.50^{a}\pm0.16$	$0.58^{a}\pm0.01$	$581.50^{a}\pm13.35$	$-44.45^{b}\pm2.58$
20	0	3	$78.68^{e}\pm0.21$	$19.22^{a}\pm0.08$	$0.45^{d}\pm0.01$	$507.34^{bc}\pm8.30$	$-29.56^{a}\pm2.90$
20	24	3	$81.54^{b}\pm0.11$	$19.35^{a}\pm0.05$	$0.59^{b}\pm0.01$	$510.58^{abc}\pm10.38$	$-30.96^{a}\pm5.21$
20	48	3	$80.52^{c}\pm0.15$	$19.55^{a}\pm0.06$	$0.60^{ab}\pm0.02$	$485.17^{c}\pm17.11$	$-31.98^{a}\pm2.17$
20	72	3	$81.27^{b}\pm0.04$	$19.03^{ab}\pm0.07$	$0.63^{a}\pm0.01$	$497.69^{bc}\pm10.14$	$-32.97^{ab}\pm2.78$
24	0	3	$79.42^{d}\pm0.06$	$19.31^{a}\pm0.21$	$0.53^{c}\pm0.01$	$512.03^{bc}\pm10.46$	$-33.04^{ab}\pm2.92$
24	24	3	$82.28^{a}\pm0.03$	$18.97^{ab}\pm0.05$	$0.59^{b}\pm0.01$	$529.49^{ab}\pm17.61$	$-33.39^{ab}\pm5.30$
24	48	3	$80.56^{c}\pm0.21$	$18.57^{bc}\pm0.07$	$0.59^{b}\pm0.01$	$512.34^{ab}\pm14.02$	$-33.13^{ab}\pm3.09$
24	72	3	$81.63^{b}\pm0.05$	$18.38^{c}\pm0.09$	$0.60^{b}\pm0.01$	$528.55^{a}\pm18.35$	$-37.23^{b}\pm2.95$
20	0	6	$78.60^{e}\pm0.03$	$19.85^{a}\pm0.04$	$0.51^{e}\pm0.01$	$445.72^{d}\pm24.22$	$-29.43^{a}\pm6.67$
20	24	6	$80.20^{bc}\pm0.01$	$19.62^{a}\pm0.12$	$0.48^{f}\pm0.01$	$458.99^{d}\pm20.52$	$-28.43^{a}\pm5.11$
20	48	6	$81.50^{a}\pm0.01$	$19.42^{a}\pm0.14$	$0.54^{d}\pm0.01$	$534.87^{b}\pm21.24$	$-33.80^{ab}\pm2.22$
20	72	6	$81.30^{a}\pm0.28$	$18.75^{b}\pm0.06$	$0.53^{d}\pm0.01$	$627.72^{a}\pm20.10$	$-38.74^{b}\pm3.27$
24	0	6	$79.30^{d}\pm0.08$	$19.38^{a}\pm0.11$	$0.50^{e}\pm0.02$	$458.59^{d}\pm20.79$	$-30.99^{a}\pm5.28$
24	24	6	$79.60^{cd}\pm0.11$	$18.80^{b}\pm0.05$	$0.56^{c}\pm0.01$	$498.06^{c}\pm18.52$	$-33.68^{ab}\pm2.74$
24	48	6	$80.70^{b}\pm0.35$	$18.79^{b}\pm0.08$	$0.63^{a}\pm0.01$	$538.06^{b}\pm18.63$	$-34.42^{ab}\pm3.44$
24	72	6	$81.40^{a}\pm0.10$	$18.69^{b}\pm0.03$	$0.59^{b}\pm0.01$	$635.78^{a}\pm24.01$	$-35.40^{ab}\pm8.18$

*Different small letters indicate significant difference ($P < 0.05$) for the same unit weight with different stoving and WIP times

TSSC (%) increased significantly when WIP time increased from 0 h to 72 h at 20 h stoving time for the 2.5 g samples as seen in Table 3 ($P<0.05$). For these samples, sharp increase was monitored when WIP time increased to 72 h at constant 24 h stoving time. However, for the 3-gram samples, fluctuations were observed. From 0 to 24 h, an ascending trend was observed in TSSC (%) results while descending behavior was observed when the WIP time increased from 24 h to 72 h at constant stoving time. This case was valid for both stoving times (20 h and 24 h). For the 6 g samples, similar case was observed with the 2.5 g samples and TSSC (%) increased when the WIP time increased to 72 h at 20 & 24 h stoving times.

Unit weight basis analysis showed more indicative results on the effects of storage conditions of oil coated candies. If the unit weight was considered as the only factor, it was observed that as the unit weight increased from 2.5 g to 3 g and 6 g, TSSC (%) decreased significantly as seen in Table 4 ($P <0.05$). Soft candies with 2.5 g unit weight was found to have the highest TSSC (%).

