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Title: Novel Ingredients and Additives In Food Product Innovation

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Abstract

The European food market is characterized by a large number of micro, small, and medium sized enterprises (SMEs), as well as traditional, low-tech industry. Such companies operate in an increasingly globalizing market, and thus, despite the seeming controversy between tradition and innovation, the latter is more and more recognized an essential strategic tool in food production to achieve competitive advantage.

Innovation in traditional food mainly pertains to product innovations, such as packaging innovations and changes in product composition, product size and shape or new ways of using the product. In addition, it may also imply improvements in the ways industries produce and commercialize things, e.g. product changes, process changes, company reorganization or new distribution forms.

The recent advances in biotechnology, nanotechnology and preservation technology, such as dried ingredients, food additives and new processing technologies represent unique opportunities for application in the food industry. The main benefits for the industry are the lengthening of products shelf life, the easing of products storage and the reduction of production costs. At the same time, the need to ensure food safety requires the compliance with regulation, and establishing close relationships with external actors in order to access the knowledge needed.

This work will describe the development of two innovative products for the bakery and farinaceous production, namely:

- A hygroscopic mix for stuffed pasta
- A new leavening powder for bakery

and a new product for confectionery:

- Cocoa powder for sprinkling use obtained trough micro-encapsulation

The mixture of food ingredients in powder developed to improve the shelf-life of fresh stuffed pasta has guaranteed the product wholesomeness up to the end of its shelf-life, ensuring a typical aspect of fresh product without affecting the organoleptic properties compared to the traditional recipe.

The new leavening powder for bakery has been developed to provide the consumer with a finished product, be it a cake or a flat bread, with a taste as natural as possible, because sodium acid pyrophosphate, widely and advantangeously used in bakery, may impart an undesirable flavor, defined as "chemical taste" to the finished food product.

This new formulation has a fast acting leavening and is especially suitable for all applications that require an immediate rising.

Finally, the Cocoa powder for sprinkling use obtained trough micro-encapsulation is an example of how a new process step can improve the product performance, because it allows to have dessert covered with cocoa fresh longer, due its low moisture absorption.

Thus, the present work confirms the importance of powder raw materials, food additives and technologies such as micro-encapsulation as innovation drivers for food industry. The competitiveness of food producers is indeed more and more correlated with their ability to integrate new processes and new ingredients in traditional food chains, thus combining state-of-the art technology and products with an artisanal ethos.

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Preface

This work will be organised in four main sections.

A general introduction will define the main concepts and tools related to product innovation in the food industry.

Chapter 1 will describe the research work carried out for the development of a new formulation for tortelli stuffing.

Chapter 2 will concern the New Leavening Powder for bakery.

Finally, in Chapter 3, the development of a New Cocoa Powder for sprinkling use obtained trough micro-encapsulation will be treated.

Process And Product Innovation: Overview

Introduction

During the 1960s, like many other industries, the food industry benefited from the investments that resulted in the technological boom of the post- Sputnik era. The rapid expansion in technology developed in the universities was adapted and converted into new products by the food industry.

At the time many major food manufacturers had comprehensive research programs that spanned from discovery research to product development programs (Smith et al., 2000).

Throughout this era universities developed high-quality basic science which found its way to the ‘‘basic’’ research in industry. In the late 1970s and into the 1980s, corporate America began to retrench and emphasize much more short-term financial return versus longer-term development programs. For this reason, companies change their target from product development to thedevelopment of ‘‘now’’ products, in order to remain competitive. Although product development was cut back relative to sales in the food industry, it survived by applying technologies that were in the pipeline or by developing line extensions. The longer-term work became minimal. The technology pipeline was beginning to run dry because industry was moving away from basic research and many universities were often finding more support in applied areas (Crawford, 1997).

It became evident that a balance needed to be reestablished between fundamental research and product development for the food industry.

During the age of mass production and huge production lines, well-defined jobs were necessary for production and assembly of goods. Frequently, research and development organizations in the food industry evolved to be somewhat like production lines. The product was developed, handed off to a pilot plant, and later to production. Microbiology and quality assurance were brought in later in the development phase to prevent disasters. The product was passed to packaging and then to distribution to deliver for sales, sometimes after production was initiated. (Smith et al., 2000).

Innovation In Traditional Food Products In Europe

The food category of “traditional food products” is not yet extensively analyzed from the chain perspective, with some notable exceptions. The integration of chain partners in the innovation process enhances the capacity to innovate and reduces the risks involved in implementing innovation. From this perspective, incorporating the opinion of customers and consumers further supports the realization of competitive advantage through innovation implementation (Bianka et al., 2010).

The agri-business sector is characterized by a large number of micro, small, and medium sized enterprises (SMEs), and by a low-tech industry, especially in the “traditional food sector”. Thereby, traditional food products are food products (1) whose their key production steps are performed in a specific area at national, regional or local level, (2) which are authentic in their recipe (mix of ingredients), origin of raw material and/or production process, (3) which are commercially available for about 50 years and (4) which are part of the gastronomic heritage. The majority of these traditional food products do not possess any form of origin label (Gellynck & Kühne, 2008).

In the current increasingly globalizing food market, innovation is an essential strategic tool for SMEs to achieve competitive advantage. This also applies for traditional food products, despite the seeming controversy between innovation and tradition and the challenge this controversy involves. So far, only few published studies focus particularly on innovations in traditional food products. Innovations in the traditional food sector strengthen and widen the market of traditional food products in accordance to the emerging problems, such as poor imitations and changing preferences and eating patterns towards more manufactured foods and convenience. Innovations in traditional food mainly pertain to product innovations, such as packaging innovations and changes in product composition, product size and shape or new ways of using the product (Trichopoulou et al., 2006).

Process innovations are less common, given their impact on the authentic identity of the product and its production process.

Feasible applications relate to improving the production process in order to assure quality and traceability. Finally, the implementation of market and organizational innovations can be valuable for traditional food products but their potential is not yet realized or recognized by all chain actors in the traditional food sector (Bianka et al., 2010).

Further, for the successful introduction of innovations in traditional food products, it is also important to have a good understanding of consumers' perceptions, expectations and attitudes towards traditional food products and of consumers' attitudes towards innovations in traditional food products (Linnemann et al., 2006). Consumer research prior to developing and introducing innovation is generally acknowledged to be useful, given that public perceptions of innovations and related risks associated with innovations are likely to differ from the technical estimates provided by experts. In addition, the controversy between tradition and innovation can cause confusion at consumer level when used in combination. Recent efforts to assess the pan-European consumer interpretation of the concept of traditional food products through qualitative research in six European countries (i.e. Belgium, Italy, Norway, Poland, France and Spain) resulted in the following definition (Guerrero et al., 2009): "A traditional food product is a product frequently consumed or associated with specific celebrations and/or seasons, normally transmitted from one generation to another, made accurately in a specific way according to the gastronomic heritage, with little or no processing/manipulation, distinguished and known because of its sensory properties and associated to a certain local area, region or country."

Traditional foods are important components of the dietary patterns of many populations, including those of modern developed societies. Consumer demand for traditional food increases in many western countries, even though the growing globalization of the food markets has promoted the production of large volumes of uniform and cheap food products (Trichopoulou et al., 2007). The increased demand is in line with the general positive image consumers have of traditional foods, and it is related to the fact that food represents a significant part of the cultural, historical and geographical identity, as historians and sociologists have repeatedly reported. Food consumption patterns were mainly shaped, until a recent time, by local and durable cooperation among a variety of socioeconomic actors operating in a given territory.

This cooperation has left its imprint over time on current individual and group food preferences. Thus, despite the food standardization propensity that characterized the 20th century, a great diversity of dietary patterns can be observed from the international diet (Naska et al., 2006). In the last decades, both food companies and public authorities have shown a growing interest in traditional foods. The traditional food sector, as any other sector of the food industry, faces the need to continuously innovate and develop its produce, in order to maintain or expand market share and profitability. One of the principal challenges identified was innovation which can be roughly defined as improvements in the ways industries produce and commercialize things, e.g. product changes, process changes, new ways of organizing the company or new forms of distribution. However, food marketing literature indicates that few innovations are widely accepted by consumers. In a series of works, Rozin et al. (2004) have shown that people increasingly display a preference for natural entities which have been produced without human intervention in a number of domains, especially food. This preference may be associated with the fact that consumers are increasingly averse to risks induced by food innovation resulting from human intervention (Verbeke et al., 2007). Although there is no substantial evidence for an overall advantage of natural products, most European consumers are likely to endorse the idea that natural entities are safer than human-produced entities.

More than for other food categories, the level of novelty and its conceptualization will be critical for the acceptance of innovations. Stolzenbach et al., (2013) stated in this perspective that introducing changes without a clear understanding of the implications on consumer perception and acceptance can potentially do more harm than good to the base traditional products (Vanhonacker et al., 2013).

Innovation has become of particular interest also in the food sector, even if the extant literature traditionally regards this industry as a sector with low research intensity. It is the largest manufacturing sector within the EU driving its economy, and contributing to both economic results and employment opportunities (Avermaete et al., 2002; Menrad, 2004; Traill et al., 2002). The European Confederation of the food and drink industry (CIAA) concludes from a benchmark report on competitiveness of the industry, that its innovative potential should be enhanced if it is to remain competitive in the years to come (CIAA, 2008).

Consequently, companies may adopt new types of product development process, that necessarily involves creating or at least adopting innovative technological solutions and new business models.

In the majority of food companies these new product development processes are still based on internal innovation factor.

The recent advances in biotechnology, nanotechnology and preservation technology represent unique opportunities for application in the food industry. At the same time, the need to ensure food safety requires the compliance to regulation, and establishing close relationships with external actors in order to access the knowledge needed. All these factors provide considerable evidence that open innovation is the appropriate paradigm for addressing the new challenges of the food industry (Bigliardia et al., 2013).

Product Development Strategy

Food processor/marketers have made and will continue to make significant investments to improve their new product development process.

For these companies creativity and speed were removed from the process in favor of analysis and in cases, paralysis.

The world of food product development has changed. It has shifted into high gear. Based on our organization's audits of the major world markets, we estimate that globally a new product is launched every twenty minutes. Speed is the fuel that drives the process. Speed to market, speed versus the competition, speed of consumers' evolving changing needs, and speed necessary to produce new products to meet the needs of the company bottom line. Short-term gains and short term losses versus long-term gains and losses and the balance that goes along with it are important to the product development team (Robinson L., 2000).

What corporations tend to forget is that on a basic level, the process should consist of essentially four key components:

- assessing senior management's commitment to new product development
- finding the right idea
- developing the business case for the product
- development and commercialization of the concept

The important thing to remember is that all of these are important for success.

It is easier to be successful if an organization understands management's commitment to the new product process.

On one hand, food and beverage product development requires the experienced analysis of complex, detailed consumer information that must be synthesized with business and financial information, as well as product development information, technological assessments, and manufacturing practicalities.

On the other hand, new food and beverage product development requires a fresh perspective, one that might provide a new twist to an old idea.

There are two schools of thought on the best design for product development teams. One believes that a single team should work on a product from the ideation stage through commercialization and the launch. The second believes that there should be two teams involved. The first team, which works on design and development, while the second team works on commercialization. The theory is that design and development requires a different skill set than commercialization. Although this is true, the downside risk is that in the transition from team to team, there is a risk that there will be a shift in the objectives or a change or modification of some critical element of the product.

Last, there is a potential for conflict of interests between the groups. In either case, the key is to create a team and an environment where all of the players feel involved and committed to the overall success of the product, whether they are from packaging, R&D, sensory, manufacturing, finance, marketing, or consumer research. Their involvement wilt keep the project grounded in reality and increase the product's chance for success (Gruenwald, 1992).

In order to optimize the product and maximize its potential, the new product development team needs time to create, refine, and test the product and money to provide the necessary resources to do their jobs correctly. Cutting short on either of these two things can seriously jeopardize the successful launch of the product by compromising the product's potential (Crawford, 1997).

The first step is to undertake a strategic assessment of what platforms should provide the basis for new food/beverage product development. It is absolutely critical to understand the core competencies and the unique strengths that the company has. A company's core competencies could be formulation ability, manufacturing and production capabilities, legal, distribution, sales, and/or marketing prowess.

After identifying consumer trends and determining the company's strengths and competencies, a company is ready to develop products. First, establish clear benchmark introductory criteria for each phase of food/beverage product development.

Criteria should be based on tangible measurable markers such as purchase intent, household penetration, revenue potential, or payout potential (Cooper, 1993).

New products play several roles for the organization. They help maintain growth and thereby protect the interests of investors, employees, suppliers of the organization. New products help keep the firm competitive in a changing market. The consequences of product development have a direct impact on competitiveness. (Robinson, 2000).

Product Development And Sustainability

Research in new product development (NPD) has been of interest for several decades. NPD attracts researchers being interested in engineering (Perrone et al., 2010), collaboration aspects (e.g. Emden et al., 2006), with regard to globalization efforts and green NPD.

