

**DEVELOPMENT OF NOVEL PET FOOD
INVOLVING DRYING STEP**

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**by
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ABSTRACT

DEVELOPMENT OF NOVEL PET FOOD INVOLVING DRYING STEP

Most of the pet parents prefer dry petfood since it is easy to portion, has longer open-shelf life and doesn't have an unappealing smell compared to wet petfood. However, wet petfood more closely resembles the fresh meat that pets crave. It helps maintaining hydration and digestion since it includes a high water content. In the petfood industry, it is an open new area to produce dry petfood that can be easily rehydrated and turned into a wet product to provide the benefits of both dry and wet petfood. Considering current food products in the market, a pasta production line can be shown as a good example of manufacturing such products. It consists of dry powder and wet material. Having this analogy in mind, a novel textured wet petfood development was investigated. A Microwave vacuum dryer is used as a novel drying technology in petfood industry. In laboratory trials, die plates of different sizes were tested and the effect of surface area on rehydration was investigated. In pilot plant trials, different raw materials were tested and their effect on the process and on the rehydration time were evaluated. Results showed that the increase in the surface area had a significant effect on the rehydration time. Also, it was observed that the use of blood plasma powder affected the formulation positively and the rehydration negatively. The products made by using poultry meal which were cooled twice after cooking, showed the highest efficiency by causing the least formation of fines during cutting.

ÖZET

KURUTMA İŞLEMİ İÇEREN YENİ EVCİL HAYVAN MAMALARININ GELİŞTİRİLMESİ

Evcil hayvan sahiplerinin çoğu, porsiyonlanması kolay, açık raf ömrü daha uzun ve ıslak mamaya göre hoş olmayan bir kokuya sahip olmadığı için kuru mama satın almayı tercih etmektedir. Bununla birlikte, yaş evcil hayvan maması, evcil hayvanların can attığı taze ete daha çok benzemektedir. Kuru gıdalara kıyasla yüksek su içerdiğinden hidrasyonun ve sindirimin korunmasına yardımcı olur. Mama endüstrisinde, hem kuru hem de yaş mamanın faydalarını içermesi için kolayca rehidre edilebilen ve yaş ürüne dönüştürülebilen kuru mama üretimi yeni bir açık alandır. Piyasadaki gıda ürünlerine bakıldığında makarna üretim hattı bu tür ürünlere güzel bir örnek olarak gösterilebilir. Ağırıklı olarak tambur irmiği ve su olmak üzere kuru toz ve yaş malzemeden oluşmaktadır. Bu benzetme akılda tutularak, kuru proteinlerden ve et ürünlerinden yeni bir yaş evcil hayvan maması gelişimi araştırıldı. Mikrodalga vakumlu kurutucu, yeni bir kurutma teknolojisi olarak kullanılmaktadır. Laboratuvar denemelerinde farklı boyutlarda kalıplar denenmiş ve yüzey alanının rehidrasyona etkisi araştırılmıştır. Pilot tesis denemelerinde farklı hammaddeler denenmiş ve bunların proses ve rehidrasyon süresi üzerindeki etkileri değerlendirilmiştir. Sonuçlar, yüzey alanındaki artışın rehidrasyon süresi üzerinde önemli bir etkiye sahip olduğunu göstermiştir. Ayrıca pilot fabrikada yapılan deneylerde kan plazması kullanımının formülasyonu olumlu, rehidrasyonu olumsuz etkilediği gözlemlendi. Farklı malzemelerin kullanılması rehidrasyon süresinde önemli bir değişikliğe neden olmamasına rağmen, pişirme sonrası iki kez soğutulan kanatlı unu kullanılarak yapılan ürünler en az ürün kaybına neden olarak en yüksek verimi göstermiştir.

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LIST OF ABBREVIATIONS

BG - Beef greaves (85%) powder

BP – Blood plasma powder

w/o BP – Without blood plasma powder

CL – Chicken liver frozen

PG – Poultry greaves powder

PM – Poultry meal powder

MVD – Microwave Vacuum Dryer

CHAPTER 1

INTRODUCTION

In this section, current products in the pet food industry and the reasons for developing a new product are explained. Then, correlation between pasta process and the novel pet food product is explained by giving information about drying and rehydration process in foods.

1.1. Problem Statement

Water intake is a pivotal concern for cats. It is recommended that a cat requires about 50 mL of water per kg of bodyweight daily, so this translates into 200-250 mL per day for a cat weighing 4-5 kg. This water requirement can be covered by free water from drinking and eating, or from metabolic water. Metabolic water is generated from the oxidation of macronutrients (fats, carbohydrates, and protein). The requirement of fluid for cats can be provided with wet petfood that has an 80% water content (at an average requirement of 250-300 g) (Handl & Fritz, 2018). Many of the cat owners rely on dry petfood as their pets primary source of nutrition since it is the easiest to feed and to manage portion size (Schleicher et al., 2019). The major concern with feeding dry-only diets is the lack of moisture. Dry cat food typically contains less than 10% water, compared to at least 80% water content in wet food. Dry petfood also contains high amounts of carbohydrates, which is unnatural in a cat diet. There are concerns from veterinarians that feeding a dry-only diet can lead to chronic dehydration and health issues, especially obesity (Bermingham et al., 2011; Pierson, 2013). Pet owners generally prefer to add water on dry food to supply the required daily intake of water for cats since they do not prefer wet food because of product stickiness and its intense smell (Schleicher et al., 2019). Some veterinarians suggest that feeding cats with wet food ensures that they are obtaining the required water intake to promote good health and prevent urinary tract

issues. ((NRC), 2006; Pierson, 2013). In addition to that, feeding cats with wet food may support maintaining a healthy bodyweight as compared to feeding dry or freeze-dried foods. Cats also showed a preference for wet food compared to the other petfood products. (Wei et al., 2011). In order to combine the benefits of both dry and wet petfood, this thesis investigated a novel dry petfood product that can be easily rehydrated.

1.2. Objective of the study

The aim of this thesis study is to develop a novel production process for a wet proposition to shape, de- and rehydrate meat analogs while ensuring product attributes such as nutrition, palatability and shelf life. A microwave vacuum dryer is investigated as novel drying process for petfood industry. The shape of the product is one of the important parameters to manage drying and rehydration time of the chunks. To decrease required time for the process, effects of shape of product on processing time are also investigated.

1.3. State of Art

1.3.1. Pasta Production

Pasta is an ancient food which could be defined as a type of dough extruded or stamped into various shapes for cooking. It is economical, easy to prepare, has a longer shelf life and is consumed all over the world in many different ways (Sanni et al., 2007). The main ingredients of pasta are durum wheat semolina and water. 18–25% water is added to dry raw durum semolina and mixed for 10–20 min to produce a dough with an average moisture content of 30–32% (Wrigley, 2004). After the dough is mixed, it is transferred to an extruder. In this process, extruder forces the dough through the die, also it kneads the dough into a homogeneous mass, controls the rate of production, and affects the overall quality of the end product (Pagani et al., 1989) . For forming or extrusion-pressing the dough, three basic approaches are possible (Dawa, 2001):

- (1) Small scale, batch mixers and extruders.
- (2) A continuous process with screw presses. Here, no preliminary kneading of the material into a homogeneous dough takes place. A crumbly dough mass is produced in troughs equipped with mixing paddles. This mass is then slowly conveyed by screws to the press head. This homogenization of the crumbly dough is achieved by the high pressures of about 80 to 120 bar in the extrusion screw chamber before the die.
- (3) Continuous mixing/kneading process by means of co-rotating twin screws. Dry raw materials and the added liquid ingredients are mixed and kneaded into a homogeneous dough. The homogenized, kneaded and moist plastic dough is extruded through the die in a continuous stream. At the discharge of the die, a blower immediately dries the surface of the dough strands in order to eliminate their stickiness. Just below the die there may be rotating knives which cut the preshaped strands to the desired length.

In pasta production, bronze dies are traditionally used. However, dies made of Teflon have recently been introduced to elongate the lifetime of the die by reducing wear and improve the appearance of dried pasta by obtaining a smoother pasta surface. (Dawa, 2001). Pasta prepared using bronze dies has lower density and breaking strength, higher porosity and a larger effective diffusion coefficient of water during drying than that prepared using Teflon dies. (Lucisano et al., 2008). After the forming step, pasta is dried to reach a moisture content of approximately 11% by weight (Lagoudaki et al., 1993). Although some dried foods, such as instant noodles, are processed using superheated steam, pasta is dried by using hot air (Ogawa & Adachi, 2017). A typical drying curve for pasta is concave, (moisture content rapidly decreases during the early stage of drying, and gradually decelerates to become very low at the later stage) (Ogawa et al., 2012). Pasta is dried after forming and rehydrated by cooking before consumption (Ogawa & Adachi, 2017). On the other hand, cooking time decreases in case of instant pasta products such as noodles. (Kim, 1996). These products are precooked, dried and commercially packed. They are simply soaked in hot boiling water for a few minutes before they are eaten (Sanni et al., 2007).

For the development of easily rehydratable dry cat food, examining and learning from how pasta is produced is regarded as a starting point for this study.

1.3.2. Wet Petfood Production

In this section, wet petfood production is explained considering 9 main steps: chopping, mixing, emulsification, cooking, cooling, cutting, gravy/jelly preparation, filling and sterilization. Schematic diagram of wet petfood production is shown in Figure 1.1.

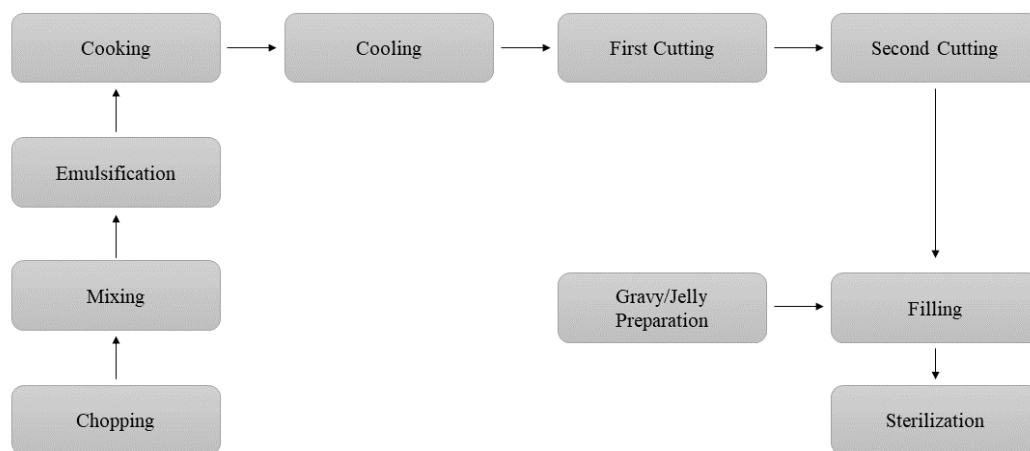


Figure 1.1. Schematic diagram of wet petfood production

Chopping: Meat used in the petfood industry is usually a mixture of high-quality animal parts/organs (livers, kidneys, hearts) and other by-products (lungs, stomach, intestines, skin, bone, blood, etc) that are derived from healthy animals as confirmed by veterinarians during the slaughter process. Meats are stored as frozen blocks at -18°C until production. Prior to mixing, frozen meat is chopped to reduce its size.

Mixing: Frozen and fresh meat materials, water and dry components (vitamins, flour etc.) are added and mixed in a mixer, until reaching the temperature described in the process specifications of the production site.

Emulsification: The aim of emulsification is to achieve a homogenous product and to reduce the size of the particles of the mixture, applying a mechanical treatment using different knives and die plates.

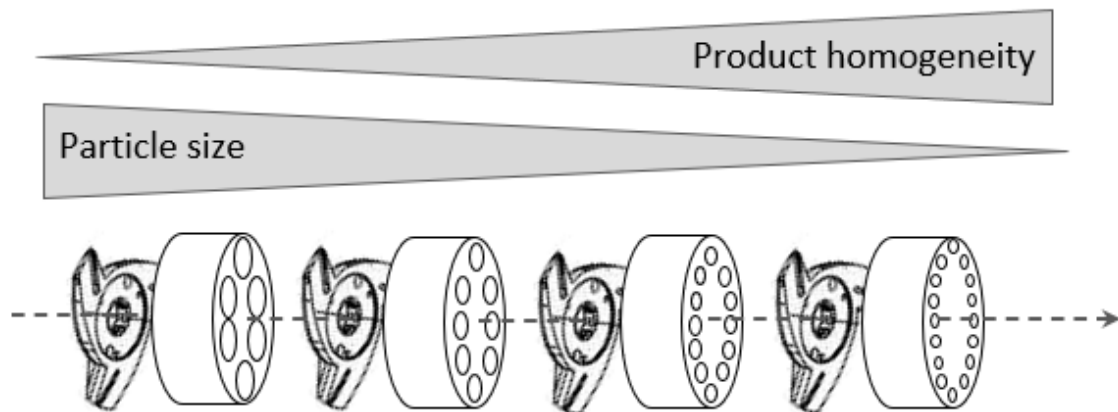


Figure 1.2. Example of emulsification step, using four different die plates, and knives attached between each die plate

Gravy / Jelly Preparation: Viscosity, finished product consistency, pH control, flavors, vitamins, and color are pivotal considerations for gravy/jelly products. In this section all components are mixed to ensure high mixing quality before hydration of hydrocolloids and viscosity development occurs.

