Baking characteristic, color and rheological properties of frozen pizza dough under different freezing conditions by using a superheated steam (SHS) oven

A thesis presented

by

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Submitted to the Graduate school Agricultural and Life Sciences, the University of Tokyo in partial fulfillment of the requirement for the degree of

Master of Agriculture

July, 2016

International Program in Agricultural Development Studies (IPADS)

Acknowledgement

I want to deliver my full gratefulness to all the people who have been sharing with me laughter, depression, sunshine and loneliness, to every encouragement and assistance that kept pushing me moving forward, to each single minute which has been telling me that this would be the very unique period of life of mine, in this well-organized, graceful, fascinating but also silently stressed city, Tokyo.

It is hard to believe that one day I may forget the time spent here. The very first moment when I stepped out of the plane from Narita Airport, after which a fat middle-aged Japanese man who was the headmaster in the Japanese language school that I attended before I came Todai, spoke purely Japanese that stressed me so much for it was nearly the first time I had ever talked in Japanese with a real native-speaker. I will not forget all those architectures, sceneries, neon light and gigantic posters outside of those buildings on the walls. They were so strangely exciting for me, meanwhile I was remotely unsettled for them. On earth, I was merely an inexperienced man who was expecting to see a bigger world while not knowing what was in front of him, from a remote small town where people had seldom heard of.

But an unbelievable thought, or better to use the word “dream”, was actually rooted in my heart. I came here for the pursuit of a university which was named, Todai, even though the vice-headmaster in the Japanese language school who had listened to my reason of why I came Japan, only left me with a few words emotionlessly but in a very certain kind of tongue-“There is no way”.

Not only one time had I been still recalling to the memory on that day, when I was dressed in suit, standing on the top of Second B Annex, looking down at the green leaves

dancing in the wind, while grabbing the bag and documents that were all prepared for that interview with Araki sensei with my sweated hand. I was questioning myself, “Would I had any chance to come again?”

The next time when I stepped into this campus once again, I just felt everything, everything I experienced had made all worth of it. All the effort, stress, failure and self-questioning that many people may need to undergo unwillingly, did not beat me down but had taught me a good lesson-“Never give up”.

With no question the life in Todai has been way better than it was in the Japanese language school, although I came here still with some uncomfortable disbelief that was marked deeply in my mind due to those stressed, not-too-good memories in the past, none of which deserved to be written here, however. It only turned out that none of my pitiful worries was necessary.

Araki sensei, in the first place, was so considerate in so many things that you even gave me the permission to do my job-hunting back in my country for over three months. Without that opportunity, I would have enough reasons to be continually stressed up till the day I find one. Not to mention your help and concern in other places because it will take too long to cover them all. Special thanks to the night sensei, Liu san and I shared those topics and alcohol just as “can’t-be-closer” friends. It was an amazing night. I think it will be an unforgettable night for Liu san as well.

Sagara sensei then, who behaves just like my old grandparent, offered me so much not just help, but also warmness. I was honored being trusted to share your office room’s key, your snacks in the freezer, your beer and wine (bought only for me because I do not enjoy the flavor of beer as a matter of fact) and your toilet paper (lol). I was treated so

many times either in those Izakaya or some other restaurants. And thanks for your invitation and payment of the transportation fee for attending the conference, which only turned out to be my personal trip around Kansai area. I will always remember those conversations between us, in which you spent so much time and effort on educating me.

Okada sensei, Kobayashi sensei and Takahashi sensei who have been taking so much care of IPADS students, thanks for the nice courses inside the paddy field, taking part in wheat harvesting, wine trip to Nagano and Yamanashi. They have all become parts of my colorful life in Todai.

Thanks for Kouno san and Hikaru san, who helped me very much in my research. Thanks for your precious time squeezed out from other works. I appreciate all the cooperation offered from Mayekawa Manufacturing Company, your time, support and staff canteen had been of great help.

I know many people like writing a lot "thanks" to their friends in acknowledgement, but some of them were as if in a tongue like saying goodbye forever. I do not want to write in that way because I have solid confidence we will meet in the future, in this smaller and smaller world again, and not only once. Members in our lab room, Morita san, Jaki, Sasao san, Keren, Xiuming, Xueting, Xilin, Hosoe san, "Mr. good man", Malica, Ishiwata san, Matsuki san, Manish, Sato san, Kishida san, Morikuchi san.. Members outside our lab, Nigel, Jade, Naho, Mariko, Mai, Javid, Jason, Mihray, Yimon, Aung, Nuwan, Mie san, Ichimura san, Kitamura san, Takada san, Hoang. We had so many good memories, you guys really have colored my life in the lab.

I still remember the very precious email which Araki sensei wrote to me, one of the sentence was "One best thing you can do in your life, is to keep learning in this long

run”. When a person really becomes one that does not need to be pushed hard on the back to study, not only for tests, not only for money but more for his real development, one day he grows up. If I need to tell what I think is the essence in the education I get from Todai, I think that will answer.

Yes, life is long, or short. One part of my life ends up here with this university permanently, another part, however, is just going to start off on the way.

Best wishes to everyone, to everything, to the old days and to tomorrow.

Regards,

Lingke Meng

Baking characteristic and subsequent properties of frozen pizza dough under different freezing conditions by using a superheated steam (SHS) oven

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A new processing method of utilizing superheated steam (SHS) on one of the most highlighted frozen food-frozen pizza product is tested, based on the advantages of using SHS as a processing medium such as lower energy cost, higher heat transfer coefficient compared with its counterpart hot air (HA) medium. This research aims to give a better understanding on the evaluation for the baking characteristic and subsequent quality of frozen pizza, including thawing rate, baking time (till 100 ℃), drying rate, crust colour and rheological properties. HA is used as the comparison baking medium for comparison together with SHS throughout the whole research steps.

Materials and methods

Samples ingredients include: Rustica pizza flour (100%), salt (1.74%), sugar (2%), olive oil (3%), dry yeast (0.5%) and water (58%).

The flow of the research steps follows such an order: 1) mixing (7 min), 2) dough preparation (divided into 75g per sample, moulding), 3) freezing (-5, -15, -25, -35 and -45 ℃), 4) frozen storage (1, 15, 25 and 35 days), 5) sample transportation, 6) baking (HA and SHS, 260 ℃), 7) instrumental measurement (color and rheological properties including creep test, fractural test and TPA test) and 8) data analysis (R programming language: Student t test, ANOVA and TukeyHSD). An experimental SHS oven, by which both SHS and HA baking environment are available, is developed and used in this research. In the creep test and the fractural test, the crumbs and crusts of the samples were tested separately.

Results and discussion

Freezing characteristics

Freezing rate was higher in the samples frozen under lower freezing temperatures. Nonetheless, throughout the whole research, different freezing temperature did not have significant influence on either color or rheological properties in the samples baked by HA

or SHS, except thawing rate due to the temperature difference between frozen pizza dough and baking medium.

Baking characteristics

Compared with the HA baked samples, the samples baked by SHS showed significantly higher thawing rate due to higher heat transfer properties. Apparent correlation was found between baking time and freezing temperature, whereas such correlation was not found in the SHS baked samples. Higher drying rate of the SHS baked samples were observed, which was due to higher heat transfer properties and easier vapor diffusion environment in SHS filled space.

Colour change

Crust colour of the samples baked by SHS had significantly darker, yellower and redder outcomes than the HA baked ones, which was attributed to the accelerated Maillard reaction due to higher heat transfer properties of SHS.

Rheological properties

In the SHS baked samples, crusts were found to be more viscoelastic than crumbs whereas this was exactly opposite to the results from the HA baked samples. In fractural test, no breaking points were found except for the samples frozen under -5 ℃ and 15 days of frozen storage. This was because of the extremely dried condition inside the samples, indicating that fractural test is suitable for the samples in this research. No significant difference was found in hardness, cohesiveness or gumminess in comparisons between the crumbs of the samples baked by HA and SHS.

Conclusions

As a new processing method compared with HA, SHS baking showed the potential to give frozen pizza products higher thawing rate, less baking time, higher drying rate, quick browning ability and different viscoelastic properties between crumbs and crusts. The combined effect of initial condensation, drying rate especially at the top layer of pizza, crust thickness and water redistribution from crumbs to crusts might have played the most important role which differentiated samples baked by HA and SHS. Different freezing temperatures from -5 ℃ to -45 ℃ did not seem to cause any significant difference in colour or rheological properties in the samples baked by either HA or SHS.

Chapter 1. Introduction

1.1. Superheated steam (SHS)

1.1.1. Brief introduction to SHS

Superheated steam (SHS), namely the saturated steam that is “super” or “over” heated, is able to reach higher temperature than its vaporization (boiling) point at the absolute pressure where the temperature is measured. SHS has a history over 100 years, but only received serious attention during the past 20-30 years, such as in industrial engine working and drying area. Superheated steam drying systems begin to gain industrial acceptance with 15 systems operating in several European countries and recently, two superheated steam driers have been installed in the United States to dry sugar-beet gulp (Bruce & Hulkkonen, 1998). However, utilization of SHS is still not well spread yet due to a lack of knowledge about the process and effects of SHS on product quality (Pronyk, Cenkowski, & Muir, 2005).

1.1.2. Advantages and disadvantages

Currently SHS is still utilized mainly in industrial drying process, as it has several advantages in playing as a drying medium as were pointed out by (Lane & Stern, 1956) (Devente & Heijmans, 2001) (Shitata & Mujumdar, 1994) (Tang & Cenkowski, 2000):

1) Able to recover latent heat that was supplied to SHS, thus reducing cost.
2) When drying food, nutrients inside might be well preserved due to a lack of oxygen within SHS drying system.
3) Higher drying rate possible.

4) Toxic and organic liquids can be recovered easily by condensation, such as wood drying.
5) Sterilization, deodorization, blanching, boiling and cooking can be performed simultaneously.
6) Comparatively safer drying operation available (no fire or explosion hazard because of oxygen-free environment created).

Note that sterilization is considered as a benefit here, whereas this aspect of characteristic would be adverse for other occasions, for instance in certain varieties of food stuffs that essential microbial reactions are desired.

Aside from advantages, disadvantages were also reported by (Mujumdar & Devahastin , 2008):

1) More complex than hot air method.
2) Initial condensation is hard to avoid.
3) Products that may melt would undergo glass transition.
4) Industrial experience is limited

1.1.3. Comparison between steam, saturated steam and SHS

Commonly for comparison, the counterpart drying or baking medium is hot air, which is same within this research, since hot air (HA) and SHS could be considered as extreme situation within the inner baking environment (Yoshida & Hyodo, 1963).

Steaming (not saturated), is believed being able to improve the quality of bakery products such as color and moisture loss. The difference between steam and SHS is that steam causes vapor condensation on the surface of the baked material, forming a thin film

which separates material and baking medium from direct contacting, whereas in SHS processing, no such vapor condensation happens except for the initial condensation due to the overlarge temperature difference between materials and SHS. In a sense, steaming process delays the whole baking process and even delays the crust formation. Additional moisture absorbs heat from the oven, thereby lowering the surface temperature from the materials and thus greatly impacts the quality of products (Mahini, Tarzi, & Ardebili, 2013).

As was reported by (Lalonde, accessed on 13th, June, 2016), saturated steam is often more preferable when utilized to obtain better heat transfer capacity, since the temperature of saturated steam is uniform, no temperature profile can appear between steam and heating surface. When heating with saturated steam, a thin layer of condensate also forms on top of the products. The thickness of condensation film is relatively even over the entire heating surface and the flow equalizes the temperature of the film and prevents the formation of a temperature profile, which helps main the heat transfer between steam and liquid to a maximum.

Compared with saturated steam, SHS is actually a heat insulator which conducts heat badly than saturated steam, even though it is hotter and contains more energy. One of the superior merits of introducing SHS as the drying system is that a drop in temperature will not result in condensation of the steam as long as the temperature is still greater than the saturation temperature at the processing pressure (Pronyk, Cenkowski, Eng., & Muir, 2004) (Mujumdar & Devahastin , 2008). Also, no condensation exists except for the initial condensation due to the distinct temperature difference between the products and SHS at the beginning stage where baking starts.

1.1.4. Working principle

The working principle of SHS drying is sensible heat transferred from superheated steam to the products to be dried. During the whole process, SHS stays above its condensation temperature (depends on the drying system pressure and products temperature). During drying, SHS plays not only the drying medium but also as a heat source. Water removed from the product during baking process becomes part of the medium whereas in hot air drying, the moist air must be replaced by fresh air heated to the desired temperature (Moreira, 2001). During drying, the excess steam, as well as steam evaporated from the product, can be collected (if possible) and can be used elsewhere, in other processing part or plant, hence a more efficient usage of energy is feasible. Emissions during drying, such as polluted or toxic substances, mixed with drying medium, can be effectively removed through condensation in liquid form and then be separated and drained. Pollutants that can be collected by condensation, can be burned to clean.

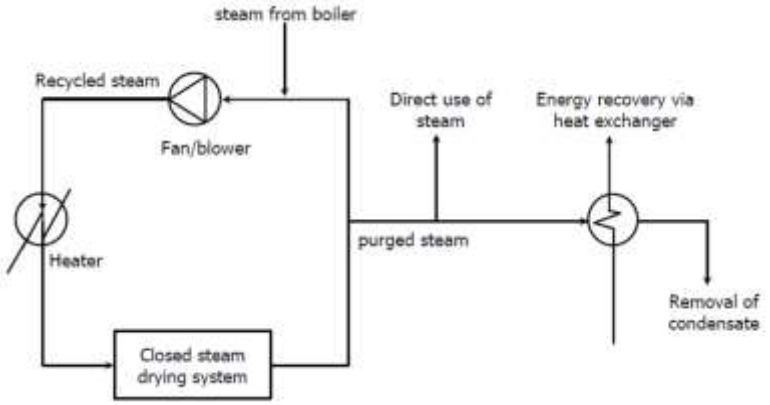

Figure 1-1 Principal scheme of the SHS drying process (Mujumdar & Devahastin , 2008)

1.1.5. Previous research on SHS

1.1.5.1. Utilization of SHS on food stuffs

SHS has been proved to be beneficial as a drying medium in many materials, especially in non-temperature-sensitive materials such as coal (Moreira, 2001) (Li, et al., 2011). It has also been suggested that the future utilization of SHS may not always be in the form of strictly drying, but also decontamination and product processing. Although SHS is still mostly utilized in drying processing industrially, some researches had been conducted on its usage in food stuffs as well. Food such as sugar-beet, pulp, potatoes, Asian noodles, brewers and distillers' grain were test as research samples. It was shown initial condensation was not able to be avoided because of the comparatively cold temperature of food at the beginning when processing started. Higher steam temperature was found to lead to decreased drying time and an increase in drying rate. Some quality of processed products could be beneficially influenced such as an improvements in recovery, adhesiveness and gumminess of Asian noodles, starch gelatinization and dietary fiber contents of spent grains. On the other hand, SHS was shown not always beneficial as a processing medium because some parameters like maximum cutting stress, resistance to compression and noodle surface firmness of Asian noodles were deleteriously affected (Pronyk C. , Cenkowski, Muir, & Lukow, 2008).

1.1.5.2. Drying characteristics of SHS

The difference between SHS and HA drying is in diffusion step. In HA drying, diffusion of moisture occurs from inside the product to its surface. In SHS, evaporation of moisture occurs within the material causing cells to expand as moisture turned into steam which leads to a highly porous dried material (Sehrawat, Nema, & Kaur, 2016).

Compared with HA, SHS drying had higher vapor evaporation rate (Chu, Lane, & Conklin, 1953). The drying rate increases as the steam temperature increases for the same convective heat transfer coefficient. Higher steam temperature also results in more pores and coarser appearance, lower degree of shrinkage, higher modulus of deformation. Highly dried samples had less gelatinized (Moreira, 2001). Compared with HA drying, initial condensation was detected in SHS drying due to an overlarge temperature difference between product and SHS. This stage is very important for SHS processing because it affects drying kinetics (Bourassa, Ramachandran, Paliwal, & Cenkowski, 2015). The SHS coming in contact with the product then became cooled and condenses on the material surface as sensible heat was transferred to the material, which gave a quick rise in the internal temperature in products (Iyota, Nishimura, Yoshida, & Nomura, 2001). During the initial condensation, tested samples including sugar-beat, pulp, potatoes, Asian noodles, brewers and distillers' grain gained small amount of moisture in the first few seconds before the temperature of the products increased to the saturation temperature (100℃) (Pronyk, Cenkowski, Eng., & Muir, 2004). The condensed water even permeated into the interior and just after evaporation from the surface began, a region slightly inward from the surface had higher moisture content than its initial value, in the research tested on potato slices (Iyota, Nishimura, Yoshida, & Nomura, 2001).

1.1.5.3. Coloring ability of SHS

A color measurement research was conducted by (Iyota, Sakai, & Mamiya, 2013), sliced bread were baked under hot air (HA) and SHS method, CIE lab parameters were measured. It showed that in the case of heating with SHS, the value of L* (brightness) decreased more quickly and b* (blue to yellow) had a lower value than the counterpart

baked with HA. In this research, CIE lab parameters of samples before and after baking were also measured to check the influence between the two baking method.

Another research of using SHS on the processing of oat groats (Head, Cenkowski, Arntfield, & Henderson, 2010) found that after processing with SHS, groats were brighter, yellower and redder than the unprocessed groats. Also, Color of oat groats processed with SHS changed significantly ($p<0.05$) during the initial exposure to SHS, but no major changes in the color were noted after around 1 min of SHS processing.

1.1.5.4. Sterilization ability of SHS

Another research was conducted revealing the microbial decontamination characteristics of SHS processing method on wheat grain (Hu, Nie, Hu, & Li, 2016). Different steam temperature, processing time and SHS velocities were used to be compared. It showed that decontamination was efficient at higher SHS velocity, with a distinct decrease in residual levels of bacteria. After SHS treated at 200℃, with 15.0 m^3/h for 80s, approximately 99.9% of total bacteria and 81.8% of Bacillus spp. were reduced. In this, research, although the velocities of SHS were not measured, the air temperature was controlled at around 260℃ more baked with baking time much longer than only 80s, meaning an almost extinction in microorganisms in samples.

1.2. Frozen pizza products

1.2.1. Chance and challenges for frozen food

Shifts in global economic, social and demographic trends will continue to put pressure on food supplies, creating new challenges for food manufacturers and consumers. These changes will significantly reshape the context for food and perceptions from consumers. Along with the global populations growing and increasing average incomes,

diet will change to include more meat and dairy products in countries especially those in the developing world such as China and India. To ensure all the needs for the diet change in the emerging market in the future, frozen food could be crucial due to their advantages for preservation, i.e. long shelf life of the product. So there is a growing desire to understand more about the frozen food we eat, including the quality, safety and nutritional aspects and so on (Board Bia & The Futures Company, 2013).

The commercial production of frozen bakery products mainly began during the 1950s, started to gain popularity ever since (Virginia & Tzia, 2007). Despite additional costs for freezing, transportation and frozen storage, the use of frozen dough can be attractive, especially when producing freshly baked products of high add-values at relatively expensive locations (Asghar, Anjum, Butt, Tariq, & Hussain, 2007). Use of frozen dough permits large scale centralized dough production, distribution and storage of dough in the frozen form and relatively small scale point-of-scale baking (Phimolsiripol, Siripatrawan, Tulyathan, & Cleland, 2008). Frozen dough should have 16 weeks shelf-life if the dough has not been temperature abused during transportation and storage. As various specialty food products such as frozen pizzas have been developed, their popularity has increased even further, so that at the present time frozen pizzas and like products are very important commercially.

1.2.2. Over view of global market of pizza

An overview report of the situation for global pizza industry was given by (PMQ Pizza Magzine, 2014). Still concerned as western food, pizza industry in Western Europe, North America and Latin America occupied the largest market shared globally (See *Figure* 1-2). It was safe to say the pizza restaurant industry had entered the mature stage

of its life cycle in these regions, especially in the US. Many towns and cities had reached the limit of pizza restaurants that their populations could support. That made it harder for operators to open new stores and for new operators to get into the business.

However, as an emerging market, pizza industrial growth in Asia Pacific regions was the most remarkable highlight (27%) among all other regions(see *Figure 1-3*), leading the global pizza market growth rate. The global diet transition, had brought the popularity of western food like pasta and pizza to other areas including Asia Pacific, Middle East, Africa and East Europe. When pizza becomes more and more accepted, consumers may want to try to make their own pizzas as well. Frozen pizza thus came into the sight of consumers step by step, offering not only convenience, but also various flavour by varied toppings, well-preserved nutrients inside, cheaper price and longer preservation availability.

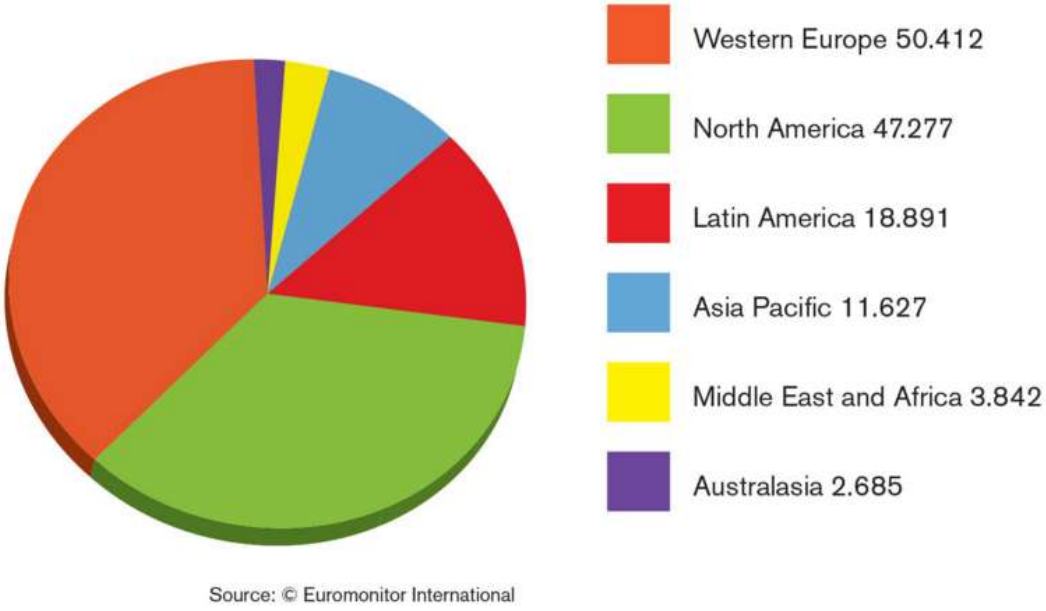

Figure 1-2 World pizza market (Billions of dollars) 2014

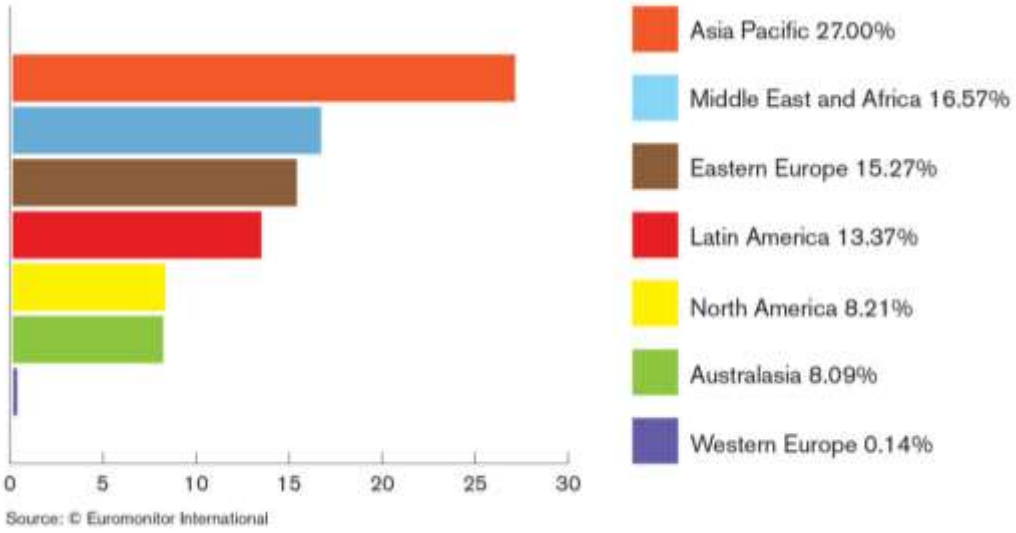

Figure 1-3 Pizza industry international growth rates 2014

Considering the skill-requiring, time-consuming or other restrained conditions in the kitchen, the introduction of frozen pizza, including frozen ready-to-eat pizza products as well as frozen pizza crust and dough, might be beneficial and thus get serious attention.

Frozen bakery products including pizzas have a bright prospect in market growth annually as shown in *Figure* 1-4 and *Figure* 1-5.

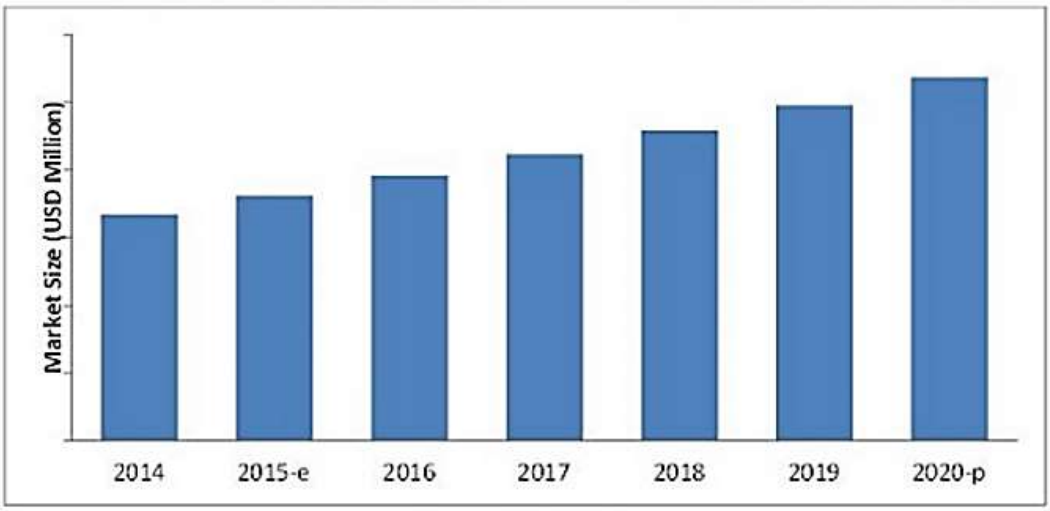

Figure 1-4 Frozen bakery products market size trend, 2013-2020 (USD Million).

Source: Industry Journals, Related Publications, Company Publications, and MarketsandMarkets Analysis.

Within all frozen prepared foods, frozen pizza has the greatest penetration as much as 57 percent to American households. Demographically speaking, frozen pizza consumption is highest among people 18-44 years of age, with consumption dropping in age levels 45 and up in America. Asian-Americans are less likely to consume frozen pizza than whites and blacks (Holcomb, 2003).

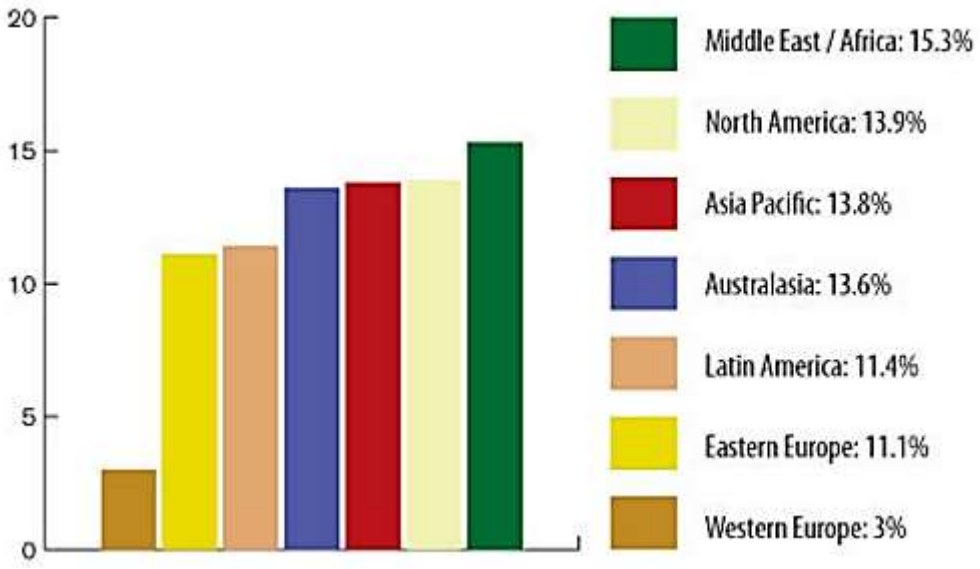

Figure 1-5 World frozen/chilled pizza growth (compound annual growth rate) Period: 2007-2012. Source: Euromonitor International

In all, the simplistic nature of frozen pizza has provided convenience to consumers for decades. The various options for toppings have allowed frozen pizza to transcend the traditional image of Italian food and provided the variety that consumers desire.