New product development indicates a transformation of a market opportunity and a set of assumptions about a product technology into a product available for sale with cross-functional integration and quick development cycles. Following a market opportunity is essential, which nowadays is asking for products with sustainable characteristics (Bevilacqua et al., 2007).

Sustainable products, however, require internal and external interaction and collaboration in new product development. Consequently, collaboration in NPD processes across companies may provide long-term advantages for a new product development (Moreno et al., 2011).

Researchers describe that processes of product development within a company are linked to other internal processes thus requiring collaboration. Collaboration is herewith extended across companies. Cross-functional collaboration among departments is seen a success factor for sustainability in NPD (Petala et al., 2010). However, in order to achieve as sustainable product development, the social aspect needs to be addressed as well. It was already mentioned in the 1970s that social aspects of sustainability efforts need to be considered. Yet, the concentration on it decreased due to the strong focus on environmental improvements .

Nowadays the importance of social factors for products and production is of greater interest than it used to be (Aguilera et al., 2007). However, it is still said that the social dimension has often been neglected (Mu et al., 2011).

Risk Management

New product development is inherently linked to taking and managing risks (Baba etal.,1995;Kwak and LaPlace,2005), as most activities can be interpreted as a structured reduction of uncertainty. Studies of customer needs and market trends reduce the uncertainty surrounding requirements; technology development, testing and evaluation create certainty regarding the capabilities and cost of new technologies, and the improvement and standardization of NPD processes within the organization increases there liability of executing the development process (Mu etal.,2009). Reducing risks in new product development can also increase customer value, and can be used as a lens to analyze and optimize product development processes (Oehmen and Seering,2011). More generally, improving the “risk-return balance” is a central tenant of business decision.

A number of prior studies have pointed out that risk management in general is an important contributor to new product development program success: For example,(Mu etal.(2009) show that risk management strategies targeting technological, organizational and marketing risk factors improve NPD performance individually and interactively. The study by Jacob and Kwak (2003) highlights the positive contribution of risk management to improve project selection, review and resource allocation of NPD projects. In their investigation of over 100 technology-related projects, Raz et al.(2002) show that the use of risk management practices contribute to project success. In a recent large scale survey of 700 project managers on risk management in general project management, Zwikael and Ahn (2011) showed that even moderate risk management efforts increased project performance. Also, a recent study carried out by one of the authors indicates that a lack of proper risk management is one of the 10 major challenges that plague large-scale new product development programs, and that conversely the introduction of efficient risk management practices is a contributor to increased performance (Oehmen et al.,2012).

Implicit in the scale-up and trial production of the new food product is a total quality program that continuously identifies, analyses and controls risk. The risk controlling process begins with the identification of all potential hazards and proceeds through the screening, analysis ranking, quantification, and evaluation stages, ultimately to the controlling of the risks (Rudolph, 2000) (Figure 1).

A hazard analysis critical control point (HACCP) matrix is a useful tool for identifying and prioritizing hazards which may affect food product quality. Such a matrix has the following elements:

- identification of critical control point
- evaluation of hazard potential
- assignment of degree of concern (low, medium, high)
- development of criteria for hazard control
- preparation of monitoring/verification procedures
- designation of corrective action alternatives that may be required

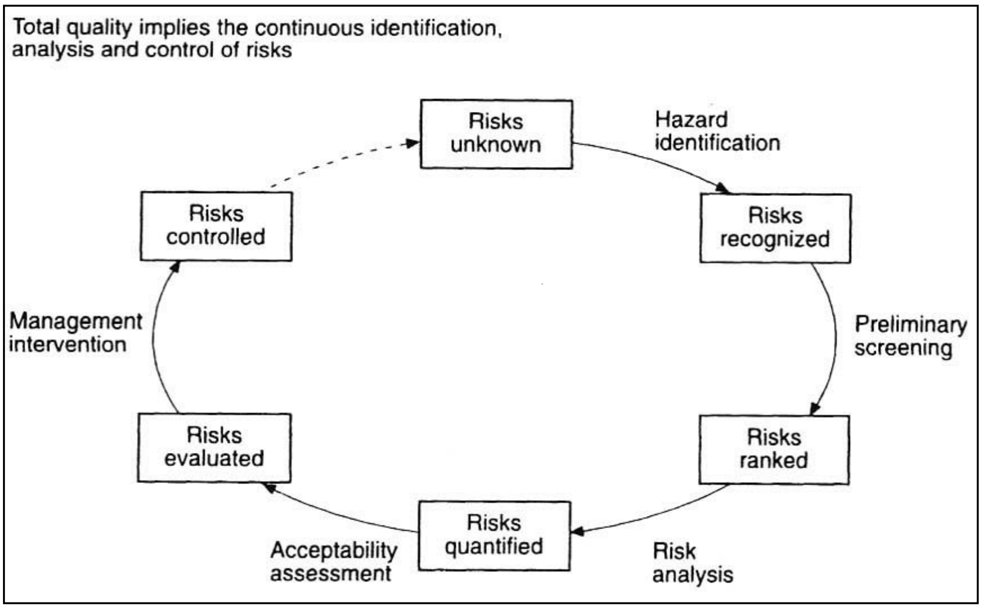

FIG 1.Food industry risk controlling process.

Because food safety is always of paramount concern, new products often linger or die at this point in the process if the issues cannot be resolved satisfactorily (Morris, 1993).

The product introduction milestone is led by sales but supported through all other functional areas, especially marketing and distribution. Field trials have been completed and the product is designed to meet the needs of the consumer. The product has been packaged and priced appropriately to convey the correct messages of quality and value. Packaging for transport has been tested and the product has been distributed in a timely and correct fashion so that it flows through the distribution system without impediments.

This phase is perhaps the most exciting and anxious, where customers see the product for what it is. Their initial response generally reveals the potential for success or failure of the product (Rudolph, 2000).

Example Of a Consolidated Food Product: Nutella Ferrero

With a turnover of more than 7 billion euro and a workforce of about 22,000 people in its various facilities in Italy and abroad, the Ferrero Group is a leading player in the international confectionery industry. Every year, investments are made in research and development, including by updating production plants. Apart from innovation, research and development, the largest share of investments goes into energy and environment sustainability, with special focus on production plants in Italy, Germany, Poland and Belgium. The Group also continues to implement initiatives in the lesser-developed Countries by means of the Ferrero social enterprise model.

Among the Group's guiding values stands out its constant focus on consumer needs, a factor at the bottom of a corporate policy which aspires to utmost transparency and quality excellence (Ferrero Group Online Archive).

The development of Nutella spread dates back to the Second World War.

In the hungry months after the end of World War Two, a young confectioner has a vision of an affordable luxury made of a small amount of cocoa and lots of hazelnuts. His name: Pietro Ferrero.

The way the family tells the story, it's a modern fairytale. Pietro was a humble man who lived in an enchanting region famed throughout the land for its delicious and abundant hazelnuts. Times were hard and chocolaty delights were not for the common people. Still, he dreamed of a magic formula that would enable everyone to enjoy his sweet treats.

There's a happy ending, too. Ferrero's tiny business in the picturesque town of Alba goes on to become the fourth most important international group in the chocolate confectionery market, with an annual turnover of more than 8bn euros (£6.5bn; $11bn r) (Mitzman, 2014).

FIG 2.The first chocolate laboratory in Alba of Ferrero Group.

When Pietro had his vision, the Piedmont region of Italy, and its capital Turin, was already famed for its chocolate industry. It was the birthplace of Gianduja, a creamy combination of chocolate and hazelnuts. But only the rich could think of buying it.

But in 1946 his grandfather launched Giandujot, or Pasta Gianduja. Produced as loaves wrapped in aluminium foil, it was a sort of solidified Nutella that had to be cut with a knife. The first spreadable version - Supercrema - came a few years later (Figure 3).

"This was a big success," says Giovanni. "It was the first brand that allowed people to enjoy confectionery at a very accessible price, even if it was not fully confectionery. This is how everything started."Spreadability meant that a small amount went a long way, helping to break down the perception that chocolate was, as Giovanni puts it, "only for very special occasions and celebrations like Christmas and Easter".

It could also be eaten with bread, which formed a big part of the diet at the time. People who never ate chocolate got the Supercrema habit.

FIG 3. Examples of product presentation.

The name gave the product instant international appeal. It said nuts. It also said Italy - "-ella" being a common affectionate or diminutive ending in Italian, as in mozzarella (cheese), tagliatella (a form of pasta), or caramella (Italian for a candy).

Fifty years on, Nutella is a global phenomenon, produced in 11 factories worldwide, and accounting for one fifth of the Ferrero Group's turnover, along with other products such as Kinder and Ferrero Rocher chocolates. The company is the number one user of hazelnuts in the world, buying up 25% of the entire world production (Mitzman, 2014).

As described so far is not always attributable to the process of innovation characterizing small and medium-sized enterprises, for which very often all that is left to the providers themselves; the latter are in most cases to launch new ideas and similarly to outline the new market trends. To mediate between the two different ways in which we decline the concept of innovation, between the large and the small and medium enterprise, there is the innovation understood as "problem solving", or develop a new product starting from the demands of the market and in particular customer.

In these three years of research carried out in a small enterprise in addition to solving and responding to problems reported by client companies, it was possible to develop three new products, which I will discuss below, through an approach that went beyond the "problem solving", and then studying in more detail the features, functionality, and all possible fields of application, in addition to the one from which the 'idea started.

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Chapter 1

A Hygroscopic Mix For Stuffed Pasta

ABSTRACT

A potential cause of degradation of food products is represented by changes in humidity, due to transfers to and from the atmospheric environment or to the interior of a package. However, migration of water inside the food may cause unwanted alterations, though not compromising the safety of food products. The addition of ingredients or mixtures of ingredients with hygroscopic activity can avoid these phenomena of mass transfer between heterogeneous phases within the food. The aim of this work was to obtain the selection of formulations with this capacity, by the estimation of some key parameters for their prior analysis. Measurements of initial Water Activity (Aw) and of water retention capacity (WRC) were taken on different ingredients (taken individually and diversely mixed), and finally adsorption isotherms were built.

We examined different powdered ingredients, grouped into STARCHES (obtained from different backgrounds and from different production processes), FIBERS (pea, bamboo, wheat, potato) and DAIRY (milk powder, caseinates, proteins whey and whey). The results obtained from the determinations listed above allowed to select the most suitable ingredients for this study, and to identify the best combinations with hygroscopic properties. Two different powder formulations have been developed, called MIX MILK and MIX STARCH, and they were subsequently used in an experiment conducted at the industrial level. Within the experiment, the formulations were added in stuffings with ricotta and spinach for the preparation of tortelli. The aim was to evaluate the ability of the above formulated in slowing the transfer of moisture from the filling to pastry. During such testing, which lasted for about two months, it was observed that the two experimental MIX are able to retain water within the filling for a longer time, compared to what was observed in stuffed pasta control, ie without both formulations. The slowdown of the passage of water from the filling to dough involves a series of positive effects throughout the shelf-life of the product examined. In particular, it is possible to observe the maintenance of a greater creaminess and softness of the filling, maintaining a turgid appearance of stuffed pasta and a right moisture level of the dough; on the contrary the ravioli control, in the same period of observation provided by the experimentation, show a marked wrinkling and their filling is more dry and hardened.

The preventive estimation of the hygroscopic properties of ingredients and additives for food has thus proved a valuable experimental approach, which allows to "predict" what ingredients are most indicated to act as modulators of the transfer of moisture between food and environment or between different phases of the same food, but also what formulations can be prepared for doing so.

Introduction

Fresh Stuffed Pasta

Typically, pasta is a noodle made from an unleavened dough of a durum wheat flour mixed with water and formed into sheets or various shapes, then cooked and served in any number of dishes. It can be made with flour from other cereals or grains, and eggs may be used instead of water. Pastas may be divided into two broad categories, dried and fresh.

The History Of Pasta

There are a lot of theories concerning the origin of the pasta product. Some researchers place its discovery in the XIII Century by Marco Polo, who introduced pasta in Italy returning from one of his trips to China in 1271. On chapter CLXXI from the "Books of the World's Wonders", Marco Polo makes a reference to the pasta in China. In our opinion, the pasta dates much further back, maybe to ancient Etruscan civilizations, which made pasta by grinding several cereals and grains and then mixing them with water, producing a blend that after cooking became a tasty and nutritious food product.

When the Greeks founded Naples, they adopted a dish made by the natives, made up by barley-flour pasta and water dried to the sun, called "macaria". We also find references to pasta dishes in the ancient Rome, which date back to the III century before Christ. In fact, the Roman emperor Cicero himself speaks about his passion for the "Laganum", the "laganas", which are strips of long pasta (wheat-flour pasta shaped as wide and flat sheets). During that time, the Romans developed instruments, tools, procedures (machines) to manufacture the pasta for lasagna. Ever since, cereals have exhibited great facilities for both its transportation as well as its storage. It was the Roman expansion and dominion which fostered the harvest of cereals in the whole Mediterranean basin.