Table 4. Effect of unit weight on Total Solid Content & Moisture Content and Water Activity results for oil coated jelly candies

Type (g)	Total Solid Content (%)	Moisture Content (%)	Water activity (a_w)	Hardness (N)	Stickiness (N)	T_1(ms)	$T_{2monoexp}$ (ms)
2.5	$82.35^{a}\pm1.16$	$17.48^{b}\pm0.16$	$0.56^{a}\pm0.02$	$560.33^{a}\pm9.36$	$-38.93^{b}\pm2.34$	$47.58^{a}\pm0.32$	$0.71^{a}\pm0.03$
3	$80.74^{c}0.08$	$19.05^{a}\pm0.24$	$0.57^{a}\pm0.03$	$509.89^{c}\pm7.07$	$-32.65^{a}\pm1.24$	$44.83^{b}\pm0.57$	$0.72^{b}\pm0.04$
6	$82.32^{b}\pm0.03$	$19.16^{a}\pm0.26$	$0.54^{a}\pm0.03$	$524.73^{b}\pm9.53$	$33.11^{a}\pm1.95$	$47.67^{a}\pm0.45$	$0.64^{c}\pm0.04$

*Different small letters indicate significant difference ($P < 0.05$) considering the unit weight as the only factor

Stoving time and WIP time that is necessary to obtain jelly candies with stable network can be different depending on different unit weights due to the existence of different surface areas that contacted with starch bed. Larger samples had larger surface area in starch bed moulds. So, time to reach equilibrium takes longer time as compared to smaller samples.

Moisture Content

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

In this study, stoving time levels (12, 16, 20, 24 hour) were found effective on moisture content (MC) for non-coated samples having 2.5 g unit weights and gradual decrease in MC was monitored as the stoving time increased, as seen in Table 1 ($P < 0.05$). This was an expected result since TSSC (%) of these samples increased significantly as it was mentioned in the previous section. According to Delgado and Banon's study (2015), TSSC also increased when moisture content decreased from 12 h to 24 h for gummy products as in the case of our study (Delgado and Bañón 2015). In the study of Delgado et al. (2015) average unit weight of gummy candies was reported as 2.13 g which is very similar to unit weights that was used for this study (2.5 g). Therefore, observing such an obvious decreasing trend in MC was not surprising. However, when the unit weights of gummy candies were changed, scenario was different. For the non-coated gummy candies with 3 and 6 g unit weights, significant decrease in MC was only observed while switching from 20 h to 24 h stoving time as seen in Table 1 ($P < 0.05$). As it was illustrated in Table 2, if the unit weight was considered as the only factor for these non-coated samples, it was seen that gradual and significant increase was observed in MC as the unit weight was increased ($P < 0.05$). Since moisture removal from the relatively larger sample was difficult, necessary drying time for these samples were relatively longer.

After stoving, samples were coated with oil and kept on the WIP area. As seen in Table 3, increasing WIP time did not affect the MC of the 2.5 g samples ($P > 0.05$) at each constant stoving time (20 h & 24 h). Most probably, oil found on the surface of these samples kept the moisture inside the gel matrix and did not allow the moisture outside to the gel resulting in constant moisture content even WIP time was increased to 72 h. Therefore, it could be concluded that increasing WIP time was not helpful for the jelly candies having 2.5 g unit weight. Similar case was also valid for the 3 g samples that were exposed to 20 h stoving time. For these samples, increasing WIP time did not also lead to significant decreases in MC result ($P > 0.05$). However, a different scenario occurred for the 3 g samples which were exposed to 24 h stoving time. For these samples, it was elucidated that, increasing WIP time to 72 h led significant and gradual decrease in MC ($P < 0.05$). For 6 g samples that were exposed to 20 h stoving time, detectable decrease in MC was

observed only when the samples were kept at 72 h conditioning room (P <0.05). On the other hand, for 6 g samples that were exposed to 24 h stoving time, noticeable decrease in MC was observed when they were exposed to 24 h WIP time while after 24 h, increasing WIP time did not lead a detectable change and MC of these samples remained constant (P >0.05) (Table 3). As it was illustrated in Table 4, if the unit weight was considered as the only factor for these oil coated samples, it was seen that, significant increase in MC was detected as the unit weight was increased from 2.5 to 3 g (P <0.05). However, steadiness in MC was detected when the unit weight was increased to 6 g.

In summary, moisture content did not change significantly until 20 h; but, it decreased slowly for all non-coated samples from 20 to 24 h. Moreover, for the coated samples, only 3 g and 6 g samples were mostly affected from being kept in the WIP area whereas 2.5 g oil coated samples were not affected from the WIP area, so it was confirmed that they were able to lose the whole free water inside and reached equilibrium totally during stoving.