1.2.3. Previous studies

1.2.3.1. Effects of frozen storage on the quality of wheat flour doughs

Several problems arising from the production made from frozen dough have been described, including 1) gradual loss of dough strength, 2) decrease in the retention capacity of CO_2, 3) longer fermentation time, 4) reduced yeast activity, 5) lowering of loaf volume, 6) deterioration in the texture of the final product.

The influential factors are containing:

1) The formula ingredients (Salas-Mellado & Chang, 2003) and specifically the effect of: emulsifiers, shortenings, enzymes or yeast (Ribotta, Leon, & Anon,

2001) (Mandala & Sotirakoglou, 2005) (Rosell, Rojas, & Barber, 2001) (Wolt & D' Appolonia, 1984) (Autio & Sinda, 1992). The dough formulation significantly influence the specific volume and crumb hardness. The type of wheat flour used for frozen dough production is crucial in imparting desirable baking characteristics after prolonged frozen storage and the influence of flour type is evident. Supplement of improvers such as gluten and guar-gum improved the specific volume and form ratio but this was not decisive because excessive use of hydrocolloids might cause destabilizing or firmer texture of dough due to the interference with gluten proteins or starch polymers matrix. The problems of reduced carbon dioxide production or the damage of the gluten network may be overcome by adding improvers. Nevertheless, no one particular improvers resolve all the issues pertaining to products made from frozen dough.

2) The process parameters such as freezing rate, storage time were announced by (Yi & Kerr, 2009) (Giannou, Kessoglou, & Tzia, 2003) (Giannou & Tzia, 2007) (Salas-Mellado & Chang, 2003). Ice crystal formation and recrystallization, water redistribution, yeast destruction, damage to gluten network and starch granules possibly are the direct reasons lead to the change to dough properties (Berglund, Shelton, & Freeman, 1991) (Varriano-Marston, Hse, & Mahdj, 1980). Dough frozen at relatively slow freezing rates and stored at higher temperature (-10 ℃ to -20 ℃), was softer, had higher specific volume and was lighter in color, yet had greater propensity for staling. In the frozen dough industry, protecting against the effects of freezing on yeast cellular viability is a main focus of attention. Therefore, slow cooling in a conventional freezer is the preferred technology instead of

cryogenics, which gives much more rapid cooling but is supposed to be more detrimental to the survival of yeast cells in dough pieces (Neyreneuf & Delpuech, 1993).

1.2.3.2. Experimental pizza crusts with toppings

By far the studies on pizza products is still limited, due to its difficulties to standardize the samples for experimental purposes. Pizza is a flatbread generally topped with tomato sauce and cheese and baked in an oven. It is commonly topped with a selection of meats, vegetables and condiments. The term was first recorded in the 10th century, in a Latin manuscript from Gaeta in Central Italy. The modern pizza was invented in Naples, Italy, and the dish and its variants have since become popular in many areas of the world. It will be very difficult and complicated to conduct research on the pizzas with toppings. A comparison between the doughs cooked by itself and the doughs cooked with toppings on the top of the crust was asserted by (Mckee, 1997). The toppings would act as a thermal insulator for the raw dough as heat was driven into the top of pizza. Whereas, when without toppings, more heat would be driven into the top layer of pizza, which would result in a tougher crust. Also, toppings are always with at least a certain moisture vapor permeability which would act as a barrier to the escape of moisture vapor driven from the top of the pizza during the cooking procedure. On the contrary, when without toppings, the dough would be drier. On the other hand, the weight of the toppings also increases the difficulties of researching pizza. In a word, it remains hard to research on the properties of pizza with toppings before all the influential factors are well controlled.

While conducting organoleptic evaluation of pizza products, pizza toppings were carefully selected by either a thin dressing on the top of crust by tomato sauce but not covering the crust flavor (Delahaye, Jimenez, & Perez, 2005), or clearly listing the ingredients and their proportion inside the toppings (Asghar, Anjum, Butt, Tariq, & Hussain, 2007)

1.2.4. Pizza baking methods

Historically, pizzas have been baked in deck ovens for a time sufficient to bake the crust and to bake, heat and melt the ingredients atop the crust. Typically the required baking time would be around 15 to 25 minutes per pizza (depends on the baking temperature). This too long waiting time for baking lowered satisfaction of customers. Likewise, decreased the efficiency and profit for pizzerias (undesirable occupation of time and space) (Cox, et al., 1995).

One improvement to the art of baking pizzas have been the development of the conveyor oven. Through the use of conveyor ovens, it has been possible to improve the consistency of baking, ease of operation and utilization of available floor space. For high volume pizzerias, conveyor ovens have been the industry standard.

Another improvement, air-impingement ovens, are used for baking and cooking such as pizza crust, tortilla and potato chips, pretzels, crackers, cookies, bread and cakes (Moreira, 2001). Compared with convection ovens, impingement drying methods generally bake faster and more uniformly, due to its faster air flow speed as was mentioned in (Pizza Today, 2012).

1.3. Objective of this research

By far, using SHS as baking medium for frozen pizza crust has not been a well-known method and almost there was no previous trials on pizza products in this way. Based on the unique advantages of SHS processing as was noted above, a new baking method of utilizing SHS on frozen pizza product is conducted in this research. As a potential processing medium, SHS is still lack of industrial knowledge and experience. Aside from the advantages, it is also important to investigate on feasibility of the baked products in this method, such as physical properties, nutritional and healthful aspects, sensory scores, environmental impact as well as cost and profit.

This research mainly focus on the two major points in order to give a better understanding of SHS processing on frozen pizza products:

1) Baking characteristics of pizza crust frozen under different temperature and storage duration by SHS, including thawing rate, baking time and drying rate，color change of SHS baked samples and rheological properties of SHS baked samples.
2) Comparison of quality of baked samples between HA and SHS baking methods is simultaneously conducted.

Chapter 2. Materials and methods

2.1. Sample ingredients

Rustica pizza flour (Nisshin Seifun Group), salt, sugar, olive oil, dry yeast and water were used as ingredients for making pizza sample in this research (as shown *Figure* 2-1). These ingredients follows the basic pizza making method from the research done by (Asghar, Anjum, Butt, Tariq, & Hussain, 2007) .

Figure 2-1 Sample ingredients

2.2. Flow chart of research steps

In *Figure* 2-2, the flow of the main research steps was listed in a flow chart. Samples underwent mixing, dough preparation, freezing, frozen storage, transportation, baking, instrumental measurement and obtained data were analysed. Details for each of the steps will be depicted in following sections.

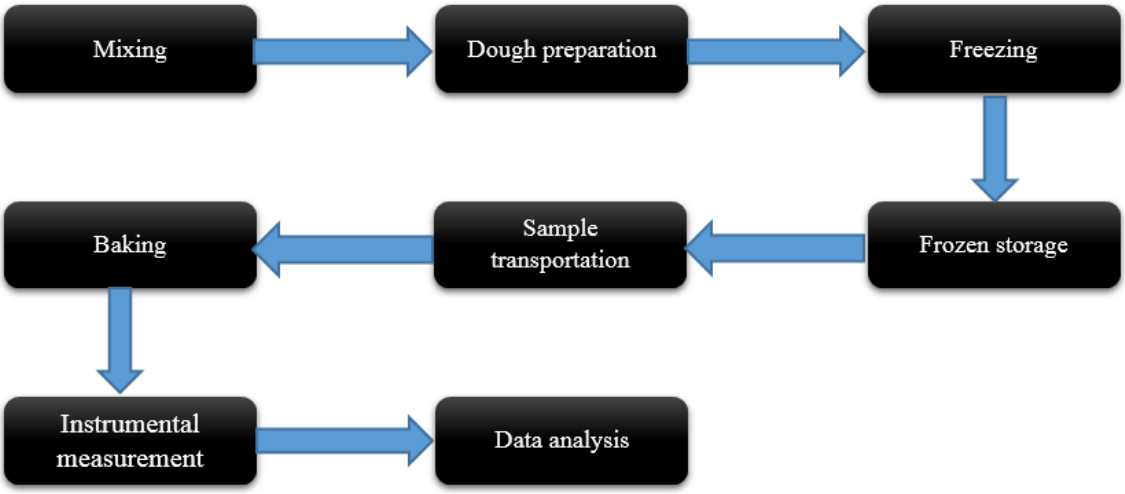

Figure 2-2 Flow chart of the research steps

2.3. Mixing

Figure 2-3 Kitchen-aid heavy duty mixer

All ingredients were mixed according to a proportion as listed in the *Table* 2-1, by a mixer (Kitchen-aid heavy duty plus stand mixer, see *Figure* 2-3). The order of materials put in the mixing bow l was 1) flour, 2) yeast melted in 40℃ water, 3) salt, 4) sugar, 5) olive oil. All the materials was slowly mixed for 1 minute and after the dough

stuck together the mixing speed was turned to medium-slow for another 6 minutes. The mixing time of dough could be very significant, as pointed out by (Dobraszczyk & Salmanowidcz, 2008) that dough film rupture could be associated with how well the dough is kneaded. Under or over kneading might lead to easier dough film rupture, which no longer is able to withstand pressure and thus restricted the growth of gas cells within dough. There are many sayings about the standard of mixing time, according to what (Correll, 2011) suggested, mixing time for pizza dough was initially based on the mixing time for bread dough. However, if so, it would made the mixed pizza dough soft enough while not stiff, which is also one of the desired characters of pizza dough. Soft dough, medium-soft, medium, medium-stiff, stiff were five rankings for mixed pizza dough evaluation and among them medium level which required around 6 to 9 minutes of mixing was selected in this research. After mixing was completed, the dough was taken off from the hook of the mixer and was rested for ten minutes, with an aluminium paper covered for a short-time pre-proofing.

Table 2-1 Sample ingredients proportion

	Proportion/%	**Weight in each pieces/g**
Flour	100	45.4
Salt	1.74	0.8
Sugar	2	0.9
Olive oil	3	1.4
Dry yeast	0.5	0.2
Water	58	26.3

Total mass	75

2.4. Dough preparation

Once the dough was pre-proofed, they were divided into 75 g for each pieces, then shaped and rounded by a sample mould (with a 100 mm diameter and 10 mm height, cut from plastic container),as shown in *Figure* 2-4.

Figure 2-4 Samples shaped by a simple mould

Sample in which the freezing temperature needed to be measured, was fixed in a sample holder which was designed for this research. Three iron needles (シリンジ.ノズル UD 15G*150mm), which were hollow inside, had holes opened right in the middle from either side. Thermocouples ((Brand RS, K type, PFA twin twisted pair thermocouple cables, single core strand diameter 0.5 mm, minimum operating temperature: – 75 ℃, maximum operating temperature: 280 ℃) were then inserted and penetrated out of the pipes through the opened holes, by which three exact points were fixed for recording the temperature in samples during freezing in the upper, core and lower layers, respectively.

The distance of centre point between upper to centre and centre to lower needles in the sample holder were equally 5 mm, as shown in *Figure* 2-5.

Figure 2-5 Samples with thermocouples inserted in three layers, separately

2.5. Freezing

Hitachi thermostat chamber, low temperature series EC-33LTP, provided by Mayekawa manufacturing company (see *Figure* 2-6), was used in this research for freezing process. After all samples were ready to freeze, they were put on stainless steel net, frozen under -5 ℃, -15 ℃, -25 ℃, -35 ℃ and -45 ℃, separately. Initial temperatures in all samples before freezing were around 25 ℃. During freezing, inner temperature of samples were measured using thermocouples (Brand RS, K type, PFA twin twisted pair thermocouple cables) connected to a data logger (midi LOGGER GL220, GRAPHTEC, see *Figure* 2-7).

Figure 2-6 Hitachi thermostat chamber, low temperature series (EC-33LTP)

Figure 2-7 Graphtec data logger

The temperature of upper, centre and bottom layers in all samples were measured and freezing was stopped when the centre temperature in samples reached down to the freezing air temperature. In order to capture a better freezing performance also prevent too much moisture content loss, surface of samples were slightly covered with preservative film while not sealed too well, so that cold air generated from the refrigerator was ensured to have a good contact with samples (see *Figure* 2-8). After freezing process completed, samples were packaged by zip-loc.

Figure 2-8 Research samples freezing process

2.6. Frozen storage (Appendix 2, additional studies)

After the core temperature had reached down to the desired temperature, samples were kept frozen in storage refrigerator (SANYO, MDF-U536D). Groups of samples were separated in terms of 1 day, 15 days, 25 days and 35 days frozen storage, under temperatures same with their freezing temperatures, respectively. Among them, 1 day frozen samples were triplicated, whereas due to availability, samples frozen for 15 days, 25 days and 35 days were not repeated, thus 3 samples each for all 5 different freezing temperature (-5 ℃, -15 ℃, -25 ℃, -35 ℃, -45 ℃), under 1 day frozen storage. 1 sample each for 5 different freezing temperature under 15 days, 25 days and 35 days frozen storage was used for additional studies which was introduced in *Appendix* 2. Samples frozen under -5 ℃ were frozen up to 15 days due to its difficulty to maintain the comparatively higher freezing temperature. Frozen dough should have 16 weeks shelf-life if the dough has not been temperature abused during transportation and storage (Phimolsiripol, Siripatrawan, Tulyathan, & Cleland, 2008). However, due to the

availability in the schedule, samples were only remained as long as 35 days frozen storage to see if there had any significant difference generalised by frozen storage duration.

2.7. Transportation

Figure 2-9 Cooler box containing dry ice for frozen sample transportation.

When frozen samples reached the desired storage date, they were taken out from the storage refrigerator, transferred to a cooler box (I-BEAM, IJSSEL, *Figure* 2-9). Dry ice were used to prevent temperature rise inside. Samples were posted from Mayekawa manufacturing company to the University of Tokyo. Because the usage of dry ice, temperature of samples descended further from their original frozen temperature. Hence, samples were waited to recover to their original frozen temperature before baking started.

2.8. Baking

Baking experiment was conducted using an oven developed that was able to generate superheated steam (see *Figure* 2-10.) Pipes connecting faucet and the oven

allowed tap water flew into the heater and was boiled. Boiling of water kept until water turned into saturated steam and then was superheated via the super heater (as shown in *Figure* 2-11). To achieve superheated steam, a preheating was necessary. The temperature of superheated steam in this research was 105 ℃.

Figure 2-10 Superheated steam oven

An electric scale (Shimadzu UW4200H) was put on top of the oven, while separated from the oven by a heat protection space, where an electric fan was also used for heat dissipation. This was very important because the very sensitive electric scale could be severely influenced by heat from the oven and thus led to inaccurate data. Heaters were installed both in upper and lower layer in the oven and heat-durable thermocouples were tied around heaters to check their temperature. Another thermocouple was used to check the air surrounding the sample holder. During preheating, temperature of heaters was adjusted so as to ensure the air temperature surrounding the

samples to be approximately same and stable for each trials. In this research, surrounding air temperature was adjusted to around 260 ℃ for both hot air (HA) and superheated steam (SHS) baking methods, since HA and SHS could be considered as extreme situations within the inner baking environment (Yoshida & Hyodo, 1963).

Samples that were baked were inserted with thermocouples approximately into core part in the samples. Thus it was able to measure the inside temperature of samples during baking. To avoid samples under or over baking, according to the baking curves shown later in the results section, baking were stopped immediately when inner temperature reached up to 100 ℃, which was a threshold because temperature of samples would not rise above it. During baking, weight and temperature of samples were measured simultaneously.

After baking was completed, samples were allowed to cool down till the inner temperature was down to room temperature before wrapped with preservative films to prevent moisture loss. Three samples frozen under each temperature under 1 day frozen storage were baked in this research. Samples frozen over 15, 25 and 35 days were not triplicated due to the availability, however (see *Appendix* 2).

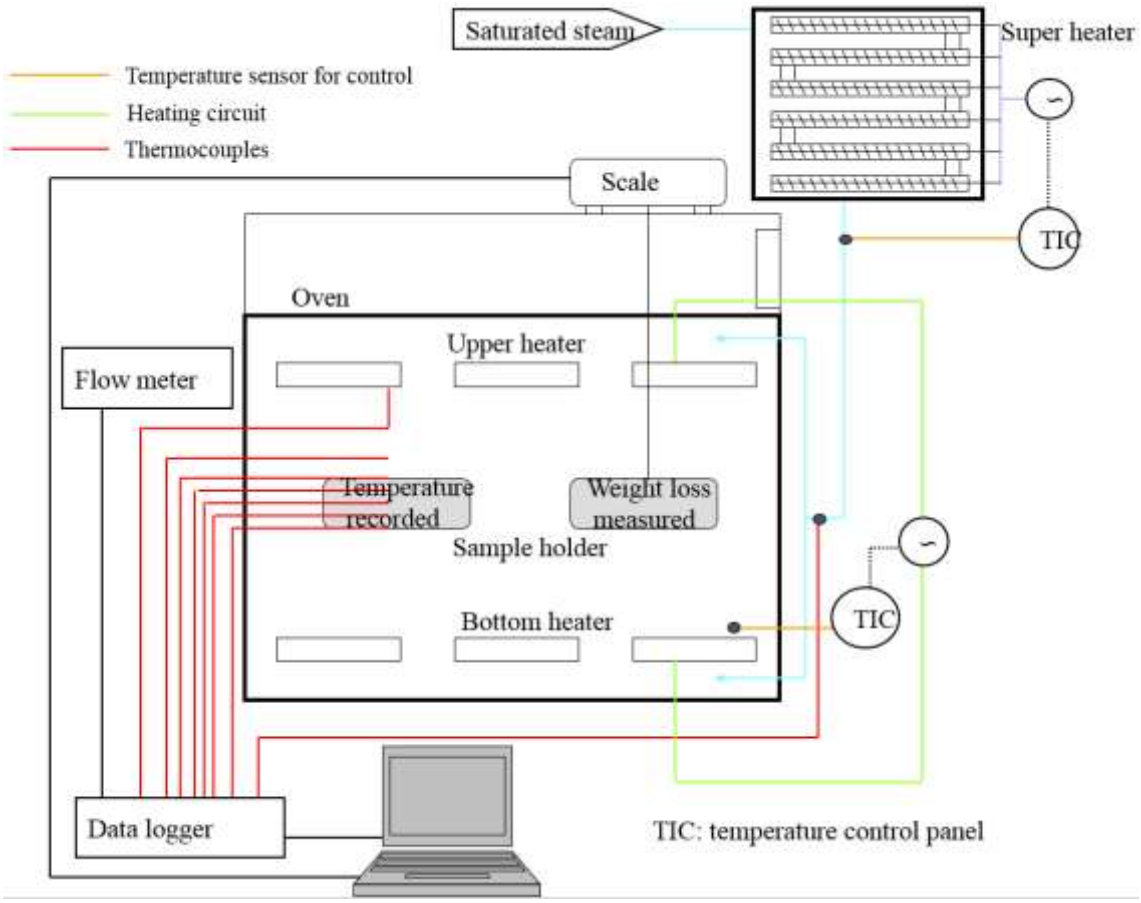

Figure 2-11 SHS oven working principles

2.9. Colour measurement

Figure 2-12 Handy spectrophotometer

Colour of samples was measured using a handy spectrophotometer NF-333 (Nippon Denshoku, Ind.Co.Ltd) as shown in *Figure* 2-12 and colour was presented in

CIE Lab colour space shown in *Figure* 2-13. L (lightness from 0 to 100), a (+ red, - green) and b (+ yellow, - green) were recorded. Three places near the center area of the sample (see *Figure* 2-15) were measured.

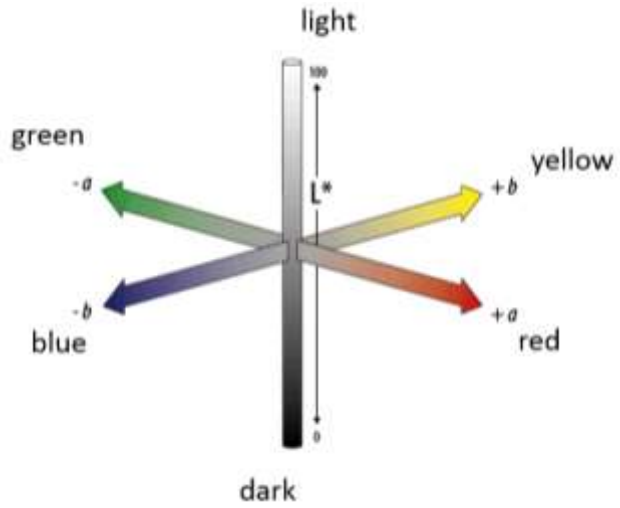

Figure 2-13 The CIE Lab colour space

(http://dba.med.sc.edu/price/irf/Adobe_tg/models/cielab.html)

After L, a, b values were recorded, Delta E (total colour variance before and after baking) was calculated, using Equation 2-3-1

$$\Delta E^*_{ab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2} \quad (2\text{-}1)$$

where L_1, a_1, b_1 stand for L, a, b values of samples before baking; L_2, a_2, b_2 values stand for CIE Lab values of baked samples.

2.10. Moisture content measurement

Three cylindrical small samples (diameter 6 mm, height 10 mm) taken from the middle part of the samples by cork borer, were dried by a drying oven (F0-60W, Japan, see *Figure* 2-14) under 105 ℃ over 24 h. Samples moisture content M [%] was measured by dry basis method (Singh & Heldman, 2014) through Equation 2-2

$$M = \frac{W_w - W_d}{W_d} 100\% \tag{2-2}$$

where W_w is the wet base weight, W_d is the dry base weight.

Figure 2-14 Drying oven F0-60W, Japan

2.11. Rheological properties measurement

2.11.1. Sample specimens and devices

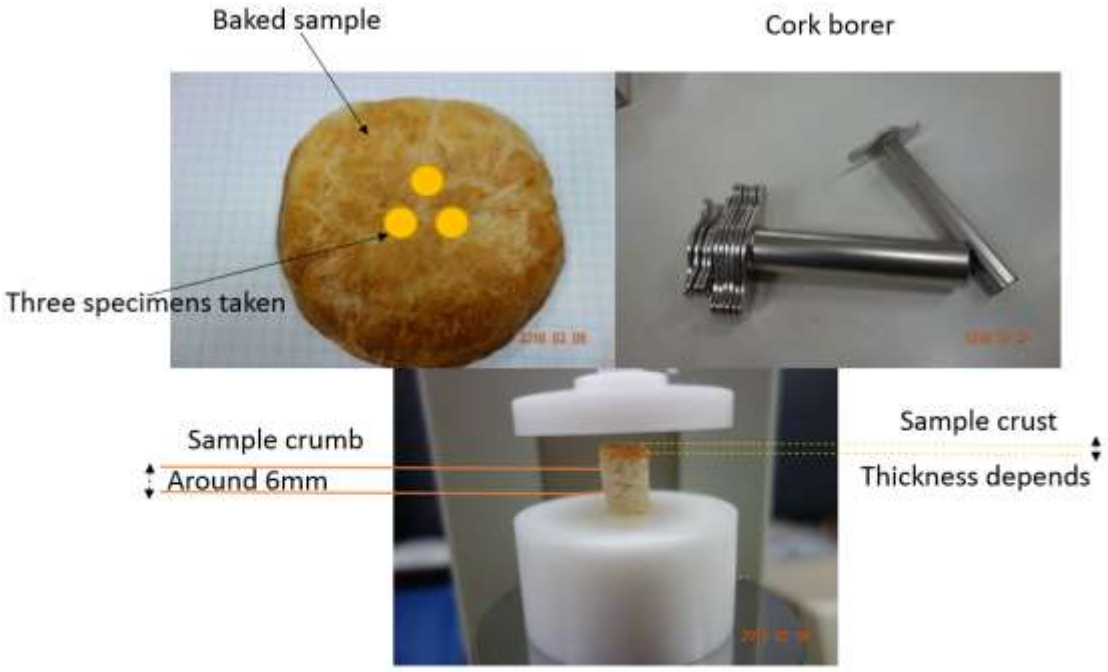

Figure 2-15 Specimens taken from baked samples

As baked sample had been cooled down to room temperature, three places around the centre area in the sample were taken by cork borer (Kenis, cork borer, size = 3, diameter = 6mm) as shown in *Figure* 2-15. Crusts and crumbs of samples were taken and measured separately based on visual textural difference (Westerlund, Theander, & Aman, 1989), as it still remains hard to set a precise definition of the spatial domain comprising crust due to the difficulty to define a boundry during the gradual change in crust properties (Vanin, Lucas, & Trystram, 2009). Before rheological properties measured, thickness

[mm] of crusts were measured with the build-in thickness measuring function of the creep meter.

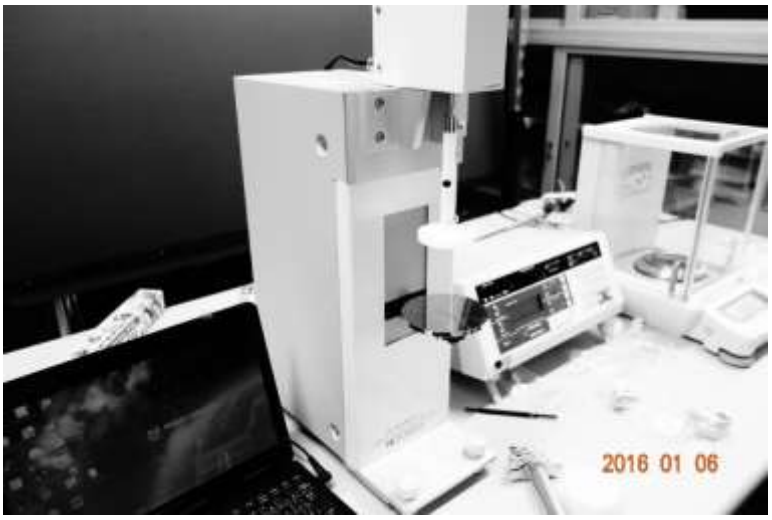

Figure 2-16 Creep meter

A creep meter (RE2-33005C, YAMADEN corp., Japan) was used for rheological properties of samples (see *Figure* 2-16). A schematic graph of creep meter was listed (see *Figure* 2-17) (Morita, et al., 2015). This experimental instrument system was constituted from viscoelastic measurement part, thickness measurement gauge of specimen, data recorder and a control and operating computer.

As shown in *Figure* 2-17, when samples were compressed by a plunger during the up and down movement of the sample holding stage according to a certain speed, the load cell sensed and recorded the corresponding force. In this research, two large deformation test (fractural test, textural test) and a small deformation test (creep test) was conducted and they were explained in the following sections, respectively.

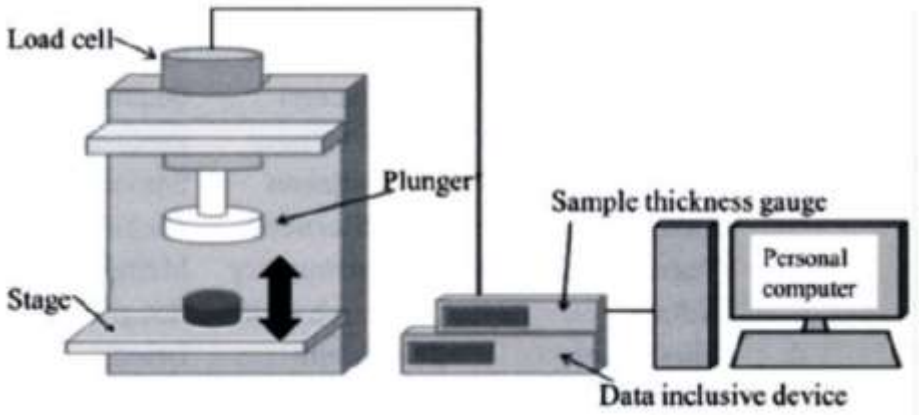

Figure 2-17 Schematic of creep meter working principles

2.11.2. Fractural test (Tensile test/large deformation)

A dedicated software for the creep meter (Ver. 2.3 (BAS-3305)) was employed to analyse the mechanical properties of crumbs and crusts from baked samples. In fractural test, breaking force [N], the force recorded at the breaking point in the curve, was used as the parameter to evaluate samples physical properties through fractural test. Plunger diameter was 53 [mm].

During fractural test, the compression was conducted up to 100% distortion rate in a speed of 5 mm/s. Force-strain curve was obtained (as shown in *Figure* 2-18) with a load cell (200N maximum) in 10 times the voltage magnification (20N), where the rupture force was taken as the maximum force peak height required to break the sample.

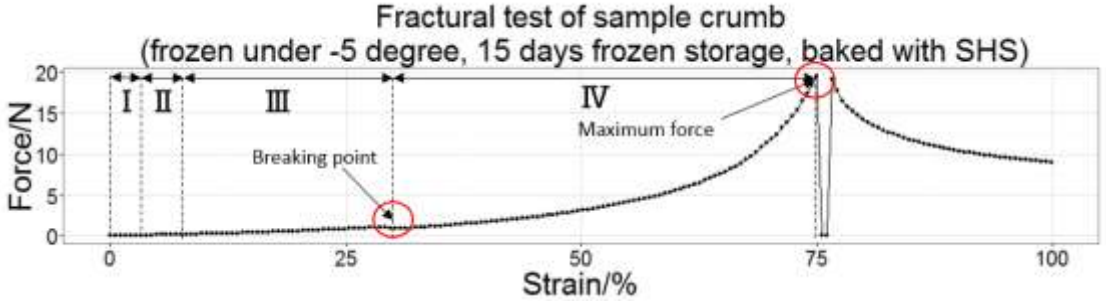

Figure 2-18 Typical fractural test curve

Aside from the direct results in fractural test, four stages in fractural curve were shown. The first stage (stage Ⅰ, strain rate around 0%~5%) was the short moment when sample surface got touched by the plunger and sometimes an unconstant deformation occurred which might be due to the uneven surface contact between the sample and plunger. In the second stage in the fractural curve, there was a near-linear line area (stage Ⅱ, strain rate around 5%~10%). Linear viscoelasticity is when the function is separable in both creep response and load and is usually applicable only for small deformations.