Sicily was Rome's main granary, but cereals were also "imported" from the Mediterranean strip of Africa and the Lebanon, and later on, during Augustus' empire, from Spain, Sardinia and Syria. About 400,000 tons per year are estimated. The guaranty that each inhabitant could receive the necessary amount of cereals depended on the maritime traffic, subjected to storms, shipwrecks and inexperience and it also depended on the storage capability.

The term macaroni, with which a type of long pasta is currently referred to, was cited in Roman writings since the first centuries of our era. Platina, curator of the Vatican library, wrote in the XII century that macaroni with cheese were a legacy from the kitchens of Genoa and Naples. In a book called "The Cooking Pan" (XIII century), it was reported that lasagna were eaten as pasta strips in enriched broths. The term "laganas" is found in the book named "Of the Culinary Art" from Apicio. In a Codex from the XIII century, which can be found in the University of Bologna, we can reed the description on how lasagna is made.

On the other hand, it very likely that pasta reached Naples from Greece; Naples was once a colony of the Greek empire. In modern history pasta was already widely accepted in Spain, most of all starting from the XVI century, the era of the Spanish viceroyalty in Naples.

Hence, fresh pasta was used and appreciated in Italy since the time of the Etruscan and the Romans. During centuries, pasta continuously appears on the Italian tables with different names: for example, in the 1400 pasta was called "lasagna" and pasta manufacturers were referred to as "lasagnari". In the 1800 the name was changed and they were referred to as "vermicellai". But between the 1400 and the 1800 between the "lasagne" and the "vermicelli" the "fidelli" were born; these were pasta thread with a cylindrical shape. In this way, the pasta manufacturers also became the "fidellai".

At the onset of the XVII century in Naples the first and elementary machines for the pasta production were developed; but a decidedly improvement in the pasta production process was reached in Gragnano, a small town few kilometers far from the city of Partenopea. In this site the drying pasta process was developed, thanks to the particular climate conditions created by the alternation of the dry Ponentino winds and the warm and humid Vesubiano winds, which generated the conditions for an excellent manufacturing process. The extension of the kneading machine and the invention of the press, made it possible to diminish the costs of producing pasta.

In 1740, in the city of Venice, Paolo Adami, obtained the license to open the first pasta factory. A hundred years thereafter, in Amalfi, water mills and stone grinders for the separation of semolina from the bran started to be used, contributing to the improvement of the product quality.

At the beginning of the XIX century, pasta dishes triumphed on the tables of the nobility, and little by little they became a gastronomic habit in the upper classes. During this century the dry pasta consumption spread quickly among the whole Italian society and its offering to guests became a sign of distinction. Up until now, pasta was eaten by hands, but the complexity reached by the recipes make this way of eating pasta no longer the most adequate one. Hence, fork started to be used at the tables of the upper classes. When eating pasta with tomato sauce became a general issue, the fork was then adopted as an everyday tool, but a new fork specifically made to eat pasta was developed, with four curved tips with a length not larger than twice its total width.

In 1878, the Marsellais Purifier was invented; the name sprang from the inventor himself, and it was used to improve the semolina and, hence, to improve the pasta. First, the pasta was seasoned with tomato sauce and olive oil, then it was enriched by the creativity of housewives, chefs and gourmets, who started to mix it with typical Italian products such as mozzarella cheese, Parmesan cheese, ham, and cured swine meat, in addition to other types of cheese, meats and fish. During the present century, pasta was consolidated as a main ingredient of the Italian cuisine.

Since 1914, the artificial drying process allowed the pasta to be available in all Italian regions. The great development of the Italian pasta at the turn of the century was tightly linked to the export, which reached a record level of 70,000 tons, many of which were sent to the United States of America. Later on, importing countries started to produce machines to manufacture their own pasta products, and these managed to conquer the world, and this phenomenon was called “The Industry of Pasta”.

Starting from the egg pasta, also in the Middle Ages the various regional traditions of stuffed pasta came to life, which will take the name of agnolini, in Lombardy, agnolotti in Piedmont, anolini in Parma and Piacenza, tortellini between Modena and Bologna, cappelletti in Romagna, ravioli in Rome and Lazio. The tortelli, cappelletti, anolini are mentioned in the Work of Scappi, private cook of Pope Pius V. The book is the most respected and comprehensive treaty of Renaissance gastronomy. At first, it fully confirmed that tortellini, cappelletti and anolini have a common origin. Moreover Scappi reports in his work the recipes of tortelletti, a mixture of meat, pulp capon, beef marrow or tortelletti of grass or peas that do not contain meat, but vegetables or legumes, cheese, eggs and spices and are similar our green tortelli. The literature of the history of fresh stuffed pasta preceding the work of Scappi is not so easy. Ten years before the publication of the Opera (1570) the Venetian publisher Michele Tramezzino published "The Singular Doctrine" of M. Domenico Romoli, a learned dissertation which together with the Scappi’s work represents the benchmark of the sixteenth century Italian cuisine (The History of PASTA).

Pasta Market In European Union

Most categories of pasta in the E.U. are forecasted to continue growing in the next five years, with the exception of canned pasta (Figure 1.1).

Several factors should drive pasta sales between 2008 and 2013. Consumers are moving to cooking and entertaining at home to save money, thereby presenting an opportunity for pasta meals. Convenience and easy preparation are the main features of many pasta products. Many efforts by manufacturers, like inventing new recipes and packaging formats for chilled/fresh products and the continued introduction of value added product formulations in dried pasta, will create demand in the future.

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EU 27	2006	2007	2008	2013	03-08 CAGR	08-13 CAGR
Pasta	7,771	8,009	8,361	10,138	2.5%	3.9%
Canned/preserved pasta	422	408	395	371	-3.0%	-1.2%
Dried pasta	5,350	5,488	5,722	6,871	1.8%	3.7%
Chilled/fresh pasta	1,999	2,113	2,244	2,896	5.7%	5.2%

FIG 1.1 EU Retail Sales of Pasta by Subsector, Historic/ Forecast, US $ million (*Source: Euromonitor)*

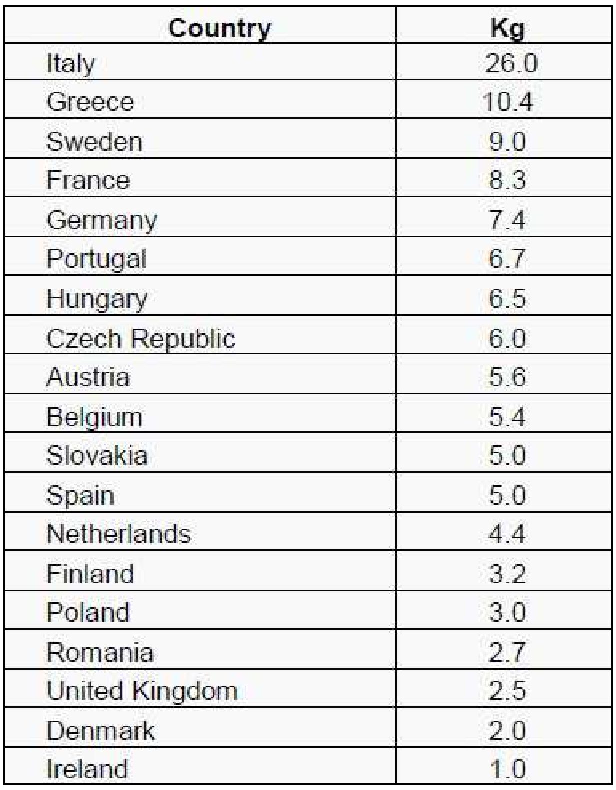
In Western Europe, value-added products, such as chilled/fresh pasta, are expected to continue to appeal the consumers, and will benefit from further efforts by manufacturers to provide new recipes and new packaging.

The value share of stuffed pasta is expected to rise sharply, given the considerable potential for innovation in this area and the innovation already initiated by manufacturers. In general, increasing numbers of single-person households will boost sales of value-added chilled/fresh products, and canned products.

In Eastern Europe, eating habits are changing to emphasize convenience, freshness and variety, all of which are offered by pasta. This trend is expected to boost sales from 2008 to 2013.

The health-conscious eating trend has also affected sales of pasta, with increased demand for whole grain pasta and pasta made with fresh ingredients. A few producers have already launched whole grain pasta, and they are expected to be followed by other players in the future.

In recent years, concepts of stuffed and flavored have sustained the development of new pasta shapes, and manufacturers are exploring such options as organic, fortified and enriched pasta (The World Pasta Industry Status Report 2012).



Country	Kg
Italy	26.0
Greece	10.4
Sweden	9.0
France	8.3
Germany	7.4
Portugal	6.7
Hungary	6.5
Czech Republic	6.0
Austria	5.6
Belgium	5.4
Slovakia	5.0
Spain	5.0
Netherlands	4.4
Finland	3.2
Poland	3.0
Romania	2.7
United Kingdom	2.5
Denmark	2.0
Ireland	1.0

FIG 1.2. Top EU Per Capita Consumption of Pasta
(Source: Unione Industriali Pastai Italiani)

In recent years, retailers and manufacturers have become aware of the great potential for chilled pasta and have intensified their efforts in terms of distribution and marketing (Figure 1.2 and Figure 1.3).

Private label sales were aided by price competition in the pasta sector since 2005, particularly in the developing markets of Western Europe. A feature of private label sales was the availability of premium products, including organic pasta, sold by gourmet and natural food outlets.

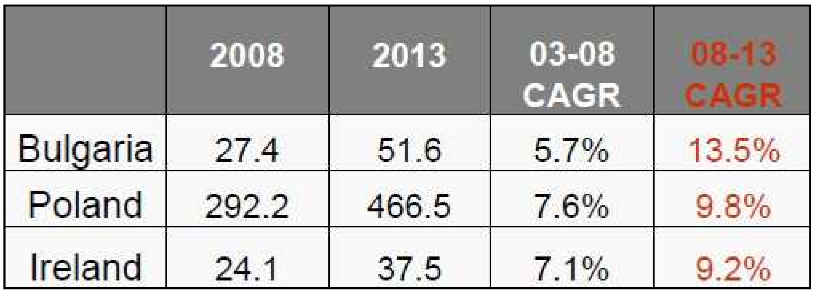
Pasta	2006	2007	2008	2009	2013	03-08 CAGR	08-13 CAGR
Bulgaria	23	25	27	32	52	6%	13%
Ireland	34	36	40	44	60	7%	9%
Finland	43	43	50	54	65	1%	5%
Slovakia	60	63	65	68	81	4%	5%
Portugal	68	70	74	77	89	2%	4%
Denmark	64	69	76	80	96	4%	5%
Austria	88	89	90	92	100	2%	2%
Hungary	108	114	118	127	156	3%	6%
Czech Republic	101	107	123	134	191	8%	9%
Greece	139	145	155	159	182	3%	3%
Sweden	145	149	156	163	187	3%	4%
Netherlands	153	157	161	169	206	2%	5%
Romania	143	157	169	184	244	10%	8%
Belgium	167	176	215	233	310	8%	8%
Poland	254	265	293	331	467	8%	10%
Spain	370	389	412	431	509	4%	4%
United Kingdom	887	902	926	953	1,050	2%	3%
France	1,120	1,133	1,156	1,179	1,247	1%	2%
Germany	1,294	1,331	1,364	1,402	1,550	3%	3%
Italy	2,511	2,588	2,691	2,809	3,295	2%	4%

FIG 1.3. Retail Sales of Pasta by Region, Historic/ Forecast, US $ million
(Source: Euromonitor)

Dried pasta is available in many sizes and shapes. It is factory made and fully dried before it is packaged. It rarely contains eggs, so it has a long shelf life requiring no refrigeration or freezing. Its firmness also allows it to be stored easily without damage.

Most consumers prefer dried pasta with no added flavour, although coloured dried pasta seasoned with spinach, beetroot or tomato is also popular. Traditional varieties such as spaghetti, rigatoni and macaroni, are the most popular forms.

Product innovation, value-added product and low cost will contribute to positive growth for dried pasta (Figure 1.4). Sales value of dried pasta, accounted for 68% of the overall category in 2008, and are expected to grow by 3.7% from 2008 to 2013.



	2008	2013	03-08 CAGR	08-13 CAGR
Bulgaria	27.4	51.6	5.7%	13.5%
Poland	292.2	466.5	7.6%	9.8%
Ireland	24.1	37.5	7.1%	9.2%

FIG 1.4. Fastest Growing Markets for Dried Pasta, US $ Million
Source: Euromonitor

Fresh pasta is available in a variety of sizes and shapes, but not to the extent of dried pasta. Fresh pasta most often contains eggs and has high water content, making refrigeration or freezing necessary to keep it from spoiling (Pasta Market in EU. April 2010).

In comparison with other types of pasta, the chilled/fresh format benefited from positive consumer perceptions of its high quality, taste and health value, combined with an easy preparation. Consumer willingness to pay premium prices for such products boosted sales in key markets such as Italy, Germany and the United Kingdom (U.K.) .

It is expected that chilled/fresh pasta will grow by 5.2% in sales value from 2008 to 2013 (Figure 1.5).