Cooking: After emulsification process, chunk processing can be done by using conventional steam or high moisture extrusion. For the chunks that are made by using steam, firstly, strands or ropes of mixture enter steam tunnel or conveyor belt. Then meat is cooked with the help of steam. When cooked mixture arrived at the end of steam tunnel, chunks will be cooled at the cooling unit. Afterwards, it is cut in pieces at the cutting unit. On the other hand, high-moisture extrusion process is used to make product with fibrous texture like animal meat by using vegetable protein. The mechanism of high moisture extrusion is strongly related with the pressure and the flow of material, which depend on the feed moisture content, cooking temperature, and the screw rotational speed. For the extrusion process, the required barrel temperature exceeds 130°C at which vegetable

proteins are denatured by the shearing force of screw rotation inside the barrel of the extruder. Then, because of the effects of moisture and temperature, three-dimensional protein structure is destroyed, and amino acid chains connected by peptide bonds are unfolded. The formation of crosslinks between denatured protein chains by means of amide, disulfide, and hydrogen bonds begins in the barrel near the die. As the protein melt with cross-linked bonds passes through the cooling die, it is texturized by the formation of laminar flow with the protein matrix in the longitudinal direction of the die (Ryu, 2020).

Cutting: Once temperature has been decreased to below 40°C, ropes are cut into chunks. Depending on the brand, the recipe and the production site, chunks can be cut one or two times

Filling: The filling of the chunks in gravy and in jelly products is done by using piston fillers or in a two step filling process. Controlling the gravy viscosity is then a key for a successful filling, which is achieved by a combination of gels and thickeners in the gravy.

Sterilization: The aim of this step is to obtain commercial sterility, giving enough heat treatment to destroy the heat resistant spores of the pathogens. Together with closing, this is the most critical operation in the manufacture of heat sterilized wet pet foods.

1.3.3. Dry Petfood Production

Pet foods are categorized based on their moisture content by The Association of American Feed Control Officials (AAFCO). Dry petfood moisture content is around 8%–10% by weight and their water activity is lower than 0.6 (Dzanic, 2003). They are microbiologically stable considering their water activity, no additional sterilization step is required during production (Brannen, 1971).

Weighing: For each raw material family, there is a weighing section, and it has different capabilities according to each scale. Flowability of the raw material is a key factor for dry petfood processing.

Grinding: Materials are ground to reduce particle size for an extrusion process. Heating phenomenon due to friction may affect thermosensitive ingredients. Also, the particle size

distribution has an important effect on kibble aesthetics, digestibility and palatability. A larger particle size causes a rougher surface. Small particles of starch granules support a better cooking in the extrusion process to increase the digestibility of products as a result of good gelatinization.

Mixing: To obtain a homogeneous blend of all ingredients, a mixing process is applied after grinding. Ground raw materials, powders and liquid injections are mixed in this section.

Extrusion: The extrusion process is divided into 3 sub-categories: Dosing, preconditioning and extrusion. Following the dosing of ingredients a preconditioner is used to mix, hydrate and heat the starch granules to increase the degree of cooking, and to increase gelatinization. Starch gelatinization is an irreversible swelling of starch granules caused by heat and moisture. The objectives of the extrusion process are mixing, cooking, killing microorganisms and forming/cutting. Because of the high friction inside the extruder, starch granules break down before hydration. At the end of the extruder, a die plate serves as a flow restriction to control bulk density, textural development, size and shape of the kibble. Flash evaporation of the water in the hot product when leaving the process is caused by the pressure difference inside and outside of the extruder.

Drying: The objective of the drying is to control product moisture. This is a pivotal parameter in terms of shelf life, palatability and product texture. Vertical dryers are used with counter flow principle. Firstly the product accumulates in an accumulator deck then passes through 3 drying zones. Finally the product is cooled and subjected to a coating step.

Coating: This process section is required to apply liquid aromas and adjust the fat level of the kibbles. The liquids are applied via nozzles or distribution plates under vacuum or atmospheric pressure. At atmospheric pressure, air can move through the wholes inside the kibbles, therefore, after coating, fat granules could not reach the inner part of the product. To avoid this, vacuum is used to support the uniformly distribution of the coating materials.

Cooling: The product must be cooled sufficiently to avoid any condensation in the bags. The temperature between the product at the cooler exit and packaging area must be lower than 10°C.

Packaging: In this process section, kibbles are packed according to the required amount. At the top of the packaging device, small scales are used to more precisely control the weight of the finished products.

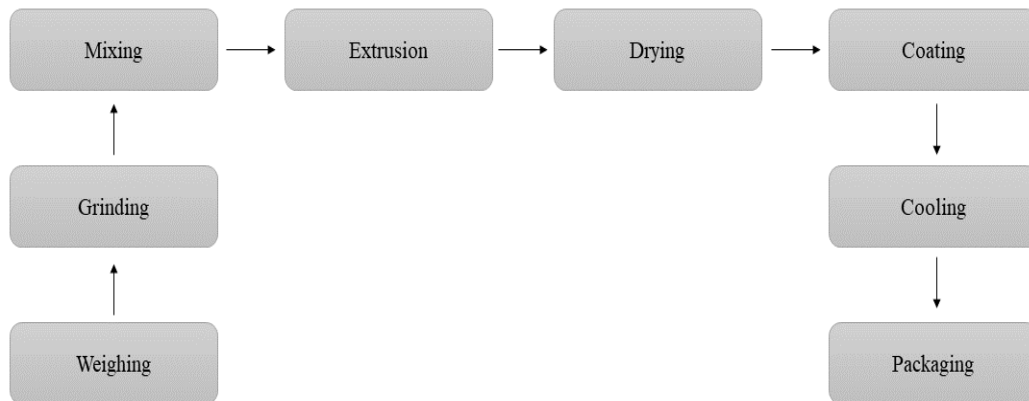


Figure 1.3. Schematic diagram of dry petfood production

1.3.4. Fundamentals of drying process

Drying is among the most ancient and pre-eminent physical methods for food preservation. It is the process of decreasing the moisture content of food and generally is used for foods which have high moisture content (80%) such as fruits, vegetables and other products (Changrue et al., 2006). Moisture classification can be done according to the water content in food. Bound water is that fraction of water present in food materials which is either physically or chemically attached with other compounds and solid structural matrix. Water in excess of bound water is called free water (Joardder et al., 2019). When a product is subjected to thermal drying, two processes occur simultaneously:

1. Energy transfer from the surrounding environment to evaporate the surface moisture

2. Internal moisture transfer to the surface of the solid and its subsequent evaporation due to process 1

Energy transfer as heat from the surrounding environment to the product can occur as a result of conduction, convection or radiation and in some cases as a result of a combination of these effects. Industrial dryers can be different depending on the principle of heat transfer employed. Generally, heat is transferred to the surface of the product and then to the interior (Mujumdar, 2006).

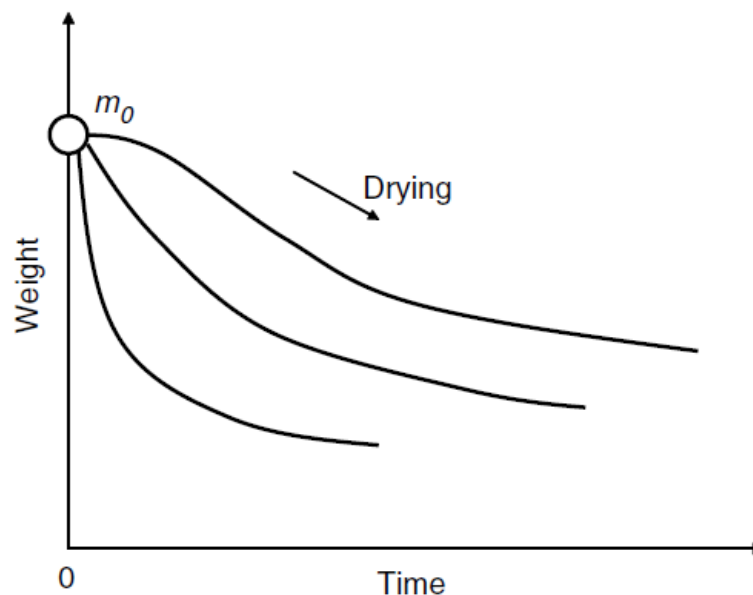


Figure 1.4. Weight loss curves as drying proceeds

(Source: X. D. Chen & Mujumdar, 2009)

The water activity (a_w) of a food is the ratio between the vapor pressure of the food itself, when in a completely undisturbed balance with the surrounding air media, and the vapor pressure of distilled water under identical conditions. The water activity increases with temperature. By measuring and controlling the water activity in food, it is possible to:

- 1) predict which microorganisms will be potential sources of spoilage and infection,
- 2) maintain the chemical stability of foods,
- 3) minimize nonenzymatic browning reactions and spontaneous lipid oxidation reactions,
- 4) prolong the desired activity of enzymes in food,
- 5) optimize the physical properties of foods, such as texture and shelf life (Fontana, 2000).

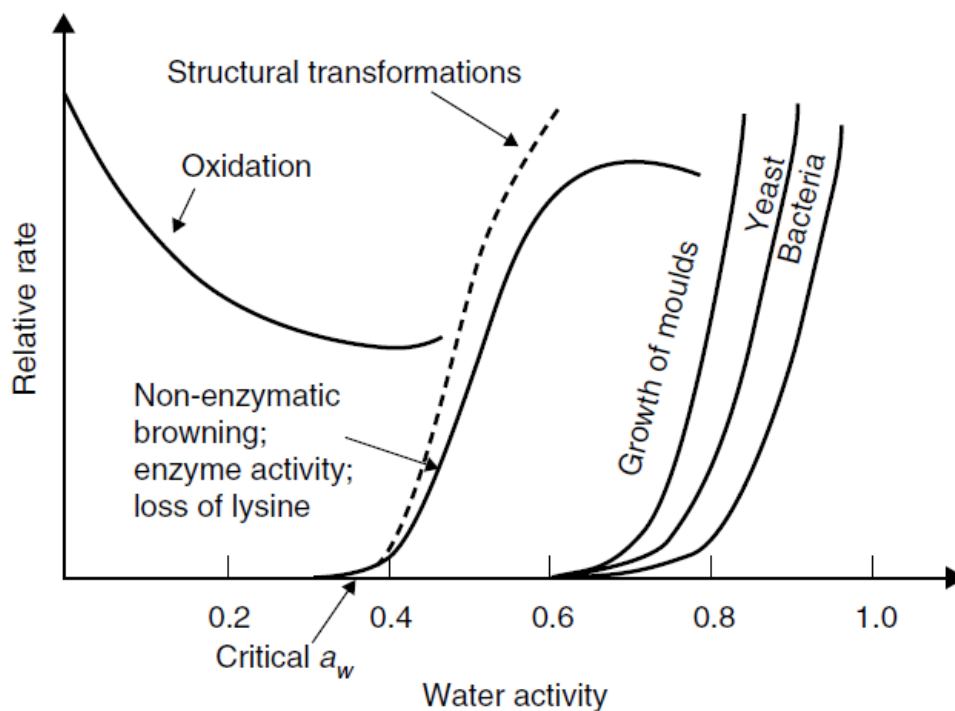


Figure 1.5. Chemical and biochemical reaction rates as functions of water activity

(Source: X. D. Chen & Mujumdar, 2009).

Moisture sorption isotherms are a graphical representation of the thermodynamic equilibrium between moisture content and the water activity at a given temperature and pressure (Iglesias and Chirife 1982). The shape of the moisture sorption isotherms changes with temperature, composition, pressure, physical state of the components, and

the process of dehydration/humidification used (Goula et al. 2008; Vega-Ga'lvez et al. 2009). Moisture sorption isotherms describe how water molecules are adsorbed by a specific material and are used to predict the shelf life of food and pharmaceutical products due to moisture gain (adsorption) or loss (desorption), and also to define storage, transport, and process conditions, or to estimate the energy requirements of a dehydration process (Arlabosse et al. 2003; Iglesias and Chirife 1982; Lewicki 2000).