Water Activity

The amount and rate of moisture migration that occurs between the components of a multi-domain system such as jelly candies strongly depend on the thermodynamic forces of the system (water activity) and factors influencing the diffusion rate (Troutman et al. 2001). Therefore, water activity (a_w) is an important thermodynamic parameter to explain the moisture migration behavior of the food systems during drying. It could be defined as the ratio of partial pressure of water vapor ratio to pressure of pure water at a specified temperature (Mathlouthi 2001).

As seen in Table 1, for the non-coated 2.5 g samples, the MC and a_w values validated that further dehydration did not occur from 20 h onwards. Similar case was also observed in previous studies. Delgado et al. (2015) examined the a_w and MC of gummy candies having 2.13 g unit weight and, in their study,, it was also mentioned that, after 20 h, candies also did not dehydrate further. Since the unit weight utilized in their study was very similar to our study, this outcome was not surprising. It is also worth to mention that, for the non-coated samples owing 2.5 g unit weight, while water activity changed around 0.45-0.53 from 12 h to 16 h on oven, moisture content did not change significantly and remained almost constant (P >0.05). Around 20 h, MC decreased distinguishably whereas at the same time a_w values did not change so much. This behaviour was similar to Type II behaviour of intermediate moisture products. In the study of Ergun et al. (2010), it is stated that many type of candies would have sorption isotherms which follow either Type II or Type III. In this study, this case was also valid and jelly candies that were used in this study also followed a similar sorption isotherm trend. On the other hand, for the 3 g and 6 g non-coated samples, noticeable decrease in MC and a_w were observed even after 20 h validating the on-going dehydration. This case might be explained with relatively bigger sample size compared to 2.5 g samples. As it was illustrated in Table 2, if the unit weight was considered as the only factor for these non-coated samples, it was observed that, unit weight was an important factor itself that caused changes in a_w values significantly (P <0.05).

After coating, samples were kept in the conditioning area. It was observed that a_w of oil coated jelly candies were relatively higher compared to their non-coated counterparts (Table 3). As seen in Table 3, for the 2.5 g oil-coated samples, increasing WIP times did not lead to any significant changes in the a_w values of the samples (P >0.05). Therefore, it is important to mention that these samples fully dehydrated after the stoving step and storing them in the conditioning room did not result in further dehydration. As indicated in previous sections, MC results for these samples were also consistent with a_w results and confirmed the end of dehydration. On the contrary, for the 3 g and 6 coated samples, increasing WIP time for each stoving time (20 h & 24 h) led to detectable increments in a_w values of these samples (P <0.05).

As it was illustrated in Table 4, if the unit weight was considered as the only factor for these oil-coated samples, it was observed that different unit weight did not have a significant effect on water activity while waiting in WIP area (P >0.05).

Hardness

Hardness is directly proportional to the maximum force when analysing deformation in the first bite (Delgado and Bañón 2015). Hardness values of oil-coated jelly candies are shown in Table 3. In texture analyses, it was not logical to use 12 and 16 h stoving time data. Since, after stoving cycle, samples' surface was covered fully with starch and it was very hard to analyse texture profile because of the sticky surface (inadequate drying). As explained in the previous section, samples were taken after 20 h and 24 h separately from oven and coated with oil; then kept in the WIP area. According to the Table 3, 6 g samples that were held 24 h in oven and 72 h in WIP area had the highest hardness values. It was also observed that, 2.5 g coated samples', texture profile was not influenced from conditioning area although the total solid content had increasing trend with time (P >0.05). For the 3 g samples, hardness values did not change at constant 20 h stoving time while WIP time increased to 72h. Similarly, at 24 h stoving time, significant increase was not detected in hardness values while switching from 48 to 72 h WIP time (P <0.05). The effect of WIP time on the hardness values of 6 g samples was more apparent compared to its counterparts. Increasing WIP time resulted in gradual and significant increase in hardness values of 6 g samples (P <0.05). This increment was also valid for the TSSC of these sample as discussed previously.

If the unit weight was considered as the only factor affecting hardness, it was found that samples were significantly different from each other (P <0.05) as shown in Table 4. Similarly, this result was found to be parallel with total soluble solid content. Minimum TSSC (%) and minimum hardness values were found for the both 3 g samples.

In previous studies it was demonstrated that drying generally reduces moisture and increases the solid content, and this causes increasing hardness and change on other textural properties (Vieira et al. 2008; Nur Farah Hani et al. 2014; Delgado and Bañón 2015). In our study, this outcome was also validated. According to the Pearson's correlation results that were shown in Table 5, hardness values was correlated with the moisture content (R= -0.70; P<0.05) and total soluble solid content (R=0.70; P<0.05). According to the study of Delgado et al. (2015), changes in textural properties of gummy candies during drying could be explained with both dehydration and gelation phenomena. In their study, modest correlation coefficients were found suggesting that drying were not only in the control of dehydration phenomena but also in the control of gelation phenomena. The lack of a correlation between moisture and textural properties also reinforces this argument in their study. On the other hand, in our study, moisture and textural properties (hardness& stickiness) was found to be well-correlated (r>0.70; P <0.05) suggesting that drying of jelly candies was dehydration controlled.