	2008	2013	03-08 CAGR	08-13 CAGR
Germany	281.5	355	7.7%	4.7%
Italy	995.9	1326.7	5.5%	5.9%
U.K.	294.5	368.4	8.6%	4.6%

FIG1.5. Fastest Growing Markets for Fresh Pasta, US $ Million
(Source: Euromonitor)

Focus On Filled Pasta Production

Fresh filled pasta is a typical Italian product. This product reached remarkable interest for several years, not only in Italy, but also in other countries, such as the United Kingdom, Germany, France and United States of America.

Several constituents of this food can be identified, bearing in mind that some products should be consumed in a few days, while others have shelf-life of 120 days. Such significant differences result from different production processes and technologies.

From the scientific point of view, pasta being starchy, presents problems due to products associated with any food of animal origin, including eggs, as used in dough and due to the presence of meat and/or milk ingredients.

The factors that make the product as potentially "at risk", from a microbiological point of view , include composition, relative high pH, and high water activity values.

The production technology, conditioning, and conservation utilized make it "safe" from the sanitary point of view. The thermal treatment leads to a reduction of the microbial populations and also influences the residual microbiological activity due to the water activity reduction and to the structure conferred to the pasta. This treatment reduces the microbiological populations to level of absolute safety, while maintains sensorial and nutritional characteristics of the fresh product (Zardetto et al., 2007).

The basic production process is, with some variations, substantially as follows (Figure 1.6):

- components of the pasta production recipe are assembled (automatic or manual)
- mixing
- sheeting-roll or extrusion of the pasta and addition of stuffing in the case of filled pasta
- pasteurisation of the unpacked product: (1) first thermal treatment
- superficial drying
- cooling
- packing under a modified atmosphere
- optional pasteurisation of the packed product: (2) second thermal treatment

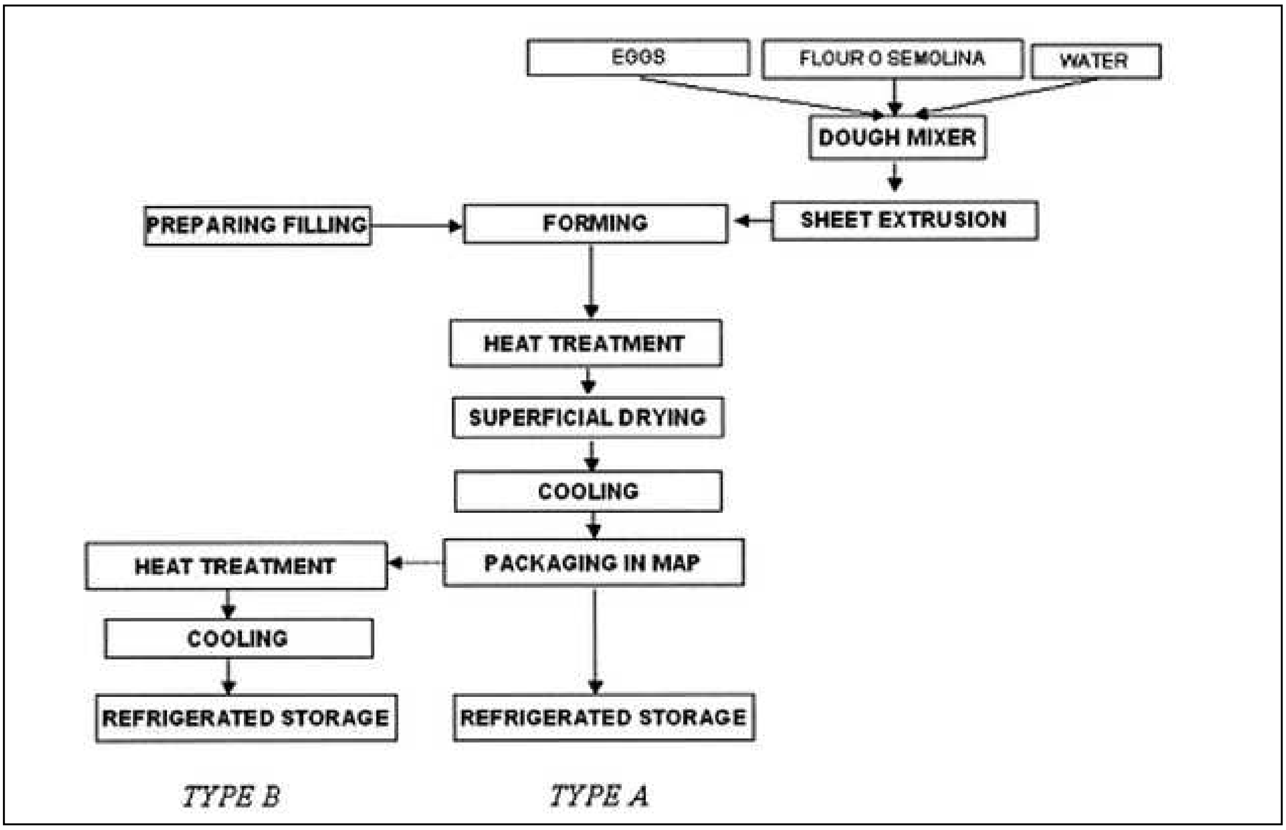


FIG 1.6. Flow chart relative to the production of two types of filled pasta subjected to different treatments.

The addition of other stages in the basic manufacture process and the different ways that they are combined also should be considered due to product diversification.

From the flow chart in Figure 1.6 it can be seen that the product can be subject to two thermal treatments. A single thermal treatment (product type A) mantains the sensorial and nutritional properties of the product and also its image of a "fresh" product. Instead, double pasteurisation (product type B) is used with the aim to obtain a more efficient thermal distribution and permits omission of sterile conditions in the downstream stages, even though good hygienic standards must in any case be observed. The second thermal treatment prolongs the product shelf-life by up 90 days or more, but produces a loss of sensorial characteristics and there is a significant increase in manufacturing costs (Zardetto et al., 2007; Batisti et al., 1995).

Food Preservation And New Product Development

Food preservation involves several actions whose aim is to maintain ad long as possible with the initial quality of the food products. Food preservation, however, must comply with the concepts of sustainability and safety. The United Nations World Commission on Environment and Development defined sustainable development as “meeting the needs of the present generation without compromising the ability of future generations to meet their own needs.” A sustainable way of designing and developing food products stands to appeal to consumers, and provides a point of differentiation from competitors and a perfect platform for a range of positive public relations activities (French, 2004). Innovation is vital to maintain progress in technology and engineering. Food safety is now the first priority of the food production and preservation industry, incorporating innovation and sustainability. The industry can compromise with some quantities such as color to some extent, but not with safety.

The preservation and processing of food is not as simple or straightforward as it was in the past. A number of new preservation techniques are being developed to satisfy current demands of economic preservation and consumer satisfaction in nutritional and sensory aspects, convenience, safety, absence of chemical preservatives, price, and environmental safety. Understanding the effects of each preservation method on food has therefore become critical in all aspects (Rahman, 2007).

Food Preservation

Preservation methods start with the complete analysis and understanding of the whole food chain, including growing, harvesting, processing, packaging and distribution; thus an integrated approach needs to be applied. It lies at the heart of food science and technology, and it is the main purpose of food processing. First, it is important to identify the properties or characteristics that need to be preserved.

One property may be important for one product, but detrimental for others. For example, collapse and pore formation occur during the drying of foods. This can be desirable or undesirable depending on the desired quality of the dried product, for example, crust formation is desirable for long bowl life in the case of breakfast cereal ingredients, and quick rehydration is necessary (i.e., no crust and more open pores) for instant soup ingredients. In another instance, the consumer expects apple juice to be clear whereas orange juice could be cloudy (Rahman, 2007).

Why Preservation?

The main reasons for food preservation are to overcome inappropriate planning in agriculture, produce value-added products, and provide diet variation (Rahman, 1999).

Value-added food products can give better-quality foods in terms of improved nutritional, functional, convenience, and sensory properties. Consumer demand for healthier and more convenient foods also affects the way food is preserved. Eating should be pleasurable to the consumer, and not boring. People like to eat wide varieties of foods with different tastes and flavors. Variation in the diet is important, particularly in underdeveloped countries to reduce reliance on a specific type of grain (i.e., rice or wheat) (Rahman, 2007).

In food preservation, the most important points under consideration are:

- The desired level of quality
- The preservation length
- The group for whom the products are preserved

After storage of a preserved food for a certain period, one or more of its quality attributes may reach an undesirable level. Quality is an illusive, ever-changing concept. In general, it is defined as the degree of fitness for use or the condition indicated by the satisfaction level of consumers. When a food product is deteriorated to such an extent that it is considered unsuitable for consumption, it is said to have reached the end of its shelf life. In food products shelf life studies , it is important to measure the rate of change of a given quality attribute (Singh, 1994). In all cases, safety is the first attribute, followed by other quality indexes. The product quality attributes can be quite varied, such as appearance, sensory, or microbial characteristics, but ultimately the final use must provide a pleasurable experience for the consumer (Sebranek, 1996).

The major quality-loss mechanisms and consequences are shown in Table 1.1 and Figure 1.7. The required length of preservation depends on the purpose. In many cases, very prolonged storage or shelf life is not needed, which simplifies both the transport and marketing of the foodstuff.

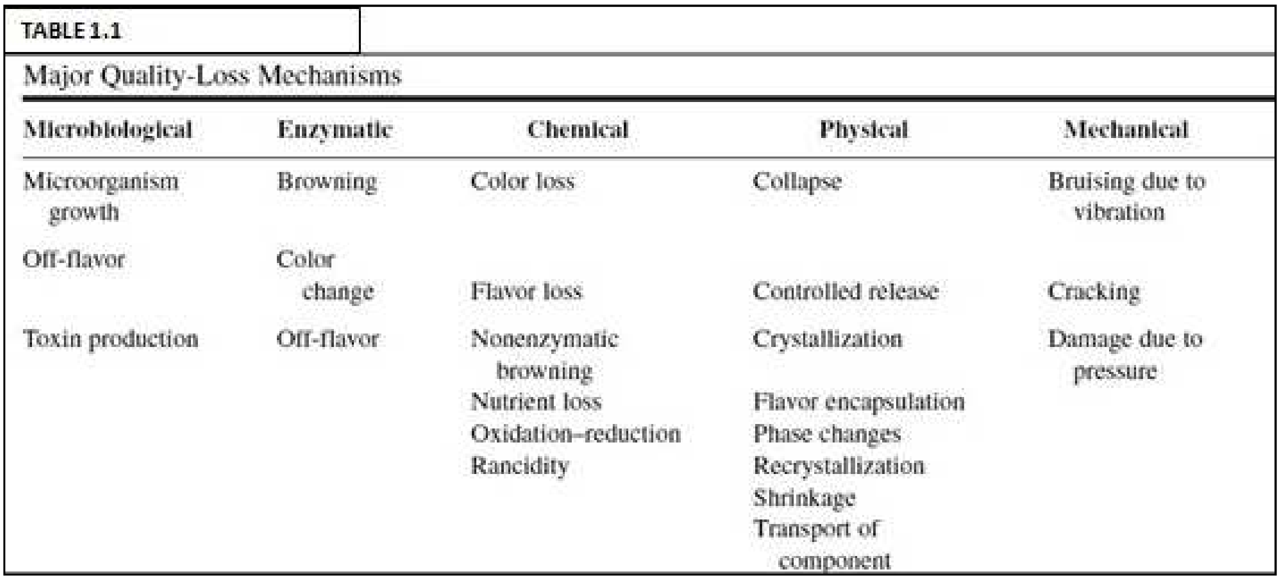


TABLE 1.1

Major Quality-Loss Mechanisms

Microbiological	Enzymatic	Chemical	Physical	Mechanical
Microorganism growth	Browning	Color loss	Collapse	Bruising due to vibration
Off-flavor	Color change	Flavor loss	Controlled release	Cracking
Toxin production	Off-flavor	Nonenzymatic browning	Crystallization	Damage due to pressure
		Nutrient loss	Flavor encapsulation	
		Oxidation–reduction	Phase changes	
		Rancidity	Recrystallization	
			Shrinkage	
			Transport of component	

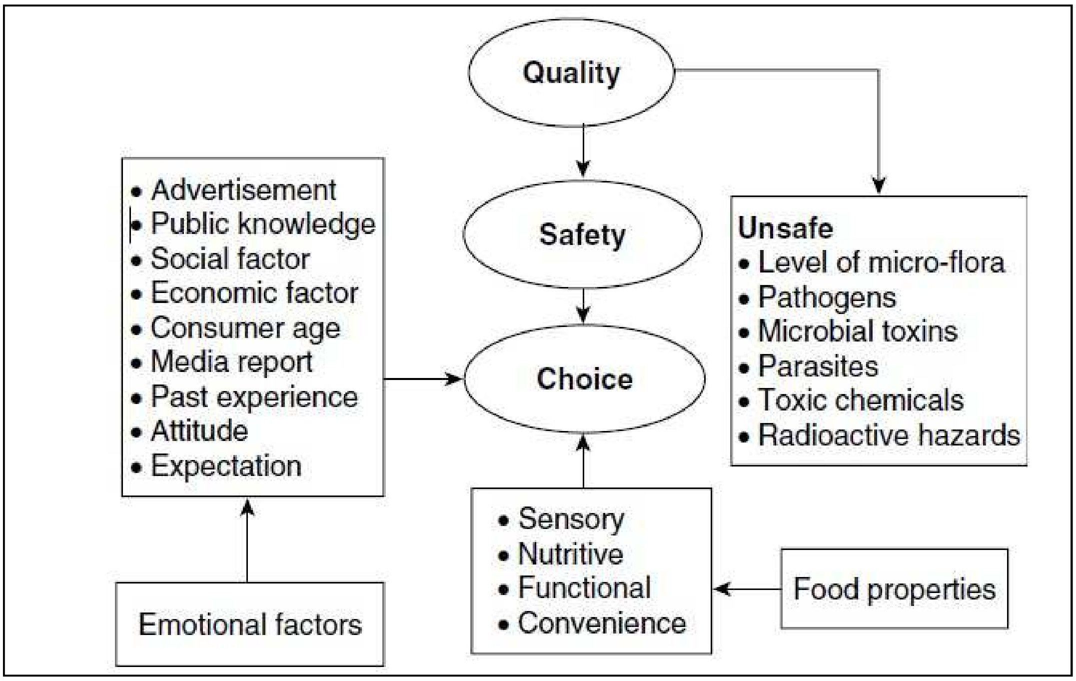


FIG 1.7. Factors affecting food quality, safety, and choice.