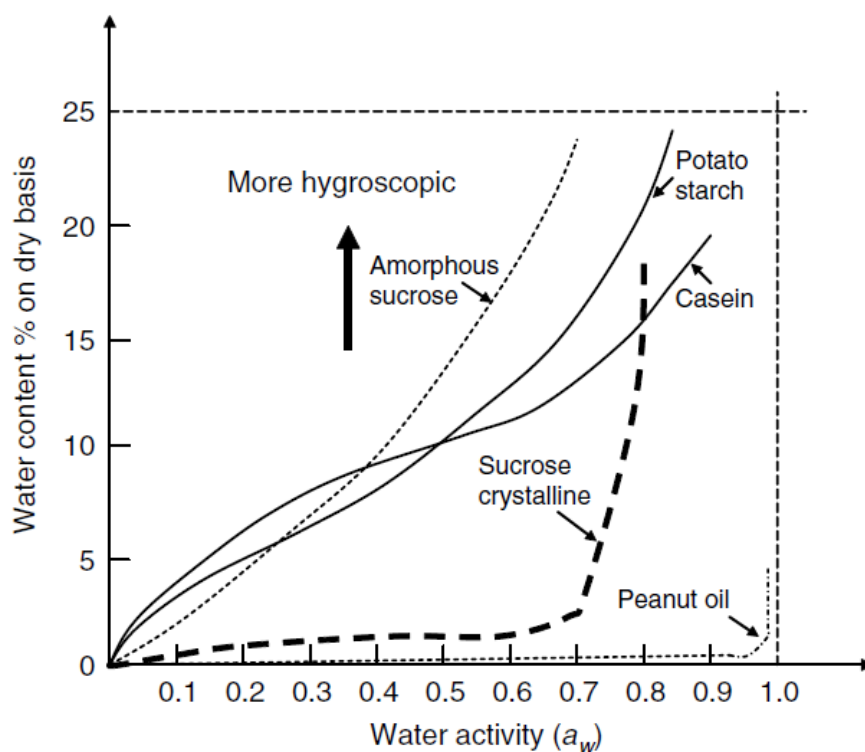


Figure 1.6. Typical sorption isotherms for some food materials

(Source: X. D. Chen & Mujumdar, 2009).

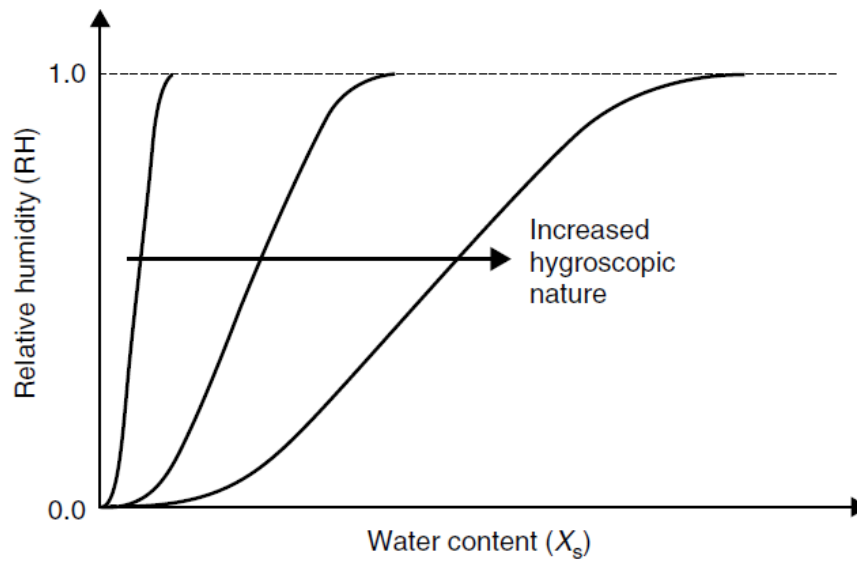


Figure 1.7. Qualitative equilibrium isotherms for various types of food materials

(Source: X. D. Chen & Mujumdar, 2009).

1.3.4.1. Industrial Drying Methods

Drying process is an effective method to inhibit growth of microorganisms, minimize quantities of moisture-mediated degradation reactions, and reduce transportation mass (Jin et al., 2014). Conventional hot air drying (HAD) is the most commonly used commercial technique for drying vegetables and fruits, in which heat is transferred from the hot air to the product by convection, and evaporated water from product is transported to the air also by convection. Generally, it causes the degradation of bioactive constituents with heat sensitivity, loss of color and transformation of food structures (Lewicki, 2006). Sun drying (SD) is another conventional drying method for reducing the moisture content of products under the sun. The solar radiation heats up the products as well as the surrounding air and thus increases the rate of water evaporating from them. Sun drying tends to be labor-intensive, has limited capacity and hard to control temperature during process (Doymaz, 2004). Freeze drying (FD) has been considered as an most effective drying method to obtain superior quality products. However, it requires higher energy compared to the other drying processes and it is time-consuming (Nail et

al., 2002). Consequently, novel drying methods are emerging to deal with the weakness of conventional HAD, SD and FD and create high-quality products. Microwave drying (MD) is an energy efficient drying technology, which could save drying time, accelerate drying rates and improve certain quality of products. However, one of its disadvantages is the non-uniformity of the electromagnetic field and it causes hot spots on product surface (Zhang et al., 2006). Thus, MD has been combined with other drying techniques such as HAD, FD, vacuum drying (VD), and intermittent power application to acquire more effective drying and higher quality products.

1.3.4.1.1. Microwave Drying Principles and Microwave-Vacuum Drying (MVD)

Microwave energy is used for food thawing, preheating, pasteurization, sterilization, cooking, and drying. In microwave dryers, the electromagnetic waves (in 300–300,000 MHz frequency range) are transmitted to the product and cause molecular movement by two mechanisms: migration of ionic parts and rotation of dipolar parts. Dipolar water molecules in food product are realigned a million times per second in electric field direction depending on the microwave frequency applied. Friction takes place due to this movement of water molecules and the food is heated by volume. Volumetric heating occurs as a result of the absorption of microwave energy in food and the conversion of this energy into heat (Schiffmann, 2020). When a material is exposed to electromagnetic (EM) radiation such as microwaves, part of the energy is reflected and part of it is transmitted through the surface, where a fraction of this latter portion is actually absorbed (Venkatesh & Raghavan, 2004). The proportions of energy that fall into these three categories are defined by the dielectric properties of the material, which explain its behavior when EM radiation is applied (Sosa-Morales et al., 2010; Venkatesh & Raghavan, 2004). Specifically, the dielectric constant (ϵ') refers to the EM-field distribution in the material and indicates the ability of a material to couple with microwave energy, and the loss factor (ϵ'') expresses the loss interactions or the material's ability to dissipate electromagnetic energy. These parameters may change with frequency of the applied field, temperature, composition, moisture content and particle density of the material (Venkatesh & Raghavan, 2004).

Penetration depth (d_p) is also an important parameter when considering the application of EM energy to food products, where most fresh foods have a penetration depth of approximately 0.6–1 cm at 2,450 MHz (Venkatesh & Raghavan, 2004). The penetration depth is defined as the distance into the material at which the power has dropped to $1/e$ (or 36.8%) of its incident power (Orsat et al., 2007).

Several factors affect dielectric properties of food: sample composition; sample density, because the amount of load per unit volume will determine how much material is present to interact with the electromagnetic field; temperature, where at high frequencies the loss factor (ϵ'') will decrease with temperature and vice versa at low frequencies; and storage time, because composition of food may change during storage and this may affect its dielectric properties (Orsat et al., 2007). Temperature uniformity is one of the main issues in microwave drying techniques because the standing wave pattern within the microwave cavity can produce hot spots within the cavity where the sample will rapidly heat to very high temperatures and damage the samples. The overall temperature and temperature uniformity of the sample can be controlled in several ways. Suitable power density is critical to minimize the possibility of hot spots. Decreasing magnetron output, increasing sample size and cavity size have important impact on reducing power density (Alibas, 2007).

The application of vacuum during microwave drying is a good solution for preventing physical damage during microwave drying such as scorching, off-color production, and uneven heat distribution (Gunasekaran, 1990). Vacuum is applied to reduce absolute pressure to vaporize water at lower temperatures than under atmospheric conditions and it helps to limit quality degradation by maintaining the product at much lower temperatures (Yongsawatdigul & Gunasekaran, 1996). Moreover, because air is excluded during drying, oxidation reactions are minimized (Gunasekaran, 1990). All of these contribute to a dried product with better texture, color and flavor, making it advantageous to use this process despite high installation and operating costs (Yongsawatdigul & Gunasekaran, 1996). Important parameters for a successful microwave drying are shown in Table 1.1.

Table 1.1. Advantages of microwave vacuum drying

Criteria	Necessary actions
Cost savings	Energy savings Increased throughput Labor reduction Reduction in heat load in the plant speedup of the process Operational efficiencies Reduced maintenance costs
Improved quality	Prevention of structural defects Reduction of bacterial contamination
High yield	The instantaneous control of temperature allows better control of drying and provides a lower level of rejects. Moisture leveling effects avoid over or under drying of products

MWV drying is an advantageous technique since it reduces volatile loss, accelerate moisture removal, and slow heat transfer to the solid phase due to the absence of convection (Drouzas & Schubert, 1996). In the literature, there are several studies that used MWV for apple (Erle & Schubert, 2001), banana (Mousa & Farid, 2002), honey (Cui et al., 2008), grape (Clary et al., 2007), potato (Bondaruk et al., 2007) and pineapple (Corrêa et al., 2011) among others. Also, the drying kinetics of turkey breast meat with different drying techniques was investigated and reported that microwave-drying as shorter process time compared to freeze-drying and hot air techniques because of volumetric heating (Elmas et al., 2020) . In another study in which beef samples, chicken and fish were dried at different microwave powers, as a result drying time decreased as microwave power increased from 90 W to 360 W, and fish fillets were dried in a shorter time than the others [(Kipcak & \.Ismail, 2021). In another study, microwave-vacuum drying provided the highest rehydration rate and the lowest b^* , hardness, springiness, and chewiness values compared with hot air and sun drying.(F. Chen et al., 2014). These studies indicates that microwave drying especially combined with vacuum could be used as an energy-efficient and time-saving technology to prepare dried meat-based products of higher quality.

1.3.5. Rehydration Characteristics of Food Products

Rehydration is one of the most pivotal quality parameters of dehydrated foods. The quick and complete process of rehydration can lead to a reduction of labor costs and to improving the efficiency of production (Deng et al., 2014). In addition, some of the dried food products are consumed after rehydration process such as fruit juices (Krokida and Philippopoulos 2005; Segui, Fito, and Fito 2013). In the rehydration process, the dried product is expected to interact with the liquid and recover the pre-drying properties. (Maldonado et al., 2010). During the described process, the following steps take place simultaneously: absorption of liquid by the dehydrated product, swelling of the rehydrated material and leaching of the solutes such as (vitamins, minerals and sugars) from the product to the rehydrating medium. The mass transfer kinetics depends on the rehydrating liquid (Giraldo et al., 2006; Moreira et al., 2008). Rehydration can be considered as a measure of the degree of change that occur during the drying process because pre-drying, drying and rehydration may cause negative change in the product structure and composition (Lewicki, 1998a, 1998b).

The effect of different parameters of drying and rehydration on rehydration characteristics of food products has been widely investigated in the literature. The effect of convection, freeze and freeze-convection drying on carrot and pumpkin rehydration was investigated by Kumar et al. (2001) whereas Giri et al. (2007) investigated the influence of convection and microwave-vacuum dehydration on rehydration of button mushrooms. Convection, convection-explosion and puffing drying on rehydration of pitaya was also determined (Yi et al., 2016). Giri et al. (2007) evaluated rehydration rate of microwave-vacuum dried button mushrooms at different pressures. Rhim et al. (2011) studied the effect of freezing temperature on rehydration characteristics of freeze-dried rice porridge. The effect of drying air temperature on rehydration characteristics was investigated for apple, red bell pepper and parboiled rice. (Rafiq et al., 2015; Vega-Gálvez et al., 2008; Wang & Chao, 2003).

The influence of the rehydrating medium on rehydration characteristics has also been evaluated in the literature. Oliveira et al. (1999) was investigated the rehydration characteristic of dried apple in milk and water, whereas Prothon et al. (2001) immersed apple in water and yogurt. Rehydration rate of candied mango fruit in the sucrose

solutions at different concentrations was also investigated (Giraldo et al., 2006). Chemical composition of the food products affect the ability of liquid absorption. Kaptso et al. (2008) investigated the rehydration kinetics of the cowpea and bambara seeds. The differences observed during the rehydration process underline the differences between the varieties and species. Markowski et al. (2006) studied the effect of six varieties of carrots on the water absorption of dried carrots. According to the results, it was noticed that variety was significantly affected the process of rehydration. Ciurzyńska et al. (2011) evaluated the influence of variety on rehydration properties of vacuum-dried strawberries. Results showed that fruit variety cause differences in higher-final water content of strawberries.