Stickiness

Stickiness is a very important parameter for the jelly candies. Stickiness is the force necessary to overcome the attractive forces between the surface of the product and the surface of the material (the probe) with which the product comes in contact. In jelly products, stickiness, hardness, poor crusting, poor chewiness are considered to be important quality defects (Ergun et al. 2010a).

As seen in Table 3, changing WIP times generally did not lead to changes in the stickiness values of the oil-coated samples. For the samples having 2.5 g unit weight that exposed to 24 h stoving time, only detectable change was observed when the WIP time was increased from 48 h to 72 h (P <0.05) while there was no significant change for the stickiness values of 3 g samples. For the 6 g samples, similar stickiness values were found for the WIP times 0 h, 24 h and 48 h. Although significant decrease was observed in stickiness values at 72 h WIP time compared to the samples that expose 0 and 24 h WIP times, they were found to be similar at 48 h and 72 h WIP times.

On the other hand, increasing WIP times did not result in any changes in the stickiness values at 24 h stoving time.

If the unit weight was considered as the only factor affecting stickiness, the lowest stickiness value was observed on the samples having 2.5 g unit weight (P <0.05) while relatively higher and similar stickiness values were found for the samples having 3 g and 6 g unit weight. According to Table 5, stickiness values were correlated with moisture content (r=0.90; P<0.05) and TSSC (%) (r=-0.89; P<0.05) like hardness value (P<0.05). Stickiness and hardness values were also found to be correlated (r: -0.770; P<0.05). This result was not surprising since it was known from the previous studies that moisture caused a sticky feeling in products. Hani et al. (2014) studied the dehydration of watermelon rind candies and in their study, it was also observed that, as the drying time increased, moisture content of the products decreased leading to reduction in stickiness values. Moisture-stickiness relation was also indicated in the study of Ergun et al. (2010). In their study, it was demonstrated that when the moisture migration occurred from surrounding to the confectionary product, due to the slow diffusion of water molecules into candy, surface layer with elevated moisture content arose first leading to increased stickiness of the candy (Ergun et al. 2010b).This phenomena was also valid for our study since high correlation was found between the moisture and stickiness values (r=0.90; P <0.05).

Table 5. Pearson's correlation between moisture content, total soluble solids, water activity and textural properties

		TSSC	Moisture	A_W	Hardness	Stickiness	T1
Moisture	R	-0.890					
	p	0.000					
AW	R	0.211	-0.137				
	p	0.322	0.522				
Hardness	R	0.701	-0.701	0.201			
	p	0.000	0.000	0.346			
Stickiness	R	-0.885	0.903	-0.220	-0.770		
	p	0.000	0.000	0.303	0.000		
T_1	R	0.401	-0.406	-0.125	0.250	-0.360	
	p	0.052	0.049	0.562	0.240	0.084	
T_2	R	0.0145	-0.021	0.020	0.051	-0.064	-0.453
	p	0.498	0.924	0.925	0.811	0.768	0.026

T_1 (Spin-Lattice Relaxation) Times

T_1 (Spin-Lattice Relaxation) time is also known as the longitudinal relaxation time and it refers the time which is necessary for spins to give back the energy that they obtained from the radio frequency pulse for turning their initial state (Ozel et al. 2017b). From previous studies, it was known that, T_1 relaxation time strongly depended on mobile protons of the free water (Ozel et al. 2017b). Therefore, it is worth to mention that, T_1 (spin-lattice relaxation time) is a great tool to detect the moisture distribution of food samples (Pocan et al. 2019a). In this study, T_1 relaxation times of oil coated gelatin based soft candies with 2.5 g, 3 g and 6 g unit weights which were exposed to different stoving and WIP times were determined and results are given in Table 6. T_1 relaxation times were expressed by using a mono-

exponential model. In order to examine the correlations between textural properties (hardness, stickiness), NMR relaxometry data were only discussed for the oil coated ones like the previous experiments.

Table 6. Effect of WIP and stoving time on the relaxation times and relative areas (% RA) of oil coated jelly candies