For Whom To Preserve?

It is important to know for whom the preserved food is being produced.

Nutritional requirements and food restrictions apply differently to different population groups. Food poisoning can be fatal, especially in infants, pregnant women, the elderly, and those with depressed immune systems.

The legal aspects of food preservation are different in case of foods produced for human and for animal consumption. Thus, it is necessary to consider the group for whom the products are being manufactured (Rahaman, 2007).

Causes Of Food Deterioration

Mechanical, physical, chemical, and biological reactions are the main causes of food deterioration and spoilage. Damage can start at the initial point by mishandling of foods during harvesting, processing, and distribution; this may lead to ultimate reduction of shelf life. Other examples of deterioration can be listed as follows: (i) bruising of fruits and vegetables during harvesting and postharvest handing, leading to the development of rot, (ii) tuberous and leafy vegetables lose water when kept in atmospheres with low humidity and, subsequently, wilt, and (iii) dried foods kept in high humidity may pick up moisture and become soggy.

During storage and distribution, foods are exposed to a wide range of environmental conditions (Bengtsson, 2003).

Environmental factors such as pressure, temperature, humidity, oxygen, and light can trigger several reactions that may lead to food degradation. As a consequence of these mechanisms, foods may be altered to such an extent that they are either rejected by or harmful to the consumer (Singh, 1994). Condensation of moisture on foods or a damp atmosphere favors microbial growth, occasionally promotes insects development, and may indirectly lead to deterioration, resulting in destructive self-heating (Borgstrom, 1968).

Mechanical damage (e.g., bruises and wounds) is conducive to spoilage, and it frequently causes further chemical and microbial deterioration. Peels, skins, and shells constitute natural protection against this kind of spoilage (Borgstrom, 1968).

Each microorganism has (i) an optimum growing temperature, (ii) a minimum temperature below which growth takes place no longer, and (iii) a maximum temperature above which all development is suppressed.

Microbial growth in foods results in food spoilage with the development of undesirable sensory characteristics, and in certain cases the food may become unsafe for consumption. Microorganisms have the ability to multiply at high rates when favorable conditions occur.

Microbial or chemical contaminants are also of concern in food deterioration. Chemicals from packaging materials may also be a source of food contamination.

In addition to temperature, other environmental factors such as oxygen, water, and pH induce unwanted changes in foods that are catalyzed by enzymes (Singh, 1994).

Food Preservation Methods

Based on the mode of action, food preservation techniques can be categorized as (1) slowing down or inhibiting chemical deterioration and microbial growth, (2) directly inactivating bacteria, yeasts, molds, or enzymes, and (3) avoiding recontamination before and after processing (Gould, 1989; Gould 1995). A number of techniques or methods from the above categories are shown in Figure 1.8.

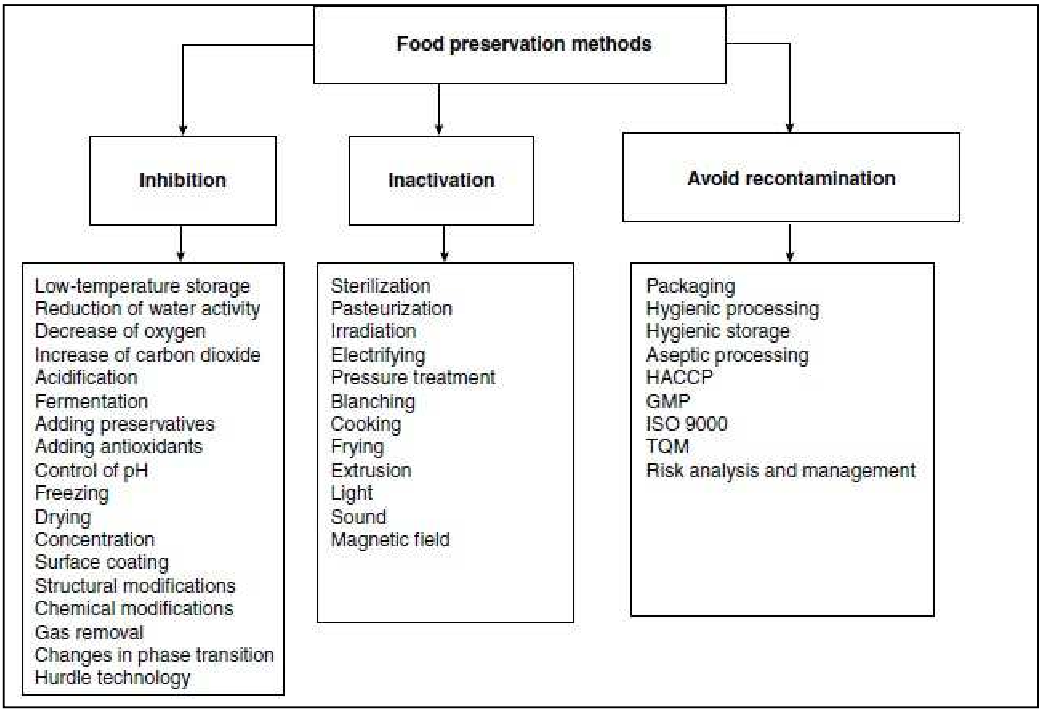


FIG 1.8. Major food preservation techniques

The methods based on inhibition include those that rely on control of the environment (e.g., temperature control), those that result from particular methods of processing (e.g., microstructural control), and those that depend on the intrinsic properties built into particular foods (e.g., control by the adjustment of water activity or pH value (Gould, 1995).

Water Activity And Food Preservation

Basics Of Water Activity

Water is an important constituent of all foods. Why water activity and not water content? In the middle of the nineteenth century, scientists began to discover the existence of a relation between water in a food and its relative tendency to spoil (Rahman et al., 2007). They also began to realize that the *active water* could be much more important to the stability of food than the total amount of water. Scott, 1953 (Scott, 1957) clearly identified that water activity of a medium correlated well with the deterioration of food stability due to the growth of microorganism. Thus, it was possible to develop generalized rules or limits for the stability of foods by using water activity. This was the main reason why food scientists started to emphasize water activity rather than water content. Since then, the scientific community has explored the great significance of water activity in determining the physical characteristics, processes, shelf life, and sensory properties of foods. The water activity of fresh foods, as shown by Chirife and Fontan (Chirife et al., 1982), is 0.970–0.996.

Water Activity

Water activity (a_w) is defined as the ratio of vapor pressure of water in a system and the vapor pressure of pure water at the same temperature, or the equilibrium relative humidity of the air surrounding the system at the same temperature. A number of methods have been reported in literature to measure or estimate the water activity of foods. Water activity measurement methods include the following: (i) equilibrium sorption rate method (isopiestic method), (ii) vapor pressure measurement method, and (iii) hygrometric instrument method. In addition, water activity can be predicted from other thermodynamic properties such as freezing point. The accuracy of most methods lies in the range of 0.01–0.02 water activity units (Rizvi, 1995). Details of the different measurement techniques are described by Labuza et al. 1976, Rizvi, 1995, Rahman, 1995, Rahman and Sablani, 2002, Rahman et al. 2001, Fontana, 2001, and Sablani et al., 2001.

Water activity can be lowered or controlled by several methods such as separating out of water and adding solutes.

Processes that can be used to remove water are drying, concentration, and dewatering by centrifuge.

Other unit operations such as baking, extrusion, and frying also reduced the water activity to some extent. Solutes can be added to foods to reduce water activity as well as improve the functional and sensory properties of foods, for example, adding salt to meat and fish, and adding sugars to fruits. When only solutes are used to reduce water activity, then the specific antimicrobial effects and the cost of solutes or humectants should be considered for food product formulation. The factors affecting the selection of humectants are summarized in Table 1.2.

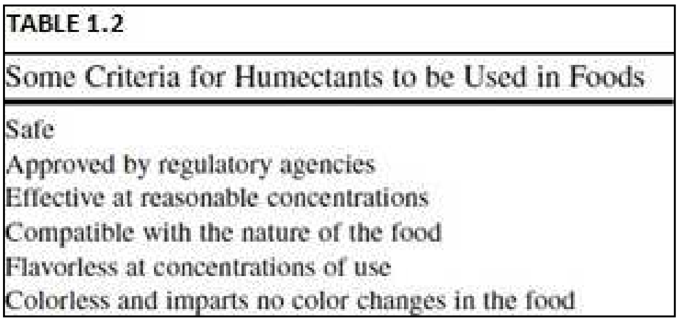


TABLE 1.2

Some Criteria for Humectants to be Used in Foods

Some Criteria for Humectants to be Used in Foods
Safe
Approved by regulatory agencies
Effective at reasonable concentrations
Compatible with the nature of the food
Flavorless at concentrations of use
Colorless and imparts no color changes in the food

Food Properties Which Influence Water Activity

Properties of the food affect the way that water interacts with its components. The three major physical effects that lower water activity are discussed below.

Colligative effect

From a purely kinetic perspective, if particles are added to a solvent, such as water, and they were the same size and density as the water without chemical interactions, the particles would interfere with the kinetic motion of water. At the surface of the solvent, as shown in figure 8, the particles wuold reduce the escaping tendency of the solvent. Thus, at equilibrium, there will be less solvent molecules in the vapor space as compared to a system consisting of pure solvent (Bell et al., 2000).

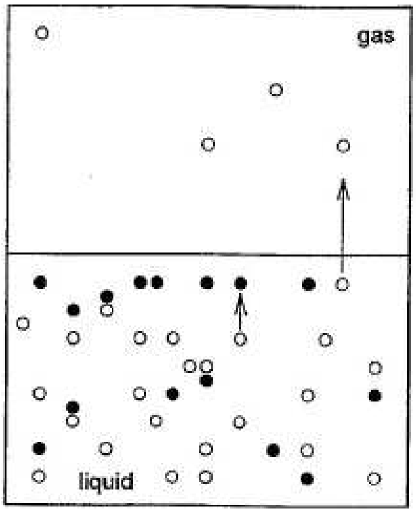


FIG 1.8. Effect of particles (•) on the vaporization ability of a solvent(o).

The relative activity of the solvent is reduced as shown by

$$\mathbf{a_s=f_s/f_o}$$

where $\mathbf{a_s}$ is the activity of the solvent, $\mathbf{f_s}$ is the fugacity of the solvent containg particles, $\mathbf{f_o}$
is the fugacity of the pure solvent.

Fugacity, which has units of pressure, is similar to partial pressure, except that fugacity is applied to non-ideal systems (Levine, 1988).

When a solute dissolves in water, it interacts with the water through dipolar, ionic and hydrogen bonds, resulting in a further decrease in the escaping tendency and thus chemical potentetial of water, and water activity is lowered even more.

The properties of solvent (e.g., water) influenced by the interaction with solutes are called " colligative properties" (Levine, 1988).

Capillary effect

Water activity value is also slowed down by the capillary effect. Food contains a lot of pores and capillaries in which water exists (Bluestein and Labuza, 1972; Karel,1973). The vapor pressure of water above the curved liquid meniscus in the pores is less than that of pure water, because of changes in the hydrogen bonding between water molecules. On this curved capillary surface, more water molecules interact with each other, which results in a lower a_w.

Surface interaction

In food products, water can react directly with other chemical groups on molecules through dipole-dipole forces, ionic bonds (H_3O^+ or OH^-), dipolar-ionic interactions, van der Waals forces, and hydrogen bonding. These chemical interactions contribute to diminish the value of the water activity and this effect occurs over the whole range of moisture contents but is more pronounced at low a_w (Bell et al., 2000).