CHAPTER 2

MATERIALS AND METHODS

2.1. Raw materials

In this study, chicken liver had been used as a wet model raw material that came from slaughter operation for human consumption. After a veterinarian determined that the chickens were healthy, the livers were collected and frozen. It had been delivered to the European Innovation Center (EIC) as frozen blocks that were later-on cut by a meat cutter with the following dimensions: 25 mm x 25 mm x 25 mm. After cutting process, it was used within 72h since modification or loss of meat functionalities started to appear.

4 different materials were used as dry model material in the form of a dried powder:

Poultry greaves is obtained after cooking certain raw materials and separating the fat from them before drying. Greaves meal is a valuable by-product produced during melting the raw fats from multispecies. It is a high protein product with low ash and good digestibility.

Beef greaves is produced from fresh slaughterhouse by-products and meal by-products. The origin of the meal is from animals that have been slaughtered for human consumption. The meal is heated, dried and defatted by decanting and/or fat pressing.

Poultry meal is an animal protein, produced from fresh poultry by-products from EU-registered slaughterhouses. The origin of the poultry meal is from birds that have been slaughtered for human consumption. This meal is produced by carefully heating and drying the poultry by-products. The meal is used as a protein source for petfood, aqua feed and fur feed.

Blood plasma is the fluid component of blood that remains after removal of blood cells. In slaughter facilities, blood is collected and maintained in liquid state, then plasma is isolated from liquid. After that the plasma is dried and resulting in a creamish-colored

powder. It has a texturing capacity because of the protein fraction which includes albumin and immunoglobulins (Beynen, n.d.).

2.2. Methods of Chunk Preparation

2.2.1. Particle Size Distribution

For laboratory scale production, analysis was performed to understand the effect of particle size of poultry greaves meal on product surface. Sieve machine VE1000 (RETSCH GmbH, Germany) was used with 2mm - 1mm - 710um - 500um - 250um - 100um sieves. Analysis was carried out at 1.20 mm amplitude for 10 minutes. Distribution curve were drawn according to the results.

For larger scale production, all dry materials are sieved through a 1.520 um sieve by using sieving machine to make sure that larger particles do not clog the dieplate during extrusion.

2.2.2. Chunks Preparation at Laboratory Scale

Preliminary trials were carried out to determine the recipe that can make smooth chunks at the lowest moisture. According to the pre-trials, 200g poultry greaves meal was set for all experiments. It was sieved with 500um sieve to make more smooth shapes. Wet and dry materials was mixed and diluted with 35g water in Simac mix700 Pasta Machine to simplify the forming step. As a matter of fact, it showed that the forming step was challenging to carry out a proper shape with the mixture without dilution since the pressure of the pasta machine is insufficient. The mixing time for kitchen trials was fixed at 5 minutes for 435g mixture.

In order to understand the effect of chunks' shape on rehydration and drying time, dieplates with surface areas varying between 80-800 mm² were used. In order from smallest to largest: Orechiette, Torchietti, Riccioli, Conchigliette and Curvo quadro were

used. After extrusion process, chunks were cut to size 2-2.5 cm. After extrusion process, chunks were cut to size 2-2.5 cm.

2.2.3. Pilot plant scale tests

Petfood production is carried out in pilot plant by scaling up the recipe that is used in the kitchen lab trials and by using the production process is described in this section.

Meat grinding: The meat stored as big blocks (100x50x10cm) at -18°C was ground into smaller pieces (25 mm x 25 mm x 25 mm) for easier defrosting. Grounded meats kept in 4°C cabinet for thawing for 1 day before the trial.

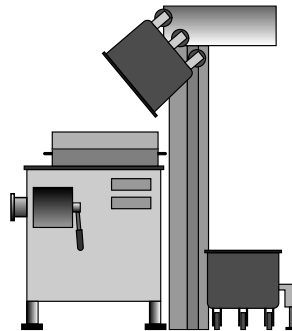


Figure 2.1. Pre-grinder

Mixing: Meats and powders were mixed using mechanical and thermal energies. For further size reduction of the meat and to get rid of ice crystals at the center of the meat pieces, it passed through a set of high-speed rotating blades until the desired size and the degree of mixing have been achieved. The mixing temperature should be as low as possible during the process to avoid microbial growth. Therefore, the mixture temperature was controlled with a probe at certain time points.

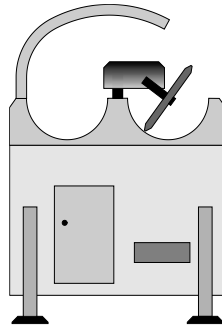


Figure 2.2. Bowl chopper

Extrusion: Extruder (Händle GmbH, Germany) with a maximum pressure capacity of 50 bar was used to form meat emulsion. The screw with a diameter of 80 mm had 3.5-35 rpm rotation capacity. Volumetric flow rate of the process was 100 L/h. A proprietary special designed die plate was used to shape the meat emulsion.

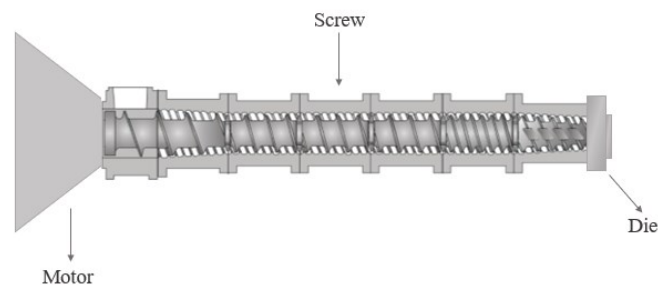


Figure 2.3. General overview for the extruder

Cooking: The aim of this step is to cook ropes with condensing steam in the steam tunnel. The middle point of ropes should be reached to 90 °C to be microbiologically safe. The time of the processing was 500 seconds and the steam supply is was 70 kg/h at 90 °C. After heating the ropes were cooled for 50 seconds and conveyed to the cutter.

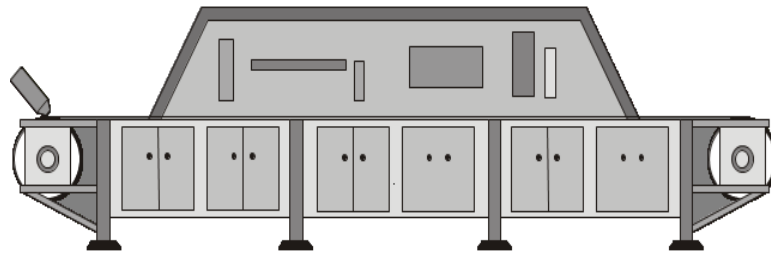
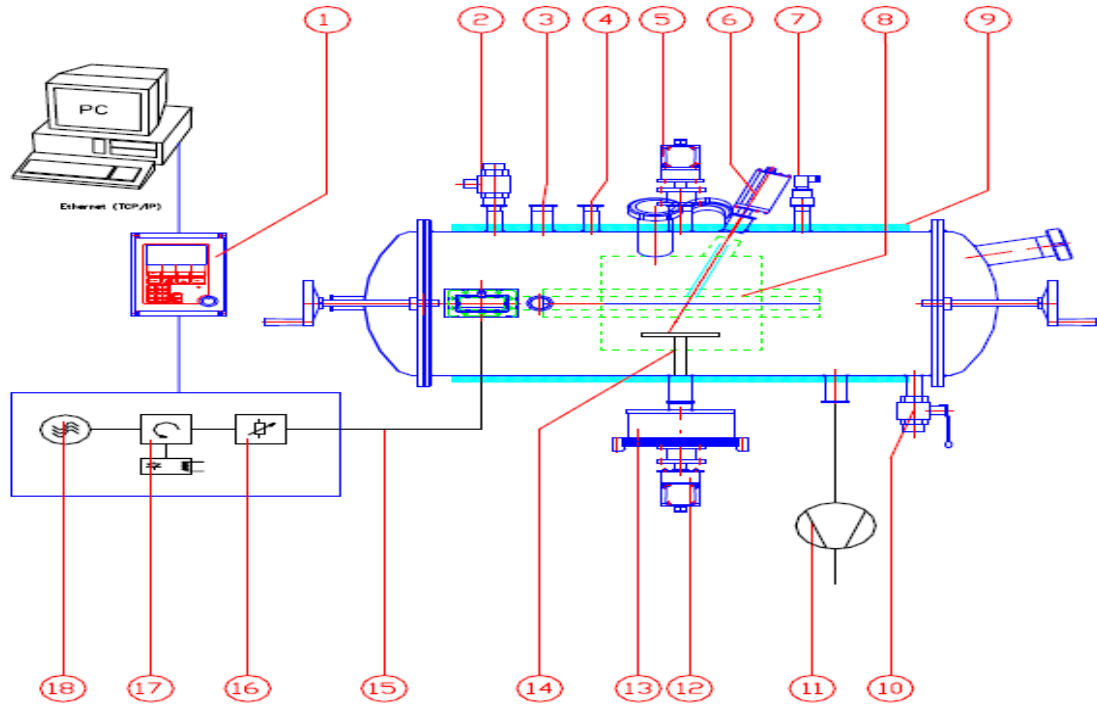


Figure 2.4. Steam oven

Cutting : In the first cutting step, ropes were cut into 25*50*5 mm with rotary knife. The setpoint for cutting roller had been set as 50 U/min. After that, disc cutter was used to make smaller chunks to reach target dimensions for cat petfood's. Blade separations were set as 6mm and 4mm, respectively.

Drying: In this study, μ WaveVac0650 microwave vacuum dryer (PÜSCHNER GmbH, Germany) was used for the purposes of the present investigation and is depicted in Figure 2.5. Turntable containing the chunks to be dried was put into the interior of the chamber to operate smaller scale experiments and half open drum was used for larger scale operations. For laboratory scale, initial load and microwave energy was set as 100g and 0.8 kW, respectively. For pilot plant scale, initial load and microwave energy was set as 1.5kg and 1.5 kW, respectively. Pressure was set as 30 mbar for all experiments. Temperature, pressure and reflected energy was controlled during the process continuously.



1. PLC System with connection to WINDOWS PC
2. Magnetic False Air Valve for Pressure Control
3. 3Auxiliary DN 40 Port for e.g. manual valve, differential Pressure Measurements, fibre optical Wires, Vacuum Pump connection etc.
4. Auxiliary DN 25 Port 1
5. Mode stirrer
6. IR-Camera 0-200°C
7. Pressure Transducer
8. Variable Applicator for different Applications like Horn Antenna, Slotted Waveguides, Broadside Array, Antenna, Cavities
9. Wall Heating
10. Manual Valve for Breaking the Vacuum
11. Auxiliary DN 40 Port for e.g. Vacuum Pump Connection
12. Turntable Motor
13. Online Scale 0-1/5/10kg, 10.000 digits
14. Turntable
15. Microwave Waveguides
16. Tuner
17. Circulator with Measurement of Reflections
18. Magnetron

Figure 2.5. Block Diagram of the Microwave Vacuum Dryer μ WaveVac0650

The WINDOWS™ Program μ WaveCAT is used to control and visualize parameters as well as for its data management capabilities. This software provides an easy access to all parameters, history of alarms and control the process continuously. It also allows to create program and download new process control files (profiles) in order to achieve best results.