Stoving time (h)	WIP Time (h)	Type (g)	T_1(ms)	$T_{2monoexp}$ (ms)	T_{2a} (ms)	T_{2b} (ms)	R_{A1} (%)	R_{A2} (%)
20	0	2.5	47.42^{ab}±0.11	0.74^{bc}±0.01	0.29^{ab}±0.00	1.07^{abc}±0.00	47.50^{a}±0.35	52.50^{a}±0.35
20	24	2.5	48.10^{ab}±0.31	0.77^{ab}±0.01	0.29^{ab}±0.00	1.08^{ab}±0.01	46.50^{a}±0.35	53.50^{a}±0.35
20	48	2.5	47.57^{ab}±0.16	0.71^{cd}±0.02	0.27^{ab}±0.00	0.99^{cd}±0.01	47.50^{a}±0.35	52.50^{a}±0.35
20	72	2.5	48.16^{a}±0.26	0.61^{f}±0.00	0.24^{b}±0.00	0.88^{e}±0.01	48.50^{a}±0.35	51.50^{a}±0.35
24	0	2.5	47.73^{ab}±0.21	0.72^{cd}±0.00	0.27^{ab}±0.00	1.02^{bcd}±0.01	48.00^{a}±0.00	52.00^{a}±0.00
24	24	2.5	46.86^{b}±0.18	0.67^{de}±0.01	0.26^{ab}±0.00	0.96^{d}±0.01	48.50^{a}±0.35	51.50^{a}±0.00
24	48	2.5	47.18^{ab}±0.13	0.64^{ef}±0.00	0.29^{ab}±0.02	0.88^{e}±0.00	47.50^{a}±0.35	52.50^{a}±0.00
24	72	2.5	47.66^{ab}±0.26	0.79^{a}±0.00	0.30^{a}±0.00	1.14^{a}±0.01	47.00^{a}±0.71	53.00^{a}±0.71
20	0	3	44.07^{cd}±0.48	0.83^{a}±0.02	0.26^{bc}±0.00	0.96^{bcd}±0.01	48.50^{a}±0.35	51.50^{b}±0.35
20	24	3	44.19^{cd}±0.22	0.75^{b}±0.02	0.31^{a}±0.00	1.16^{a}±0.02	46.00^{b}±0.00	54.00^{a}±0.00
20	48	3	43.80^{d}±0.10	0.76^{ab}±0.01	0.23^{d}±0.00	0.86^{d}±0.01	47.50^{ab}±0.35	52.50^{ab}±0.35
20	72	3	44.76^{bcd}±0.22	0.74^{bc}±0.01	0.26^{bc}±0.00	0.97^{bcd}±0.03	47.50^{ab}±0.35	52.50^{ab}±0.35
24	0	3	44.22^{cd}±0.12	0.72^{bcd}±0.00	0.28^{b}±0.00	1.00^{b}±0.01	48.00^{ab}±0.00	52.00^{ab}±0.00
24	24	3	45.62^{abc}±0.29	0.67^{cde}±0.02	0.28^{b}±0.00	1.04^{ab}±0.01	46.50^{ab}±0.35	53.50^{ab}±0.00
24	48	3	45.88^{ab}±0.22	0.66^{de}±0.01	0.24^{cd}±0.00	0.88^{cd}±0.01	48.50^{a}±0.35	51.50^{b}±0.35
24	72	3	46.27^{a}±0.26	0.64^{e}±0.01	0.27^{b}±0.00	0.99^{bc}±0.00	47.50^{ab}±0.35	52.50^{ab}±0.35
20	0	6	47.85^{a}±0.26	0.66^{ab}±0.01	0.24^{ab}±0.00	0.89^{a}±0.00	41.50^{a}±0.35	58.50^{a}±0.35
20	24	6	48.16^{a}±0.75	0.58^{b}±0.04	0.22^{ab}±0.02	0.81^{a}±0.07	42.00^{a}±0.00	58.00^{a}±0.71
20	48	6	48.39^{a}±0.35	0.62^{ab}±0.03	0.23^{ab}±0.00	0.89^{a}±0.02	42.50^{a}±0.35	57.50^{ab}±0.35
20	72	6	47.70^{a}±0.36	0.66^{ab}±0.00	0.24^{ab}±0.00	0.87^{a}±0.00	41.00^{a}±0.35	59.00^{a}±0.71
24	0	6	47.57^{a}±0.17	0.75^{a}±0.03	0.25^{a}±0.00	0.96^{a}±0.01	41.50^{a}±0.00	58.50^{a}±0.35
24	24	6	47.19^{a}±0.14	0.58^{b}±0.03	0.19^{b}±0.00	0.74^{a}±0.01	43.50^{a}±0.35	56.50^{a}±0.35
24	48	6	47.40^{a}±0.43	0.67^{ab}±0.02	0.25^{a}±0.02	0.93^{a}±0.02	42.00^{a}±0.35	58.00^{a}±0.35
24	72	6	47.07^{a}±0.22	0.62^{ab}±0.02	0.23^{ab}±0.00	0.83^{a}±0.03	41.50^{a}±0.35	58.50^{a}±0.35

*Different small letters indicate significant difference ($P < 0.05$) for the same unit weight with different stoving and WIP times