Foods Water Sorption

The distribution at equilibrium of a component between two contacting phases, and in particular the distribution between a solid and a gaseous phase, can be represented by several types of curves. These include isotherms, isobars, and isosteres (curves representing equilibria at constant temperature, constant pressure, and constant water contents, respectively). These curves describe the relationships among three key variables: waterpartial pressure in the gaseous phase, water content, and temperature (Karel et al., 2003; Rahman et al., 2007).

The moisture sorption isotherm is usually represented in a graphical form or by means mathematical or thermodynamic formula. Brunauer et al., 1940, classified adsorption isotherms of materials into five general types (Figure 1.9).

If water soluble crystalline components are present in foods, e.g., sugars or salt, Type III is the typical adsorption isotherm. A lot of other different foods or formulations result in sigmoid isotherm type II. The inflection point of the isotherm indicates the change of water-binding capacity or of the relative amounts of free and bound water. Type I is indicative of a nonswelling porous solid, such as silicate anticaking agents.

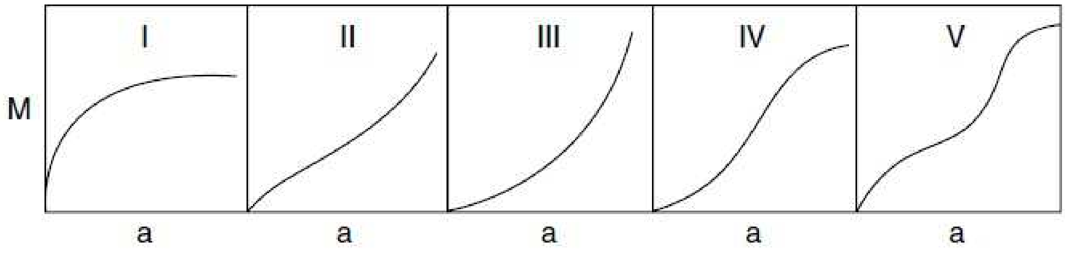


FIG 1.9. The five types of van der Waals adsorption isotherms proposed by Brunauer et al. 1940.

For practical purposes, the isotherm is presented in an empirical or theoretical model equation.

Two relations have been particularly significant in the study of sorption isotherms, namely the Brunauer–Emmet–Teller (BET) equation and the GAB equation of Guggenheim and co-workers (Van den Berg and Bruin, 1981). They are shown in Figure 1.10.

BET equation

$$m/m_1 = C\,a\,\{(1-a)\,[1+ Ca - a\,]\}^{-1}$$

GAB equation

$$m/m_1 = K'C\,a\,\{(1-C\,a)[1+K'C\,a - C\,a\,]\}^{-1}$$

FIG 1.10. The BET and the GAB equations for describing sorption isotherms.
m = water content, a= water activity, m_1 =parameter (monolayer value), K', C =parameters.

Isotherms of foods and food components are collected in several books, together with lists of BET and GAB constants values, which allow to build the adsorption isotherms of different food matrices.

Hysteresis

The difference in the equilibrium moisture content between the adsorption and desorption curves is called hysteresis and is shown in Figure 1.11. In region II of this figure, the water is bonded less tightly and it is usually present in small capillaries, whereas in region III, the water is kept loosely in large capillaries or is free (Fortes et al., 1980). Hysteresis in sorption has important theoretical and practical implications in foods. The theoretical implications evidences that of the sorption process is irreversible and states the validity of the equilibrium thermodynamic process. The practical implications deal with the effects of hysteresis on chemical and microbiological deterioration and its importance on low- and intermediate-moisture foods (Kapsalis, 1987). Strasser, 1969 and Wolf et al. , 1972, established that changes in hysteresis could be used as an index of quality deterioration, since hysteresis loops of foods change with storage time, but this is a poor method of evaluation.

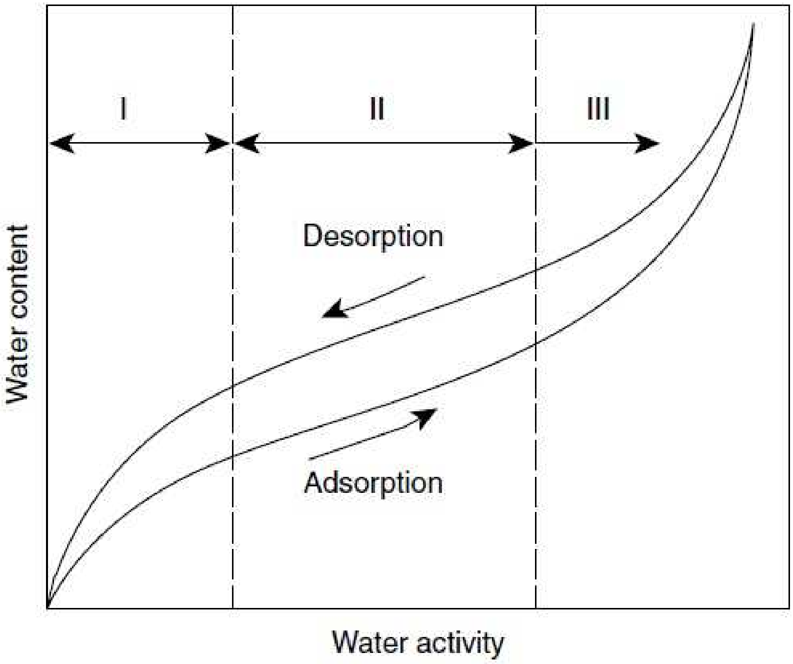


Fig 1.11: Sorption isotherm for typical food product showing hysteresis.

Total hysteresis decreases as sorption temperature increases (Weisser et al., 1982). The water activity shift caused by temperature is mainly due to the change in water binding, dissociation of water, physical state of water, or increase in the solubility of solute in water. It is widely accepted that an increase in temperature results in decreased equilibrium moisture content (Figure 1.12). Tsami et al., 1990 found similar results for the dried fruits up to a water activity of about 0.55–0.70. In that region, the curves for several temperatures intersect. At water activity values higher than 0.7, there was an inversion in the effect of temperature (i.e., equilibrium moisture content increased with temperature) due to an increase in solubility of sugars in water. The intersection (or inversion) point depends on the composition of the food and the solubility of sugars (Weisser et al., 1982).

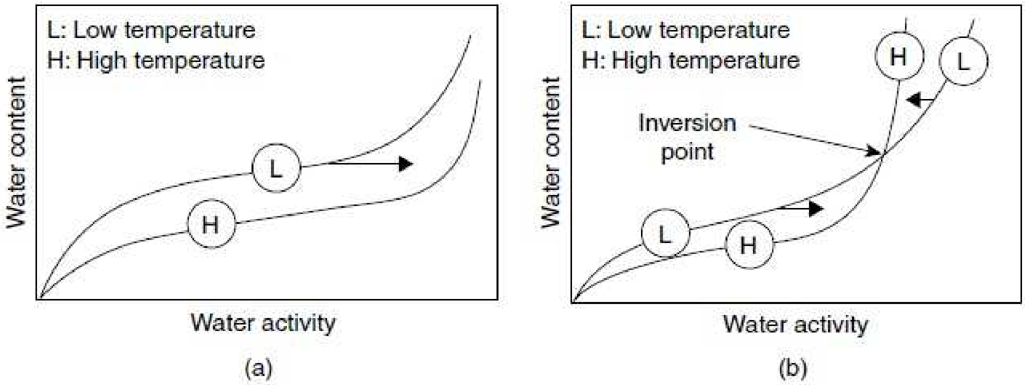


FIG 1.12. Water activity shift of food due to temperature. (a) Shift without intersection. (b) Shift showing the point of intersection or inversion point.

Food Stability Prediction With Isotherm

The moisture sorption isotherm is an extremely valuable tool for food scientists because of its usefulness in predicting food stability. Most foods have a critical moisture content below which the rate of quality loss is negligible. Quality is understood to include growth and toxin production by microorganisms as well as chemical deterioration and decrease of sensory intensity, such as crispness, hardness, caking, texture, color, flavor or aroma (Bell et al., 2000). A global food stability map is presented in Figure 1.13 (Labuza et al., 1970; Rockland et al., 1987).

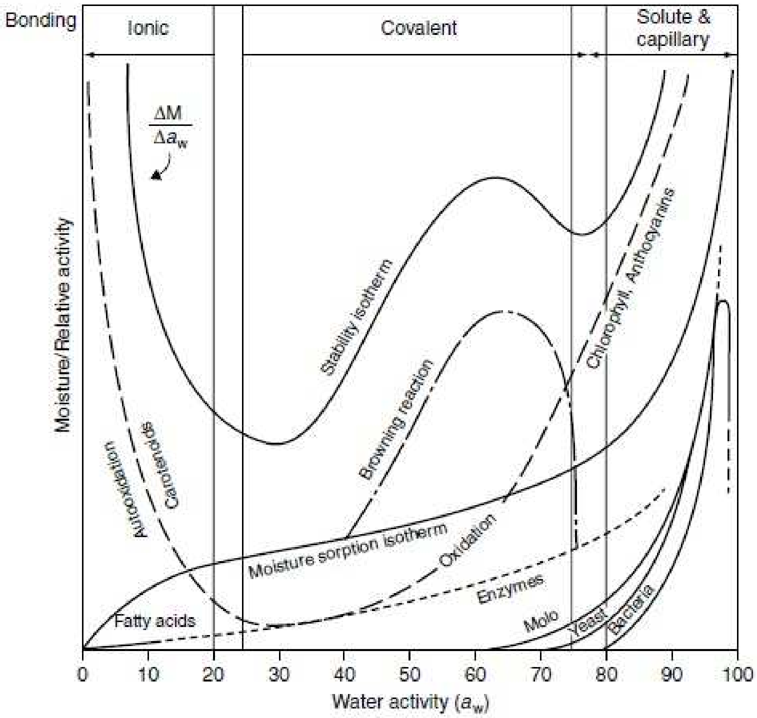


FIG 1.13. Food stability as a function of water activity.

As discussed by Labuza, 1984, the rate of quality loss begins to increase above water activity 0.2–0.3 for most chemical reactions (Figure 1.13). At this water activity, the amount of water adsorbed on surfaces and in capillaries is enough to affect the overall dielectric properties such that the water can now behave as a solvent. Thus, chemical species can dissolve, become mobile, and are reactive. The higher the water activity, the faster the reaction rate because of the greater solubility and increased mobility of the reactants. However, at some higher water activity no further species dissolve, and therefore an increase in water activity decreases the concentration of the reacting species. Since the rate of a reaction is proportional to concentration on a molecular basis, the rate should reach a maximum and then fall as in Figure 14. Between this maximum and the monolayer, a semilog plot of rate versus water activity generally results in a straight line. For most dry foods, an increase of 0.1 of water activity in this region decreases shelf life two or three times (Labuza, 1984).

Glass Transition In Food

The physical state and physicochemical properties of food components affect food behavior in processing and storage. Many of the food components can exist in the amorphous state, especially at low temperatures, and/or low moisture contents. Amorphous materials may exist in a solid-like, glassy state, or in a viscous or rubbery state (Slade and Levine, 1995).

The transitions in materials, which can exist as crystalline solids, as glasses and "rubbers," as molten liquids, and in solution, are shown schematically in Figure 1.14. The glassy state is not an equilibrium state but is determined by kinetic considerations, such as rates of cooling or dehydration.

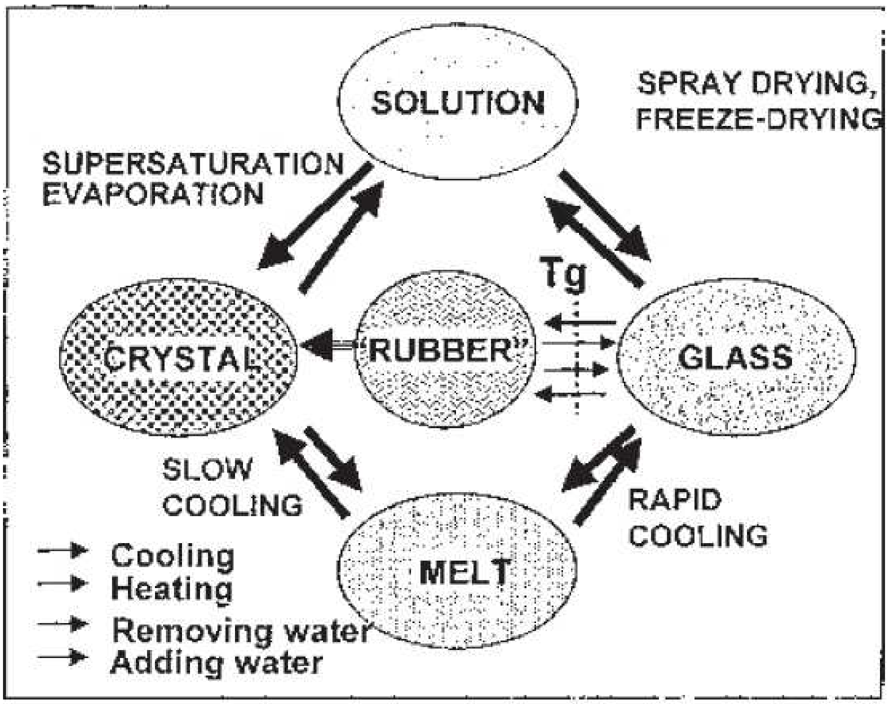


FIG 1.14. Phase transitions occurring in food components.