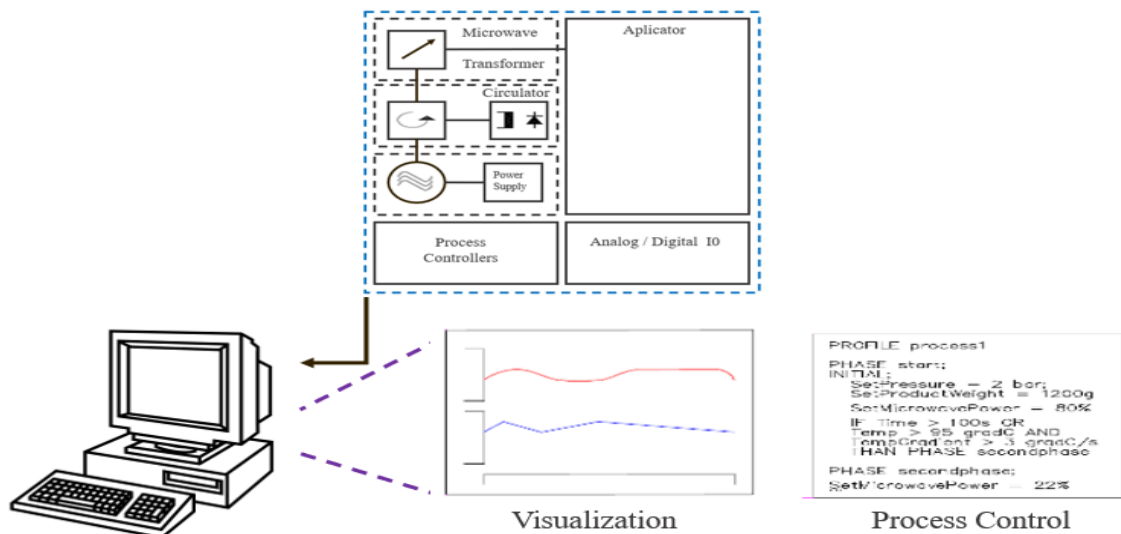


Figure 2.6. Schematic diagram for process visualization

Optimization of Microwave Vacuum Dryer: Reflected energy is an important parameter to understand how much power is absorbed by the load. Excessive reflected energy may cause plasma formation when a gas is ionized under this high energy source and it may damage the microwave chamber. To prevent this occurrence, microwave vacuum dryer was optimized for small and larger scales. The circulator (Figure 2.5.) is a three-port device that only allows a microwave signal to exit through the port directly after the one it entered. Figure 2.7. shows the working principle of circulator. The arrows represent the direction of the magnetic fields and the signal when applied to any port of these devices. A signal experiences a low loss in the direction of arrow and high loss in reverse direction while propagating through the Circulator. A signal is placed at port 1

and port 2 is well matched, the signal will exit at port 2 with low loss. If there is a mismatch at port 2, then some signal power will be reflected towards port 3 (Linkhart et al., 2014). Gap values between ports were set as 250 and 10 to reach reflected energy lower than 20% of initial microwave power.

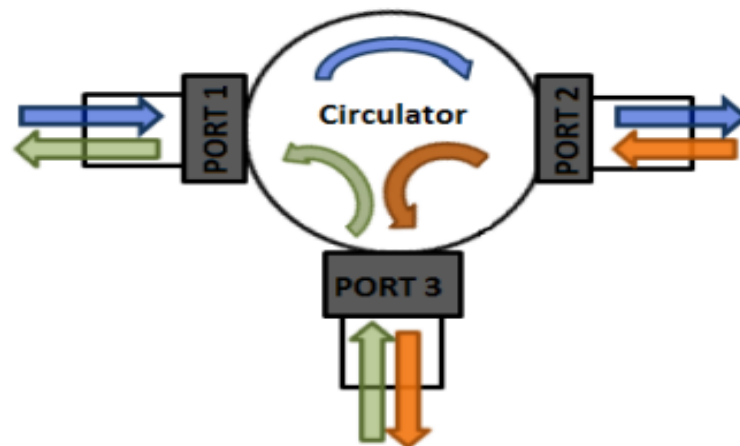


Figure 2.7. Circulator working principle

2.3. Moisture Analysis

Moisture of the chunks were measured by using SMART Track II Moisture Analyzer (CEM Corporation, North Carolina, USA). It is a thermogravimetric analysis that used microwave energy as a heating method. The principle of the method is described as the weight loss of sample that occurs as the material is heated with microwave energy. The sample weight was taken prior to heating and again after reaching a steady-state mass after drying for final moisture determination on percentage basis.

For dried chunks, 2 pads were put in the oven and tare is processed. Chunks were grinded and mixed, then 2-3 grams of sample was taken and spread on one pad. The other pad was used afterwards to cover the first one and then prepared sample was put in the moisture analyzer. Moisture content was read after 3-4 minutes processing.

2.4. Water Activity Measurement

Water activity of the chunks were measured with Water Activity Meter (AquaLab, USA). Chunks were grinded then put a 7.5 mL sample in a disposable cup. Sample was sealed with the sample chamber lid. After vapor equilibrium, results were provided within in 5 minutes.

2.5. Moisture Sorption Isotherm

Eight saturated salt solutions were prepared corresponding to a range of water activities from 0.1 to 0.98. Each solution was transferred into two separate jars in an amount to occupy a space of about 1 cm deep at the bottom. Samples of chunks were dried in a vacuum oven at about 30°C for two days. Then samples of the dried products were weighed into small crucibles of aluminum foils and placed. Each of the sets of eight jars were kept at 25°C for equilibration of samples. After equilibration, samples were analyzed for their moisture content.

2.6. Fines Analysis

After the cooking step, the products are cooled to provide the required hardness. A fines analysis was carried out to understand how the cooling process and the recipes used here affect the efficiency of the formulation of the product. After the cutting step, chunks were weighted minimum 1 kg and passed over a 2 mm sieve. The amount of fines was calculated as a percentage of the freshly cut chunks.

2.7. Rehydration Analysis

This analysis was performed to determine how long it would take for dried chunks to return to their raw moisture levels. Strainer is used to hold chunks for the experiment.

The strainer without chunks was immersed in 1500 mL of water and the water held by the strainer was measured. Then the 100 g chunks were placed into the chamber and immersed in the water for 90 seconds and the measurement was taken every 30 seconds by removing the chamber from water (Figure 2.8.). The amount of water loss is measured and water intake was calculated as percentage. The amount of water remaining on the strainer and on the surface of the product was taken into account and it was subtracted from water loss.

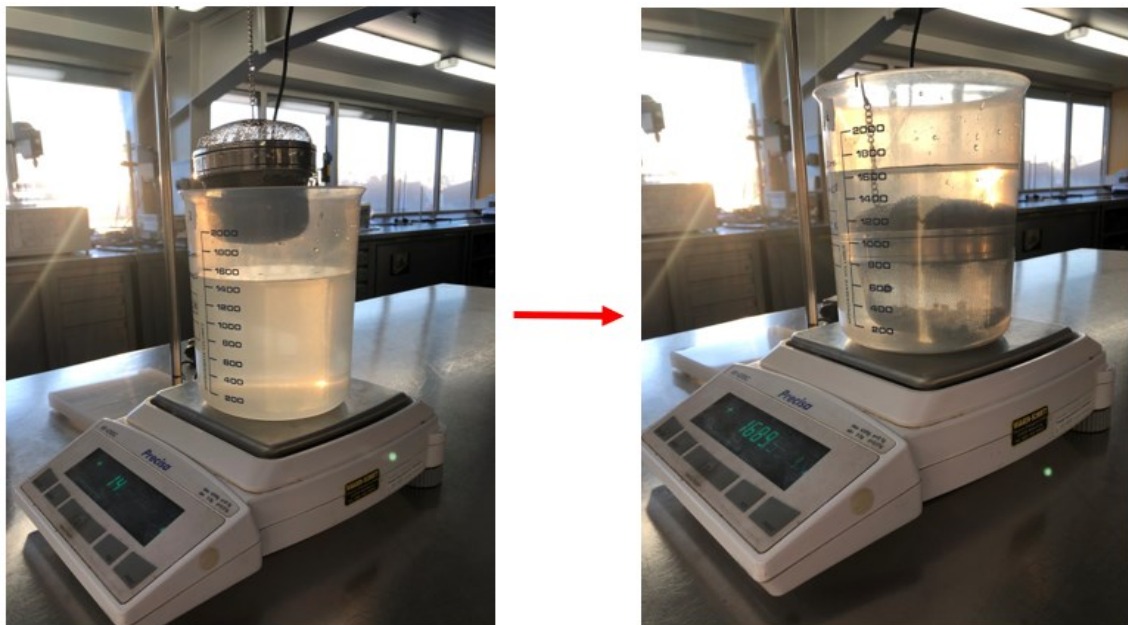


Figure 2.8. Rehydration analysis for microwave vacuum dried chunks

2.8. Statistical Analysis

The results were expressed as mean with 95% confidence interval. All analyses were performed in Minitab and figures were made in GraphPad (version 6.0). Rehydration analyses for laboratory and pilot plant production were analyzed by “Fit regression model” with 95% confidence interval.

CHAPTER 3

RESULTS AND DISCUSSION

3.1. Laboratory Scale Production

3.1.1. Chunk Formulation

In these experiments, poultry greaves and chicken liver are used as dry protein and wet materials to investigate optimum recipe formulation for laboratory scale production. Powder and liquid contents are considered as factor variables, and the moisture is considered as response variable. Table 3.1. shows description of recipes, additional unit operations and moisture contents for preliminary experiments for the product formulations. Experiments were carried out by taking the moisture level of the pasta before drying process as a reference.

Table 3.1. Recipes for laboratory scale production by containing different amount of CL and pre-treatment steps. *Abbreviations*: CL: Chicken Liver; PG: Poultry Greaves

	RECIPE 1	RECIPE 2	RECIPE 3	RECIPE 4
Powder	200g PG	200g PG	200g PG	200g PG
Liquid	200g CL	100g CL	100g CL	100g CL
Particle size reduction			Sieving <500um	Pregrinding <500um
Moisture	40.03 % ± 0.455	30.16% ± 0.982	30.15% ± 0.623	30.15% ± 0.765

Recipe 1 was carried out with 1:1 ratio of dry and wet materials. However, moisture should be reduced by decreasing amount of liquid in the mixture to reach average moisture content of 30-32% (Wrigley, 2004). Although the moisture level of the Recipe 2 was compatible with the literature, no product could be obtained from the pasta machine. This result is due to the different pressure level in the laboratory scale and plant scale extruder. Increase in pressure is required for extruding pasta dough to enhance hydration level and hydration kinetic (Manthey et al., 2004). There is a reverse correlation between particle size and hydration kinetics. The finer the particles results in the faster their sorption kinetics (Hebrard et al., 2003). To observe the effect of particle size of poultry greaves on mixture formation, sieving and pre-grinding steps were added in Recipe 3 and Recipe 4, respectively. According to the literature, particle size of materials has a pivotal impact on flowability since flow resistance increased with increasing particle size (Moreira et al., 2010) . As a result of sieving, the product at the desired moisture level (30-32%) was obtained, but still a smooth surface could not be obtained due to the low moisture level. In order to obtain a smoother surface and keep the nutritional value constant, different concentrations of water are added to the formulation (Table 3.2.).

Table 3.2. Recipes with addition of different water content for laboratory scale production. *Abbreviations:* CL: Chicken Liver; PG: Poultry Greaves

	RECIPE 5	RECIPE 6	RECIPE 7	RECIPE 8
Powder	200g PG	200g PG	200g PG	200g PG
Liquid	200g CL + 10g water	200g CL + 40g water	200g CL + 20g water	200g CL + 30g water
Particle size reduction	Sieving <500um	Sieving <500um	Sieving <500um	Sieving <500um
Moisture	40.12% ±0.342	46.13%± 0.477	41.6% ± 0.734	42.9% ± 0.351

The required structure could not be obtained with Recipe 5 due to the low-water content. The use of different raw materials causes the product not to be obtained at the moisture level used in pasta production. Therefore, the experiments were continued by using Recipe 7, where the smooth chunks were obtained with lowest moisture level among Recipe 6, 7 and 8.

3.1.2. Drying Process

In this section, to determine the parameters for the drying process and how the relationship between moisture and water activity changed according to the surface area was examined. The moisture sorption isotherm describes the thermodynamic relationship between water activity and the equilibrium of the moisture content of a food product at constant temperature and pressure. The knowledge and understanding of sorption isotherms is pivotal in food processing for the design and optimization of drying equipment (Sahin & Sumnu, 2006). Figure 3.1. was generated from desorption process in which water molecules progressively and reversibly mix together with food solids via chemisorption, physical adsorption, and multilayer condensation (Rockland & Stewart, 2013). An isotherm can be divided into three regions; the water in region A represents strongly bound water, and the enthalpy of vaporization is higher than the one of pure water. Bound water is unfreezable and it is not available for chemical reactions or as a plasticizer (Andrade et al., 2011). In region B, water molecules bind less firmly than region A and the vaporization enthalpy is slightly higher than the one of pure water. This class of constituent water can be looked upon as the continuous transition from bound to free water. In the region C, the water loosely binds to food materials (Andrade et al., 2011).