Referring back to the T_1 relaxation time data that was illustrated in Table 6, for the 2.5, 3 and 6 g samples that were exposed to 20-hour stoving time, steadiness in T_1 (spin-lattice) relaxation times were observed as the WIP time increased. Similar steadiness was also observed in moisture content results for the same samples. This point is important to mention since it is known that NMR Relaxometry is a valuable tool to detect the moisture distribution of food products and strong correlations were found between T_1 and moisture contents of sponge cakes in previous studies (Botosoa et al. 2015). In addition to this study, TD-NMR Relaxometry was also utilized for the analysis of gelatin based soft candies with different formulations and similar T_1-moisture content correlations were also found in this study (Pocan et al. 2019a). Pocan et al. (2019) indicated that as the moisture content of the samples with different formulations increased, T_1 relaxation time also increased and they mentioned that T_1 (spin-lattice relaxation) time was directly related with the mobility of water. In another study, similar results were also found. Maltitol containing gelatin based soft candies was found to be the highest moisture content leading to longest T_1 relaxation times (Efe et al. 2019). Although our study is related with drying conditions of soft candies, similar moisture content-T_1 relaxation time relation was also valid for this study.

Regarding the effect of both stoving and WIP time, Pearson correlation coefficients were found as -0.96 and 0.72 for the samples having unit weight 3 g and 6 gr respectively in our study (P <0.05).

It is worth to mention that, although the effect of moisture content was dominant in T_1 relaxation times, information related with the crystallinity could be also obtained by T_1 times. For instance, when the data were examined in Table 3, it was observed that for the 6 gram samples that were exposed to constant 24-hour stoving time but not kept in WIP area (0 hour WIP time) had significantly higher moisture content compared to the samples expose to 24-hour stoving and 72 hour WIP time (P <0.05). In terms of moisture content, it was an expected result. Increasing drying resulted in reduction in the moisture content since it was known that small amount of moisture was lost to starch and candy-air surface was dominant factor during drying process (Ziegler et al. 2003). However, it was revealed that, T_1 values of these samples did not change significantly (P >0.05). According to the previous studies, longer T_1 (spin-lattice) relaxation time resulted in more crystalline regions (Le Botlan et al. 1998). Crystallinity studies were not performed in this study but it was hypothesized that, increased WIP time might have resulted in "hard skin" formation on candies' surface leading to enhanced crystallinity.

T_2 (spin-spin) relaxation times

T_2 relaxation time is also known as the spin-spin relaxation time and the changes in this relaxation time could be attributed to the various proton related alterations such as change in moisture content, exchange of protons between compartments in food systems (Pocan et al. 2019a). Therefore, in this study, T_2 values of oil coated gelatin based soft candies with 2.5 g, 3 g and 6 g unit weights which were exposed to different stoving and WIP times were determined and results were shown in Table 6.

T_2 relaxation data is generally used to get an overall signal from the sample and it could be expressed by utilizing either mono-exponentially decaying model or higher order models like bi-exponential or tri-exponential which makes it possible to take insight information from the compartmental analysis of the samples (Yildiz et al. 2018). The results of our study revealed that while mono-exponential model (R^2=0.991) was also suitable to explain relaxation data, bi-exponential model (R^2=0.999) was found to be more ideal to give more detailed information about the compartmental analysis of jelly candies used in this study. This choice was also consistent with the literature findings. For instance, time domain NMR was utilized in many studies such as to characterize gelatin based soft candies (Efe et al. 2019; Pocan et al. 2019a) and starch-soy protein based gummy candies (Ilhan et al. 2020) and in all these studies, multi-exponential model was used to interpret the relaxation data. Due to the multi-domain structure of the confectionary systems (Troutman et al. 2001), bi-exponential model was also found to be more suitable for our system. This model also gives information about the proton density contribution of each peaks (Yildiz et al. 2018). The results of both mono-exponential and bi-exponential T_2 relaxation times and the relative areas (%) of each peaks obtained as a result of bi-exponential models was represented in Table 6. The representative decaying curve for T_2 measurements and bi-exponential fitting of this data (discrete component analysis) could be also seen in Fig. 2a and Fig. 2b respectively. In this section, T_2 relaxation times will be examined as two subtitles: Mono-exponential T_2 relaxation times and T_2 relaxation spectra.

Mono-exponential T_2 relaxation times

As it was illustrated in Table 6, for the 2.5 g samples that were exposed to 20-hour stoving time, gradual and significant decrease in mono-exponential T_2 relaxation times was observed as the WIP time increased in the range between 24-72 hours (P <0.05). A similar case was also observed for the 3 g samples that were kept for 20-hour stoving time. As the WIP time increased in the range, T_2 values decreased similar to the samples that had unit weight of 2.5 g (P <0.05). T_1 and T_2 relaxation times showed an increasing trend as the moisture content increased due to the

enhancement of water mobility in the samples (Cikrikci et al. 2018). However, in our case, a different situation was observed. For the samples T_2 values decreased although their moisture content remained same. Therefore, decrease in T_2 values was associated with a different effect other than moisture. For the same samples, it was demonstrated that total solid content increased as the WIP time increased from 0 to 72 hours (P <0.05). At this point, it was hypothesized that, increasing total solid content might have enhanced solid-solid interactions of gelatin based soft candies leading to decrease in overall T_2 values. It was also important to notice that, if the unit weight was considered as the only factor affecting mono-exponential T_2 values, all mono-exponential T_2 values were found to be significantly different as seen in Table 4. (P <0.05)