The third stage (stage Ⅲ, strain rate around 10%~30%) was the stage where sample reached to the breaking point, which was called the yield point in tensile test. A breaking point detected at the broken down moment in breaking curves were shown in *Figure* 2-18. In tensile test the breaking point referred to the yield point, before which the material deformed elastically and would return to the original shape once the stress was removed, while after which the material deformed plastically. Thus the breaking point determined the limitation of the force applied to the material without non-reversible deformation.

The fourth stage (stage Ⅳ, strain rate around 30%~75%) was the final stage where compressed samples reached to the maximum recorded force, after which the stress was stopped and removed, giving a sudden drop in the curve and further decrease of the recorded force.

2.11.3. Creep test (small deformation)

According to the stage Ⅱ in fractural test mentioned above, the material was only viscoelastic within a very small range of deformation (5%~10%), where it showed a near linear curve, indicating characteristics of Newtonian material within that range. Thus the creep test load was obtained from each of the fractural test curves of samples at the end-point from where the near-linear line turned non-linear. All operations were automatically controlled by the dedicated software (Ver.1.6 (CAS-3305)). Plunger diameter was 53 [mm].

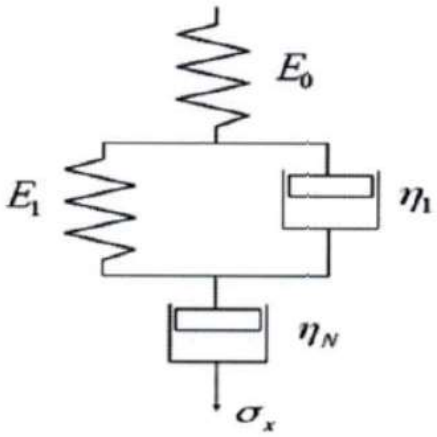

Figure 2-19 Four element Maxwell and Voigt model

In this research, a four element (instantaneous elasticity in E_0 [pa], delayed elasticity E_1 [pa], delayed viscosity η_1 [pa/s] and permanent viscosity η_N [pa/s]) Maxwell and Voigt model creep test was conducted. A spring and a dashpot were consisted to represent the elastic and the viscous part, respectively, as shown in *Figure* 2-19. *Figure* 2-20 showed typical creep test curves triplicated from one sample frozen under -5 degree and was kept frozen for 15 days. In response to a sudden deformation of the model, the spring was immediately extended, while the dashpot remained initially motionless (Stage

1). But the extended spring would be applying a steady force on the dashpot in an attempt to recoil. This caused the dashpot began to move in the direction of the spring force at a speed governed by the spring force and the spring kept a constant length all the way. As long as the force remained, the deformation remained to be in a linear style perpetually (Stage 2). After sudden release of the applied force (in this research set at 60 s), the spring began to recoil with the moving dashpot, elasticity deformation was restored while viscosity deformation remained permanently (Stage 3 and Stage 4). The total operation time was kept 120 s for all trials.

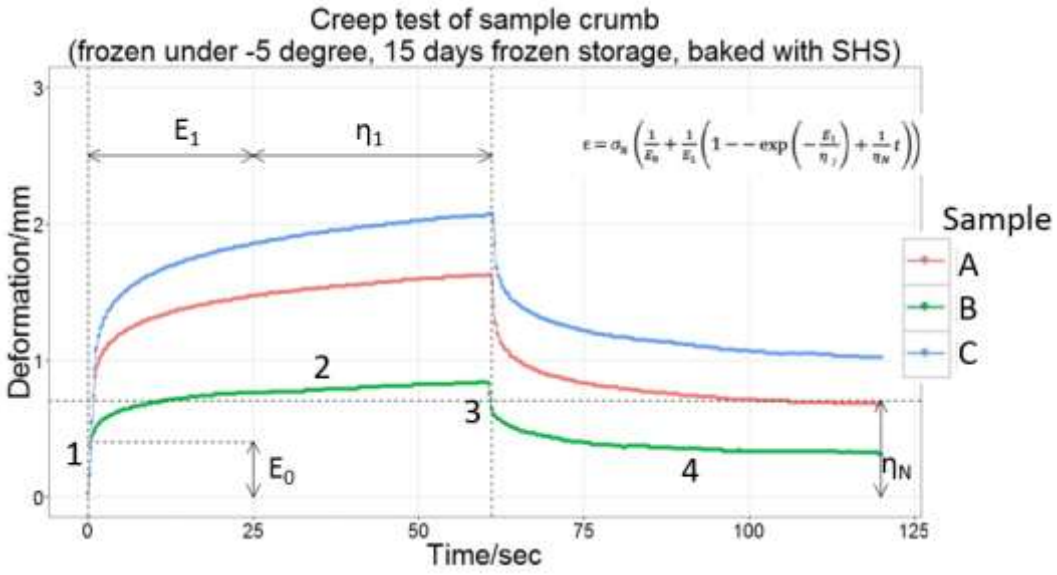

Figure 2-20 Typical creep test curve

Four elements in creep test can be calculated by Equation 2-3,

$$\varepsilon = \sigma_x \left(\frac{1}{E_0} + \frac{1}{E_1}\left(1 - -\exp\left(-\frac{E_1}{\eta_1}\right)\right) + \frac{1}{\eta_N} t \right) \quad (2\text{-}3)$$

where ε is deformation, σ_x is the loaded stress, t stands for time, exp stands for exponent retardation time constant. The exponent can be obtained by constructing a semi-log plot

of the retarding exponential approach between the end of curve which represented elastic recovery and the permanent curve which represented permanent viscosity (η_N). The semi-log plot will result in a straight line whose slope will give the time constant (Figura & Teixeira, 2007).

2.11.4. Textual test (TPA test/large deformation)

During a TPA test samples are compressed twice using a texture analyzer to provide insight into how samples behave when chewed. The TPA test is often called the "two bite test" because the texture analyzer mimics the biting action in the mouth. A two-bite TPA test was conducted and parameters such as hardness, cohesiveness and gumminess were determined and calculated automatically by the dedicated software (Ver.2.3 (TAS-3305)). Plunger diameter was 53 [mm]. In general, many food products are permanently deformed beyond the point of plastic deformation in strains of greater than 50% (Texture Technologies, accessed on 13th, June, 2016), thus 50% strain deformation was chosen as the standard. The maximum force was 20N.

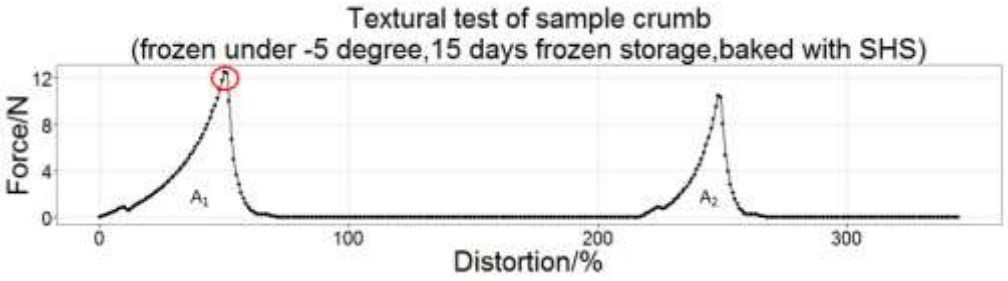

Figure 2-21 Typical TPA test curve

A textural test curve was shown in *Figure* 2-21. The mountain curve of area A_1 represented the first bite and area A_2 was the second bite. Hardness [N] values were

translated from maximum force point values, cohesiveness [-] values were calculated by Equation 2-4.

$$\text{Cohesiveness} = \frac{A_2}{A_1} \tag{2-4}$$

where A_1 is dimension within the first deformation area, A_2 stands for the dimension for second deformation area. Gumminess [N] values were determined by hardness*cohesiveness.

As a matter of fact, the textural test curve is a generalisation of all the three rheological tests in this research. It gave a food mastication process imitation by using creep test, a very small deformation happened momentarily when food was bitten, fractural test, a large deformation which completed the first bite, giving a maximum compression to the food that broke it down and another large deformation till the other one bite. The three tests were combined within one plot in order to give a better vision of the mastication process by using instrumental experiment, as shown in *Figure* 2-22.

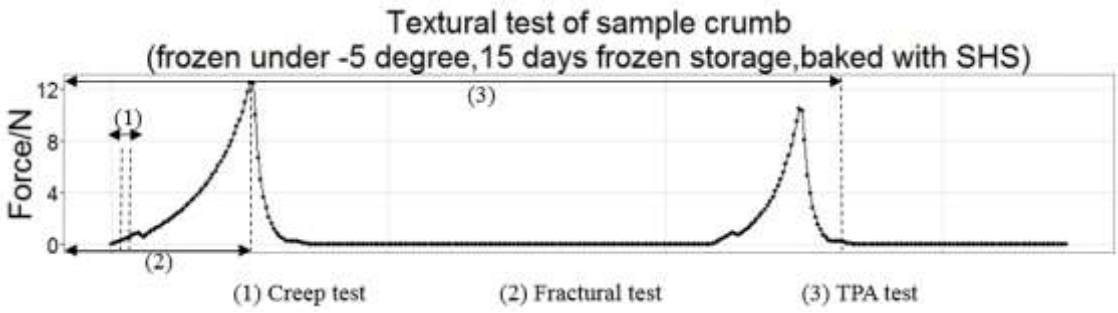

Figure 2-22 Typical force vs time curve measured during two bites

(1) Small deformation shortly after first touch

(2) Large deformation until first bite completed

(3) Large deformation until send bite completed

2.12. Research design and data analysis

2.12.1. Design of this research

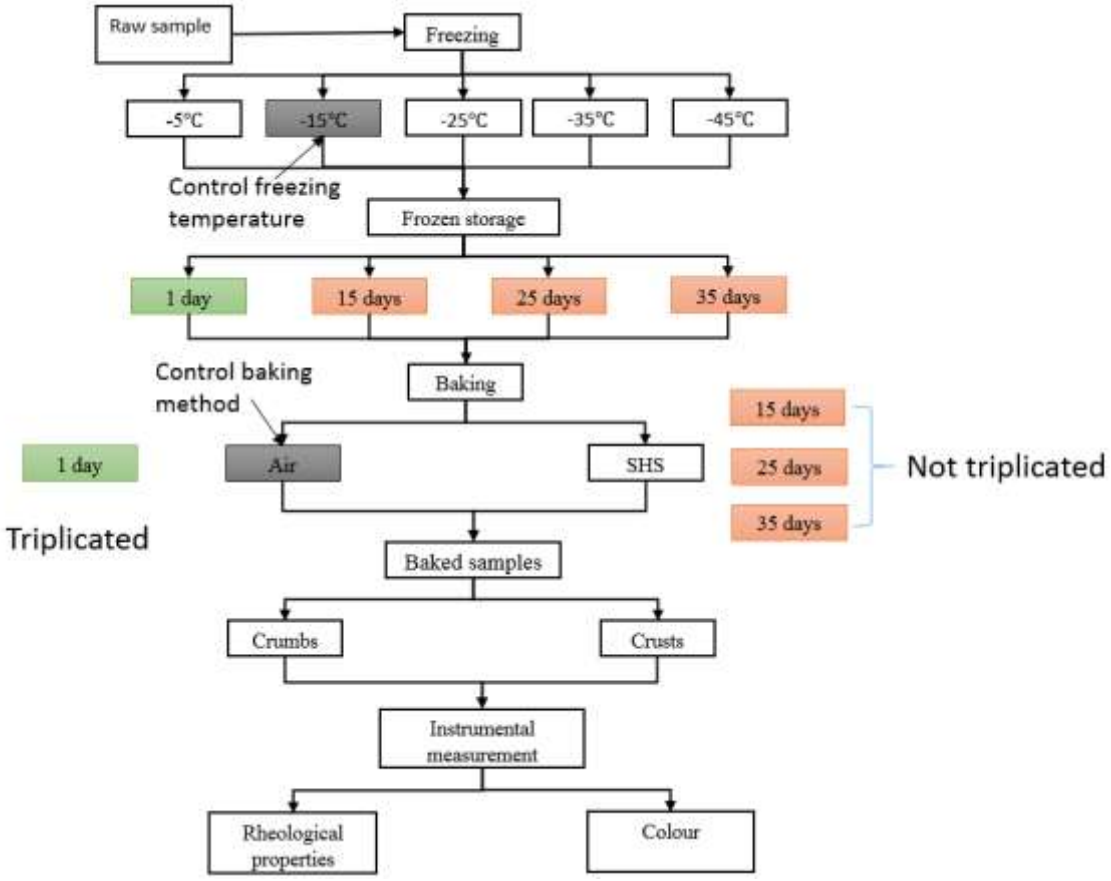

Figure 2-23 Tree diagram of research design

Figure 2-23 showed the design of this research. Among all the research steps, 1 day frozen samples were triplicated for each of the five groups of samples frozen under different temperature, from -5 ℃, -15 ℃, -25 ℃, -35 ℃, -45 ℃, and three specimens taken from each baked samples thus 9 data for each sample (colour and rheological properties measurement). Although samples kept frozen for over 15 days, 25 days and 35 days were

not triplicated in this research due to the availability, it was still expected to show some tendency and assumptions for future work.

2.13. Data analysis

R programming language was used in this research for both data analysis and graphing, together with Excel for some further handling of graphs. Packages were downloaded from CRAN (The Comprehensive R Archive Network, accessed on 13th, June, 2016),including data restructuring and aggregating package **reshape2**, data splitting package **plyr**, graphing packages such as **ggplot2**, **grid**, **gridExtra**, **scatterplot3d**, **lattice**, data reading package **xlsx** and correlation coefficient graphing package **corrplot**.

Statistical significant difference was worked out by ANOVA test if there were over two compared objectives. Student t test was used when there were only two set of data compared. If significant difference was found within multiple datasets by ANOVA test, TUKEY HSD (honestly significant difference) test was used for advanced pairwise comparison (Kono, Kawamura, Yamagami, Araki, & Sagara, 2015). Throughout this research, 95% confidence interval was chosen in ANOVA test, pairwise TUKEY-HSD test and Student t test, while 99% confidence interval was selected in finding the cross-correlation coefficient in the results.

Chapter 3. Results

3.1. Freezing characteristics

3.1.1. Freezing rate of samples frozen under different temperature

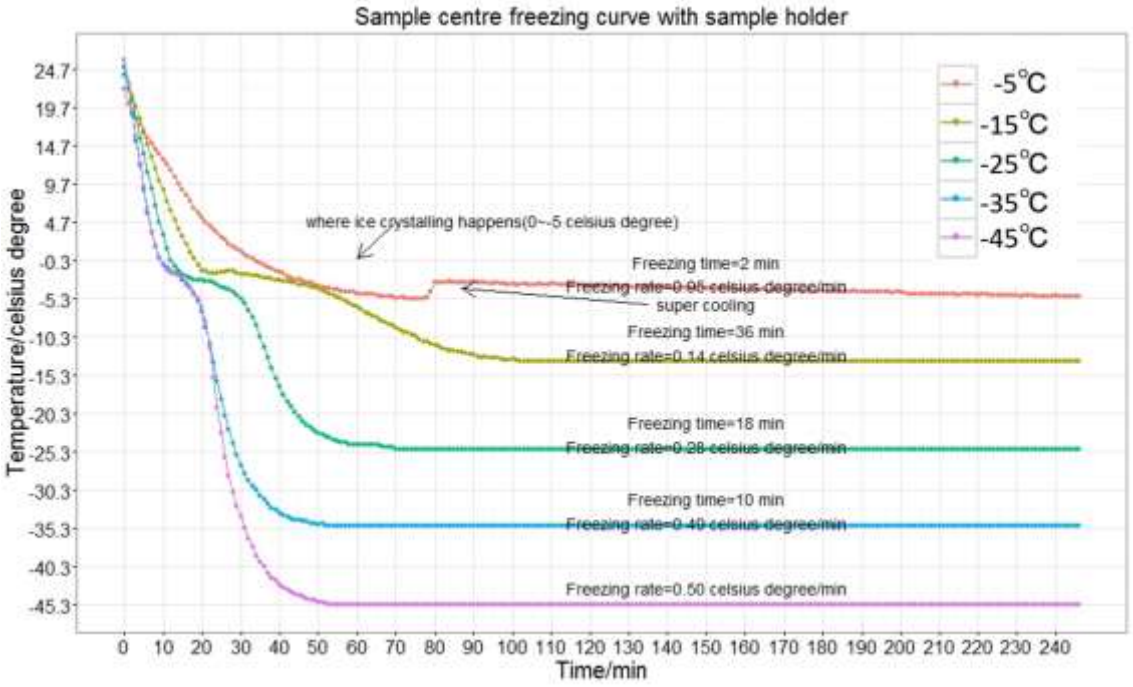

Figure 3-1 Freezing temperature curve of sample centre temperature under different freezing temperature

The plots which recorded the temperature change of upper, center and lower layers of samples frozen under five different temperature (-5, -15, -25, -35, -45 ℃) during freezing were included in the *Appendix* 1 (*Figure* A1-1, *Figure* A1-2, *Figure* A1-3, *Figure* A1-4, *Figure* A1-5*). Figure* 3-1 showed an overview of the five freezing temperature curves of center temperature in samples change during freezing process.

Initial temperature of all samples started from around 25 ℃. The freezing curves went down drastically when the temperature was above 0 ℃, while showing a distinct

change of the shape in each of the curves between the temperature ranges from around -1 ℃ to -4 ℃. It was supposed that most of the ice crystallization happened within this range. This results corresponded with the statement from (Giannou, Kessoglou, & Tzia, 2003) Freezing normally starts at −1 ℃ to −3 °C and as the temperature drops more of the water in food becomes frozen.

After a slowing down period which was required to remove the latent heat in samples, each of the freezing curves started to go down again till it reached the same temperature with the freezing air in the freezer.

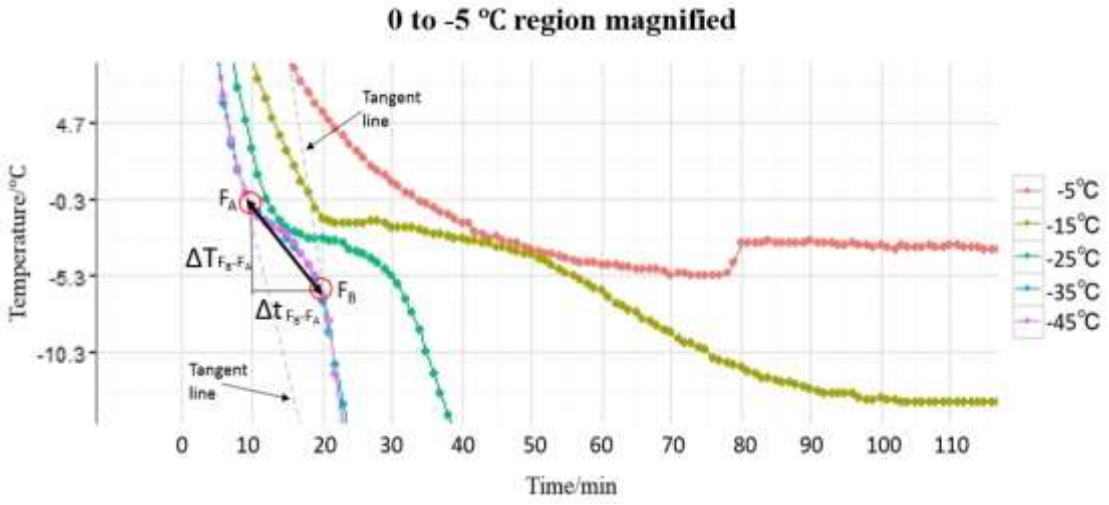

Figure 3-2 Magnified freezing temperature curve between 0 and -5 ℃ region

Freezing rate [℃/min] was calculated from the result of how fast each curves passed through the freezing zone (see *Figure* 3-2). F_A stood for the exact point where the specified freezing curve started to change the direction from which it originally was going, by drawing a tangent line. It referred to the freezing point for the sample. F_B was the exact point from where the freezing curve restarted to go down in a linear way. Freezing rate

was thus calculated as the slope of the line between F_A and F_B, as shown in Equation 3-1.

$$\text{Freezing rate} = \frac{\Delta T_{F_B\text{-}F_A}}{\Delta t_{F_B\text{-}F_A}} \quad (3\text{-}1)$$

where ΔT and Δt referred to the temperature and time difference between the two points.

Table 3-1

Freezing Rate [℃/min]	
-5℃	0.95
-15℃	0.14
-25℃	0.28
-35℃	0.49
-45℃	0.50

As a result, the lower the freezing temperature, the higher the freezing rate was, as shown in *Table* 3-1.

3.1.2. Super cooling phenomenon

However, samples frozen under -5 ℃ showed an exceptional higher freezing rate among all freezing rates calculated above. In this research, the freezing rate of samples in which super cooling phenomenon happened was calculated by how fast the temperature rose suddenly from lowest point till the point it restarted to drop again in the freezing temperature curve.

Super cooling is unpredictable and will cause a general confuse as a matter of fact. In this research, super cooling broke the trend that lower freezing temperature had higher freezing rate, because the formation of ice crystal was completed so soon that the freezing rate even doubled the freezing rates of samples that were frozen under -45 ℃. For convenience sake, in this research samples frozen under -5 ℃ were discussed separately from the other four groups of samples (see details in *Appendix* 3).

3.1.3. Moisture content after freezing and frozen storage

During freezing, a certain amount of moisture was lost in samples. In 1 day frozen samples, positive correlation was found between moisture content [%] in samples and freezing rate($r= 0.86$, p value < 0.01, $r2=0.74$), indicating that lower freezing rate and more exposure time to the refrigerating air lead to more water loss in samples, see *Table* 3-2. Samples frozen under -5 ℃ had the lowest moisture content (64.7±0.4%) and samples frozen under -45 ℃ had the most moisture content (71.1±0.5%).

Table 3-2 Sample moisture content [%] (1 day frozen storage)

Samples	Freezing Temperature/℃				
	-5	-15	-25	-35	-45
Crumb	64.7±0.4^{b}	68.1±0.8^{a}	68.5±0.1^{a}	70.0±0.4^{c}	71.1±0.5^{d}

a,b,c:Means in the same column by the same lowercase superscript letters are not significantly different($p<0.05$)

3.2. Baking characteristics

3.2.1. Overview of temperature and weight loss curves during baking

Figure 3-3 and *Figure* 3-4 showed the weight loss curve of samples, temperature of heaters and surrounding air and inner temperature within samples during baking by HA and SHS.

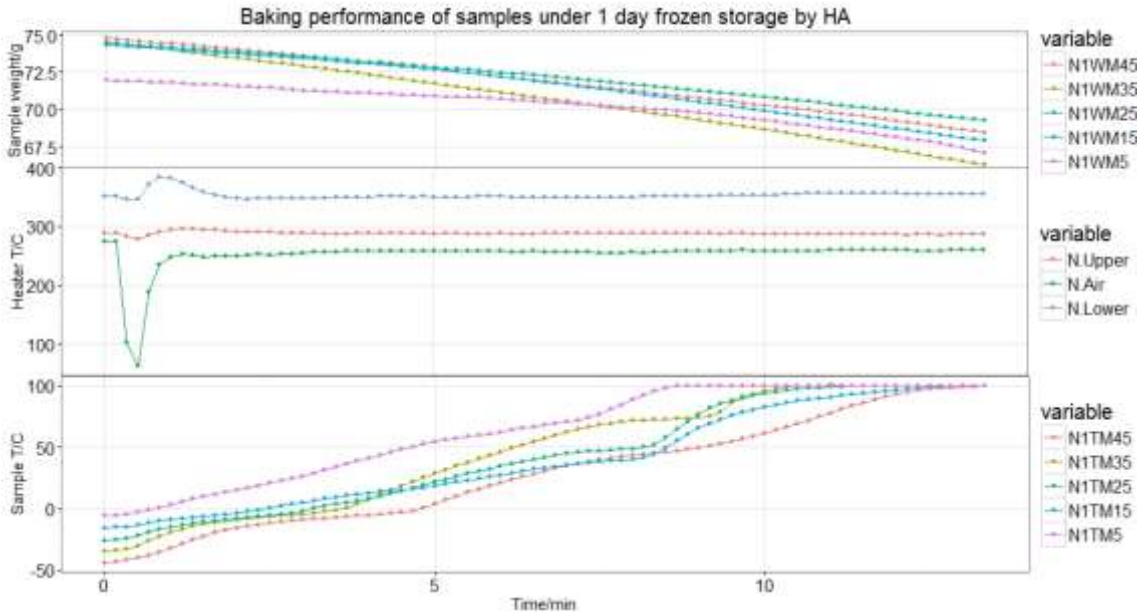

Figure 3-3 1 day frozen samples baking performance by HA method.

Note: In the legend column on the right side of graph, names of variables were assigned to be classified easily for graphing and data analysis in R programming language. N, stands for no SHS used, 1 is 1 day, T means temperature, W means weight, M means minus. N1TM45 thus indicates the inner temperature of samples frozen under -45 ℃ under 1 day frozen storage, baked without SHS. Upper, air and lower referred to upper heater, surrounding air and lower heater.

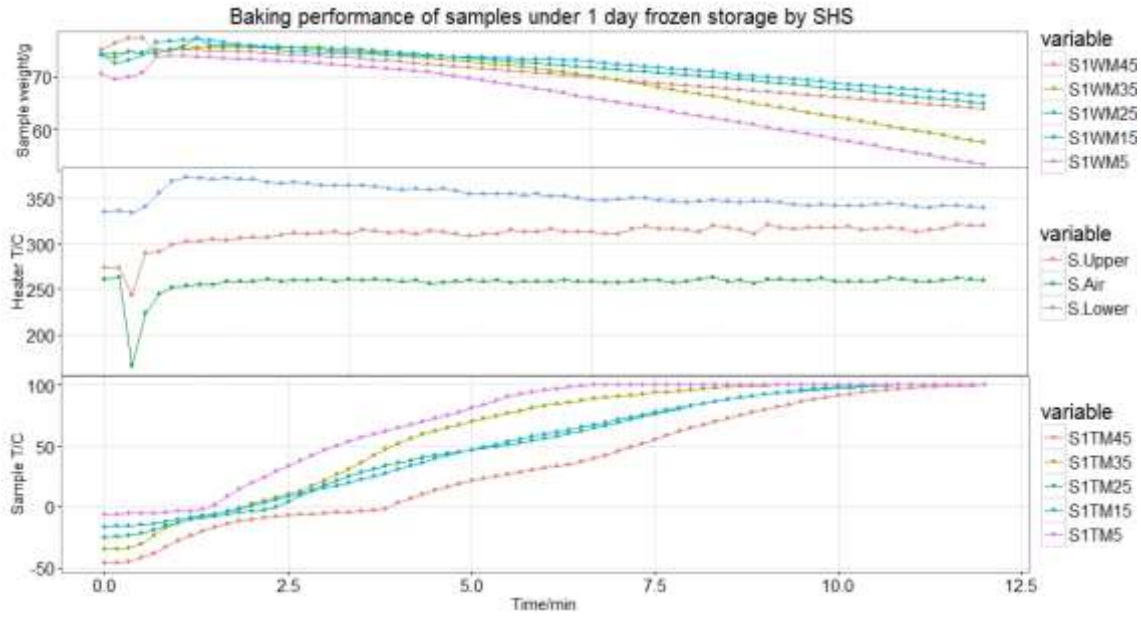

Figure 3-4 1 day frozen samples baking performance by SHS method.

Note: In the legend column on the right side of graph, names of variables were assigned to be classified easily for graphing and data analysis in R programming language. S, stands for SHS used, 1 is 1 day, T means temperature, W means weight, M means minus. S1TM45 thus indicates the inner temperature of samples frozen under -45 ℃ under 1 day frozen storage, baked with SHS. Upper, air and lower referred to upper heater, surrounding air and lower heater.

Upper part of either graph is weight loss curve of samples during baking. From weight loss curve it showed that almost all samples had a similar weight loss speed, especially in the HA baking method. In all SHS baked samples, there was a rise in the weight loss curve within a short time in the initial baking period.

Middle part of either graph stands for the temperature curves for upper and lower heaters in the oven and temperature of the air surrounding baked samples. Upper heater and lower heater were adjusted to around 350 ℃ and 290 ℃ respectively in order to allow temperature of the surrounding air to be kept around 260 ℃, for both baking methods. When SHS was added into the system, the inside temperature became less stable than barely HA. Even though, the surrounding air temperature (green line) around samples still

maintained as most stable as possible throughout the whole baking process, offering samples the same surrounding air temperature with HA.