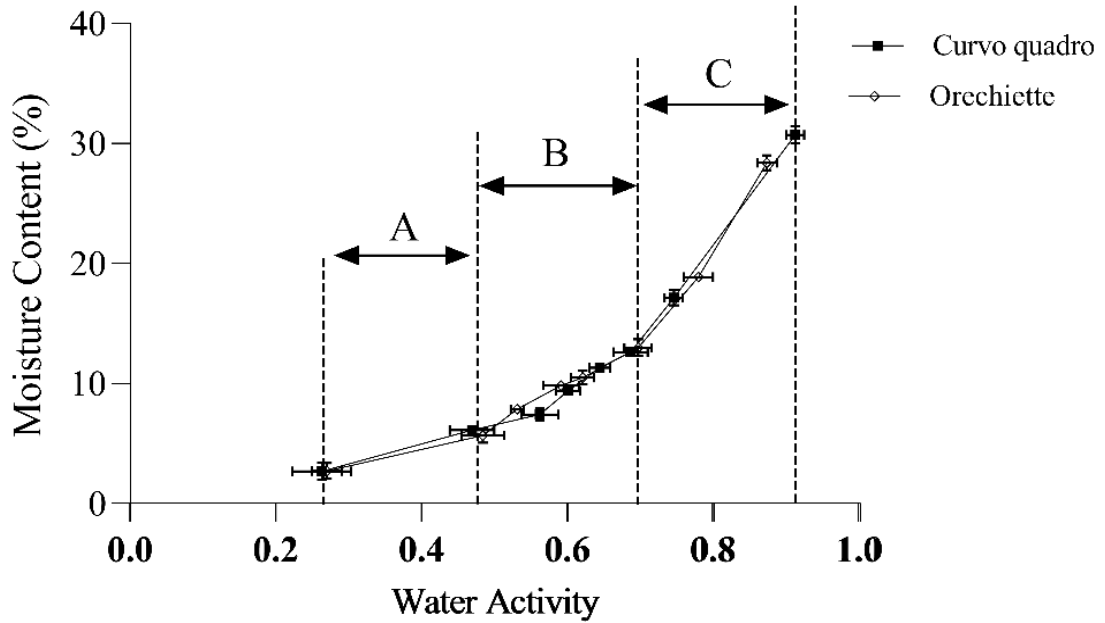


Figure 3.1. Moisture content versus water activity for Curvo quadro and Orechiette. Chunks made with Curvo quadro and Orechiette dieplates were dried at different time points and their moisture contents and water activities were measured. Values are represented as mean with 95% CI(n=3)

The a_w concept has made it possible to develop generalized rules or limits for the stability of foods and thus explain the development of preservation processes based on control or reduction of water. For most foods, the critical point is 0.6–0.7 a_w , below which no microorganisms can grow (Bell & Labuza, 2000). According to the Figure 3.1., the target moisture content for drying process is set as 8% which is a point that the water activity is below 0.6.

To determine the effects of surface area on the characteristic of moisture sorption isotherm, two extreme dieplates were used. There are not significant differences between two dieplates, so it indicates that moisture sorption isotherm characteristic of mixture does not depend on the shape. According to this result, same target moisture level (8%) was used to dry the chunks and make them microbiologically safe.

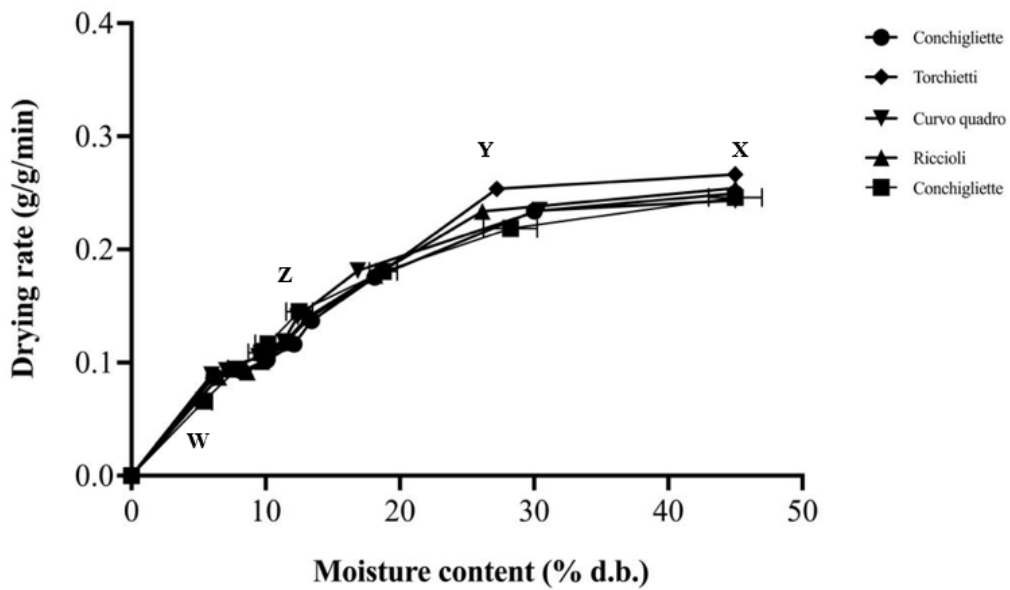


Figure 3.2. Drying rate of chunks by using various dieplates. 100g chunks is used as initial load. Values are represented as mean with 95% CI(n=3).

The drying rate curve is characterized by three main stages: (1) transient early stage, during which the product is heating up; (2) constant rate period, where the moisture is relatively easy to remove; (3) falling rate period, in which moisture is bound or held within the solid matrix. Figure 3.2. shows drying rate of chunks by using various dieplates. X-Y represents constant rate period: the presence of a thin film of water on the food is considered and there is not internal or external mass transfer resistance. In this stage, drying is controlled by external heat transfer. The constant rate period continues as long as the amount of water evaporates is equal to the amount of water supplied to the surface of the material. Y-W represents falling rate period where drying is controlled by the internal mass transfer resistance. The falling rate period can be divided into two steps. A first falling drying rate occurs when wetted spots in the surface continually diminish until the surface is dried (Point Z) (X. D. Chen & Mujumdar, 2009). Second falling rate period begins at point Z when the surface is completely dry. The plane of evaporation recedes from the surface. Heat required for moisture removal is transferred through the solid to the vaporization of moisture in the solid and the vapor moves through the solid

into air stream. The amount water removed in this period can be relatively small compared to the constant rate and first falling rate period (Maroulis et al., 1995).

3.1.3. Rehydration Analysis

In this section, effect of different dieplates on rehydration rate was investigated. Figure 3.3. shows percentage of water intake of chunks that are made in laboratory at 30, 60 and 90 seconds time period. Chunks that made by using curvo quadro dieplate has highest water intake ($82.23\% \pm 2.196$) and the lowest water intake (52.79 ± 1.774) was observed with orechiette group

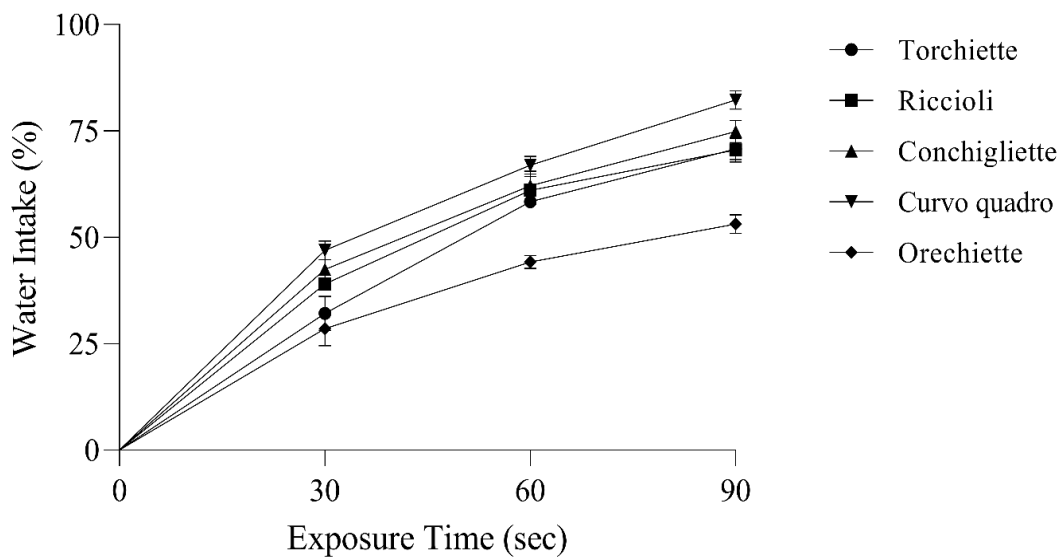


Figure 3.3. Effect of surface area on water intake during rehydration analysis. Chunks were soaked into water and the loss of water were measured at -30, -60 and -90 seconds. Fit regression model with 95% Confidence Interval was performed for statistical analysis. Values are represented as mean with 95% CI (n=3).

Table 3.3. Regression Equation for laboratory scale rehydration analysis (time in seconds)

Die plate

Conchigliette	Water Intake (%) = 12.50 + 1.139 Time - 0.004970 Time*Time
Curvo quadro	Water Intake (%) = 15.16 + 1.187 Time - 0.004970 Time*Time
Orecchiette	Water Intake (%) = 2.51 + 1.006 Time - 0.004970 Time*Time
Riccioli	Water Intake (%) = 10.56 + 1.122 Time - 0.004970 Time*Time
Torchiette	Water Intake (%) = 0.29 + 1.243 Time - 0.004970 Time*Time

In Table 3.3., Curvo quadro has highest slope (1.187) following Concighiliette. Table 3.4. shows ANOVA results and it indicates that dieplates and exposure time have significant effect on water intake since the p value for diplates (0.000) and time (0.000) lower than alpha ($\alpha < 0.05$). The p value of lack of fit (0.363) higher than alpha ($\alpha < 0.05$) and this indicates that the model is fit for the data. Results showed that larger surface area leads increase in water intake at certain time period of time and this is consistent with current findings (Aravindakshan et al., 2021).

Table 3.4. Analysis of Variance for laboratory scale rehydration analysis

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	10	11074.8	1107.48	151.54	0.000
Time	1	660.3	660.33	90.36	0.000
Die plate	4	215.3	53.82	7.36	0.000
Time*Time	1	200.1	200.08	27.38	0.000
Time*Die plate	4	167.0	41.76	5.71	0.001
Error	34	248.5	7.31		
Lack-of-Fit	4	32.4	8.11	1.13	0.363
Pure Error	30	216.0	7.20		
Total	44	11323.3			

Table 3.5. Model summary for laboratory scale rehydration analysis

S	R-sq	R-sq(adj)	R-sq(pred)
2.70335	97.81%	97.16%	96.09%

3.2. Pilot Plant Production

3.2.1. Chunk Formation

In order to scale up the recipe determined in the laboratory scale production, Recipe 9 (Table 3.6.) which is the flowable meat analogue after mixing step was selected as starting point. It was observed that there are three pivotal points to be considered when operating larger scale production:

- (1) The meat analogue should be in a flowable form rather than sticky
- (2) The shape of the ropes should be maintained after extrusion
- (3) The inner part of the ropes should reach 90C to be microbiologically safe
- (4) The product should be firm enough to operate the cutting device after the cooking process.

Table 3.6. Recipes for pilot plant scale production. Abbreviations: BG: Beef greaves; BP: Blood plasma; CL: Chicken Liver PG: Poultry greaves; PM: Poultry meal.

	RECIPE 9 (%)	RECIPE 10 (%)	RECIPE 11 (%)	RECIPE 12 (%)	RECIPE 13 (%)
Powder	42 PG	19.5 PG – 19.5 PM – 3 BP	19.5 PG – 19.5 BG – 3BP	40.5 PG – 1.5 BP	39 PG - 3 BP
Liquid	58 CL	58 CL	58 CL	58 CL	58 CL
Moisture	45.12 ± 0.680	44.13± 0.432	44.6 ± 0.451	45.13 ± 0.892	44.6 ± 0.451

For Recipe 9, separations were observed in the product coming out of the die plate, even though the mixture was flowable and had a higher humidity level than at laboratory scale. Also, with the current the product dimension, the middle part of ropes remained raw due to insufficient cooking.

By adding an additional metal apparatus to the end of the die plate, the ropes are divided into two equal parts. Ropes were cut at every 1 meter during extrusion so that a

vacuum was not formed inside the rope and the gaps would not be closed. However, the cutting process could not be performed properly due to the softness of the ropes after the cooking. Additional raw material was investigated to ensure that the shape is maintained.

Blood plasma was used as a dry material in petfood production that was characterized by its high protein content, water-holding capacity, foaming and emulsifying properties (Tybor et al., 1975) and gel strength when heated above 80 °C (HOWELL & Lawrie, 1984). 1.5 and 3% of blood plasma were added to the recipes and its effect on the rope texture after the extrusion step was investigated. 3% BP was the minimum percentage that can be used in the production efficiently.

To investigate the effect of gelation properties of blood plasma on rope firmness, the product temperature was increased above 80°C at steam tunnel during cooking. At this temperature, plasma is in liquid form and it should be cooled in the required time to make the products more rigid (Ziegler & Foegeding, 1990). However, since the cooling time used in the pilot plant was shorter than the time required for the gel to solidify, the product could not gain the required hardness. That is why other dry materials were added to improve the production performance.