However once established, the glasses are stable until the temperature exceeds the glass transition temperature T_g; T_g is a very strong function of the water content.

The major effects of water on the rates of physical changes in foods are related to effects of plasticization by water and the attendant increased mobility of amorphous components of foods (Karel et al., 2003).

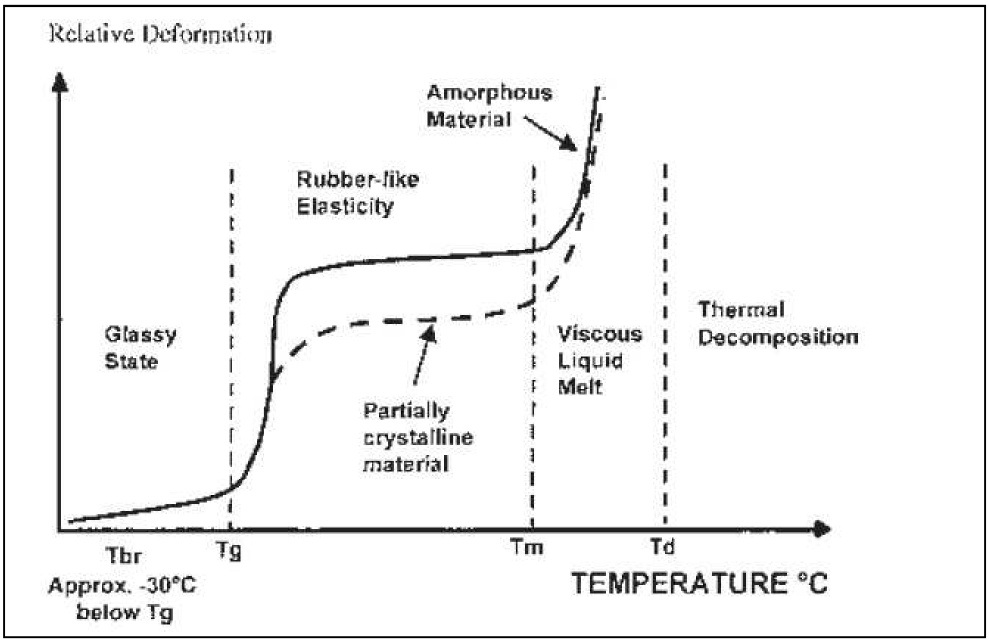


FIG 1.15. Mechanical behavior of polymers. Effect of T_g in deformation at constant stress.

Figure 1.15 shows typical changes in the deformation of polymer, at a given applied force as a result of the drop in viscosity.

The most immediate effect of glass transition is an increase in stickiness and caking (Wallack et al., 1988). Stickiness is usually measured by determining the temperature at which the force required for stirring of a powder shows a large increase, the so-called sticky point.

An important consequence of the increasing tendency of particle to stick is the development of caking. Caking of powders is an undesirable process that transforms free-flowing powders in lumps and aggregates and eventually into a sticky non-particulate mass. The caking affected by water activity changes applies to hydrophilic solids and usually is due to glass transition.

Often, the crystallization of initially amorphous components, and in particular of sugars including sucrose, lactose, and glucose is a critical physical phenomenon affecting stored water sensitive food materials (Hartel, 1993).

One characteristic of the amorphous sugars crystallization is of particular importance for food stability. When the sugars crystallizes as anhydrous crystals, they release the water associated with them in the amorphous state. As a result, the water content of the still uncrystallized amorphous sugar increases, the T_g of the glass drops, the quantity $T-T_g$ (which controls rate of crystallization) increases, and the crystallization continues to accelerate.

Crystallization inhibitors can be used to contrast this phenomenon and they include raffinose (which is an efficient inhibitor of sucrose crystallization), surfactant (which inhibit the crystallization of a variety of solutes by their effects at interfaces), and polymers (which increase viscosity) (Michael et al., 2000).

In conclusion, knowledge of the physical state and physicochemical properties of food components is advantageous in food design and formulation. The information on factors affecting the rates of different and various degradation reactions can be used to manipulate and control rates of changes that occur during food processing and to develop food products that are less sensitive to detrimental changes during storage. It is obvious that the main factors that control stability of low-moisture foods are a_w and composition. Low moisture foods are considered to be stable when they are stored in cool and dry conditions. An increase in temperature or water content may result in a significant change in the rates of deteriorative changes. Knowledge of the effects of temperature and water on the physical state and diffusion in amorphous food matrices may be used to establish relationships between food composition and storage conditions.

Traditional shelf life predictions of low-moisture foods have been based on the information on rates of deteriorative changes and loss of nutrients at various temperatures
and water contents (Karel et al., 2003).

Moisture Control In Food

Food manufacturers must consider a lot of quality and safety factors: microbial stability, physical properties, sensory properties, and the rate of chemical changes leading to loss of shelf-life. All above factors depend on the amount of moisture and the water activity value of each domain in a multi-domain food system. A multi-domain system can be at the macromolecular or molecular level. Examples of macromolecular multi-domain system are dry cereal with semi-moist raisins, frozen pizza crust with sauce, ice cream in a cone, a pastry with a fruit filling, chocolate or hard candy with liquid centers, a cheese and cracker snack, and the crust and crumb of a loaf of bread right after baking. Examples of the molecular level multi-domain systems are water within a starch granule or water in different regions (starch, protein) in a baked product.

Several factors influence the amount and rate of moisture migration in multi-domain foods (Labuza et al., 2000).

Two main factors are water activity equilibrium (thermodynamics) and factors affecting the diffusion rate (dynamics of mass transfer). To control this migration, several principles can be utilized to inhibit the change in moisture including adding an edible layer between domains, changing the water activity of the food ingredients, changing the effective diffusivity of the water, and changing the viscosity (molecular mobility) in the entrapped amorphous phases.

A possible approach to slow diffusion between two domains is to create a diffusion barrier between them.

Obviously, this has to be an edible barrier so the available barrier material is the limiting factor. Biquet and Labuza, 1988, showed that chocolate is a good barrier to moisture because of the dispersion of fat (cocoa butter) throughout the matrix. This has limited applications to candies, confectioneries, frozen bakery goods and frozen desserts. For example, a chocolate layer on the inside of an ice cream cone will help reduce the moisture pickup and thereby maintain crispness of the cone during storage. Similarly a mixed starch-lipid layer can be used between the sauce and the crust to prevent crust sogginess in frozen pizzas (Kamper et al., 1985).

Aim

The transfer of moisture between food products and the surrounding environment, where "environment" can mean both the infinite air and the finished head space of a package, represent a potential cause of food degradation. This alteration can be due to the moisture increase, which makes the product less stable from the microbiological point of view, and reduces the consumer attractiveness due to a loss in crispness / crunchiness. In addition, it can be due to dehydration, the effects of which are negative from a sensory point of view. Having to face these problems, the selection of appropriate type of packaging, as well as the use of compounds and hygroscopic ingredients, may slow down the development of mass transfer from and into the food product, extending its shelf-life. However, the transfer of moisture can take place even within the foods, in particular when they are constituted by two or more heterogeneous phases, both mixed (for example biscuits with dried fruit or chocolate chips or candy) and in contact between them (for example, the fresh filled pasta). This transfer may be of such significance that they determine a change in the short term hydration status of the two phases, causing a deterioration in the quality perceived by consumers.

The purpose of this study was therefore to develop a mix formulation that can slow down the transfer of moisture that can take place between a filling of vegetables and cheese and fresh pasta sheet that makes up the traditional meatless tortelloni / tortelli. Such transfers determine first an excess of water in the dough, which makes individual products sticky, and subsequently an unwelcome drying tortello, with an unpleasant reduction of the product volume (shrinkage). Ingredients with high hygroscopic capacity are commercially available, each one characterized by plus and minus

In order to ride out the drawbacks of the single commercial ingredients and to reach a good formulation not by trial and error, different ingredients were evaluated as follows:

1. Evaluation of their ability in reduction or control of the content of free water, by means of the measurement of Water Activity (a_w) of different vegetable fillings added with the selected ingredients

2. Measurements and drawings of water adsorption isotherms for selected preparations, which represent their Water Activity (a_w) versus a moisture content (g H2O / g d.m.)

3. Evaluation of Water Retention Capacity (WRC) or the amount of water retained by a known weight of dry matter (g water / g of dry substance) of preparations / ingredients

The preliminary phase allowed the selection of ingredients which have been suitably mixed and tested in a real production settings of ravioli stuffed with spinach and ricotta, with the purpose of assessing in time (for the duration of the commercial products) the transfer of moisture from stuffed pasta.

Materials And Methods

ROLLER DRIED WHOLE MILK POWDER: extra grade dry whole milk powders; resulting from the removal of water from pasteurized milk, with a fat content ranged between 26% and 40%, and not more than 5% by weight of moisture. It contains lactose, milk proteins, milk fat and milk minerals in the same proportions as the milk from which it was made (Danish Food Composition Databank).

DEMINERALIZED OR REDUCED MINERALS WHEY POWDER : whey with a portion of the minerals removed. The dry product cannot contain more than 7% ash. Acidity may be adjusted with safe and suitable pH adjusting ingredients (Danish Food Composition Databank).

WHEY PROTEIN CONCENTRATED: Produced by a pressure driven process known as ultrafiltration (UF). Ultrafiltration uses semi-permeable membranes; lactose and minerals are removed until desired protein content is reached. By product of this process is permeate. Higher protein WPCs require the use of water, in a process known as diafiltration (DF), to remove greater amounts of lactose and ash (Danish Food Composition Databank).

SPRAY DRIED SODIUM CASEINATE: it is the product Acid or rennet casein is insoluble in water but they may be treated with alkali to make a soluble caseinate. The most common alkali used in the manufacture of spray dried sodium caseinate is sodium hydroxide. It is mixed (as an aqueous solution with a typical concentration of 2.5 M) with a slurry of the casein curd or powder in wate (Danish Food Composition Databank).

MODIFIED CORN STARCHES: also called starch derivatives, are achieved through chemical and physical modification of extracted native starch to change its properties. Modified starch is a food additive which is prepared by treating starch or starch granules, causing the starch to be partially degraded. The purposes of this modification are to enhance its properties particularly in specific applications, such as to improve the increase in water holding capacity, heat resistant behavior, reinforce its binding, minimized syneresis of starch and improved thickening. Modified starches are used in practically all starch applications, such as in food products as a thickening agent, stabilizer or emulsifier (Abbas et al., 2010).

PREGELATINIZED CORN STARCHES: are achieved through physical modifications of extracted native starch. The pre-gelatinization process is a physical method to modify the properties of native starch, it provides the ability to form gel for native and/or modified starches. Used in instant puddings, pie fillings, soup mixes, salad dressings, sugar confectionery, and as a binder in meat products (Abbas et al., 2010).

POTATO FIBER 300 and 400: two types of soluble potato fiber with different granulations suitable for use in different food applications. The core functionality of these products is their ability to hold large amounts of water: 1 part of potato fiber is able to hold 13-14 parts of water irrespectively of the type. This can be utilized in a number of food products thanks for its unique absorption properties. The potato fibre preparation was obtained during industrial starch isolation from potato tubers (KMC Online Archive).

PEA FIBER: is a soluble fiber, highly absorptive, low carbohydrate fiber derived from specially cleaned and processed pea hulls. This fiber shows high water binding capacity, it holds 4-8 times its' own weight in water.Pea fiber improves moisture retention retains emulsions of fat and water during freezing and cooking to increase moisture retention (Tosh et al., 2010).

BAMBOO FIBER: is natural insoluble bamboo fiber obtained from fiber rich parts of the bamboo plant. The strengths of bamboo is connected with the benefits of dietary fiber to provide a unique natural fiber for various food products. This type of fiber increase dough yield of baked goods and especially freshness of meat and fish products (KMC Online Archive).

WHEAT FIBER: is natural, insoluble wheat fiber derived from wheat, after separating the straw from the grain. This fiber provides excellent water and oil retention. It is exceptional texturizer and stabilizes fine emulsified systems in food processing (KMC Online Archive).