Lower part of either graph is the inner temperature of samples curve from samples initial frozen temperature till completely baked. The maximum baking temperature in samples would not get over 100 ℃, which gave a same result as found in other research (Le-bail, et al., 2011). In order to avoid samples being under or over baked, while inner temperature of samples reached up to 100 ℃, baking was stopped immediately and thus all the subsequent properties were measured of samples that the core temperature reached exactly up to 100 ℃.

3.2.2. Thawing rate and baking time comparison between HA and SHS

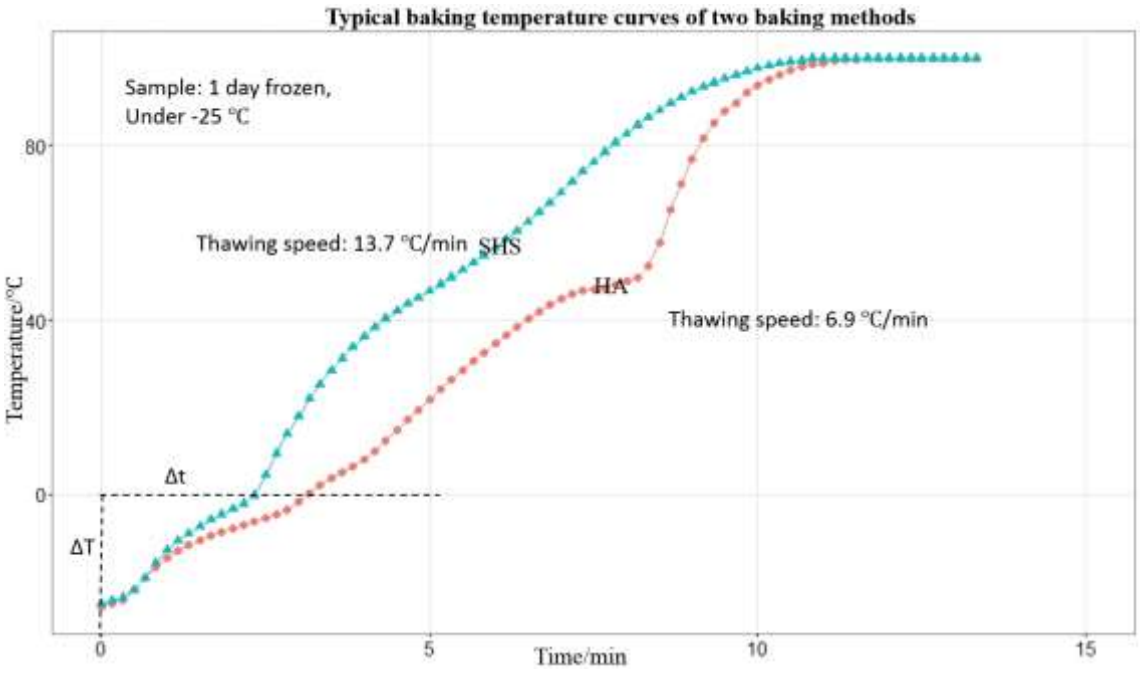

Figure 3-5 Centre temperature curve comparison of samples during baking

Figure 3-5 gave a better vision at the comparison of center temperature in samples change during baking between the two baking methods. Baking time was calculated by

the time period from initial temperature in samples till it reached up to 100 ℃. Thawing speed [℃/min] was calculated through Equation 3-1

$$\text{Thawing speed} = \frac{\Delta T}{\Delta t} \tag{3-1}$$

where ΔT and Δt are the temperature and time difference between the starting point at time 0 and the point when the curve reaches 0 ℃.

Significant difference was found between samples baking time and freezing rate, indicating lower freezing temperature, longer baking time in HA baked samples. In SHS baked samples, no such significant different or correlation was found.

HA baking method tended to give higher thawing rate to samples that were frozen with higher freezing rate (lower freezing temperature) (correlation coefficient: 0.79, $p < 0.01$, as shown in *Table* 3-7), significant difference was found, too. However, in SHS baked samples, no such correlation was found or significant difference was found.

Compared with HA baking method, SHS gave a significantly faster frozen sample thawing rate and less baking time till samples are fully baked, as shown in *Table* 3-4.

Also, negative correlation ($r = -0.67$, $p < 0.01$) was between thawing rate and baking time in SHS baked samples, i.e. faster thawing rate, shorter baking time. No such correlations was found in HA baked samples. This indicated a more consistent baking temperature characteristic from thawing stage till fully baked samples when using SHS as the baking medium.

Table 3-3 Baking time comparison between HA and SHS

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crumb	HA	$9.5\pm0.6^{c,A}$	$11.6\pm2.1^{b,A}$	$11.9\pm1.1^{ab,A}$	$12.2\pm1.2^{ab,A}$	$13.8\pm2.0^{a,A}$
	SHS	$7.0\pm0.2^{c,B}$	$8.6\pm1.7^{a,B}$	$9.8\pm1.4^{a,B}$	$8.4\pm0.6^{ac,B}$	$9.6\pm1.8^{a,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same column have no difference($p<0.05$)

Table 3-4 Thawing rate comparison between HA and SHS

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crunb	HA	$6.6\pm0.4^{ab,A}$	$5.0\pm1.2^{a,A}$	$6.9\pm1.2^{b,A}$	$8.4\pm0.9^{b,A}$	$10.8\pm2.1^{c,A}$
	SHS	$3.9\pm0.5^{a,B}$	$17.5\pm9.9^{b,B}$	$13.7\pm4.2^{ab,B}$	$37.7\pm14.0^{c,B}$	$15.1\pm4.1^{b,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same column have no difference($p<0.05$)

3.2.3. Drying rate comparison between HA and SHS

Initial condensation was mentioned in some previous studies such as (Devente & Heijmans, 2001) and an overview by (Mujumdar & Devahastin , 2008). It was reflected in the weight loss curves. As shown in *Figure* 3-6 of the weight loss comparison between the two baking methods, as soon as the baking process started, samples baked by SHS gave an immediate rise in weight, which indicated the initial condensation of SHS on the surface of the samples due to a comparatively huge temperature difference that cooled down SHS in the surrounding air.

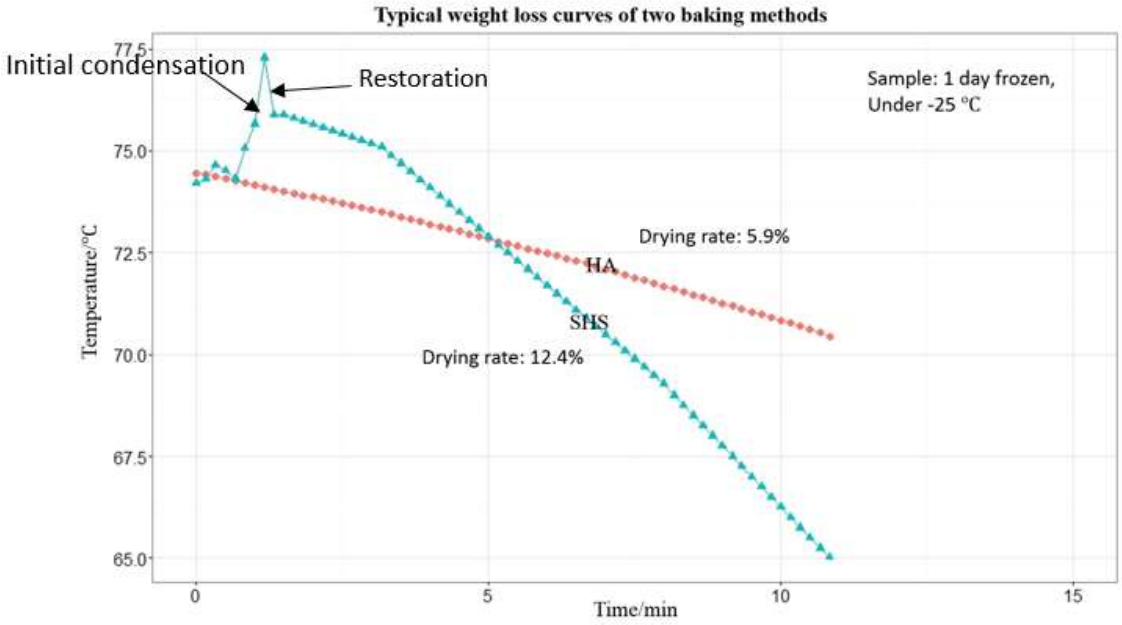

Figure 3-6 Weight loss curve comparison of samples during baking

Table 3-5 Drying rate during baking of 1 day frozen samples

Drying rate/% during baking						
Samples		Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crumbs	HA	2.8	8.4	5.9	8.5	8.5
	SHS	8.8	10.1	12.4	12.1	14.7

Table 3-6 correlation and p values between freezing rate, thawing rate and baking time

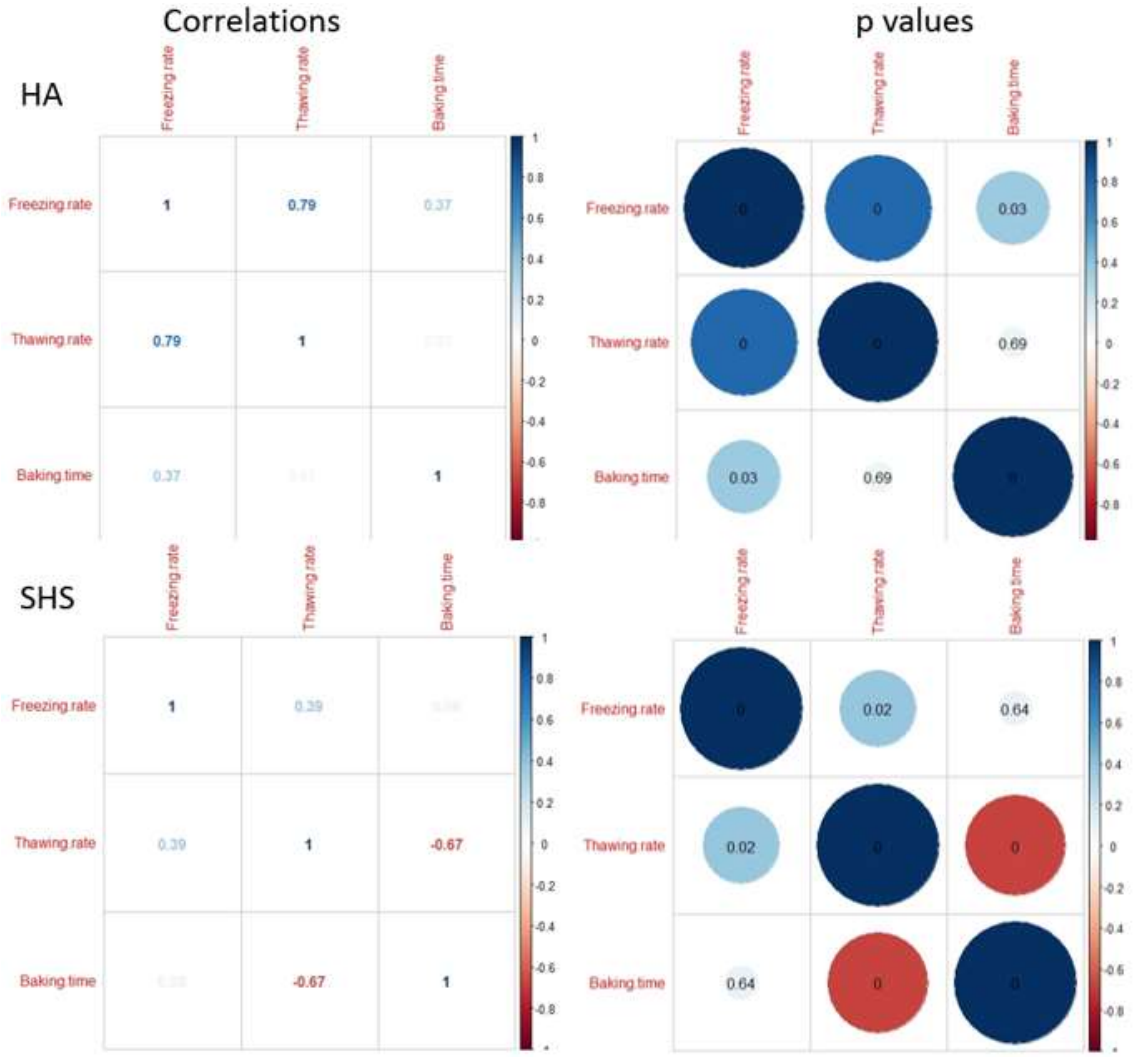

Table 3-6 showed the correlation and related p values between freezing rate, thawing rate and baking time. HA baked samples showed a positive correlation between thawing rate and freezing rate ($r = 0.79$, $p<0.01$), while SHS baked samples showed a negative correlation between thawing rate and baking time ($r = -0.67$, $p<0.01$).

3.3. Colour change

3.3.1. Lightness values

Colour is probably one of the most important parameter for food stuffs because it is always firstly evaluated by consumers (Pronyk C. , Cenkowski, Muir, & Lukow, 2008).

Table 3-7 showed CIE Lab values for 1 day frozen samples. Samples frozen under different freezing temperature showed almost no significant difference.

Table 3-7 Raw sample CIE Lab values before baking (1 day frozen storage)

Raw samples after freezing	Freezing Temperature/℃				
	-5	-15	-25	-35	-45
L_1	85.2 ± 0.8^{b}	88.9 ± 1.6^{a}	87.5 ± 1.9^{a}	87.3 ± 1.5^{a}	87.5 ± 1.6^{a}
a_1	4.0 ± 0.6^{b}	2.9 ± 0.4^{a}	2.9 ± 0.4^{a}	3.1 ± 0.5^{a}	3.0 ± 0.7^{a}
b_1	14.5 ± 1.0^{b}	12.9 ± 1.3^{a}	13.5 ± 0.9^{ab}	14.4 ± 1.1^{b}	13.4 ± 1.0^{ab}

a,b,c:Means in the same column by the same lowercase superscript letters are not significantly different($p<0.05$)

Table 3-8 listed L (lightness) values by HA and SHS baking method. Compared with HA, SHS gave samples lower L values in samples frozen under all freezing temperature. HA baked samples had no significant difference in L values under different freezing temperatures, whereas significant difference was found in SHS baked samples frozen between -25 ℃ and -35 ℃.

Table 3-8 L_2 of samples baked by two different method

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crust	HA	$80.9\pm2.0^{a,A}$	$82.5\pm4.5^{a,A}$	$82.2\pm1.7^{a,A}$	$81.4\pm2.6^{a,A}$	$81.3\pm2.3^{a,A}$
	SHS	$77.8\pm1.3^{c,B}$	$61.9\pm5.4^{ab,B}$	$54.9\pm6.1^{a,B}$	$65.7\pm5.9^{b,B}$	$60.8\pm7.7^{ab,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not significantly different($p<0.05$)

3.3.2. a values

Table 3-9 listed a_2 (- green, + yellow) values by HA and SHS baking method. Compared with HA, SHS gave samples larger a_2 values, indicating a yellower outcome. HA baked samples had no significant difference in a values under different freezing temperatures, whereas significant difference was found in SHS baked samples frozen -35 ℃ compared with other sample groups.

Table 3-9 a_2 of samples baked by two different method

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crust	HA	$3.0\pm0.4^{ab,A}$	$2.6\pm0.8^{a,A}$	$2.7\pm1.2^{ab,A}$	$2.5\pm1.1^{a,A}$	$4.5\pm2.4^{b,A}$
	SHS	$4.1\pm1.0^{c,B}$	$18.9\pm2.9^{a,B}$	$19.1\pm0.9^{a,B}$	$12.5\pm4.6^{b,B}$	$16.0\pm3.1^{a,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same column have no difference($p<0.05$)

3.3.3. b values

Table 3-10 listed b_2 (- blue, + red) values by HA and SHS baking method. Compared with HA, SHS gave samples larger b_2 values, indicating a redder outcome. In HA baked samples, significant higher b values was found in samples frozen under -45 ℃ compared with samples frozen under -15 ℃. In SHS baked samples, whereas significant

lower values was found in SHS baked samples frozen -35 ℃ compared with samples frozen under -15 ℃.

Table 3-10 b_2 of samples baked by two different method

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crust	HA	$14.6\pm0.5^{ab,A}$	$13.0\pm1.0^{a,A}$	$12.1\pm1.9^{a,A}$	$13.9\pm1.5^{ab,A}$	$16.7\pm4.8^{b,A}$
	SHS	$17.6\pm2.1^{c,B}$	$35.0\pm3.4^{a,B}$	$32.4\pm3.9^{ab,B}$	$28.2\pm5.1^{b,B}$	$30.9\pm1.9^{ab,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same column have no difference($p<0.05$)

3.3.4. Total colour variance

Table 3-11 listed ΔE* values (calculated through Equation 2-1) by HA and SHS baking method. Compared with HA, SHS gave samples larger ΔE* values, indicating more total colour variance. HA baked samples had no significant difference in ΔE* values under different freezing temperatures, whereas significant lower ΔE* values was found in samples frozen under -35 ℃ baked by SHS.

Table 3-11 ΔE of samples baked by two different method*

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crust	HA	$4.6\pm2.1^{a,A}$	$6.9\pm5.7^{a,A}$	$5.9\pm3.0^{a,A}$	$6.3\pm2.1^{a,A}$	$8.8\pm3.9^{a,A}$
	SHS	$8.1\pm2.5^{a,B}$	$38.6\pm5.4^{b,B}$	$41.4\pm5.2^{b,B}$	$27.7\pm8.2^{c,B}$	$35.6\pm8.2^{b,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same column have no difference($p<0.05$)

3.3.5. CIE Lab colour correlation with freezing temperature and baking time

Table 3-12 listed Pearson' correlation coefficients for colour values from HA baked sample crusts with freezing temperature and baking time. Only correlation coefficients that had p values less than 0.01 were listed in the *Table*. It showed that a_2, $b_2$and ΔE* had positive correlations with baking time. No correlation was found between freezing temperature and colour of samples in HA baked pizza crusts.

Table 3-12 Pearson product correlation coefficients of colour values at various freezing rate and baking time (HA baked sample crusts)

Processing variables				
HA crusts	Freezing temperature		Baking time	
	Correlation coefficient	p values	Correlation coefficient	p values
Crust thickness				
L_2				
a_2			0.72	$p < 0.01$
b_2			0.58	$p < 0.01$
ΔE*			0.41	$p < 0.01$

Only p values less than 0.01 correlations were listed

Table 3-13 listed Pearson's correlation coefficients between colour values and freezing temperature and baking time of SHS baked sample crusts. Freezing temperature showed negative correlations with a, b and total colour variance values. Baking time had negative correlation with L values, while positive correlations with a and total colour variance values. Aside from these correlations, thickness of SHS baked samples crusts was also found to have positive correlation with baking time.

Table 3-13 Pearson product correlation coefficients of colour values at various freezing temperature and baking time (SHS baked sample crusts)

Processing variables				
SHS crusts	Freezing rate		Baking time	
	Correlation coefficient	p values	Correlation coefficient	p values
Crust thickness			0.47	$p < 0.01$
L_2			-0.55	$p < 0.01$
a_2	-0.63	$p < 0.01$	0.47	$p < 0.01$
b_2	-0.56	$p < 0.01$		
ΔE*	-0.59	$p < 0.01$	0.45	$p < 0.01$

Only p values less than 0.01 correlations were listed

3.4. Rheological properties

In bar plots listed below, legends on x-axis describing each of the bars were named as follows:

N: HA/No steam

S: SHS

0, 1, 2, 3, 4 referred to the freezing temperature of specified samples in such an order: -5 ℃,-15 ℃, -25 ℃, -35 ℃, -45 ℃.

a,b,c: (HA baked samples) Means in samples by the same lowercase superscript letters are not different($p<0.05$)

A,B,C: (SHS baked samples) Means in samples by the same lowercase superscript letters are not different($p<0.05$)

3.4.1. Creep test

Firstly, the effect of freezing temperature was checked on samples baked by both HA and SHS. Analysis of Variance (ANOVA) test was run through R programming language. Significant difference were found in delayed elasticity E1 in SHS baked

sample crumbs and delayed viscosity V_1 in sample crumbs in both baking methods, as shown in *Figure* 3-7 and *Figure* 3-8.

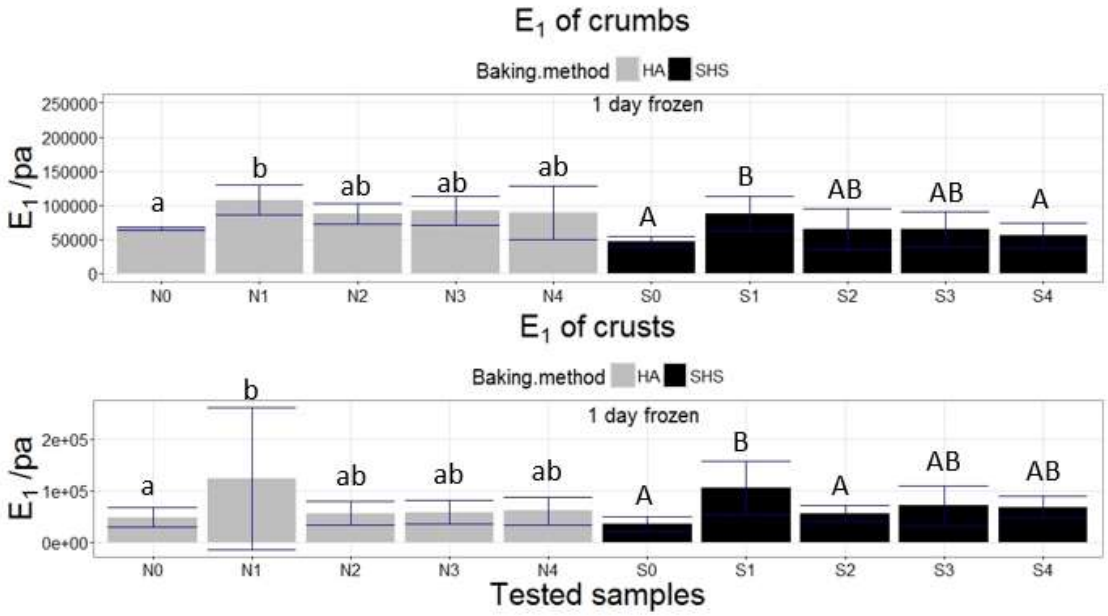

Figure 3-7 Delayed elasticity E_1 values of crumbs and crusts (1 day frozen samples)

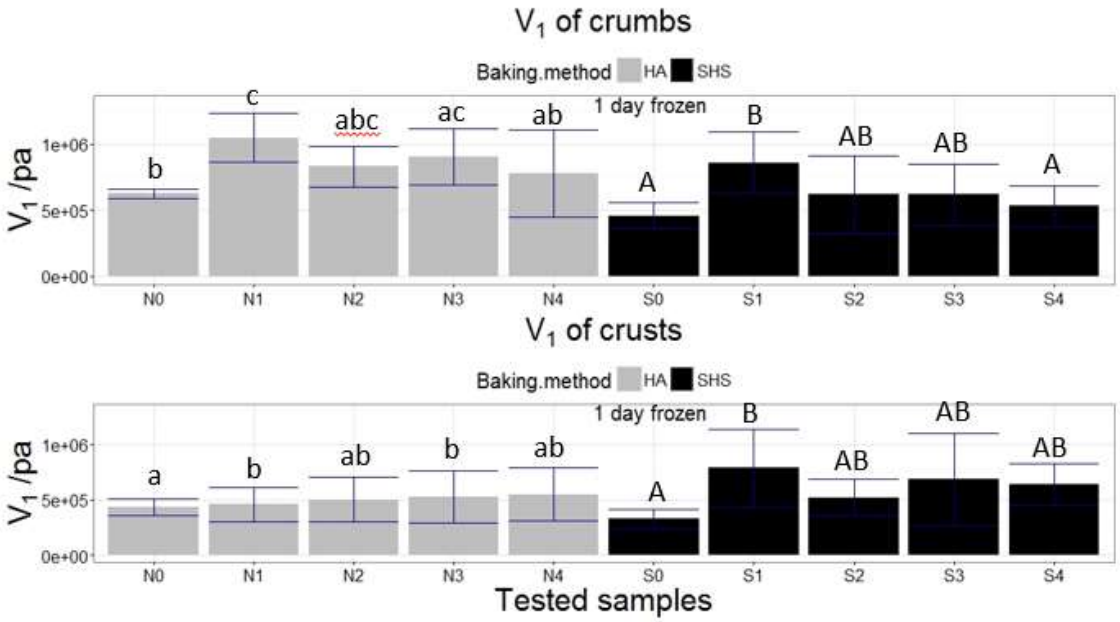

Figure 3-8 Delayed elasticity V_1 values of crumbs and crusts (1 day frozen samples)

No significant difference were found in instantaneous elasticity E_0 or permanent viscosity V_N in sample crumbs or crusts in either baking method. More information was listed in *Appendix* 1 (*Figure* A1-6).

Secondly, difference between samples baked by HA and SHS was checked through Student t.test, as shown in the *Appendix* 1 (*Table* A1-1). From the significant difference found, SHS baked sample crumbs showed less viscoelasticity than HA baked samples crusts, whereas SHS baked samples crusts had larger viscoelasticity values than HA baked samples crusts.

Also difference between samples crumbs and crusts was checked through Student t.test in both baking methods, as shown in the *Appendix* 1 (*Figure* A1-2). From the significant difference found, in HA baked samples, crumbs had larger viscoelasticity than crusts had; whereas in SHS baked samples, crumbs had less viscoelasticity than crusts had.

3.4.2. Fractural test

There was no breaking point found in fractural test curves in all samples, thus instead of breaking force, maximum force was obtained from fractural test curves of all samples (see *Figure* 2-18). There was no significant difference found in the recorded maximum force in samples frozen under different freezing temperature, or samples baked by HA or SHS. More information was listed in the *Appendix* 1 (*Figure* A1-8).

3.4.3. TPA test

No significant difference was found in hardness, cohesiveness or gumminess values of samples frozen under different freezing temperature in either HA or SHS baking method.

Also, no significant difference was found in the comparison in hardness, cohesiveness or gumminess of samples baked by the two baking methods. Detailed information was shown in the *Appendix* 1 (*Figure* A1-9, *Figure* A1-10, *Figure* A1-11*)*.

Chapter 4. Discussion

4.1. Freezing characteristics

4.1.1. Freezing rate and assumption of ice crystallization

As the results shown in *Table* 3-1, the lower the freezing temperature, the higher the freezing rate was. The freezing curve in *Figure* 3-1 showed clear freezing characteristic of pizza dough, wherein latent heat and sensible heat should be taken into account. Latent and sensible heat are types of energy released or absorbed in the atmosphere. Latent heat is related to changes in phase between liquids, gases, and solids. Sensible heat is related to changes in temperature of a gas or object with no change in phase.

In this research, lower freezing temperature caused more sensible heat between freezing air and samples, thus the freezing curve went down more rapidly. Latent heat of samples frozen under lower temperature was removed faster, indicating a faster phase transition or faster ice crystallization. According to research done by (Bevilacqua & Zaritzky, 1980), the size of ice crystals depends on the freezing rate. The faster the freezing rate, the smaller size of ice crystals were detected.

Liquids inside pizza dough frozen by crystallization, formation of crystalline solid from the uniform liquid. This was a first-order thermodynamic phase transition, which meant that, as long as solid and liquid coexisted, the temperature of the whole system remained very nearly equal to the melting point due to slow removal of heat when in contact with air, which was a poor heat conductor. Because of the latent heat of fusion, the freezing was greatly slowed down and the temperature would not drop anymore once

the freezing starts but would continue dropping once it finished. Crystallization consisted of two major events, nucleation and crystal growth. Nucleation was the step wherein the molecules started to gather into clusters, on the nanometer scale, arranging in a defined and periodic manner that defined the crystal structure. The crystal growth was the subsequent growth of the nuclei that succeeded in achieving the critical cluster size

4.1.2. Moisture content after freezing

Significant difference was found in moisture content of samples frozen under different temperature. Samples frozen under higher freezing temperature tended to had lower water content. This was due to the removed moisture from samples to the air by the refrigeration coils. Moisture left the surface of the samples and produces areas of visible damage known as freezer burn (Fellows, 1997). Picture was shown in the *Appendix* 2 (*Figure* A2-1).

4.2. Baking characteristics

4.2.1. Initial condensation (SHS)

In weight loss curves of SHS baked samples, there was a rapid rise in the beginning stage of baking, see *Figure* 3-4. This was called initial condensation and was mentioned in many researches (Bourassa, Ramachandran, Paliwal, & Cenkowski, 2015) (Mujumdar & Devahastin , 2008) (Devente & Heijmans, 2001) (Pronyk, Cenkowski, & Muir, 2005). The initial condensation was very unique to SHS as a processing medium. It occurred when a material at a temperature lower than the saturation temperature was exposed to SHS. SHS coming in contact with the material then became cooled and condensed on the material. The condensation was proved to have happened in this research according to the sudden rise in the weight loss curves of SHS baked samples in

Figure 3-4. Along with the increase of weight of samples, the momentary surface moisture content might also have increased, as proved to have happened in potato samples in research done by (Iyota, Nishimura, Yoshida, & Nomura, 2001). When the material surface temperature increased to the saturation temperature, condensation period was over and restoration period began. During restoration, the initial condensation evaporated and the moisture content of the material returned to its origin, as shown in *Figure* 3-6.