In order to understand the positive effect of the ingredients in the formulation, Recipe 9, 10 and 11 were used and the percentage of fines was measured after the cutting step. While one group of the products was passed through the cooling conveyor once, the other group was passed 2 times and the effect of the increase in the cooling time on the product quality was determined. Results indicate that changes in the formulation, cooling time and their two-way interaction have a significant effect on the amount of fines since the p values are lower than alpha ($\alpha < 0.05$) (Table 3.7.). On the model summary (Table 3.8.), there are not big differences between R-sq adjusted (88.96%) and R-sq predicted (82.46%) which indicates that the model is suitable for this data. According to the results, chunks made of Recipe 10 and passed through cooling conveyor 2 times provided the most convenient combination since it produced the least fines ($34.62\% \pm 2.81$) (Figure 3.4.). One of the reasons for this may be that the percentage of collagen in the PM is lower than PG and BG. Since, the collagen does not fall to the gelling temperature with 1 minute cooling step (1 cooling step = 50 seconds), the product will remain soft and more fines will be created during production after the cutting step.

Table 3.7. Analysis of variance (ANOVA) for fine analysis.

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Recipe	2	3255.5	1627.76	52.03	0.000
Number of cooling	1	769.9	769.88	24.61	0.000
Recipe*Number of cooling	2	415.2	207.60	6.64	0.011
Error	12	375.4	31.28		
Total	17	4816.0			

Table 3.8. Model summary for fine analysis.

S	R-sq	R-sq(adj)	R-sq(pred)
5.59307	92.21%	88.96%	82.46%

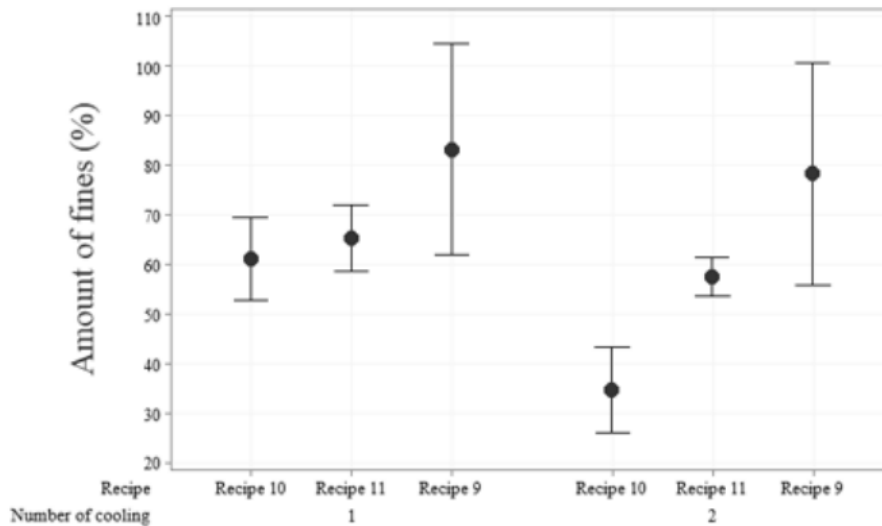


Figure 3.4. Interval plot of amount of fines. After the cutting step, chunks were sieved and amount of fines was calculated as percentage. 2-way ANOVA is performed and values represented as mean with 95% CI

3.2.2. Drying

While the drying process was carried out in the pilot plant, the initial moisture content of chunks was $45 \pm 0.824\%$. The required time for 1.5 kilograms of chunks to decrease water activity below 0.6 was determined as 23 minutes, and the energy was settled as 1.5 kW. The behavior of input and reflected power is shown in Figure 3.5. Reflected energy is one of the pivotal parameters employed to assess the efficiency of the microwave energy transfer into the material to be heated or into the formation of a plasma (Zarein et al., 2015). Figure 3.5. displays that the reflected energy peaks in the first minute then thanks to the optimization step integrated into the program, it is reduced. The reflected energy was very low during the initial phase of the drying which resulted in a higher absorption of microwave power. Following moisture reduction, the energy absorbed by the product decreased and the reflected power increased. It is observed that there is a fluctuation in the reflective energy as a result of the decrease in absorption of energy in the last step of drying.

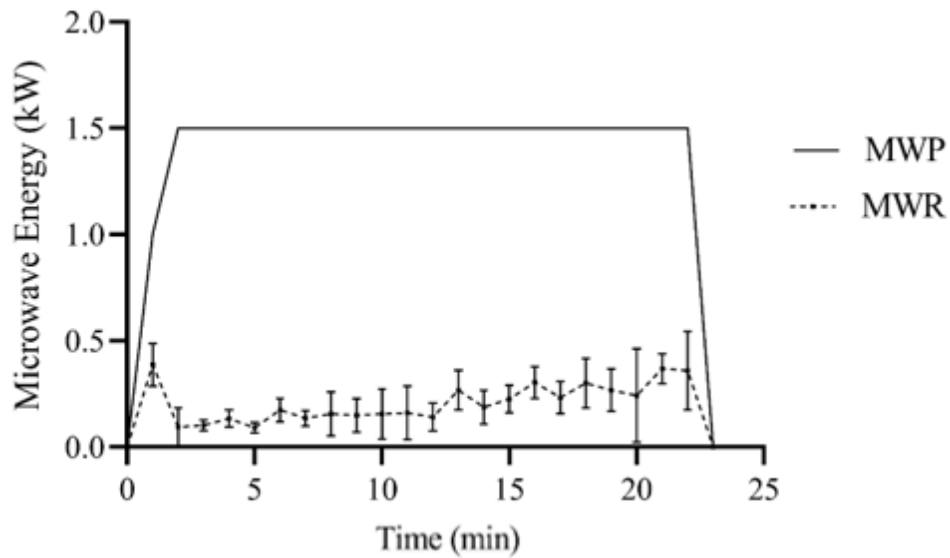


Figure 3.5. Microwave energy during drying process. Values represented as mean with 95% CI (n=3). Abbreviations: MWP: microwave power; MWR: Reflected microwave energy.

Figure 3.6. shows temperature distribution of chunks during the drying process. The temperature increases gradually, however especially at the end of the process there is a giant fluctuation due to the shaking mode of the drum to ensure uniform drying while the measurement was made with the static IR camera. A sudden temperature increase was determined in the last stage of the drying which might be caused by the changes of dielectric properties when most moisture was removed from the chunks. Similar results can be found in other studies (Andrés et al., 2004; Li et al., 2010). The cause of the large temperature fluctuations might be the high power density. As the drying was going on, the mass, moisture contents, and characters of the chunks were constantly changing, but the power levels remained the same. Hence there is a need to adjust the power levels during the microwave drying process, especially during the last drying period to eliminate fluctuation in reflected energy and temperature. On the other hand, the mean of the highest temperature is 58 °C, so this low-temperature drying allows high-quality dried products to be obtained, preserving the nutritional properties of fresh foods better than conventional drying (Santacatalina et al., 2016)

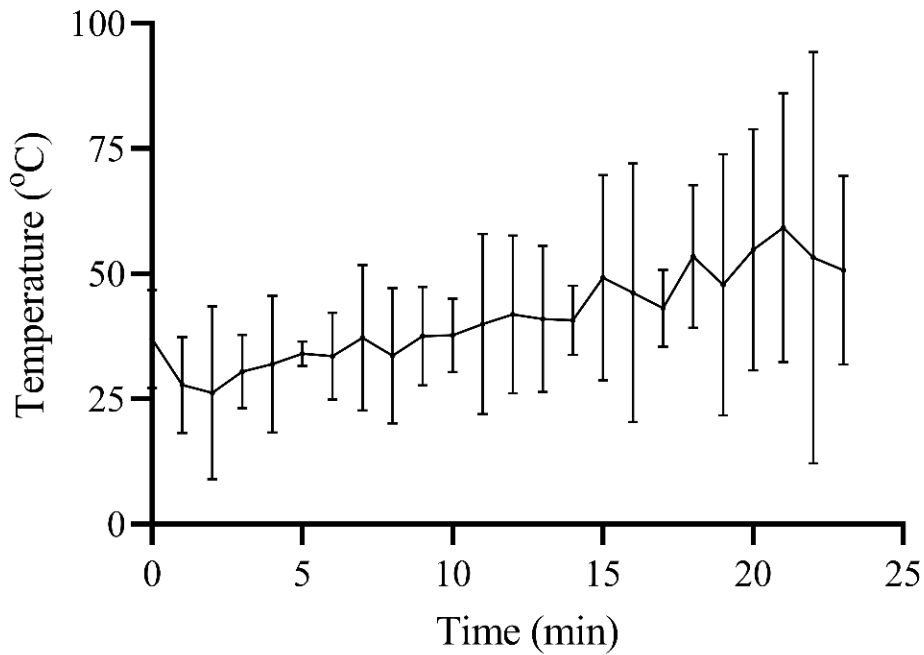


Figure 3.6. Surface temperature distribution of chunks during drying process. Values represented as mean with 95% CI (n=3).

3.2.3 Rehydration

3.2.3.1. Effect of blood plasma on rehydration time

Various concentrations of BP which was used to give texture to the chunks were tried and the impact of these concentrations on rehydration efficiency was investigated. Figure 3.7. shows percentage of water intake at 30-, 60- and 90- seconds time period. Chunks that contain 3% BP, 1.5% BP and without (w/o) BP rehydrate 94.48 ± 4.96 %, 74.86 ± 2.73 % and 63.14 ± 5.12 %, respectively after 90 seconds. Table 3.9. shows the Analysis of variance (ANOVA) results which is used to determine differences between statistical groups. Results indicate that different recipes and exposure time have significant effect on rehydration of chunks since the p value for recipes (0.018) and exposure time (0.038) below alpha ($\alpha < 0.05$). However their two way interactions is not significant since the alpha value (0.481) is higher than alpha ($\alpha < 0.05$). The model is fits the data since the p value for lack of fit (0.320) lower than alpha ($\alpha < 0.05$). Table 3.10.

shows regression equation. Recipe without plasma has the highest slope (38.90) compared to the 3% BP (18.76) and 1.5% BP (26.39) groups and this concludes that at certain time point the use of blood plasma in the formulation of the product affects rehydration in a negative way. A possible cause is that blood plasma has formed a gel with dense microstructures and this may create a barrier for water migration (Hou et al., 2019). The results are consistent with the literature since the rehydration of food is affected by pre-drying treatments, mode of dehydration, structure, composition and medium viscosity. It is therefore expected that the changes in the formulation will play important roles in the rehydration mechanism (Marabi & Saguy, 2004).

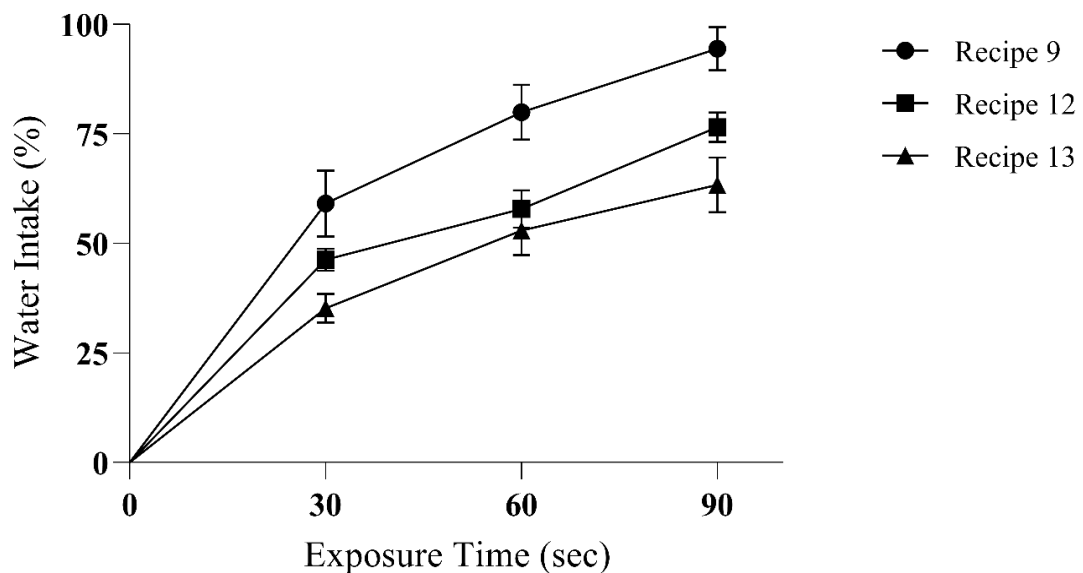


Figure 3.7. Effect of blood plasma on rehydration analysis for pilot plant scale production. Chunks were soaked into water and the loss of water were measured at -30, -60 and -90 seconds. Fit regression model with 95% Confidence Interval was performed for statistical analysis. Values are represented as mean with 95% CI (n=3).