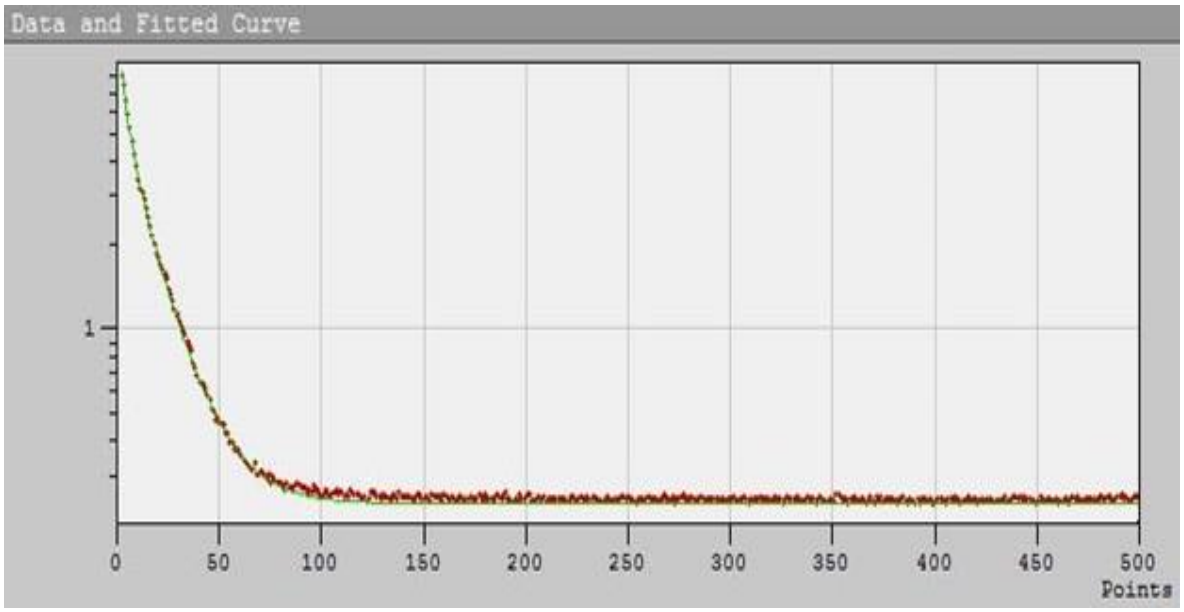

Figure 2a. Representative CPMG curve for T_2 relaxation time measurements

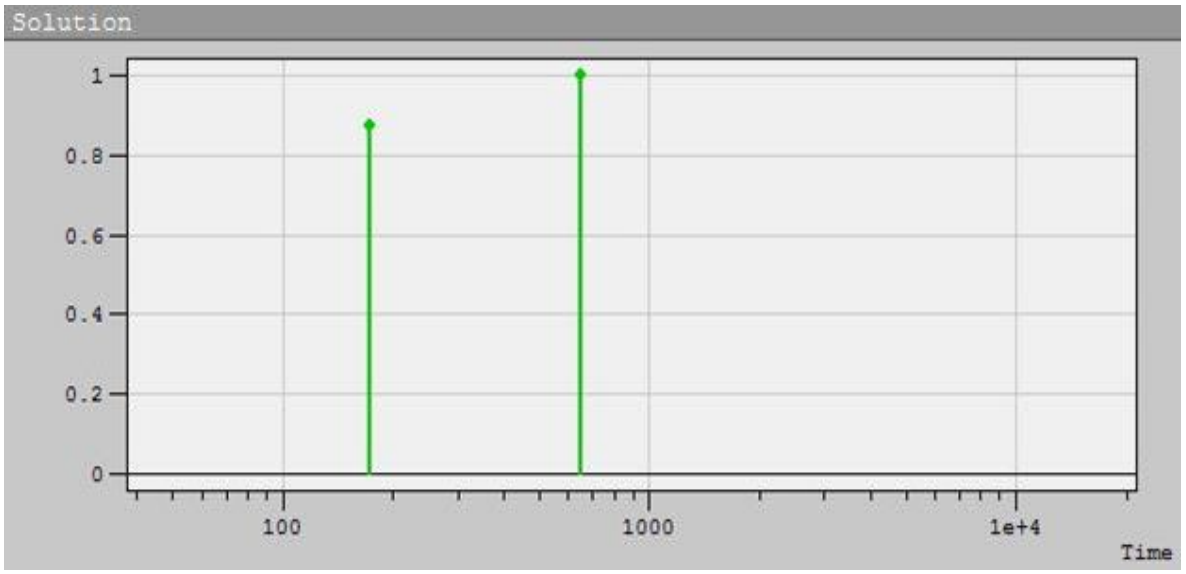

Fig.2b Discrete component analysis mode of XPFit software for a representative T_2 data

T_2 Relaxation Spectra

Discrete component analysis also validated the results of the mono-exponential fittings that described above and importance of describing the T_2 values bi-exponentially was revealed here. For the same samples that were mentioned above, a decreasing trend was observed in T_{2a} values. This was an important outcome since it was known that, compartments with the lowest relaxation times were associated with strong solid-solid interactions (Ozel et al. 2017b; Pocan et al. 2019a; Ilhan et al. 2020).