4.2.2. Temperature curves for heaters

Sudden drop in the temperature curves for heaters were detected in both baking methods, as is shown in *Figure* 3-3 and 3-4. This was due to the open of oven led to an abrupt colder air inside, which at once cooled down the surface of the heaters. However, this operation was unable to be omitted.

4.2.3. The maximum inner temperature of samples during baking

From *Figure* 3-3 and *Figure* 3-4, all samples maximum inner temperature would not transcend 100 ℃ in either baking method. This phenomenon was due to the high percentage of moisture content inside samples. As shown in *Table* 3-2, the average moisture content in most samples were over 65%.

This result corresponded with many researches for bakery products such as (Le-bail, et al., 2011) (Acar & Gokmen, 2009) (Mogol & Gokmen, 2014). During baking, inner temperature of samples rapidly rose to around 100 ℃ or near the boiling point of water according to the local atmospheric pressure and remained that temperature for certain time.

4.2.4. Thawing rate and baking time

From the results shown in *Table* 3-5 and *Table* 3-7, thawing rate of HA baked samples had positive correlation with freezing rate. Samples frozen under -45 ℃ had significant higher thawing rate than samples frozen under -15 ℃. This might be due to the more distinct sensible heat difference between -45 ℃ samples and the baking medium.

SHS baked samples did not have such correlations, significant higher thawing rate was found in -35 ℃ frozen samples compared with -15 ℃ frozen samples. However, for samples frozen under -45 ℃, the thawing rate was once again slower. The inaccurate position where the thermocouple sensor was inserted might be the reason. Compared with HA baked samples, it showed significant higher thawing rate in samples frozen under all temperatures, this was due to higher heat transfer properties in SHS than in HA at same temperature (Sehrawat, Nema, & Kaur, 2016). Initial condensation might also contribute to this result due to the condensation heat transfer which was confirmed in another research (Iyota, Nishimura, Yoshida, & Nomura, 2001). During the stage where SHS condensed on the surface of samples, instead of air, condensed water played important role of heat transfer. In free convection, heat transfer coefficient of air is 5 to 25 [W/m^2*k], whereas water would be up to 20 to 100 [W/m^2*k] (Singh & Heldman, 2014). Sensible heat transfer might have caused a faster thawing rate in this occasion.

In HA baked samples, lower freezing temperature frozen samples had longer baking time, as shown in *Table* 3-4 and *Figure* 3-7. This is easy to understand because lower temperature frozen samples had larger temperature difference before arriving at 100 ℃ and thus would generally take more time till fully baked. However, no such correlation or significant difference was found in SHS baked samples. This result showed a more complicated baking method compared to its counterpart HA baking method, as

also was indicated in (Asghar, Anjum, Butt, Tariq, & Hussain, 2007). Currently the knowledge and experience of SHS is still limited. On the other hand, in SHS baked samples, thawing rate had negative correlation with baking time. This showed a more consistent baking temperature change compared with HA, i.e. samples had faster completed thawing had faster speed of reaching to 100 ℃.

4.2.5. Drying rate

Nowadays SHS was still mainly used as drying medium, especially worked effectively in turbines due to its superior large amount of energy and specialty of avoiding droplets (Lalonde, accessed on 13th, June, 2016). The rate of evaporation of moisture present in a food material was given by Equation 4-1

$$N = \frac{q}{\lambda} = \frac{h(T_{medium} - T_{surface})}{\lambda} \quad (4\text{-}1)$$

N: Evaporation rate [kg water m^{-2} h^{-1}]

q: Heat transferred per unit area [Wm^{-2}]

ʎ: Latent heat of evaporation of water [J kg^{-1}]

h: Heat transfer coefficient [Wm^{-2}]

$T_{surface}$: Surface temperature of drying material [K]

T_{medium}: Superheated steam temperature [K]

Due to initial condensation, more time was required to dry SHS baked sample (see *Figure* 3-6). Although drying of the samples was slowed down by initial condensation, final baked samples by SHS still showed drier outcomes than HA baked ones (see *Table* 3-6). This was due to air-free environment in SHS baking method. Water vapour had no resistance to diffuse in such an environment compared with HA baking environment

(Sehrawat, Nema, & Kaur, 2016). Also, as the surface reached the dew point temperature, steam accelerated the temperature rise and water loss, especially for a relatively long baking time (Vanin, Lucas, & Trystram, 2009).

4.3. Colour change

In SHS baked samples, L_2 and a_2 values were significantly lower in samples frozen under -25 ℃, than samples frozen under -35 ℃, while a_2 values was significantly lower in samples frozen under -35 ℃. This was due to the longer baking time in samples frozen under -25 ℃ and shorter baking time in samples frozen under -35 ℃. Same results were given in sliced bread experiment (Jimenez, Vilanova, & Hernandez, 2001).

Compared with HA baked samples, SHS baked samples had significantly ($p<0.05$) lower L values, while higher in a, b total colour variance values. This result indicated accelerated surface browning by the combined effect of Maillard reaction and caramelisation (Kocadagli & Gokmen, 2016), between which the Maillard reaction took more part in colouring than caramelisation did (Deman, 1990). Previous research had found that the value of L_2 decreased more quickly by SHS than HA in the baking process of sliced bread (Iyota, Sakai, & Mamiya, 2013)by using in-situ colour measurement.

Conventional baking process contained two main stages, namely 1) development stage, 2) drying and colouring stage (Manley, 2000). In the development stage, dough temperature began to increase, causing some physical changes in dough such as fat melting, gluten softening and starch gelatinization. After certain level of thermal load, both moisture and temperature conditions became favourable for browning reactions in the latter stage. In drying and colouring stage, colour of samples crust was tanned through Maillard reaction, which essentially took place in food products containing high levels of

sugar and protein or amino acids. The rate of the reaction is proportional to the heat-treatment severity, particularly when a low water activity (Ames, Bates, & Mcdoulall, 1993)was reached. The presence of melanoidins, brown nitrogen-containing high molecular weight pigments, responded for the characteristic colour of roasted foods such as coffee, cocoa, bread and malt. Melanoidins can also be formed by sugar caramelization without participation of amino groups (Bastons, Monaro, SIguemoto, & Sefora, 2012).

In this research, baking temperature was controlled to be around 260 ℃ in either baking method. Comparatively larger amount of moisture loss to crust of samples baked by SHS might be the reason why the crust colour was more severely tanned.

The crust and crumb came from the same original dough, but their final properties differed according to a distinct local heat-moisture treatment. As soon as the dough was placed inside the oven, water evaporated from the warmer region, absorbing latent heat of vaporisation and the surface layers started drying, resulting in a much lower water content than at the centre part (Vanin, Lucas, & Trystram, 2009). Beneath this drying region, water vapour diffused through the interconnected pores towards the surface. A concomitant liquid water gradient was formed from the core to the surface. As the diffusive flow of liquid water from the core was less rapid than evaporation flow at the surface, a drying zone was developed that slowly increased in thickness and formed the crust (Wagner, Lucas, Ray, & Trystram, 2007). Compared with crumb, the heating rate of crust was higher (see *Appendix* 1, *Figure* A1-3). Also, the addition of steam reduced the water loss at the onset of baking, as shown in *Figure* 3-6. However, as the surface reached the dew point temperature, steam accelerated temperature rise and water loss for

long baking time, which differentiated with HA baked crust, showing a more tanned colour.

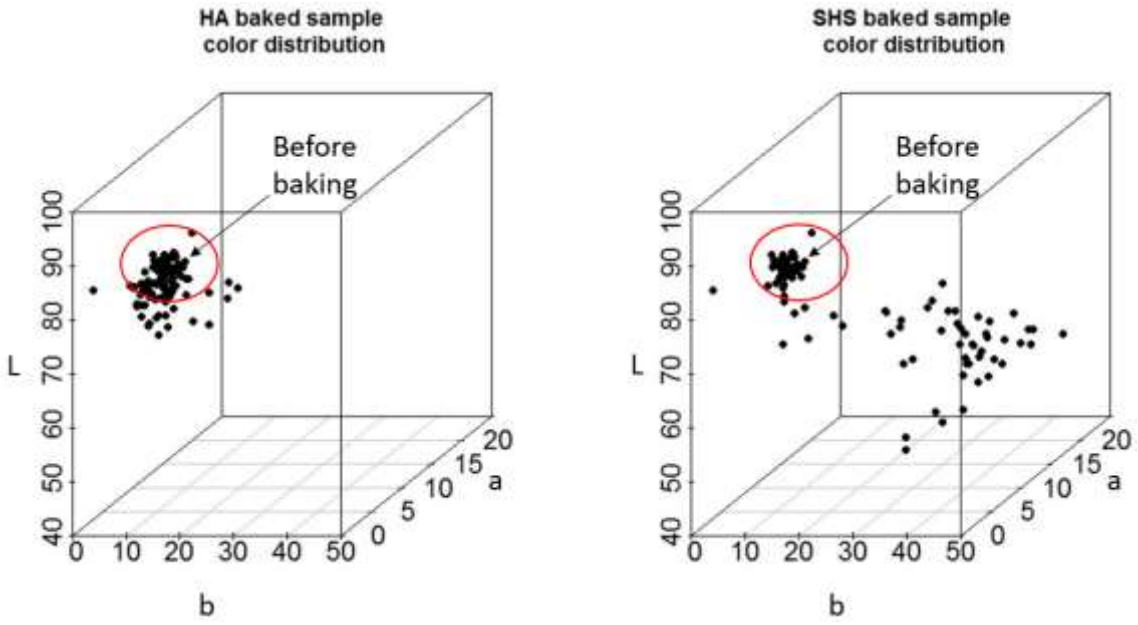

Figure 4-1 3D plot colour distribution of samples baked by HA and SHS

Figure 4-1 and *Figure* 4-2 gave the intuitive vision of the appearance comparison of crust colour between HA and SHS baked samples. L, a and b values of samples baked SHS are apparently distributed in a wider range in the colour space, indicating larger total colour change (ΔE*) values with darker, yellower and redder outcomes, even within comparatively shorter baking time.

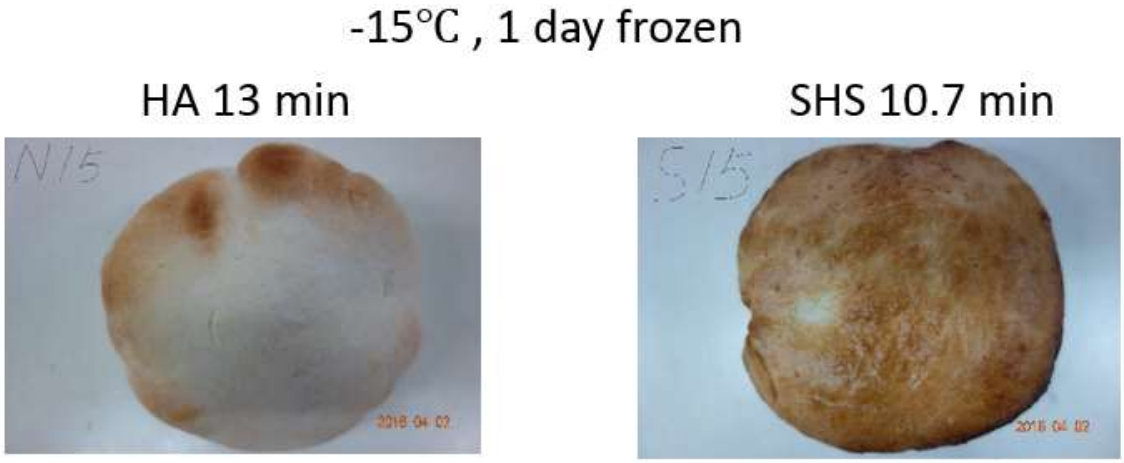

Figure 4-2 Appearance of samples baked by HA and SHS

4.4. Rheological properties

4.4.1. Creep test

4.4.1.1. Effect of freezing rate in either baking method

Different freezing rate did not have significant effect on viscoelastic properties of samples.

Although significant difference was found in delayed elasticity E_1 in SHS baked crumbs and delayed viscosity V_1 in crumbs of samples in both baking methods, as shown in *Figure* 3-9 and *Figure* 3-10, baking time seemed to have more influence on this consequence instead of freezing rate. Samples frozen under -45 ℃ and -25 ℃ took approximately same longer baking time compared with samples frozen under -15 ℃, which led to the significant lower viscoelastic properties. This result was also true in V_1 values of HA baked sample crumbs, in which samples frozen under -45 ℃ took significant longer baking time and thus rendered significant lower V_1 values compared with samples frozen under -15 ℃. On the other hand, compared with samples frozen under -15 ℃, samples frozen under -35 ℃ which had similar baking time, showed no significant difference in all four-element viscoelastic parameters. It further proved the conclusion that compared with baking time, freezing rate did not seem to cause significant difference in creep test results.

4.4.1.2. Assumptions

In viscoelasticity comparison between HA and SHS, all significant difference found was in samples baked within a shorter baking time (8.6±1.7 min, 8.4±0.6 min). This result shorter baking time, more difference in rheological properties between the two medium baked samples. This results corresponded with the other research results reported

by (Head, Cenkowski, Arntfield, & Henderson, 2010) that colour of oat processed with SHS changed significantly ($p<0.05$) during the initial exposure to SHS, but no major changes in the colour of oat groats were noted after about 1 min of SHS processing. It would be valid to assume that the initial condensation might have been the most influential factor behind these outcomes.

According to the significant difference, compared with HA baked samples, SHS baked samples were found to have lower delayed elasticity, lower delayed viscosity and lower permanent viscosity values in crumbs, whereas had higher instantaneous elasticity and higher delayed viscosity values in crusts. On the other hand, in HA baked samples, crumbs tended to be more viscoelastic than crusts whereas this result did not agree with SHS baked samples, in which crusts showed more viscoelastic behaviour than crumbs.

Starch gelatinisation might have great influence on these outcomes. The degree of gelatinisation (DSG) was shown to be directly affected by water (Burt & Russell, 1983), (Le-Meste, Huang, Panama, Anderson, & Lentz, 1992) (Jenkins & Donal, 1998) (Fessas & Schiraldi, 2000) (Cup, Abecassis, & Guilbert, 2003) and the heating rate (Donavan, 1979) (Bloksma, 1980), which again differentiated crumb and crust in one baked sample. As shown in the *Appendix* 1 (*Figure* A1-11), according to previous studies, the distinct higher heating rate in crust layer than in crumb even under different baking temperatures, as well as a much higher water loss rate, both of which contributed to the postpone of starch gelatinisation happening in crust layer. This might favour low level of viscoelasticity (Vanin, Lucas, & Trystram, 2009).

However, the introduction of initial condensation which not only delayed the drying rate but on the contrary increased crust layer moisture content, both of which might

have favoured starch gelatinisation on the surface of baked samples and thus gave an increase in viscoelasticity in crusts. Depending on the porosity of the material the condensed moisture has the ability to permeate into the interior of the material (Bourassa, Ramachandran, Paliwal, & Cenkowski, 2015). Another study done by (Iyota, Nishimura, Yoshida, & Nomura, 2001) reported there was slight moisture increase in the surface of potato shortly after the start of baking, other researches also had shown small amounts of moisture increase in the first few seconds of processing in superheated steam due to the initial condensation in sugar-beet, pulp, potatoes, Asian noodles, brewer and distiller's grain (Pronyk, Cenkowski, Eng., & Muir, 2004). Although after the temperature of dough surface reached up to the dew point, drying rate was accelerated and rendered a dryer outer layer than inside, water redistribution from crumb to crust might be the main reason that humidified the crust whereas dried the crumb. In the research done by (Baik & Chinachoti, 2000), they detected the result that during storage, bread crumb moisture content and water activity decreased significantly when stored with crust, whereas when stored without crust, the water content and water activity remained relatively unchanged.

On the other hand, case hardening might also contribute to the water redistribution from crumb to crust in SHS baked samples. Case-hardening is particularly common with foods that contain dissolved sugars and other solutes in high concentration, as pointed out by (Potter & Hotchkiss, 2012). It occurs when there is high surface temperature and unbalanced drying of the piece so that a dry skin forms quickly before most of the internal moisture has had opportunity to migrate to the surface.

Case hardening may cause sealing of the surface of food. This can be explained from various ways water may escape from a product during drying period. Some of the

water moves via cell walls or through membranes by molecular diffusion. Again, water may be heated to vapour within a food piece and escape as water vapour molecules free of solute. Water in foods rises in the voids, cracks and pores of various diameters down to minute capillary size from inside to food surface. Capillary water carries sugars, salts and other materials in solution to the surface of food during dehydration. Then at the surface, water is evaporated and the solutes are deposited, which elevated the osmatic pressure in the upper layer of samples, accelerated the water transportation from inside to outside. Although in this research, the exact moisture content in crust of samples was not measured, extremely dried and hardened surface layer of samples baked by SHS was obtained immediately after baking completed, whereas after cooling was finished, the surface regained viscoelasticity and tenderness. Another point which differentiated HA and SHS baked samples is the crus thickness (see *Appendix* 1, *Figure* A1-4). Compared with HA baked samples, SHS baked samples had thicker crusts formed during baking due to the higher drying speed the outer layer of samples, which separated the crumb and crust, substantially contributed to the organoleptic perception of the final product. Significant thicker crust bake by SHS compared with HA were found to be those under longer baking time.

4.4.2. Fractural test

No breaking point was found in crumbs or crusts in samples in either baking method. Instead of breaking force which should have been measured at the exact breaking point, maximum force was obtained from all samples in this test and there showed no significant difference within all comparisons. This might indicate that the samples in this research were not s for performing fractural test.

4.4.3. TPA test

According to the results of parameters selected in this research, hardness, cohesiveness or gumminess were not found to have significant difference in the comparison in different freezing rate or comparison between two baking methods in crumbs of samples. Due to the difficulty to conduct TPA test on crusts, comparison between crumbs and crusts in both baking methods were not available to discuss in this research. However, based on the cross-correlation plot in the *Appendix* 1(*Figure* A1-5, *Figure* A1-6, *Figure* A1-7, *Figure* A1-8*)* in both HA and crumbs of SHS baked samples, hardness values had positive correlation coefficient ($p<0.01$) with all viscoelastic parameters values. Thus an assumption that the TPA test results might show the same in the comparison between crumbs and crusts of HA and SHS baked sample is given. Further research is necessary toward this assumption.

Chapter 5. Conclusion and future work

5.1. Freezing characteristics

Freezing rate was higher in samples frozen under lower freezing temperatures. Large temperature difference might have favoured the faster speed for removing latent heat during phase transition, in which ice crystal formed. Different exposure time to the freezing air during freezing caused a moisture difference in frozen samples.

5.2. Baking characteristics

Initial condensation had been a unique phenomenon in SHS baking, a weight increase proved this phenomenon in this research. During baking, inner temperature in samples did not transcend 100 ℃ due to the phase transition.

Compared with HA baked samples, samples baked by SHS showed significant higher thawing rate in samples frozen under all temperatures, this was due to higher heat transfer properties in SHS than in HA at same temperature. Initial condensation might also contribute to this result.

Some apparent correlation was found in HA baked samples such as the longer the lower the freezing temperature, the longer the baking time. However, this did not work the same in SHS baked samples, revealing a more complicated baking method compared with HA. The knowledge and experience in SHS is still limited.

Although the results for drying rate of samples were not obtained over three times in this research, according to the existing outcome, compared with HA baked samples, SHS baked sampled showed moisture content loss. This might be due to the higher heat

transfer rate and easier diffusion environment for water vapour in SHS filled oven. Also, initial condensation might also have influence on this outcome.

5.3. Colour

Compared with HA, SHS baked samples had lower L values, larger a, b and total colour variance ΔE* values, indicating darker, yellower and redder outcomes. Colour significant difference between HA and SHS was found in samples frozen under all freezing temperature. On the other hand, different freezing temperature did not have significant influence on crust colour of baked samples by either baking method.

From Pearson's correlation coefficient results, in SHS baking method, samples frozen under higher freezing temperature had yellower and redder crust colour and more total colour variance. The longer the baking time, the darker and yellower crust colour it would be and thus more total colour variance. Also longer baking time gave SHS baked samples thicker crust.

5.4. Rheological properties

Compared with baking time, freezing rate did not seem to cause significant difference in creep test results. In viscoelasticity comparison between HA and SHS, all significant difference found was in samples baked within a shorter baking time (8.6±1.7 min, 8.4±0.6 min). On the other hand, in HA baked samples, crumbs tended to be more viscoelastic than crusts whereas in SHS baked samples, crusts showed more viscoelastic behaviour than crumbs. A combined effect of starch gelatinisation, initial condensation, crust thickness and water redistribution from crumb to crust which might have been

caused by case hardening during the cooling down period might have contributed to this result.

In fractural tests, no breaking point was found in crumbs or crusts of samples in either baking method. Instead of breaking force which should have been measured at the exact breaking point, maximum force was obtained from all samples in this test and there showed no significant difference within all comparisons. This might indicate that the samples in this research were not suitable for performing fractural test.

TPA test was only conducted on crumbs of samples due to its difficulty to conduct on the thin crust layer. No difference was found in comparison between samples that were frozen under different temperature, nor in comparison between HA and SHS baked crumbs of samples. However, due to the apparent cross-correlation coefficient between TPA test results and creep test results in crumbs in either baking method, it was assumed that hardness and gumminess might also show a positive correlation, while cohesiveness a negative correlation with viscoelastic properties of crusts.

5.5. Super cooled samples

Although the freezing temperature was the lowest (-5 ℃) of all in this research, super cooling rendered the batch of samples highest freezing rate and possibly small and homogenous fine ice crystals, which differed these samples significantly different from samples those were frozen under high freezing temperature, especially in rheological properties. Micro-structure of samples frozen under -5 ℃ should be the reason why they were differentiated in this research from other samples. Further observation of these micro-structure is necessary.

5.6. An overall wrap up of the conclusion

Compared with HA baked samples, SHS baked samples showed higher thawing rate, less baking time and higher drying rate; SHS baked samples had darker, yellower and redder crust colour; SHS baked samples were more viscoelastic in crusts, than in crumbs. Fractural test might not be suitable for measuring the samples in this research. TPA test results of samples crumbs including hardness, cohesiveness and gumminess were not found to be significantly affected by using different baking method.

Initial condensation, **drying rate** especially at the top layer of pizza crusts **crust thickness** and **water redistribution from crumbs to crusts** might have played the most important roles which differentiated HA and SHS baked products. Different freezing temperature from -15 ℃ till -45 ℃ did not seem to produce different outcomes in colour or rheological properties of frozen pizza, except for thawing rate due to the temperature difference between frozen pizza dough and baking medium.

5.7. Future work

1. In this research, using SHS as the baking medium conferred higher thawing rate, less baking time and higher drying rate on frozen pizza dough. SHS was used from the starting point of baking till baking completed. It is also important to know the optimum steaming time, velocity and pressure of steam for finding out the optimization SHS utilization on frozen pizza processing in further studies.
2. As was noted in the introduction part in this research, SHS had advantages like lower energy consumption, sterilization and so on. Further studies should also be focusing on calculating the energy cost and saved in SHS system and observations of the elimination of microorganisms.

3. More investigations should be taken to examine the sensory score of products baked by SHS, in both consumers panel and experts panel. A thin layer of tomato source toppings can be used, but note to not cover the original flavour of the crust (Delahaye, Jimenez, & Perez, 2005). Industrial survey in pizza processing factories, pizzerias, delivery-shops should be conducted to get more attitude and ideas on the feasibility of spreading SHS as a frozen pizza processing medium. It is significant to link instrumental measurement results with sensory evaluations scores in order to decide the pros and cons of utilization of SHS as a baking medium on frozen pizza products.
4. In order to capture the baking characteristics of frozen samples under different freezing conditions, samples in this research did not undergo enough fermentation except for a short time pre-proofing before freezing. Considering the capability of eliminating the microbial inside the samples by SHS, it is assumed that SHS baked frozen samples might have lower extent of oven spring compared with HA baked samples. Further studies is necessary to confirm this assumption and the associated impact.
5. Due to the availability, frozen samples had to experience a further cooling process by dry ice during transportation from Mayekawa Manufacturing Company till the University of Tokyo. The effect of dry ice on samples remains unknown. Extremely low temperature is detrimental to the vitality of yeast cells and oven spring might be negatively affected.
6. More accurate method for inserting thermocouples inside the samples for measuring the inner temperature during baking, should be developed in further studies.

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Appendix 1. Supplemental Tables and Figures