Table 3.9. Analysis of variance for rehydration analysis by using differet blood plasma concentration. Abbreviations: ExpT: Exposure Time (sec)

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	6	7899.26	1316.54	48.73	0.000
Exposure Time (sec)	1	133.27	133.27	4.93	0.038
Recipes	2	265.85	132.92	4.92	0.018
Exposure Time (sec)*Exposure Time 1 (sec)	1	6.96	6.96	0.26	0.617
Exposure Time (sec)*Recipes	2	40.99	20.50	0.76	0.481
Error	20	540.31	27.02		
Lack-of-Fit	2	64.31	32.16	1.22	0.320
Pure Error	18	475.99	26.44		
Total	26	8439.56			

Table 3.10. Regression equation for rehydration analysis by using differet blood plasma concentration

Recipes

Recipe 12	Water Intake (%)	=	26.39		+ 0.648 Exposure Time (sec)
			- 0.00120 Exposure Time (sec)*Exposure Time (sec)		
Recipe 13	Water Intake (%)	=	18.76		+ 0.613 Exposure Time (sec)
			- 0.00120 Exposure Time (sec)*Exposure Time (sec)		
Recipe 9	Water Intake (%)	=	38.90		+ 0.733 Exposure Time (sec)
			- 0.00120 Exposure Time (sec)*Exposure Time (sec)		

3.2.3.1. Effect of different raw materials on rehydration time

In this section, the effect of different raw materials (PG; PM and BG) on the rehydration rate was investigated. Figure 3.8. shows the percentage of water intake of chunks that are made in with PG, PM and BG in pilot plant scale. When the regression equation is taken into account (Table 3.12.), although recipe 13 has the highest slope (0.803) and time-dependent water intake is higher than other recipes, according to the ANOVA results, the differences in recipes do not have significant effect on water intake since the p value of recipe (0.191) is higher than alpha ($\alpha < 0.05$) (Table 3.11.). As seen in previous results, exposure time has a significant effect on water intake when different recipes are used ($p = 0.002$). Data fits for this model since the p value of lack-of-fit (0.637) is higher than alpha ($\alpha < 0.05$). Compared with the literature, it was expected that the change made in the raw material would have a significant effect on rehydration (Krokida & Philippopoulos, 2005). However, on a percentage basis, changing only 20% of the recipe may not have been enough to observe the effect of the various raw materials on rehydration (Figure 3.9.). Experiments can be repeated by making larger changes in the recipes.

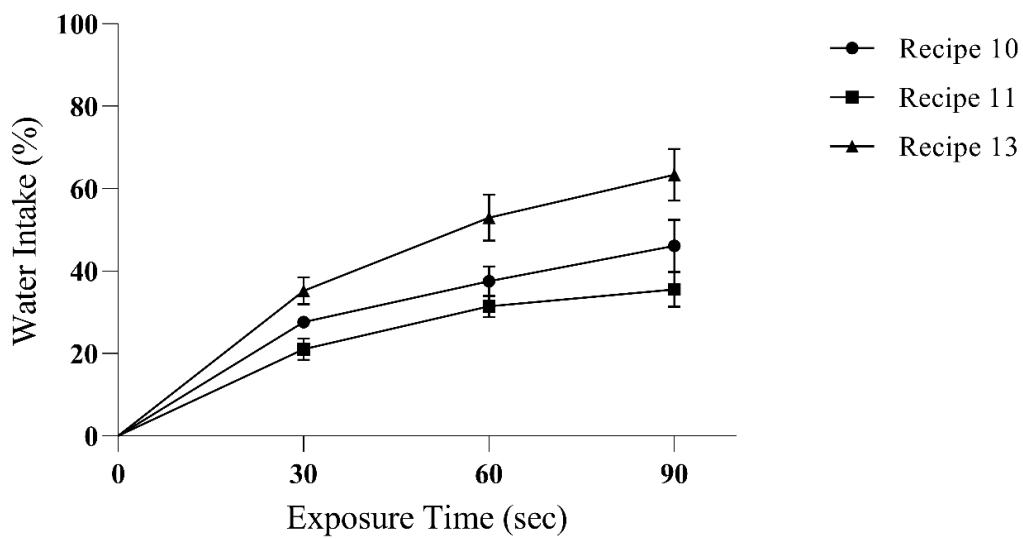


Figure 3.8. Effect of different raw materials on rehydration rate. Chunks were soaked into water and the loss of water was measured at -30, -60 and -90 seconds. Fit regression model with 95% Confidence Interval was performed for statistical analysis. Values are represented as mean with 95% CI (n=3).

Table 3.11. Analysis of variance for rehydration analysis by using different raw materials

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	6	4125.80	687.634	68.44	0.000
Exposure Time (min)	1	130.66	130.660	13.00	0.002
Recipe	2	36.20	18.098	1.80	0.191
Exposure Time (min)*Exposure Time 1 (min)	1	37.44	37.443	3.73	0.068
Exposure Time (min)*Recipe	2	148.51	74.256	7.39	0.004
Error	20	200.96	10.048		
Lack-of-Fit	2	9.84	4.919	0.46	0.637
Pure Error	18	191.12	10.618		
Total	26	4326.76			

Table 3.12. Regression equation for rehydration analysis by using differet raw materials

Recipe			
Recipe 10	Water (%)	Intake = 10.26	+ 0.641 Exposure Time (min) - 0.00278 Exposure Time (min)*Exposure Time (min)
Recipe 11	Water (%)	Intake = 6.52	+ 0.575 Exposure Time (min) - 0.00278 Exposure Time (min)*Exposure Time (min)
Recipe 13	Water (%)	Intake = 14.02	+ 0.803 Exposure Time (min) - 0.00278 Exposure Time (min)*Exposure Time (min)

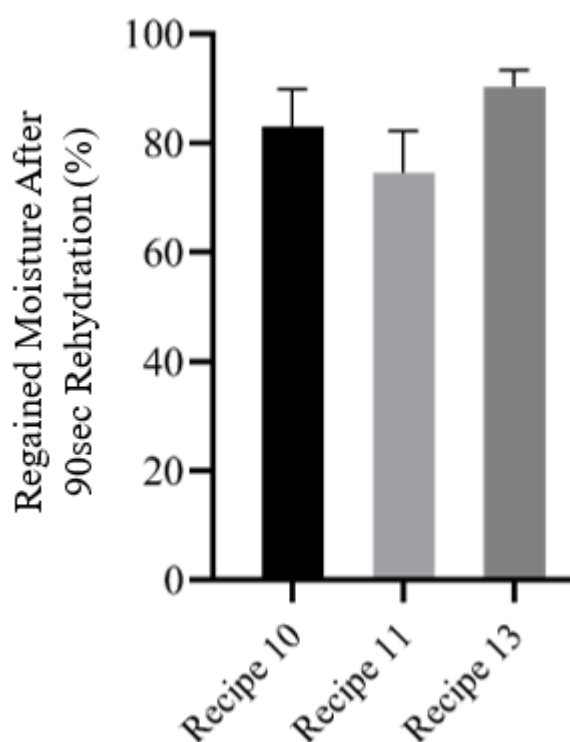


Figure 3.9. Effect of different raw materials on regained moisture content. Chunks were soaked into water for 90 seconds and the final moisture content was measured. Data shows the percentage of regained moisture content based on original moisture before drying. One-way anova was performed for statistical analysis. Values are represented as mean with 95% CI (n=3)

CHAPTER 4

CONCLUSION

Pet parents do not prefer to touch sticky wet food and do not like its strong smell, but regard wet food as a good option for pets in terms of water intake and digestibility has led to this research for a new product in the petfood industry.

In this project, a novel textured wet petfood from raw material assessment to pilot plant continuous processing line set-up was developed and the formulation and shape of the product on rehydration time was investigated. On a formulation basis, it was observed that the amount of moisture and the changes in the raw materials used significantly affect the efficiency obtained from the extruder. Experiments carried out by testing different die plates in the laboratory showed that the increase in the surface area of the product played a role in both drying and decreasing the rehydration time. As a result of this, a die plate was designed to provide as much surface area as possible for use in the pilot plant. Cooking trials showed that 4 important points should be considered during production: the wetted formulation before extrusion should be in a flowable form while passing into the extruder, the shape of the ropes should be maintained after extrusion, ropes should be cooked homogenously to be microbiologically safe and the product should be firm enough to operate the cutting device after the cooking process. The gelling feature of the blood plasma allowed the product to stay in a sufficiently rigid form and had a positive effect on the continuity of the process. Optimizations made for the drying process have kept the reflected energy at a low level throughout the drying period and ensured high efficiency from the process. However, in the final stages of drying, fluctuations in both the reflected energy and the product temperature were observed as a result of the decrease in the amount of moisture in the product and the inability to absorb microwave energy. As a result of the rehydration experiments carried out in the pilot factory, it was observed that blood plasma has a reducing effect on rehydration. Although there was no significant increase in the rehydration rate when different ingredients were tested, in general, it was determined that the recipe designed using poultry meal had a promising effect.

In future studies, recipes can be modified according to the recommendations from MARS GmbH. Rheological studies can be done to understand behavior of mixture. Also, it is suggested to make different optimizations for the microwave vacuum drying process to troubleshoot the fluctuation in reflected energy and temperature. For rehydration, different drying methods can also be used to understand effect of different technologies on rehydration properties. In addition to these, pet product performance tests in Mars Pet Center can be conducted in order to develop a product that is pleasing for pets.

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APPENDIX A. RAW DATA FOR ANOVA ANALYSIS

Time	Dieplate	Water Intake (%)			
30	Orecchiette	28.82	60	Curvo quadro	69.06
30	Orecchiette	24.48	60	Curvo quadro	64.79
30	Orecchiette	32.45	90	Curvo quadro	82.9
60	Orecchiette	42.93	90	Curvo quadro	84.44
60	Orecchiette	43.89	90	Curvo quadro	80.01
60	Orecchiette	46.01			
90	Orecchiette	54.9			
90	Orecchiette	50.75			
90	Orecchiette	53.88			
30	Torchietti	36.89			
30	Torchietti	30.77			
30	Torchietti	29.14			
60	Torchietti	59.58			
60	Torchietti	57.02			
60	Torchietti	59.16			
90	Torchietti	69.03			
90	Torchietti	70.09			
90	Torchietti	73.98			
30	Riccioli	38.99			
30	Riccioli	40.16			
30	Riccioli	38.25			
60	Riccioli	64.5			
60	Riccioli	61.2			
60	Riccioli	57.78			
90	Riccioli	70.03			
90	Riccioli	73.81			
90	Riccioli	68.09			
30	Conchigliette	39.76			
30	Conchigliette	43.83			
30	Conchigliette	43.96			
60	Conchigliette	65.98			
60	Conchigliette	59.27			
60	Conchigliette	61.48			
90	Conchigliette	77.95			
90	Conchigliette	72.91			
90	Conchigliette	74.27			
30	Curvo quadro	48.96			
30	Curvo quadro	47.53			

Time (sec)	Recipe	Water Intake (%)	Time	Recipe	Water Intake (%)
30	Recipe 9	56.22	30	Recipe 10	28.82
30	Recipe 9	53.46	30	Recipe 10	23.69
30	Recipe 9	67.67	30	Recipe 10	24.81
60	Recipe 9	82.91	60	Recipe 10	42.98
60	Recipe 9	72.74	60	Recipe 10	33.93
60	Recipe 9	84.20	60	Recipe 10	36.89
90	Recipe 9	95.11	90	Recipe 10	49.11
90	Recipe 9	99.09	90	Recipe 10	42.89
90	Recipe 9	89.23	90	Recipe 10	43.10
30	Recipe 12	46.27	30	Recipe 11	27.86
30	Recipe 12	43.84	30	Recipe 11	27.57
30	Recipe 12	48.77	30	Recipe 11	27.37
60	Recipe 12	52.98	60	Recipe 11	38.25
60	Recipe 12	60.85	60	Recipe 11	37.90
60	Recipe 12	59.77	60	Recipe 11	38.51
90	Recipe 12	73.21	90	Recipe 11	45.24
90	Recipe 12	76.51	90	Recipe 11	46.99
90	Recipe 12	79.89	90	Recipe 11	44.06
30	Recipe 13	33.77	30	Recipe 13	19.92
30	Recipe 13	39.01	30	Recipe 13	21.98
30	Recipe 13	32.91	30	Recipe 13	21.21
60	Recipe 13	48.81	60	Recipe 13	30.84
60	Recipe 13	59.35	60	Recipe 13	30.65
60	Recipe 13	50.66	60	Recipe 13	30.86
90	Recipe 13	56.57	90	Recipe 13	36.18
90	Recipe 13	68.89	90	Recipe 13	35.63
90	Recipe 13	64.77	90	Recipe 13	36.78