As seen from the Fig. 2b, for all jelly candies that was used in this study, 2 different proton pools with different relaxation times was observed. In a previous study, component analysis of gelatin based soft candies were also performed by utilizing TD-NMR and again 2 distinct proton pools related with different compartments were detected (Efe et al. 2019). It is worth to mention that, for these proton pools, the longest T_2 component observed in the jelly candies might have come from the more mobile water entrapped in the gel network (2nd peak), while the shortest component could be related with the solid-solid interactions like sugar-sugar, sugar-gelatin interactions (1st peak). (Efe et al. 2019). Relative areas (RA) of these peaks were calculated considering the magnitude of signal intensity, that was associated with each proton pool and they represented the contribution of these proton pools to the whole signal (Pocan et al. 2019a)

Considering RA (%) of peaks for all samples as seen in Table 6, changes in drying conditions (stoving and WIP times) and unit weights did not result in detectable changes in peak areas (P>0.05). For all samples, 2 distinct peaks were observed as seen in representative Fig. 2b. The first peak (P_1) and the second peak (P_2) indicated solid-solid interactions and bulk water entrapped in gelatin network, respectively as indicated previously. The RAs of these two distinct peaks were also found to be very similar showing that P_1 and P_2 contributions to overall proton population was nearly same. It was worth to mention that, especially such a high contribution of P_1 was the indication of strong solid-solid interactions that promoted well-developed gelatin network which was a desirable condition for jelly candies. The very small T_{2b} values also supported this hypothesis. Even though these T_{2b} values were related with relatively free water that was entrapped in gel network, they were found to be very short (in the range of 0.74-1.14 ms) compared to the findings of them previous studies. In the study of Pocan et al. (2019), related T_2 value was found to be in the range of 19-33 ms indicating that higher amount of water remained in the gel network most probably due to the improper drying conditions. Their gelatin based soft candies hardness values were also found to be in the range of 3-6 N whereas hardness values that was found in this study was in the range of 450-650 N. This huge difference could be explained with the well-developed drying condition that was utilized in this study resulting in enhanced solid-solid interactions and better gel network.

CONCLUSION

In this study, it was found that moisture content and total solid content were correlated with textural properties. However, T_1 and T_2 relaxation times were not directly correlated with the textural properties. Solid-solid interactions and crystallinity changes were thought to be the reason of that. On the other hand, T_1 and moisture content results were found to be correlated. Furthermore, regarding T_2 relaxation spectrum, two distinct peaks with different relaxation times were observed for all the samples and these peaks were found to be informative in terms of solid-solid interactions and bulk water found in gel matrices.

In manufacturing, performing fast and accurate analysis method is very important during production. According to the correlation results, moisture content, texture analysis and total soluble solid content could be used to control the quality of the products. However, moisture content analysis takes long time and making decision is very hard based on just moisture content while products are in oven because of the process time. Texture analysis is fast but very difficult method to make decision just after

completing stoving cycle since products have a sticky surface. So, total soluble solid content and TD-NMR results could be used together to control the quality of products with acceptance of small error and variation.

For this study, 20 h stoving time was accepted as the reference. Then, stoving time was extended 24 h in order to reach better textural properties. In the light of this study, it was recommended that 2.5 g samples' stoving cycle can be decreased 20 h again. Thus, 4 h stoving time and accordingly energy can be saved. In addition to that, all samples (2.5 g, 3 g and 6g) are kept 72 h on conditioning area before packaging and it was recommended that, 3 g and 6 g samples' texture can be better if they are not waited more than 48 h in conditioning area. This study also suggested that T_1 and T_2 relaxation times could be effectively used to monitor the changes during drying of gelatin based soft candies together as an alternative to the conventional methods such as total soluble solid and moisture content and it was believed that obtained results will pave the way for the utilization of TD- NMR relaxometry in confectionary industry.

CONFLICT OF INTEREST

The authors declare that they do not have any conflict of interest.

AUTHOR CONTRIBUTIONS

The study was the Master thesis of Ms. Kaya. Ms. Pocan prepared the 1st draft of the manuscript. Dr. Oztop and Dr Mert were the advisors of Ms Kaya and finalised the manuscript.

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