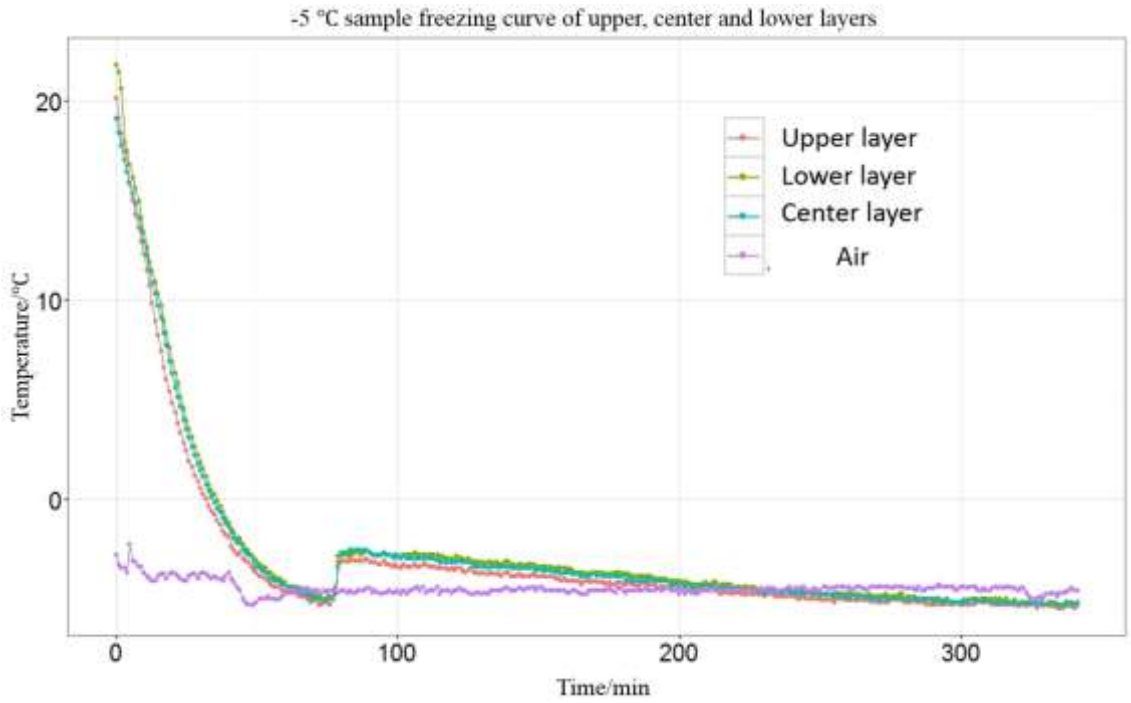


Figure A1- 1 -5℃ sample freezing curve of upper, center and lower layers

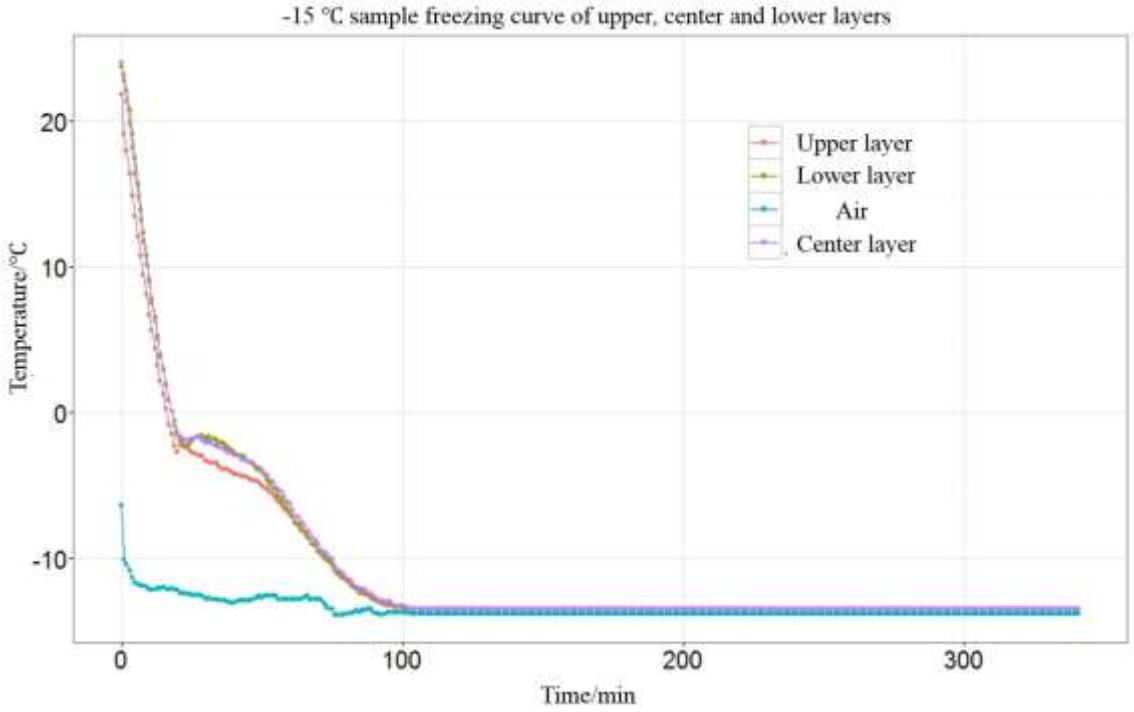


Figure A1- 2 -15℃ sample freezing curve of upper, center and lower layers

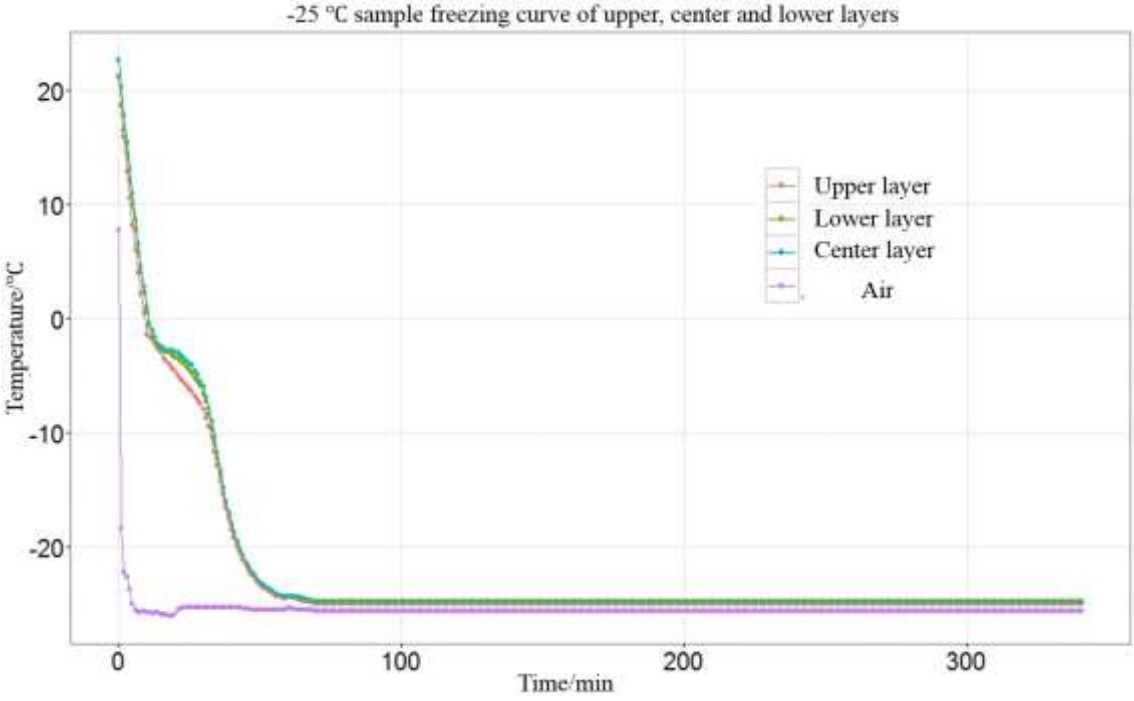


Figure A1- 3 -25℃ sample freezing curve of upper, center and lower layers

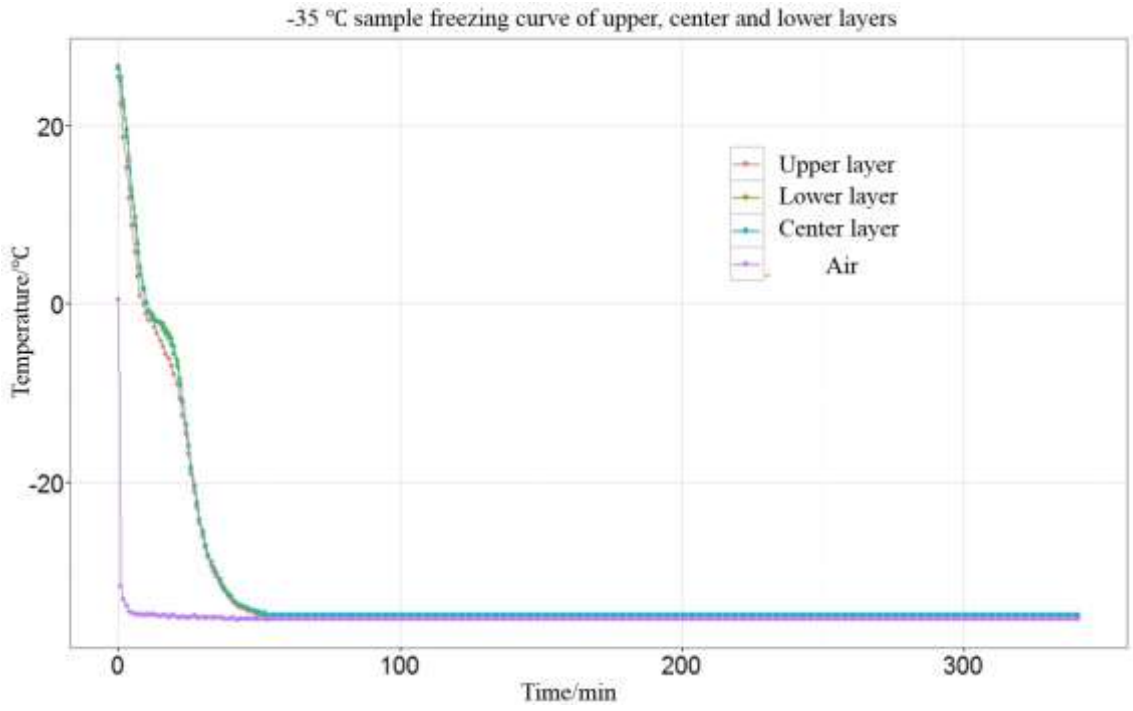


Figure A1- 4 -35℃sample freezing curve of upper, center and lower layers

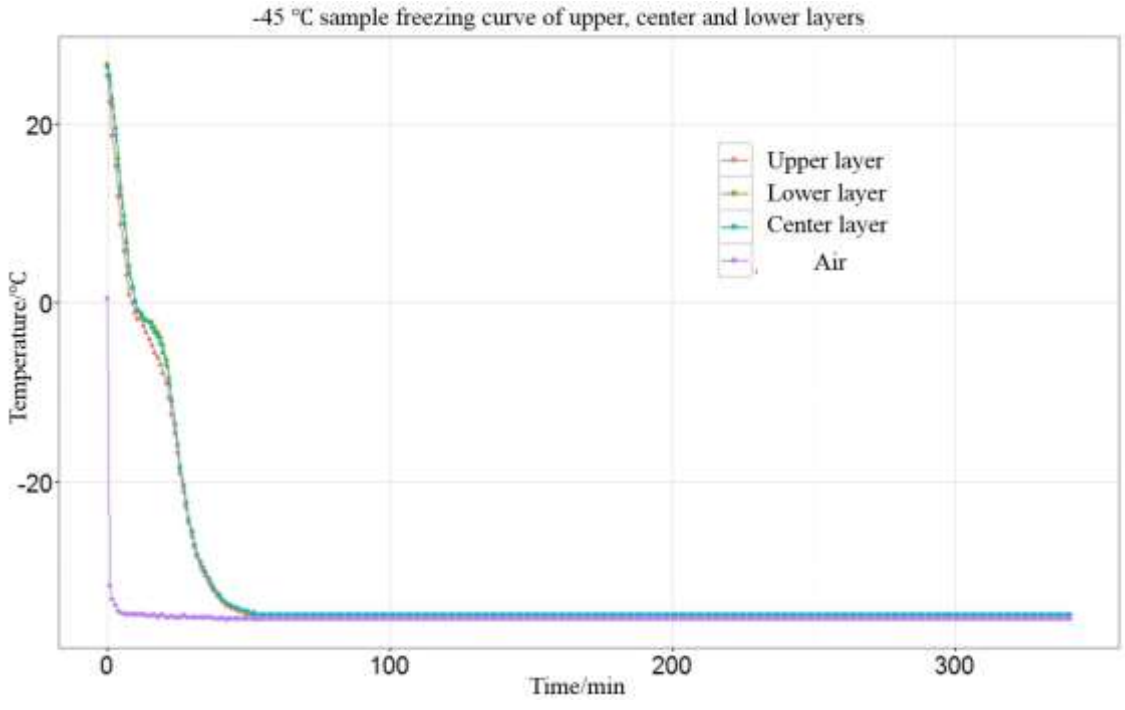


Figure A1- 5 -45℃sample freezing curve of upper, centre and lower layers

Viscoelastic properties

In bar plots listed below, legends on x-axis describing each of the bars were named as follows:

N: HA/No steam

S: SHS

0, 1, 2, 3, 4 referred to the freezing temperature of specified samples in such an order: -5 ℃,-15 ℃, -25 ℃, -35 ℃, -45 ℃.

a,b,c (HA baked): Means in samples by same baking method by the same lowercase superscript letters are not different ($p<0.05$)

A, B, C (SHS baked): Means in samples by same baking method by the same lowercase superscript letters are not different ($p<0.05$)

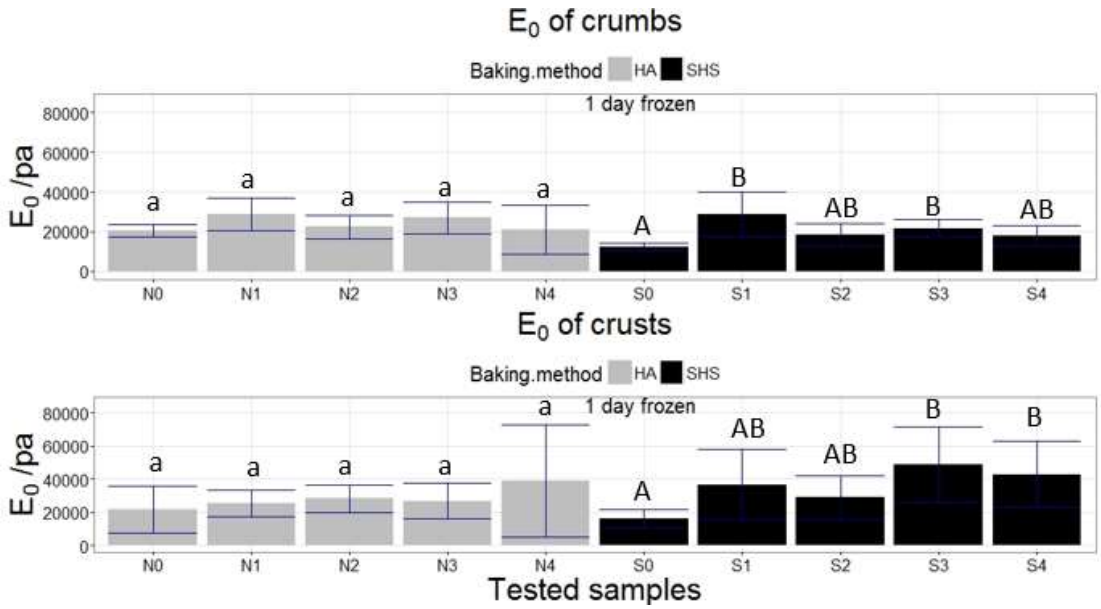


Figure A1- 6 Instantaneous elasticity E_0 values of crumbs and crusts (1 day frozen samples)

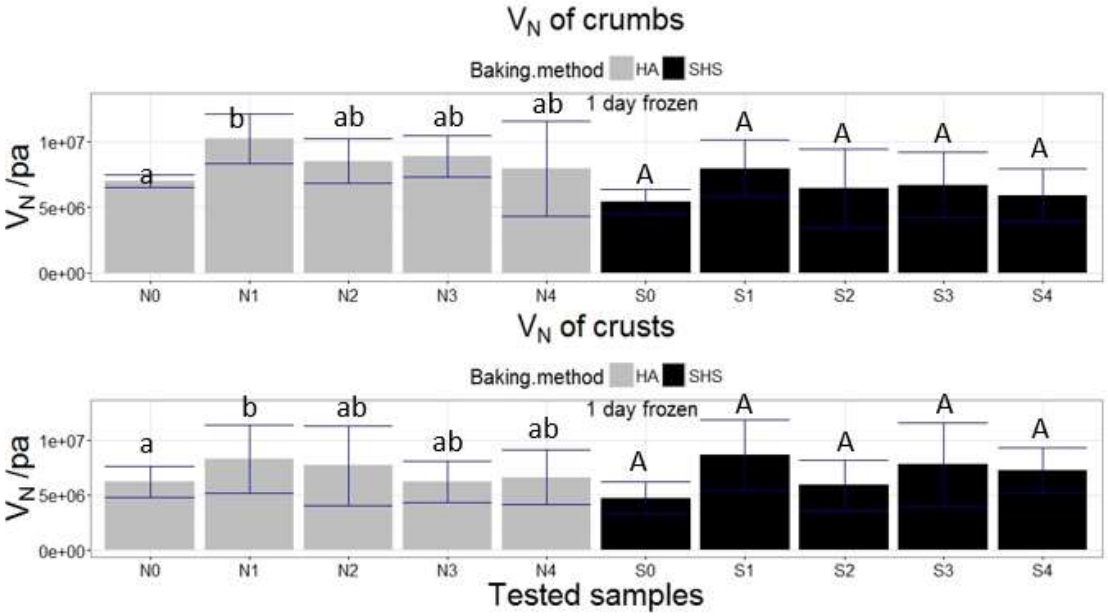


Figure A1- 7 Permanent viscosity V_N values of crumbs and crusts (1 day frozen samples)

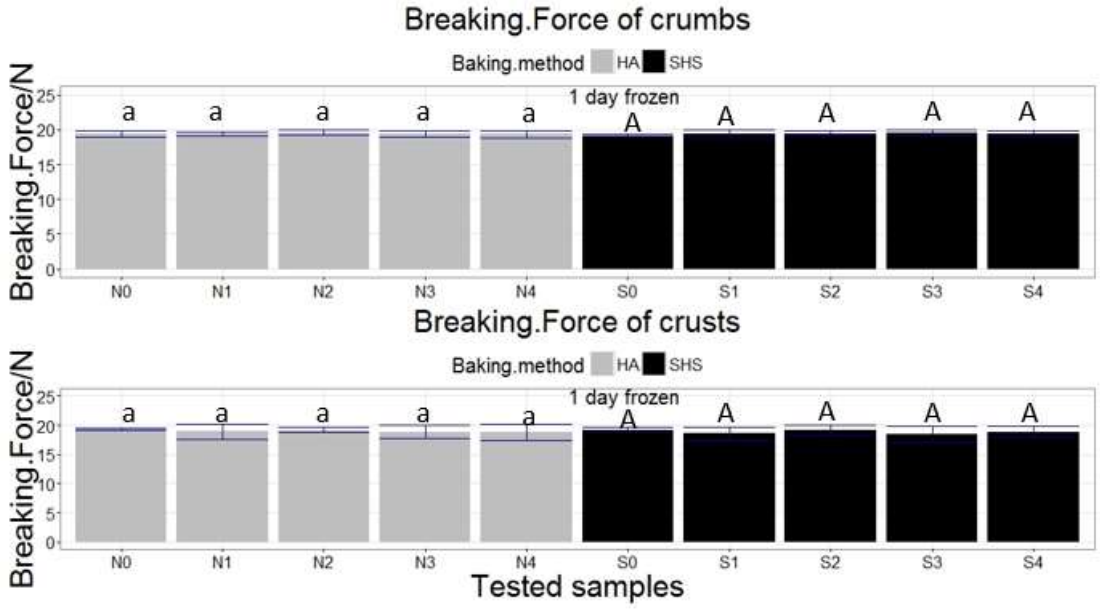


Figure A1- 8 Breaking force values of crumbs and crusts (1 day frozen samples)

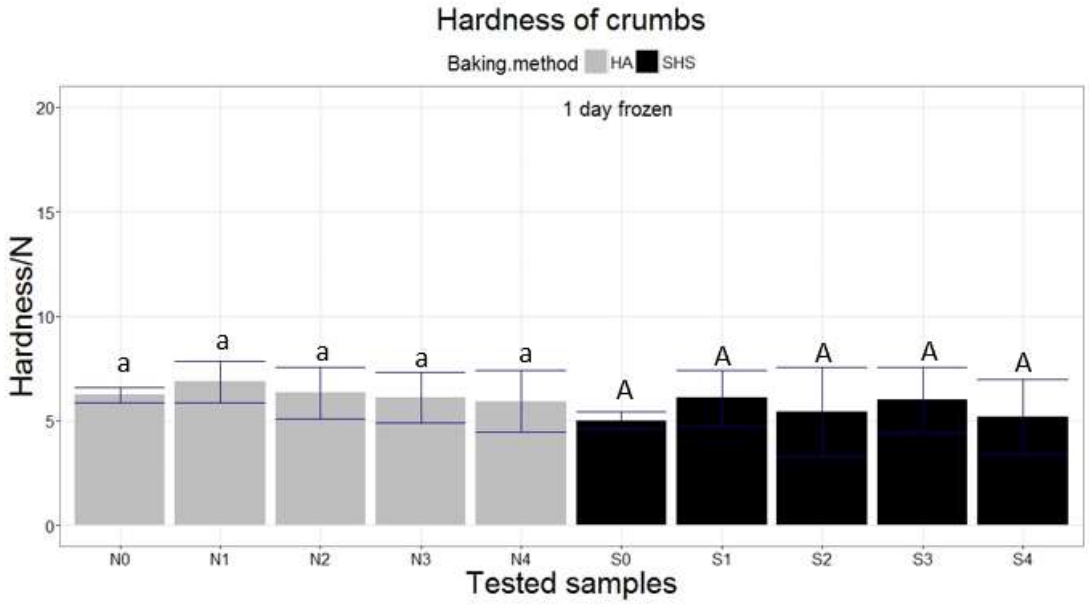


Figure A1- 9 Hardness values of crumbs (1 day frozen samples)

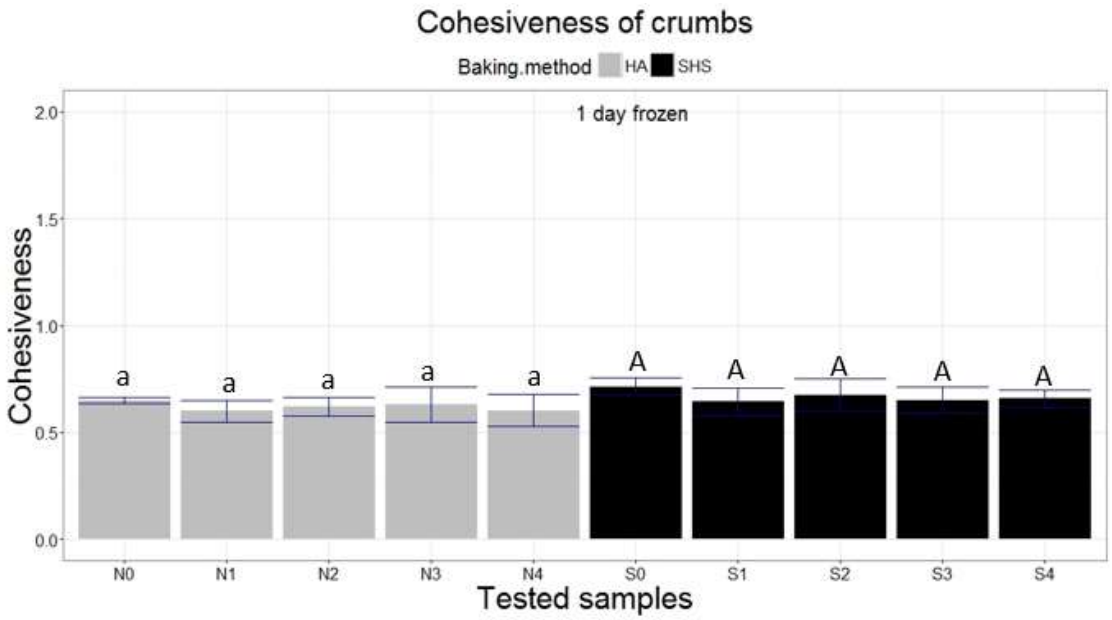


Figure A1- 10 Cohesiveness values of crumbs (1 day frozen samples)

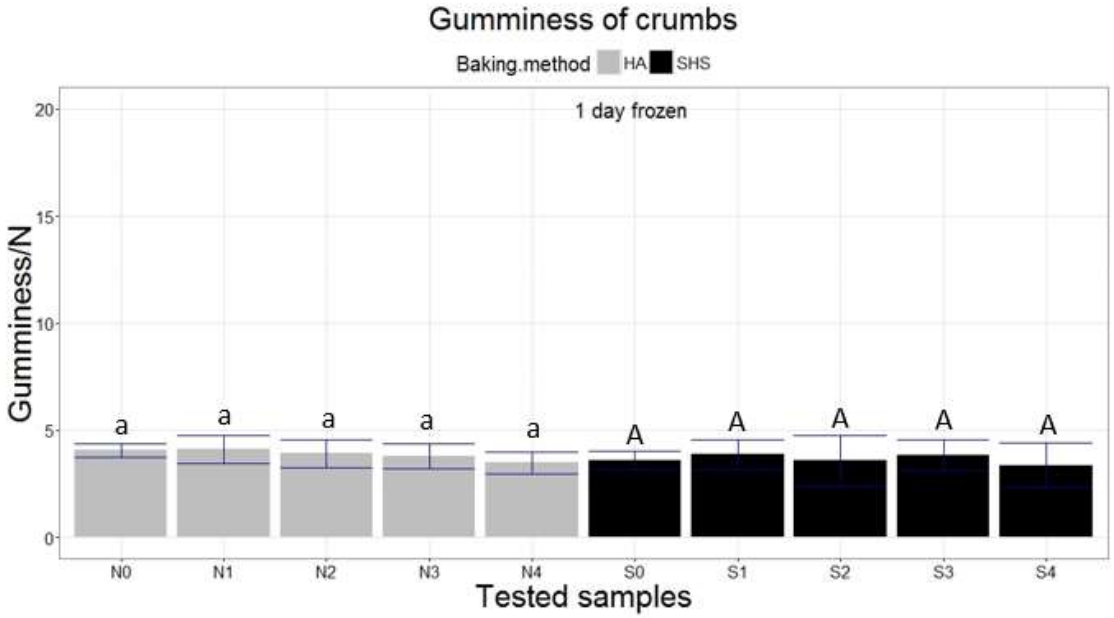


Figure A1- 11 Gumminess values of crumbs (1 day frozen samples)

Table A1- 1 Comparison of baked sample properties between HA and SHS baked samples (1 day frozen)

Instrumental data	Samples	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
E_0	Crumb	***	0.99	0.19	0.1	0.49
	Crust	0.29	0.16	0.9	*	0.4
E_1	Crumb	***	0.09	0.08	*	*
	Crust	0.13	0.73	0.87	0.43	0.36

V_1	Crumb	***	0.07	0.08	*	0.07
	Crust	*	*	0.8	0.36	0.32
V_N	Crumb	***	*	0.1	*	0.16
	Crust	*	0.78	0.23	0.29	0.45
Hardness	Crumb	***	0.19	0.29	0.87	0.34
Cohesiveness	Crumb	**	0.14	0.08	0.54	0.07
Gumminess	Crumb	*	0.46	0.49	0.89	0.76
Crust thickness	crust	0.247	0.4	***	0.24	*
L2	crust	**	***	***	***	***
a2	crust	**	***	***	***	***
b2	crust	**	***	***	***	***
ΔE*	crust	**	***	***	***	***

*,** and ***: Significant difference at $p<0.05$,$p<0.01$ and $p<0.001$ level, respectively

Table A1- 2 Comparison of baked sample properties between crumbs and crusts (1 day frozen)

Samples	Baking method	Freezing Temperature/℃				
		-5	-15	-25	-35	-45
E_0	HA	0.83	0.36	0.12	0.94	0.17
	SHS	0.1	0.34	0.06	**	**
E_1	HA	*	0.74	**	**	0.1
	SHS	0.08	0.36	0.41	0.7	0.19
V_1	HA	***	***	**	**	0.11
	SHS	**	0.63	0.41	0.68	0.2
V_N	HA	0.18	0.13	0.56	**	0.38
	SHS	0.28	0.57	0.68	0.48	0.18

, and ***: Significant difference at $p<0.05$,$p<0.01$ and $p<0.001$ level, respectively*

Table A1- 3 Heating rates for different cereal products and baking conditions at bread surface and core (reviewed by (Vanin, Lucas, & Trystram, 2009)*)*

Product	Mass of dough/batter (g)	Surfaces exposed to heat transfer	Position of temperature in dough/batter	Oven air temperature (°C)	Heating rate (°C/min)	Reference
Bread	200	Top bottom	Top surface	185	7.7	(Wagner et al., 2008a and Wagner et al., 2008b)
			Bottom surface		2.6	
			Centre		2.4	
Bread	341	All	**Top surface**	203	**11.3**	(Zanoni et al., 1993)
			Bottom surface at 1 cm beneath		9.5	
			The top surface		2.6	
Bread	–	All	**Top surface**	210	**10.3***	(Thorvaldsson, & Skjöldebrand, 1996)
			Mid-width		3.9*	
			Centre		1.8*	
Pan bread	–	–	**Surface**	220	**8.8**	(Dogan, 2002)
Francala	–	–			**14.4**	

Product	Mass of dough/batter (g)	Surfaces exposed to heat transfer	Position of temperature in dough/batter	Oven air temperature (°C)	Heating rate (°C/min)	Reference
Bread	1.5 L	All	**Top surface**	225	**6.7**	(Thorvaldsson, & Skjöldebrand, 1998)
			Bottom surface		4.4	
Bread	760	All	**Side surface**	235	**7.4**	(Marston & Wannan, 1976)
			Centre		2.8	
Biscuit	–	All	Centre	300	75.0** (400 g/kg dry air)	(Chevallier et al., 2002)
				300	61.7** (20 g/kg dry air)	
				240	44.7** (200 g/kg dry air)	
				180	19.6** (20 g/kg dry air)	
Sponge cake	400–600	Top	**Top surface**	200	**0.5**	(Lostie et al., 2002)

*"–" Refers to missing information. *Data from reheated samples. **With different relative air humidity. Bold and italic values emphasizes high surfaces values.*

Table A1- 4 Sample crust thickness (after baking, 1 day frozen)

Samples		Freezing Temperature/℃				
		-5	-15	-25	-35	-45
Crust	HA	$1.6\pm0.2^{a,A}$	$1.8\pm0.2^{a,A}$	$1.8\pm0.2^{a,A}$	$1.8\pm0.5^{a,A}$	$1.9\pm0.4^{a,A}$
	SHS	$1.5\pm0.3^{d,A}$	$2.0\pm0.3^{c,A}$	$2.5\pm0.4^{b,B}$	$2.1\pm0.3^{ac,A}$	$2.4\pm0.3^{ab,B}$

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same column have no difference($p<0.05$)

Table A1- 5 Cross- correlation coefficient (1 day frozen, HA baked crumbs)

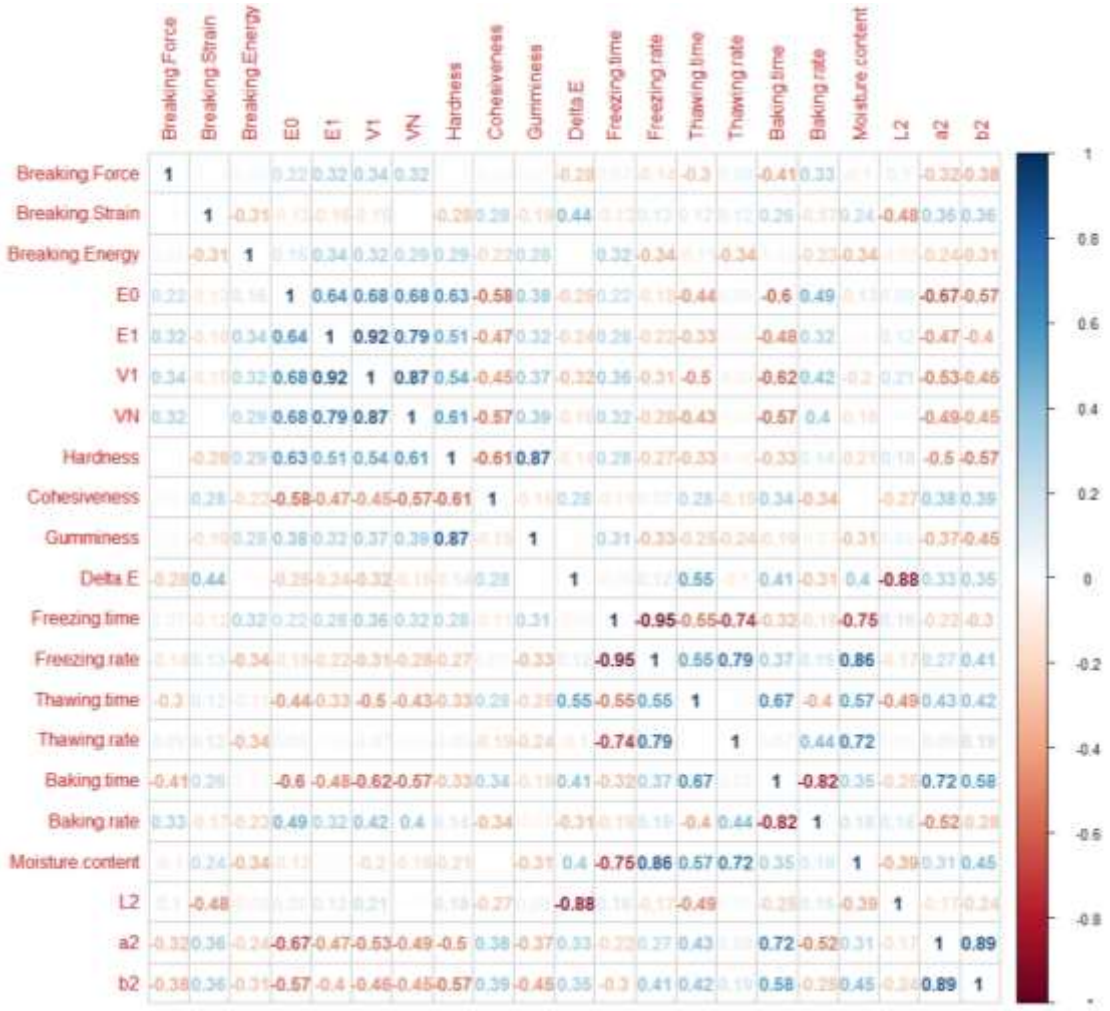


Table A1- 6 Cross- correlation coefficient (1 day frozen, SHS baked crumbs)

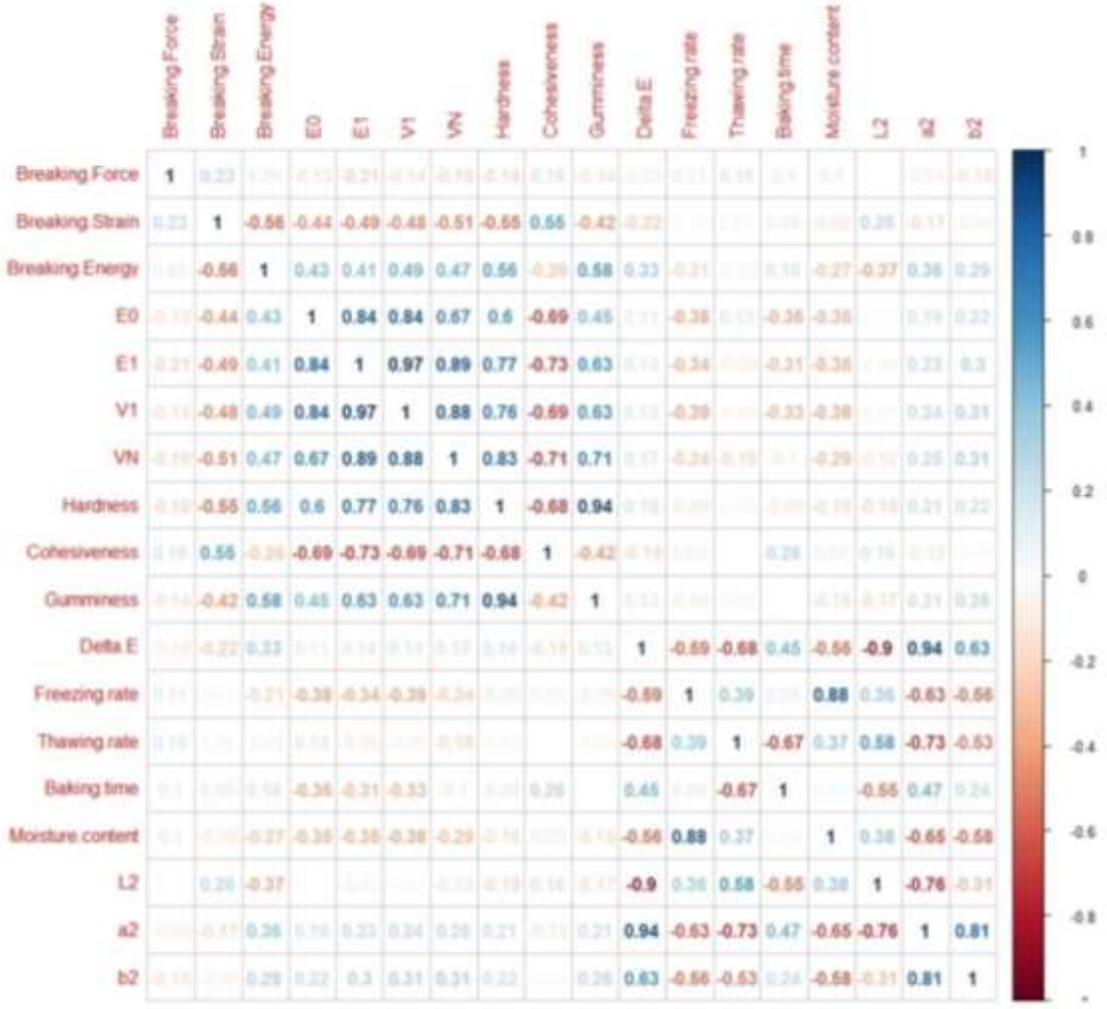


Table A1- 7 Cross- correlation coefficient (1 day frozen, HA baked crusts)

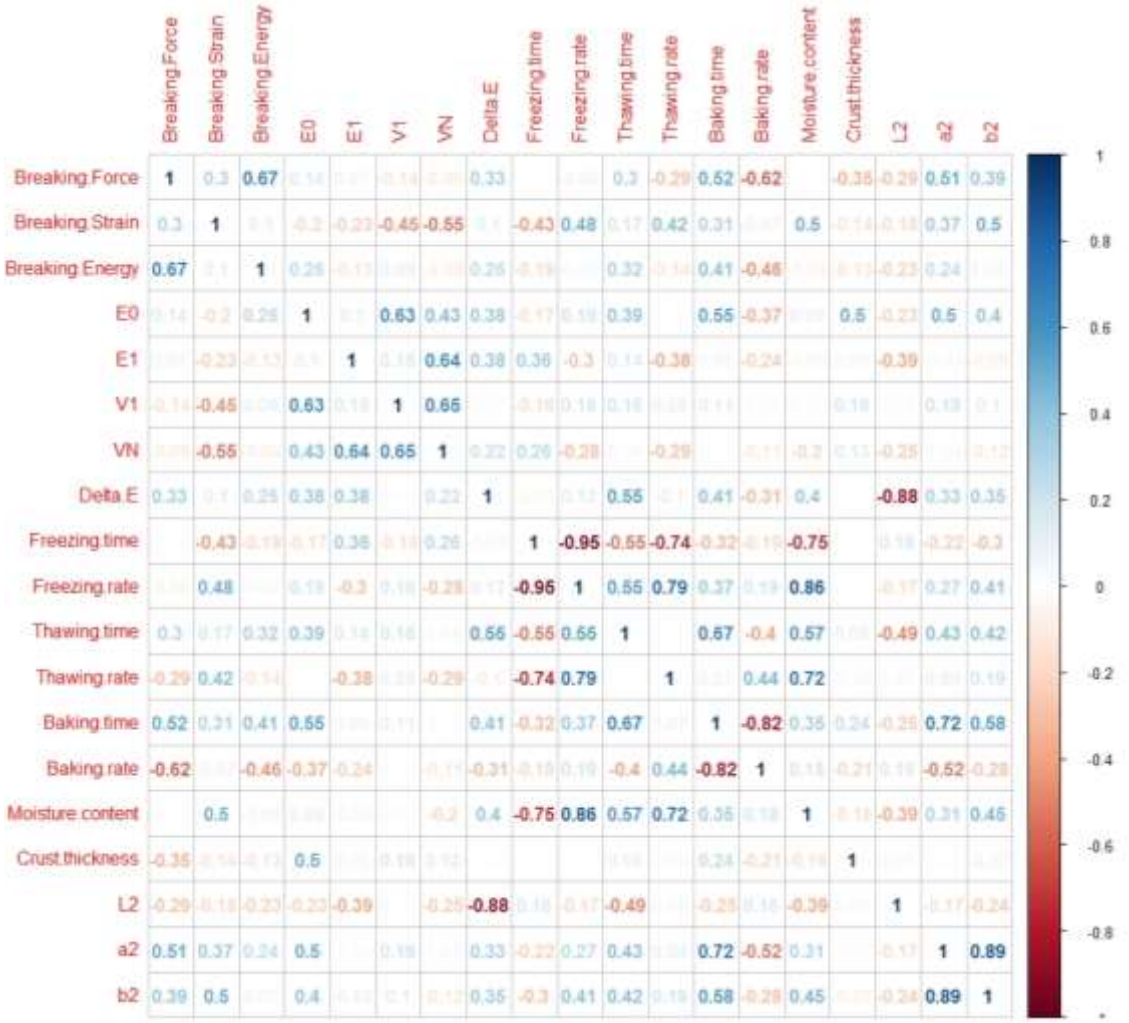
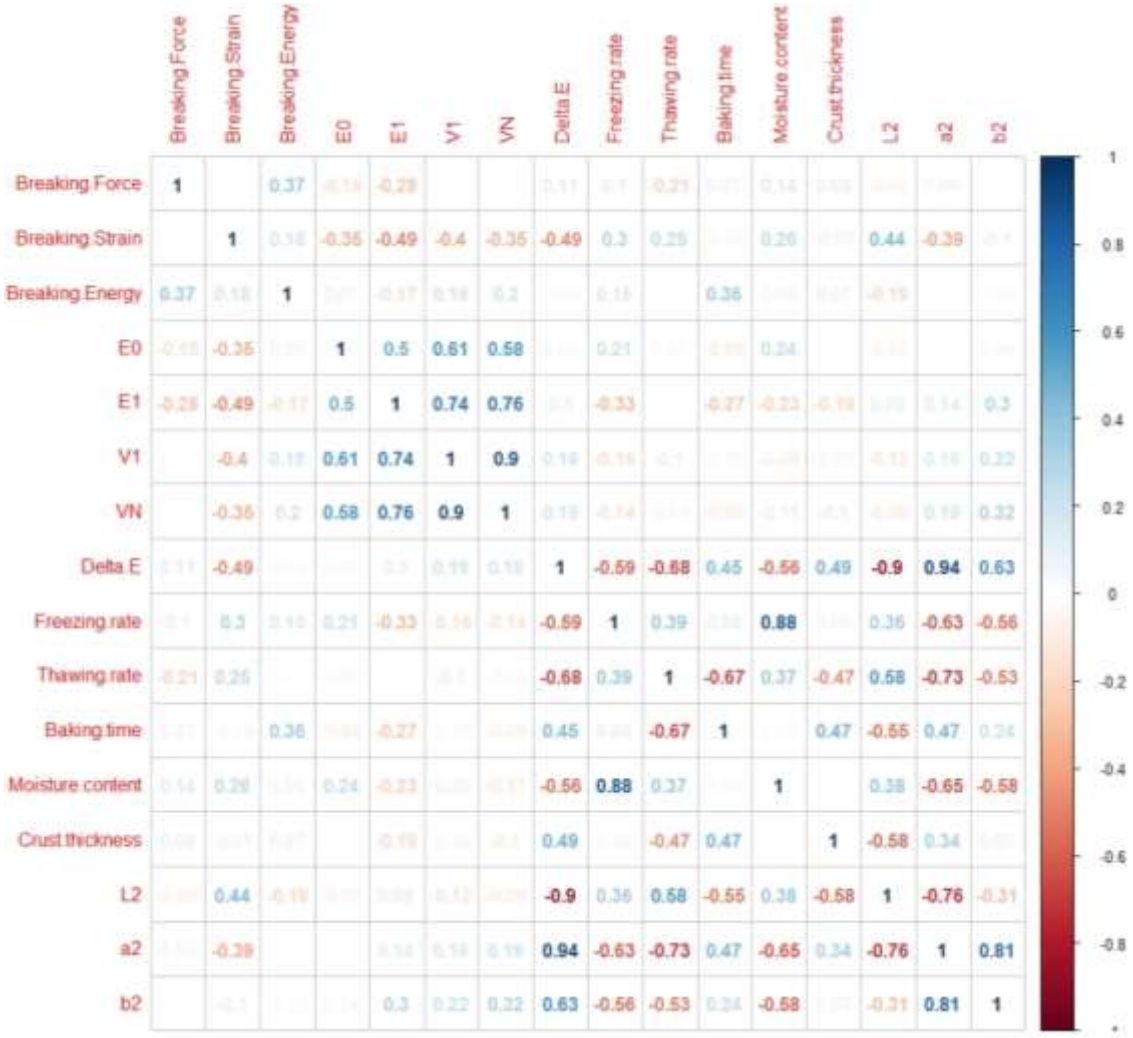


Table A1- 8 Cross- correlation coefficient (1 day frozen, SHS baked crusts)



Appendix 2. Additional tests (prolonged frozen storage)

Aside from samples frozen for 1 day, there were also trials on longer frozen storage from 15 days, 25 days till 35 days, each of which contained one sample. The research materials, methods and steps followed exactly the same with former research and were explained in *Chapter* 2.2.4.

Appendix 2.1. Moisture content after frozen storage

Longer frozen storage till 35 days did not give samples significant difference in moisture content, whereas samples frozen under -5 ℃ over 15 days gave significant ($p<0.05$)moisture content difference (49.3±1.1) from 1 day frozen samples (64.7±0.4). It indicated samples frozen under -5 ℃ over 15 days were very much dried, even close to the extent of fully dried samples moisture content (44.2±0.6).

Table A2- 1 Moisture content of raw samples after different lengths of frozen storage

Freezing Temperature/℃	Frozen storage duration/days			
	1	15	25	35
-5	64.7±0.4^{c,A}	49.3±1.1^{c,B}	NA	NA
-15	68.1±0.8^{a,A}	69.1±0.6^{ab,A}	67.8±0.6^{a,A}	68.1±0.2^{c,A}
-25	68.5±0.1^{a,A}	68.1±0.6^{b,A}	68.3±0.7^{a,A}	69.2±0.2^{a,A}
-35	70.0±0.4^{b,A}	70.1±0.3^{a,A}	69.1±0.4^{ab,A}	70.0±0.2^{ab,A}
-45	71.1±0.5^{d,A}	70.5±0.3^{a,A}	70.8±0.3^{b,A}	71.0±0.9^{b,A}

a,b,c:Means in the same column by the same lowercase superscript letters are not different($p<0.05$)

A,B,C:Numbers followed by the same uppercase superscript letters in the same row have no difference($p<0.05$)

Freezer burn was observed in sample frozen under-5 ℃ for 15 days frozen storage. Due to overlarge amount of moisture left the sample, visible damage was produced on the surface of sample, as shown in *Figure* A2-1. The serious wrinkled crust surface indicated the dried condition of the crust.

The extreme dried condition of samples frozen under -5 ℃ for 15 days frozen storage could be attributed to the freeze-thaw cycle that drew water out of the gluten matrix. Internal damage to starch granules due to ice recrystallization was noted during frozen storage and freeze-thaw cycles (Berglund, Shelton, & Freeman, 1991). Starch that was physically damaged would allow penetration of water into cracks or fractures on the surface or outer edge of the granule. (Tipples, 1969) found that damaged starch caused a linear increase in water absorption by flour since the damaged starch acts like a sponge in the presence of water. Increased damage to starch granules may be the reason to explain the water redistribution.

As more water was drew from the gluten matrix, the water was apparently concentrated in large pools recognised as ice. Subliming of the ice is the reason which caused the overlarge moisture loss.

Figure A2- 1 Freezer burnt sample (-5 ℃, 15 days frozen)

Appendix 2.2. Rheological properties analysis

In bar plots listed below, legends on x-axis describing each of the bars were named as follows:

A, B, C, D refers to the days of frozen storage for specified samples in the following order: 1 day, 15 days, 25 days and 35 days.

N: HA/No steam

S: SHS

0, 1, 2, 3, 4 refers to the freezing temperature for specified samples in the following order: -5 ℃,-15 ℃, -25 ℃, -35 ℃ and -45 ℃.

Appendix 2.3. Creep test

Creep test values for crumbs and crusts were shown in *Figure* A2-2, *Figure* A2-3, *Figure* A2-4 and *Figure* A2-5, respectively.

Negative correlation was found between viscoelastic properties and baking time ($p<0.01$) in crumbs. Frozen storage was not found to have significant correlations with viscoelasticity in samples. However, almost all significant difference were found within samples that were frozen under comparatively higher freezing rate, in either baking method. This might be deterioration in samples after prolonged frozen storage. As indicated by (Berglund, Shelton, & Freeman, 1991) (Varriano-Marston, Hse, & Mahdj,

1980). Faster freezing rates could have yielded some deterioration of the dough matrix by ice recrystallization and therefore altered the rheological and gas-retaining properties of gluten network, which directly was related with the viscoelastic properties.

Viscoelastic properties of crusts (35 days frozen) showed significant higher values, whereas no such difference was observed in HA baked sample crusts. Ice recrystallization, water redistribution from crumb to crust and case hardening might be the reason. During prolonged frozen storage, ice crystal melted and reformed, with the addition of water drew from gluten network, the total moisture redistribution from crumb to crust during cooling process caused the higher viscoelasticity in crust while lower viscoelasticity in crumb.

Likewise, individual difference should be considered because in additional studies, baked samples were not triplicated.

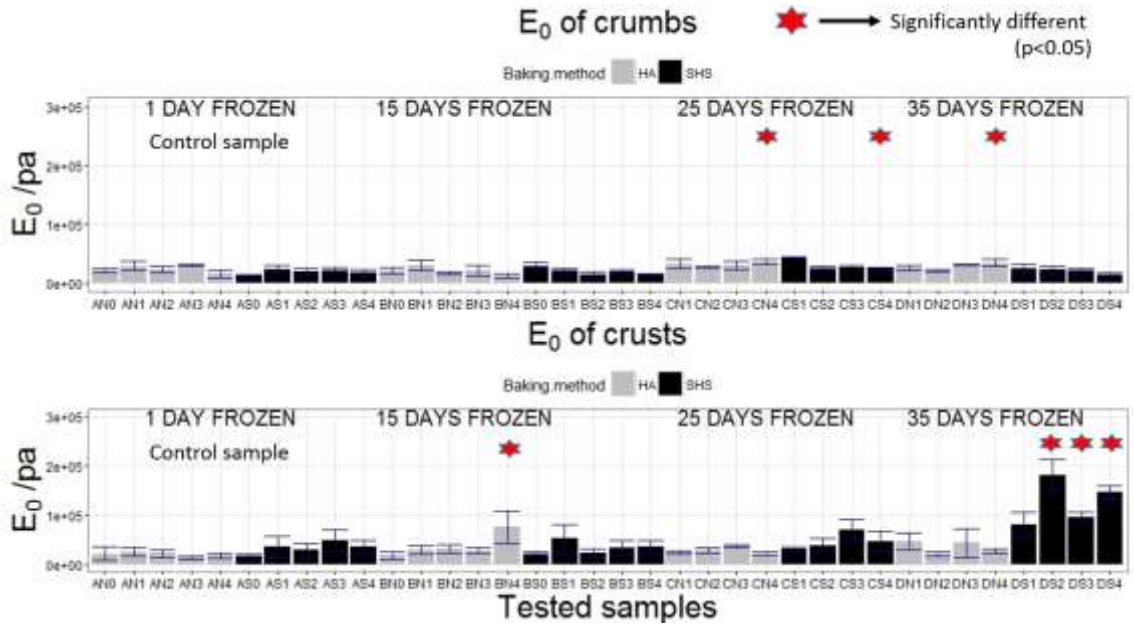


Figure A2- 2 Crumbs and crusts E_0 values baking by HA and SHS under various freezing treatments

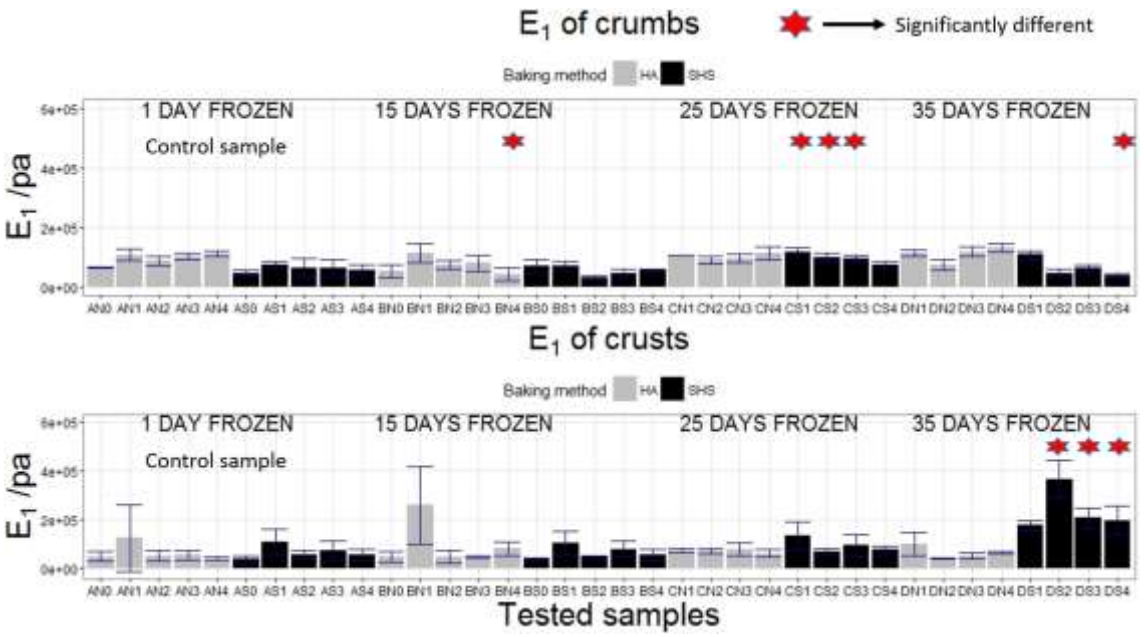


Figure A2- 3 Crumbs and crusts E_1 values baking by HA and SHS under various freezing treatments

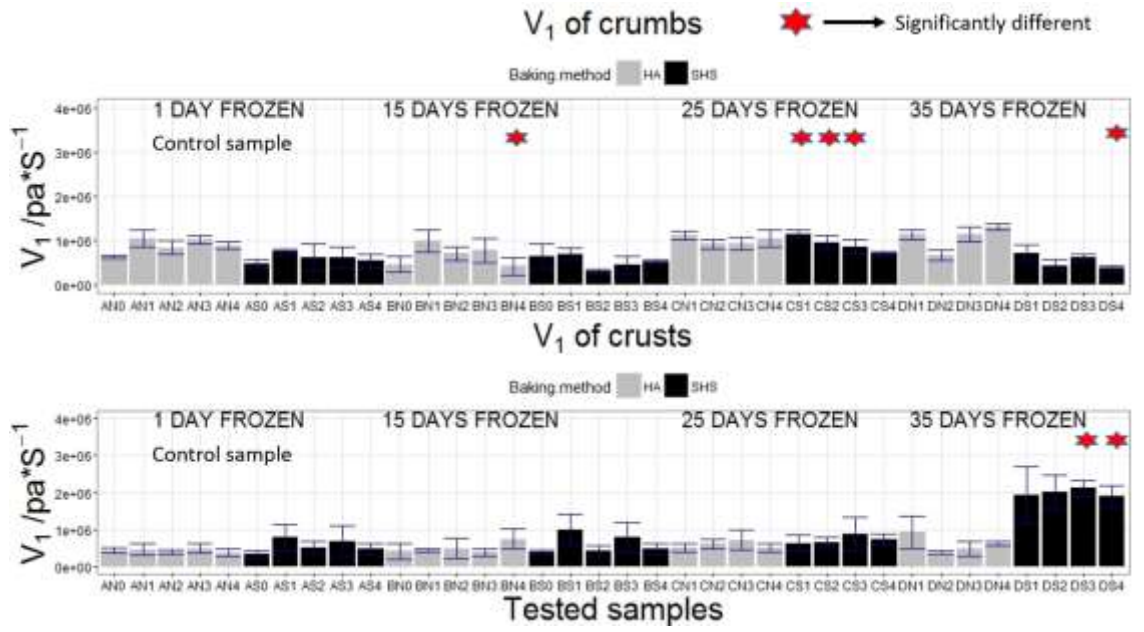


Figure A2- 4 Crumbs and crusts V_1 values baking by HA and SHS under various freezing treatments

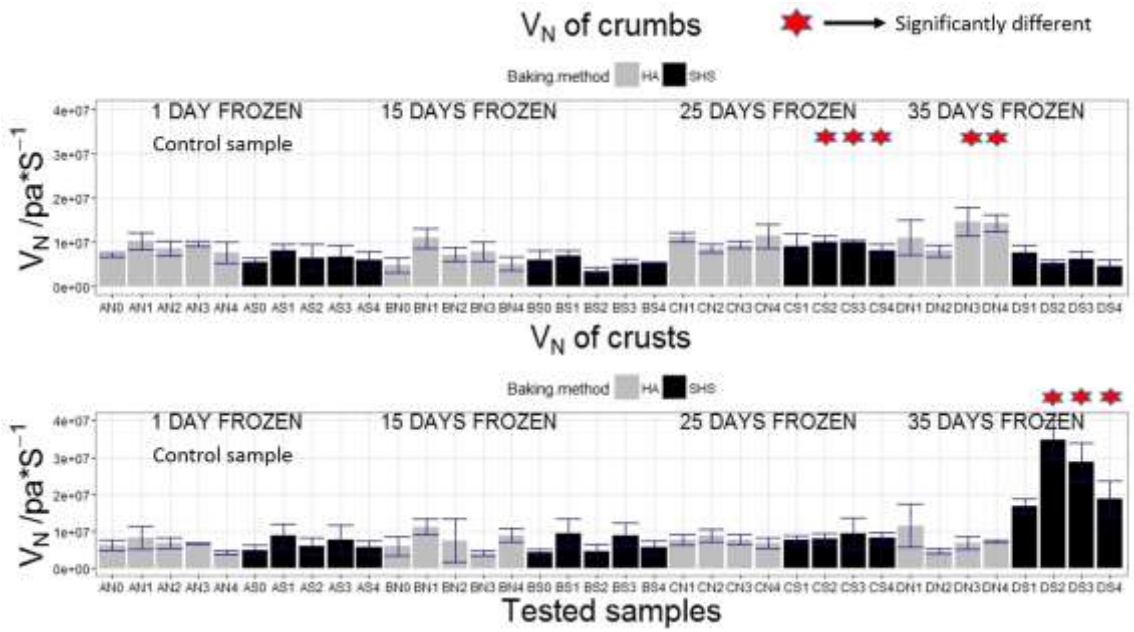


Figure A2- 5 Crumbs and crusts V_N values baking by HA and SHS under various freezing treatments

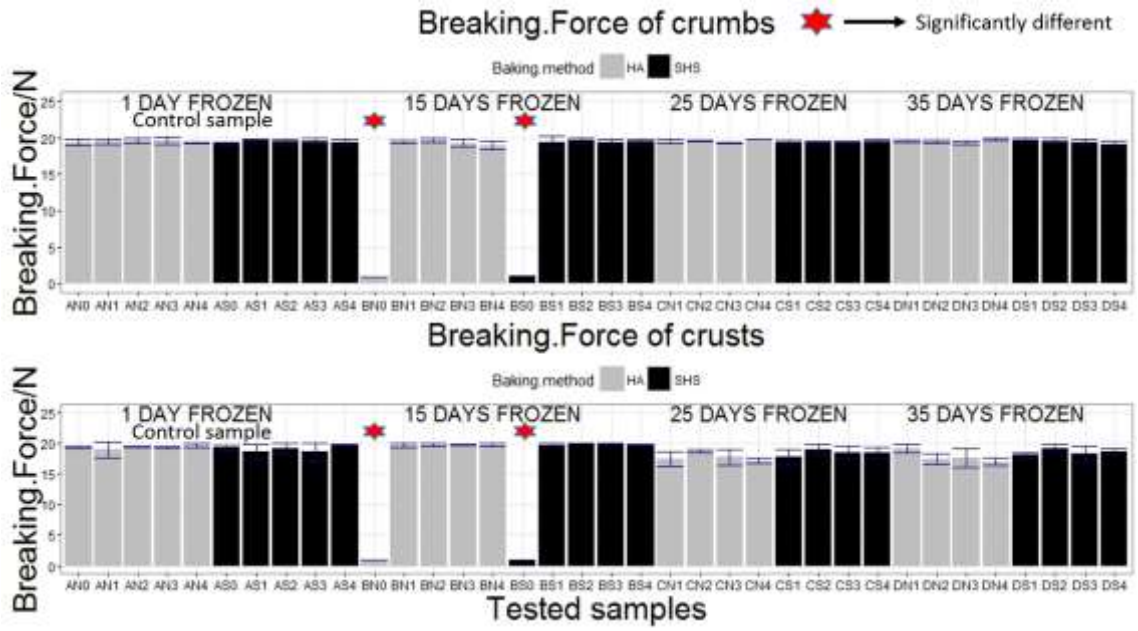


Figure A2- 6 Crumbs and crusts breaking force values baking by HA and SHS under various freezing treatments

Almost no significant difference was found under various treatments, except for samples frozen under -5 ℃, 15 days frozen storage, as shown in *Figure* A2-6. Breaking points were found in both crumbs and crusts in either baking method, which gave the distinctly lower breaking force compared with others, see *Figure* A2-7.

A crispy texture is associated with low moisture content and water activity, when starch and gluten matrix are in a glassy state making cells walls more prone to fracture (Stroke & Donald, 2000). This result further proved that fractural test might not be an appropriate way on testing pizza dough due to the wet condition of samples in the previous research, since only samples that were in a near fully dried condition were detected with breaking points.

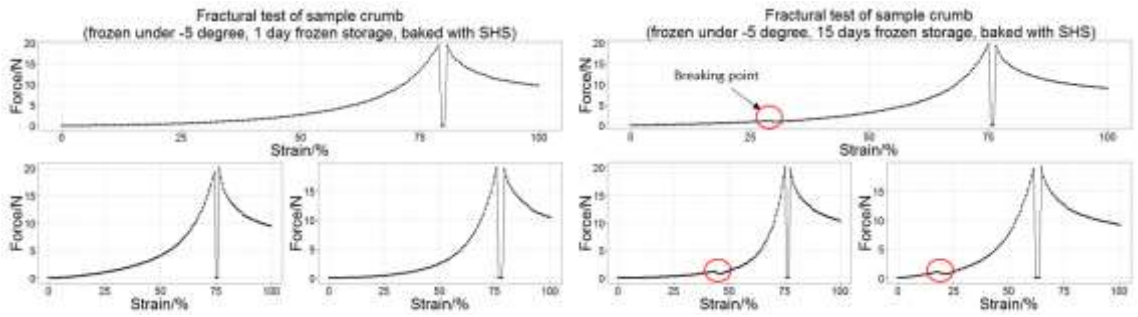


Figure A2- 7 Fractural curve comparison (samples frozen under -5 ℃, 1 day and 15 days frozen, SHS baked)

Appendix 2.4. TPA test (hardness)

Significantly ($p<0.05$) increased hardness in sample crumbs was found in samples frozen under higher freezing rates (-35 ℃ and -45 ℃) during prolonged frozen storage, in either baking method, as shown in *Figure* A2-8. The increase of dough hardness along with prolonged frozen storage was also found in other researches (Salas-Mellado & Chang, 2003) (Yi & Kerr, 2009) (Berglund, Shelton, & Freeman, 1991). Amylose

recrystallization plays an important role in the initial crumb hardness and in the first stages of staling (Hug-Iten, Escher, & Conde-Petit, 2003) (Barcenas & Rosell, 2006). Microscopic studies showed that there is a phase separation between amylose and amylopectin during baking, which leads to an accumulation of amylose inside the starch granules (Hug-Iten, Handschin, Conde-petit, & Escher, 1999). Along with prolonged frozen storage, ice crystallization causes damage to the starch granule, which allows the leaching of intracellular amylose, increasing the interaction between the intra- and inter-granular amylose and the formation of a network of amylose that increases crumb hardness. Almost no significant difference found in the hardness comparison between HA and SHS baked samples.

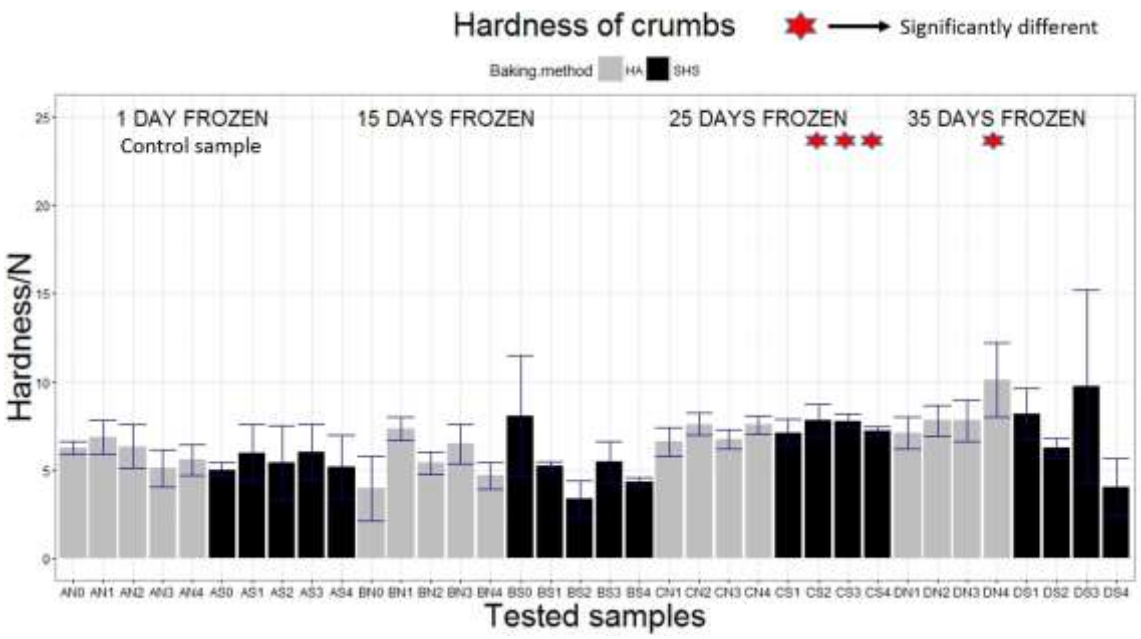


Figure A2- 8 Crumbs and crusts hardness values baking by HA and SHS under various freezing treatments

Appendix 2.5. Assumption from additional studies

Similar results were obtained in the additional studies with some previous researches. Along with prolonged frozen duration, samples frozen under higher freezing rates seemed to have increased viscoelasticity especially in the sample crusts baked by SHS. Combined effect of ice recrystallization, case hardening and water redistribution might be the reason that differentiate the two baking methods. Fractural test might not be appropriate for the analysis of samples at the breaking point for the materials used in this research. Hardness of samples increased with longer frozen storage. Using SHS did not make significant difference on the hardness of samples compared with HA method.

Appendix 3. Samples frozen under -5 ℃ (in which super cooling happened)

Samples frozen under -5 ℃ were discussed separately because super cooling phenomenon was observed.

Appendix 3.1. Freezing rate

As shown in *Table* 3-1, samples frozen under -5 ℃ was found to have the highest freezing rate compared with other groups of samples. In spite of the second law of thermodynamics, crystallization of pure liquids usually begins at a lower temperature than the melting point, due to high activation energy of homogeneous nucleation. The creation of a nucleus implies the formation of an interface at the boundaries of the new phase. Some energy is expended to form this interface, based on the surface energy of each phase. If a hypothetical nucleus is too small, the energy that would be released by forming its volume is not enough to create its surface, and nucleation does not proceed. Freezing does not start until the temperature is low enough to provide enough energy to form stable nuclei. In presence of irregularities on the surface of the containing vessel, solid or gaseous impurities, pre-formed solid crystals, or other nucleators, heterogeneous nucleation may occur, where some energy is released by the partial destruction of the previous interface, raising the super cooling point to be near or equal to the melting point. The melting point of water at 1 atmosphere of pressure is very close to 0 °C (32 °F, 273.15 K), and in the presence of nucleating substances the freezing point of water is close to the melting point, but in the absence of nucleators water can super cool to −40 °C (−40 °F, 233 K) before freezing (Lundheim, 2002) (Franks, 2003).

Appendix 3.2. Moisture content after freezing

Samples frozen under -5 ℃ had the lowest moisture content (64.7±0.4%) compared with other groups of samples, as shown in *Table* 3-2. This was due to the longer total freezing time than groups of samples.

Appendix 3.3. Thawing rate and baking time

Thawing rate and baking time were both significantly lower or less than samples frozen under -45 ℃. This indicated that the difference in starting temperature or the difference in sensible heat the baking period contributed more than different freezing rate.

Appendix 3.4. Drying rate

Although weight loss of samples before and after baking was not measured over three times, based on the results, it is still assumed that less baking time caused the lower drying rate of samples frozen under -5 ℃.

Appendix 3.5. Colour results

In spite of less baking time, HA baked samples showed no significant difference in L, a, b values compared with other groups of samples. However, in SHS baked samples, the comparatively baking time must have conferred more influence on samples colour values because samples frozen under -5 ℃ were less browned compared with other groups of samples (see *Table* 3-9, *Table* 3-10, *Table* 3-11 and *Table* 3-12). The severity of Maillard reaction could be considered to be much more in SHS environment than in HA.

Appendix 3.6. Rheological tests results

In either baking method, crumbs and crusts of samples frozen under -5 ℃ showed almost no significant difference in viscoelasticity with samples frozen under -35 ℃ or -45 ℃, whereas significant difference was found within the comparison between super cooled samples and samples frozen under -15 ℃.

This results suggested that rheological properties for super cooled samples were similar to those frozen under conventionally lower freezing temperatures. This might be due to their comparatively similar microstructure or ice crystal size (Kobayashi, Kimizuka, & Watanabe, 2015).

No significant difference was found in fractural test or TPA test between super cooled samples and other groups of samples.

Appendix 3.7. Conclusion

Although the freezing temperature was lowest (-5 ℃) of all in this research, super cooling rendered the batch of samples highest freezing rate and possibly small and homogenous fine ice crystals, which differed these samples significantly different from samples those were frozen under high freezing temperature, especially in rheological properties. Micro-structure of samples frozen under -5 ℃ should be the reason why they were differentiated in this research from other samples. Further observation of these micro-structure is necessary.

Appendix 4. R programming code examples

Appendix 4.1. Personal defined settings

N: HA/No steam

S: SHS

5, 15, 25, 35, 45: Specified samples frozen under -5, -15, -25, -35, -45 ℃, respectively

Crumb: Crumbs of samples

A: Data analysis

Appendix 4.2. 1 day samples ANOVA and TukeyHSD tests codes (comparison examples of CIE Lab L values, samples frozen under difference freezing rate)

```
setwd("C:/Users/LINGKE/Desktop/R/Research/1 day samples")

N5crumb1 <- read.csv("N5crumb1A.csv")

N15crumb1 <- read.csv("N15crumb1A.csv")

N25crumb1 <- read.csv("N25crumb1A.csv")

N35crumb1 <- read.csv("N35crumb1A.csv")

N45crumb1 <- read.csv("N45crumb1A.csv")

S5crumb1 <- read.csv("S5crumb1A.csv")

S15crumb1 <- read.csv("S15crumb1A.csv")

S25crumb1 <- read.csv("S25crumb1A.csv")

S35crumb1 <- read.csv("S35crumb1A.csv")

S45crumb1 <- read.csv("S45crumb1A.csv")

N5crumb1.L <- N5crumb1$L


N15crumb1.L <- N15crumb1$L


N25crumb1.L <- N25crumb1$L


N35crumb1.L <- N35crumb1$L


N45crumb1.L <- N45crumb1$L
```

```
S5crumb1.L <- S5crumb1$L

S15crumb1.L <- S15crumb1$L

S25crumb1.L <- S25crumb1$L

S35crumb1.L <- S35crumb1$L

S45crumb1.L <- S45crumb1$L

df.Ncrumb.L<-

data.frame(N5crumb1.L,N15crumb1.L,N25crumb1.L,N35crumb1.L,N45crumb1.L)

df.Ncrumb.L <- stack(df.Ncrumb.L)

Anova.Ncrumb.L <- aov(df.Ncrumb.L$values~df.Ncrumb.L$ind)

summary(Anova.Ncrumb.L)

TukeyHSD(Anova.Ncrumb.L)

df.Scrumb.L<-

data.frame(S5crumb1.L,S15crumb1.L,S25crumb1.L,S35crumb1.L,S45crumb1.L)

df.Scrumb.L <- stack(df.Scrumb.L)

Anova.Scrumb.L <- aov(df.Scrumb.L$values~df.Scrumb.L$ind)

summary(Anova.Scrumb.L)

TukeyHSD(Anova.Scrumb.L)
```

Appendix 4.3. 1 day frozen samples Student t test (comparison examples between samples by HA and SHS)

```
t.test(N5crumb1.L,S5crumb1.L)
t.test(N15crumb1.L,S15crumb1.L)
t.test(N25crumb1.L,S25crumb1.L)
t.test(N35crumb1.L,S35crumb1.L)
t.test(N45crumb1.L,S45crumb1.L)
```

Appendix 4.4. 1 day samples cross-correlation (package "corrplot" required)

```
setwd("C:/Users/LINGKE/Desktop/R/Research/1 day samples")
require(corrplot)
Ncrumb.correlation.coefficient <- read.csv("1 day HA crumb.csv")
Ncrust.correlation.coefficient <- read.csv("1 day HA crust.csv")
Scrumb.correlation.coefficient <- read.csv("1 day SHS crumb.csv")
Scrust.correlation.coefficient <- read.csv("1 day SHS crust.csv")
 cor.mtest <- function(mat, conf.level = 0.95){
   mat <- as.matrix(mat)
   n <- ncol(mat)
   p.mat <- lowCI.mat <- uppCI.mat <- matrix(NA, n, n)
   diag(p.mat) <- 0
   diag(lowCI.mat) <- diag(uppCI.mat) <- 1
   for(i in 1:(n-1)){
     for(j in (i+1):n){
       tmp <- cor.test(mat[,i], mat[,j], conf.level = conf.level)
       p.mat[i,j] <- p.mat[j,i] <- tmp$p.value
       lowCI.mat[i,j] <- lowCI.mat[j,i] <- tmp$conf.int[1]
       uppCI.mat[i,j] <- uppCI.mat[j,i] <- tmp$conf.int[2]
     }
   }
   return(list(p.mat, lowCI.mat, uppCI.mat))
 }
```

```
res1 <- cor.mtest(Ncrumb.correlation.coefficient,0.95)

res2 <- cor.mtest(Ncrumb.correlation.coefficient,0.99)

p.Ncrumb.correlation.coefficient <- cor(Ncrumb.correlation.coefficient)

corrplot(p.Ncrumb.correlation.coefficient,addrect = 2,title = "Ncrumb coefficient correlation",method="number" ,mar = c(1,1,1,1))

p.Ncrust.correlation.coefficient <- cor(Ncrust.correlation.coefficient)

corrplot(p.Ncrust.correlation.coefficient,method = "number",addrect = 2,title = "Ncrust coefficient correlation",mar = c(1,1,1,1))

p.Scrumb.correlation.coefficient <- cor(Scrumb.correlation.coefficient)

corrplot(p.Scrumb.correlation.coefficient,method = "number",addrect = 2,title = "Scrumb coefficient correlation",mar = c(1,1,1,1))

p.Scrust.correlation.coefficient <- cor(Scrust.correlation.coefficient)

corrplot(p.Scrust.correlation.coefficient,method = "number",addrect = 2,title = "Scrust coefficient correlation",mar = c(1,1,1,1))
```

Appendix 4.5. Centre temperature curve of samples during freezing (package "ggplot2", "grid", "gridExtra", "lattice", "reshape2" required)

```
require(ggplot2)

require(grid)

require(gridExtra)

require(lattice)

require(reshape2)

getwd()

setwd("C:/Users/LINGKE/Desktop/R/Research")

sampleholder.freezing.curve <- read.csv("Freezing curve generalisation with sample

holder.csv")
```

```
time.sampleholder <- sampleholder.freezing.curve$Time

STM5 <- sampleholder.freezing.curve$Minus5

STM15 <- sampleholder.freezing.curve$Minus15

STM25 <- sampleholder.freezing.curve$Minus25

STM35 <- sampleholder.freezing.curve$Minus35

STM45 <- sampleholder.freezing.curve$Minus45

Sdata <- data.frame(time.sampleholder,STM5,STM15,STM25,STM35,STM45)

Sdata <- melt(Sdata,id.vars = "time.sampleholder", value.name = "Temperature")

Sdata

plot.sampleholder <- ggplot(Sdata,aes(x=time.sampleholder,y=Temperature,
color=variable))+geom_line()+geom_point()+theme_bw()+ggtitle("Sample centre
freezing curve with sample holder")+theme(plot.title=element_text(size =
20))+xlab("Time/min")+ylab("Temperature/celsius
degree")+scale_x_continuous(breaks = round(seq(min(Sdata$time.sampleholder),
max(Sdata$time.sampleholder), by = 10),1)) +scale_y_continuous(breaks =
round(seq(min(Sdata$Temperature), max(Sdata$Temperature), by =
5),1))+annotate("text",x=150,y=-11,label="Freezing time=36 min\nFreezing rate=0.14
celsius degree/min",size=5)+annotate("text",x=150,y=-23,label="Freezing time=18
min\nFreezing rate=0.28 celsius degree/min",size=5)+annotate("text",x=150,y=-
33,label="Freezing time=10 min\nFreezing rate=0.49 celsius
degree/min",size=5)+annotate("text",x=150,y=-43,label="Freezing rate=0.50 celsius
```

```
degree/min",size=5)+annotate("text",x=150,y=-2,label="Freezing time=2
min\nFreezing rate=0.95 celsius
degree/min",size=5)+theme(legend.text=element_text(size=15))+annotate("text",x=95,y
=5,label="where ice crystalling happens(0~-5 celsius
degree)",size=5)+geom_segment(col="black",x=70,y=4.7,xend=60,yend=0,arrow =
arrow(length =unit(0.5,"cm")))+geom_segment(col="black",x=135,y=-
6,xend=87,yend=-3.9,arrow = arrow(length =
unit(0.5,"cm")))+annotate("text",x=150,y=-6,label="super
cooling",size=5)+theme(text=element_text(size = 20))

plot.sampleholder <- plot.sampleholder+scale_x_continuous(breaks =
round(seq(min(Sdata$time.sampleholder), max(Sdata$time.sampleholder), by = 10),1))
+scale_y_continuous(breaks = round(seq(min(Sdata$Temperature),
max(Sdata$Temperature), by = 5),1))

plot.sampleholder
```

Appendix 4.6. Baking characteristics of samples by SHS (package "ggplot2", "grid", "gridExtra", "lattice", "reshape2" required)

```
require(ggplot2)

require(grid)

require(gridExtra)

require(lattice)

require(reshape2)
```

```
getwd()

setwd("C:/Users/LINGKE/DESKTOP/R/Research")

S1curve <- read.csv("S1baking performance.csv")

S1curve.samples <- read.csv("S1 baking performance samples.csv")

S1curve

str(S1curve)

xS1 <- S1curve$Time.S1.

xS1 <- round(xS1*(1/60),2)

xS1

xS2 <- S1curve.samples$Time.S2.

xS2 <- round(xS2*(1/60),2)

xS2

S1TM45 <- S1curve.samples$S1TM45

S1TM35 <- S1curve.samples$S1TM35

S1TM25 <- S1curve.samples$S1TM25

S1TM15 <- S1curve.samples$S1TM15

S1TM5 <- S1curve.samples$S1TM5

S1WM45 <- S1curve$S1WM45

S1WM35 <- S1curve$S1WM35
```

```
S1WM25 <- S1curve$S1WM25

S1WM15 <- S1curve$S1WM15

S1WM5 <- S1curve$S1WM5

S.Upper <- S1curve$Upper.heater.S.

S.Air <- S1curve$Surrounding.air.S.

S.Lower <- S1curve$Lower.heater.S.

S1df1 <- data.frame(xS1,S.Upper,S.Air,S.Lower)

S1df2 <- data.frame(xS2,S1TM45,S1TM35,S1TM25,S1TM15,S1TM5)

S1df3 <- data.frame(xS1,S1WM45,S1WM35,S1WM25,S1WM15,S1WM5)

S1dfa <- melt(data=S1df1,id.vars="xS1",value.name = "Heater.T")

S1dfb <- melt(data=S1df2,id.vars="xS2",value.name = "Sample.T")

S1dfc <- melt(data=S1df3,id.vars="xS1",value.name = "Sample.W")

WS1 <- qplot(x=xS1,y=Sample.W,data=S1dfc,col=variable,xlab="",ylab = "Sample
weight/g",main="Baking performance of samples under 1 day frozen storage by SHS")

WS1 <- WS1+theme_bw()+theme(plot.margin=unit(c(0.7,-
0.5,0,0.4),"cm"))+theme(axis.title.y=element_text(size=15))+theme(axis.title.x=element
_text(size=15))+geom_line()+geom_point()+theme(axis.text.x=element_blank())+geom
_point()+theme(plot.title=element_text(size=20))+theme(text=element_text(size=20))

PS1 <- qplot(x=xS1,y=Heater.T,data=S1dfa,col=variable,geom = "line",xlab = "",ylab =
"Heater T/C")
```

```
PS1 <-
PS1+theme_bw()+theme(axis.text.x=element_blank())+theme(axis.title.y=element_text
(size=15))+geom_point()+theme(plot.title=element_text(size=30))+theme(plot.margin=
unit(c(-0.9,-0.2,0,0.2),"cm"))+theme(text=element_text(size=20))

TS1 <- qplot(x=xS2,y=Sample.T,data=S1dfb,col=variable,xlab="Time/min",ylab =
"Sample T/C")

TS1 <- TS1+theme_bw()+theme(plot.margin=unit(c(-0.9,-
0.3,0,0.2),"cm"))+theme(axis.title.y=element_text(size=15))+theme(axis.title.x=element
_text(size=15))+geom_line()+geom_point()+theme(text=element_text(size=20))

+scale_x_continuous(limits = c(0,15))

grid.arrange(WS1,PS1,TS1)
```

Appendix 4.7. 3D scatterplot of CIE Lab colour space (package "scatterplot3d" required)

```
require("scatterplot3d")

getwd()

setwd("C:/Users/LINGKE/DESKTOP/R/Research")

Ncolor <- read.csv("Ncolor.csv")

LN <- Ncolor$Delta.L

aN <- Ncolor$Delta.a

bN <- Ncolor$Delta.b
```

```
Scolor <- read.csv("Scolor.csv")

LS <- Scolor$Delta.L

aS <- Scolor$Delta.a

bS <- Scolor$Delta.b

xlimN <- range(0,50)

ylimN <- range(0,25)

zlimN <- range(40,LN)

zlimS <- range(40,LS)

par(mfrow=c(1,2))

scatterplot3d(bN,aN,LN,pch=19,highlight.3d = FALSE,xlim=xlimN,ylim = ylimN,zlim

= zlimN,main = "HA baked sample \ncolor distribution",cex.axis = 1.5)

scatterplot3d(bS,aS,LS,pch=19,highlight.3d = FALSE,xlim=xlimN,ylim = ylimN,zlim

= zlimN,main = "SHS baked sample \ncolor distribution",cex.axis = 1.5)
```

Appendix 4.8. Rheological properties graphics (instantaneous elasticity/$E_{0,}$ package "ggplot2", "grid", "gridExtra", :lattice", "reshape2", "matrixStats" required)

```
require(ggplot2)

require(grid)

require(gridExtra)

require(lattice)
```

```
require(reshape2)

require(matrixStats)

setwd("C:/Users/LINGKE/Desktop/R/Research/1 day samples")

N5crumb1 <- read.csv("N5crumb1A.csv")

N15crumb1 <- read.csv("N15crumb1A.csv")

N25crumb1 <- read.csv("N25crumb1A.csv")

N35crumb1 <- read.csv("N35crumb1A.csv")

N45crumb1 <- read.csv("N45crumb1A.csv")

S5crumb1 <- read.csv("S5crumb1A.csv")

S15crumb1 <- read.csv("S15crumb1A.csv")

S25crumb1 <- read.csv("S25crumb1A.csv")

S35crumb1 <- read.csv("S35crumb1A.csv")

S45crumb1 <- read.csv("S45crumb1A.csv")

N5crust1 <- read.csv("N5crust1A.csv")

N15crust1 <- read.csv("N15crust1A.csv")

N25crust1 <- read.csv("N25crust1A.csv")

N35crust1 <- read.csv("N35crust1A.csv")

N45crust1 <- read.csv("N45crust1A.csv")
```

```
S5crust1 <- read.csv("S5crust1A.csv")

S15crust1 <- read.csv("S15crust1A.csv")

S25crust1 <- read.csv("S25crust1A.csv")

S35crust1 <- read.csv("S35crust1A.csv")

S45crust1 <- read.csv("S45crust1A.csv")

N5crumb1.E0 <- N5crumb1$E0

N15crumb1.E0 <- N15crumb1$E0

N25crumb1.E0 <- N25crumb1$E0

N35crumb1.E0 <- N35crumb1$E0

N45crumb1.E0 <- N45crumb1$E0

S5crumb1.E0 <- S5crumb1$E0

S15crumb1.E0 <- S15crumb1$E0

S25crumb1.E0 <- S25crumb1$E0

S35crumb1.E0 <- S35crumb1$E0

S45crumb1.E0 <- S45crumb1$E0

N5crust1.E0 <- N5crust1$E0

N15crust1.E0 <- N15crust1$E0

N25crust1.E0 <- N25crust1$E0

N35crust1.E0 <- N35crust1$E0
```

```
N45crust1.E0 <- N45crust1$E0

S5crust1.E0 <- S5crust1$E0

S15crust1.E0 <- S15crust1$E0

S25crust1.E0 <- S25crust1$E0

S35crust1.E0 <- S35crust1$E0

S45crust1.E0 <- S45crust1$E0

df.crumb.E0 <-
data.frame(N5crumb1.E0,S5crumb1.E0,N15crumb1.E0,S15crumb1.E0,N25crumb1.E0,
S25crumb1.E0,N35crumb1.E0,S35crumb1.E0,N45crumb1.E0,S45crumb1.E0)

mean.df.crumb.E0 <- colMeans(df.crumb.E0)

mean.df.crumb.E0 <- as.matrix(mean.df.crumb.E0)

sd.df.crumb.E0 <- as.matrix(df.crumb.E0)

sd.df.crumb.E0 <- colSds(sd.df.crumb.E0)

column.crumb.E0 <- c("N0","S0","N1","S1","N2","S2","N3","S3","N4","S4")

Baking.method <- gl(2,1,10,labels = c("HA","SHS"))

d.crumb.E0 <-
data.frame(column.crumb.E0,Baking.method,mean.df.crumb.E0,sd.df.crumb.E0)

d.crumb.E0
```

```
p.crumb.E0 <-
ggplot(d.crumb.E0,aes(x=column.crumb.E0,y=mean.df.crumb.E0,fill=Baking.method))
+
  geom_bar(stat = "identity",position = position_dodge(1))+
  theme_bw()+
  ylab(expression(paste(E[0], " /pa")))+
  ggtitle(expression(paste(E[0], " of crumbs")))+
  theme(axis.title.x=element_text(size=30))+
  theme(axis.title.y=element_text(size=30))+
  theme(plot.title=element_text(size=30))+
  scale_fill_manual(values=c("grey","black"))+
  geom_errorbar(aes(ymin=mean.df.crumb.E0-sd.df.crumb.E0,
ymax=mean.df.crumb.E0+sd.df.crumb.E0),position=position_dodge(width=.9),
color="darkblue")+theme(legend.position="top")+annotate("text",x=6,y=85000,label="
1 day frozen
",size=7)+theme(axis.title.x=element_blank())+theme(text=element_text(size=20))
print(p.crumb.E0)
```

```
df.crust.E0 <-
data.frame(N5crust1.E0,S5crust1.E0,N15crust1.E0,S15crust1.E0,N25crust1.E0,S25crus
t1.E0,N35crust1.E0,S35crust1.E0,N45crust1.E0,S45crust1.E0)
mean.df.crust.E0 <- colMeans(df.crust.E0)
mean.df.crust.E0 <- as.matrix(mean.df.crust.E0)
sd.df.crust.E0 <- as.matrix(df.crust.E0)
sd.df.crust.E0 <- colSds(sd.df.crust.E0)
column.crust.E0 <- c("N0","S0","N1","S1","N2","S2","N3","S3","N4","S4")
Baking.method <- gl(2,1,10,labels = c("HA","SHS"))
d.crust.E0 <-
data.frame(column.crust.E0,Baking.method,mean.df.crust.E0,sd.df.crust.E0)
p.crust.E0 <-
ggplot(d.crust.E0,aes(x=column.crust.E0,y=mean.df.crust.E0,fill=Baking.method))+
  geom_bar(stat = "identity",position = position_dodge(1))+
  theme_bw()+xlab("Tested samples")+
  ylab(expression(paste(E[0], " /pa")))+
  ggtitle(expression(paste(E[0], " of crusts")))+
  theme(axis.title.x=element_text(size=30))+
  theme(axis.title.y=element_text(size=30))+
```

```
  theme(plot.title=element_text(size=30))+

  scale_fill_manual(values=c("grey","black"))+

  geom_errorbar(aes(ymin=mean.df.crust.E0-sd.df.crust.E0,
ymax=mean.df.crust.E0+sd.df.crust.E0),position=position_dodge(width=.9),
color="darkblue")+theme(legend.position="top")+annotate("text",x=6,y=85000,label="
1 day frozen ",size=7)+theme(text=element_text(size=20))

print(p.crust.E0)

grid.arrange(p.crumb.E0,p.crust.E0)
```