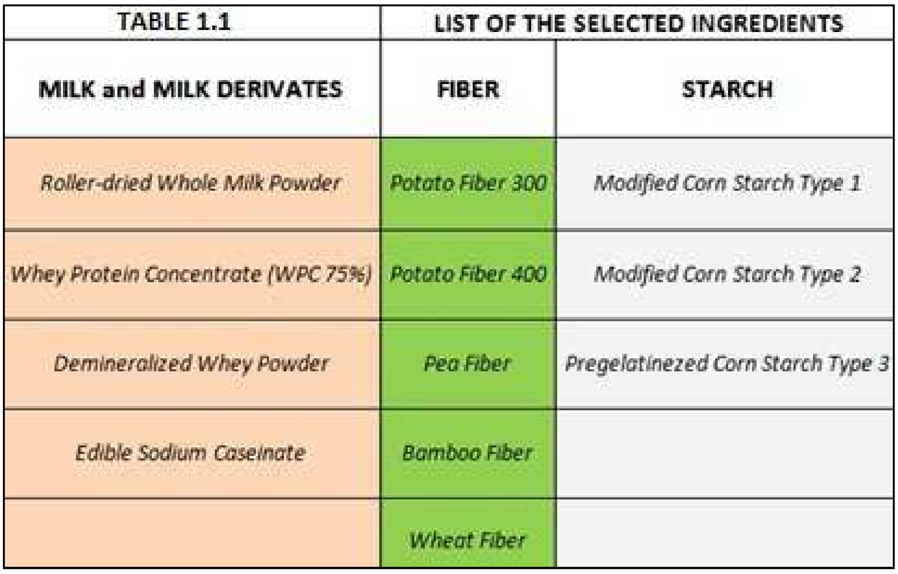


TABLE 1.1	LIST OF THE SELECTED INGREDIENTS	
MILK and MILK DERIVATES	**FIBER**	**STARCH**
Roller-dried Whole Milk Powder	*Potato Fiber 300*	*Modified Corn Starch Type 1*
Whey Protein Concentrate (WPC 75%)	*Potato Fiber 400*	*Modified Corn Starch Type 2*
Demineralized Whey Powder	*Pea Fiber*	*Pregelatinezed Corn Starch Type 3*
Edible Sodium Caseinate	*Bamboo Fiber*	
	Wheat Fiber	

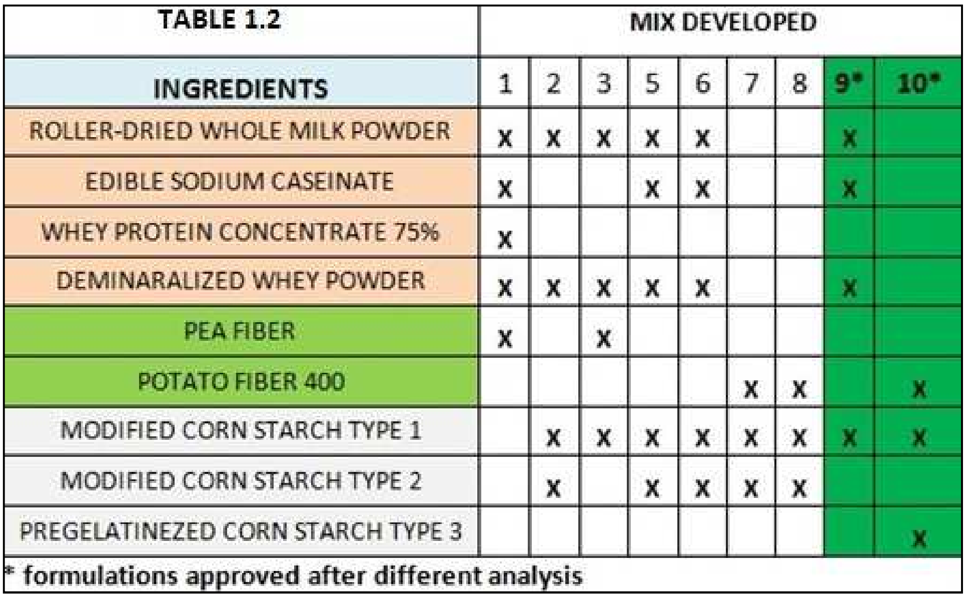


TABLE 1.2	MIX DEVELOPED								
INGREDIENTS	1	2	3	5	6	7	8	9*	10*
ROLLER-DRIED WHOLE MILK POWDER	X	X	X	X	X			X	
EDIBLE SODIUM CASEINATE	X			X	X			X	
WHEY PROTEIN CONCENTRATE 75%	X								
DEMINARALIZED WHEY POWDER	X	X	X	X	X			X	
PEA FIBER	X		X						
POTATO FIBER 400						X	X		X
MODIFIED CORN STARCH TYPE 1		X	X	X	X	X	X	X	X
MODIFIED CORN STARCH TYPE 2		X		X	X	X	X		
PREGELATINEZED CORN STARCH TYPE 3									X

* formulations approved after different analysis

Water Activity Measurement

The measurements were made by means the equipment LabMASTER-a$_w$ (Novasina) (Figure 1.16) at 25°C.

FIG.1.16 LabMASTER a$_w$-Novasina

Protocol:

1. Switch on the LabMASTER-aw and after reaching the chamber temperature, the instrument is ready for measuring the sample.
2. Fill the sample cup to approximately 2/3 with the product to be measured.
3. Open the instrument cover and check the chamber inside. This must be clean an absolutely dry.
4. Make sure an appropriate cell protection filter is installed in front of the measuring cell unit, should the product sample require such protection measures.

5. Insert the open sample cup containing the sample into the measuring chamber (left) and close the cover immediately. Press the cover completely down, otherwise the analyzing function does not start.
6. The Novasina LabMASTER-a_w continuously measures the water activity and the temperature and displays the momentary values. If an analyzing of a probe will be finished, the system will fix the result.
7. Open the instrument cover after ending of the analyzing and after writing down the result
8. Remove the sample cup from the measuring chamber carefully and close the sample dish cover.
9.The instrument cover shall be closed now.

Water Or Moisture Content Determination

This method is used to determine the percentage of water in a sample by drying the sample to a constant weight.

The water content is expressed as the percentage, by weight, of the dry sample (AOAC 2000).

Protocol

1. Weigh moisture sample immediately and record as “wet weight of sample”
2. Dry the wet sample to a constant weight, at a temperature not exceeding 239º F (115°C) for 2h using the suitable drying equipment.
3. Allow the sample to cool.
4. Weigh the cooled sample again, and record as the “dry weight of sample”.

Calculation

The moisture content of the sample is calculated using the following equation:

$$\boxed{\%W = \frac{A-B}{B} \times 100}$$

Where:

%W = Percentage of moisture in the sample,

A = Weight of wet sample (grams), and

B = Weight of dry sample (grams)

Sorption Isotherm Curve Determination

The relationship between moisture content and water activity is the Sorption Isotherm Curve, which is product and temperature specific.

It is very important an absolutely constant temperature during all measurements.

For such purpose it is used a set in combination with a LabMASTER/PARTNER-a_w (LabMASTER/PARTNER a_w Operating Instructions).

Protocol

In order to start, the substance must be dry, so first it must be totally desiccated in a oven for 2 h on 102°C.

1. Close the glass container before taking it out from the drying oven.
2. Weigh the probe afterwards with the closed weighing glass on a precision balance and define the net weigh of the probe.
3. Put first the SAL-T 6 in the deeper measurement chamber and afterwards the metallic sample cup containing the cooled sample over it and close the chamber.
4. After about one hour write the actual a_w value. It is not necessary to wait for the equilibrium inside the chamber.
5. Take our the probe and put it immediately in the glass container.
6. Weigh the probe again and write the net weigh a table and calculate the difference between the weights before and after humidification.
7. Put the next SAL-T into the measurement chamber and perform the same operation at point 2,3 ect. up to the SAL-T 97 humidity standard.

Moisture content calculation

Each sample should be treated individually to calculate moisture content (Bell et al., 2000).

$$\boxed{m = \frac{g\ H_2O}{g\ solids} = \frac{(w_f - w_{i,d})}{w_{i,d}}}$$

where w_f is the mass of the product after moisture gain and $w_{i,d}$ is the initial mass of the dried product. For an initial sample of mass w_i having some initial moisture content ($\%H_2O$) on a wet basis,

$$M = \frac{[wf - wi] + \frac{\%H20}{100} * wi}{wi * \left[\frac{100 - \%H20}{100}\right]} = \frac{gH20}{gSS}$$

Based on the acquired data it is possible define the Adsorption curve; dry basis moisture is plotted on the Y axis as function of a_w, on the X axis.

Water Retention Capacity (WRC) Assay

It is the amount of water retained by a known weight of substance under the conditions used (Tosh S. M. et al., 2010).

Protocol

1. Hydrate the probe, 3g, in water, 30ml for 18 h.
2. Centrifuge the sample 3000xg for 20 minutes.
3. Drain and dry the sample.
4. Weigh the dry pellet.

Calculation:

The Water Retention Capacity (WRC) of the sample is calculated using the following equation:

$$\text{WRC} = \text{g water/g dry pellet}$$

Stuffing Testing

Recipe filling

- 62.2% ricotta
- 11.15% parmiggiano reggiano
- 15.5% spinach
- 11.1% potato flakes

(S. G. Food srl recipe)

Preparation of filling

1. Weigh all ingredients.
2. Cook the spinach in a pan and then let it cool after cooking.
3. Chop the spinach with the robot.
4. Transfer the ingredients in the planetary mixers.
5. Mix until the filling becomes homogeneous.
6. Aliquot filling for tests.

(Figure 1.17)(E)

FIG 1.17 Filling based ricotta and spinach prepared with kitchen robot.

Test Of Funtional Mix On Industrial Stuffed Pasta

The two functional preparations, MIX MILK (formulation n°9) and MIX STARCH (formulation n° 10), are tested on industrial stuffed pasta process.

In this experimental phase the water transfer from the filling to the dough was determined by calculating the moisture total loss of stuffed pasta throughout the shelf-life (60 days form production date).

This analysis is performed on three type of samples :

- tortelli with MIX MILK
- tortelli with MIX STARCH
- tortelli standard without mix

Protocol

1. For each type of sample (ie tortello with MIX and without) unpack and take three tortelli at three different points.
2. Cut the edges of the dough of the tortelli, not in contact with the filling.
3. Separate the stuffing from the dough, using a spatula.
4. Transfer the filling into a special glass container; repeat the same operation for the dough (Figure 1.18).

FIG 1.18 A special glass container used in the analysis described above.

5. Weigh the glass container with filling or dough with precision balance and record the values of the gross weight fresh.
6. Determine the net weight of the fresh sample by subtracting from the gross weight fresh of tare.
7. Transfer all containers in a oven on 105°C overnight.
8. Take out from the oven all containers and transfer the containers in a dryer for 15 minutes to avoid that the sample absorbs moisture (Figure 1.19).

FIG 1.19 Dryer

9. Weigh all containers and record their gross weight dry.
10. Determine the net weight of the dry sample by subtracting from the gross weight dry of tare.

Calculation

Based on the data collected for all samples calculate the moisture content:

$$\boxed{m = g\ H_2O\ /\ 100g\ \text{of sample}}$$

Repeat this operation for three rates of each type of sample and mediate the three values obtained.

Results And Discussion

The following chapters have been organized according to the sequence of Technology Readiness Levels (TRL), which are widely used measures used to assess the maturity of evolving technologies (devices, materials, components, software, work processes, etc.) during their development and in some cases during early operations. In particular, the classification used in the present work is the one adopted by the European Commission in the 2014-2020 Horizon 2020 Programme for Research and Innovation (www.ec.europa/research/horizon2020), and is reproduced below:

- TRL 0: Idea.
- TRL 1: Basic research.
- TRL 2: Technology formulation.
- TRL 3: Applied research.
- TRL 4: Small scale prototype built in a laboratory environment.
- TRL 5: Large scale prototype tested in intended environment.
- TRL 6: Prototype system tested in intended environment close to expected performance.
- TRL 7: Demonstration system operating in operational environment at pre-commercial scale.
- TRL 8: First of a kind commercial system.
- TRL 9: Full commercial application, technology available for consumers.

Idea

In this work the ability of certain food ingredients to regulate the moisture migration in stuffed pasta from the filling to the dough has been studied (Figure 1.20).

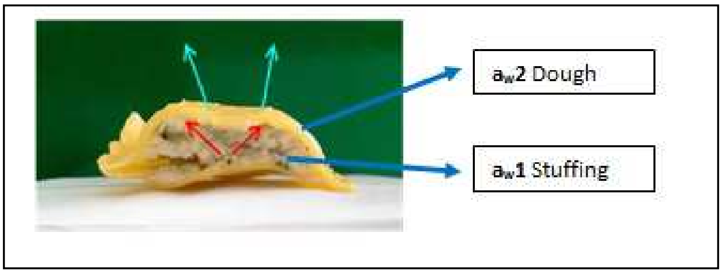


FIG 1.20 Moisture migration in stuffed pasta.

A large number of ingredients with high hygroscopic capacity, each with its positive and negative prerogatives, are now commercially available; trial took into consideration ingredients and preparations based on starches (possibly also formulated to the diet of celiac) and dairy products, with different properties and different cost also.

Basic Research

Study Of Hygroscopic Property Of The Ingredients Selected

The preliminary phase allowed the selection of ingredients which have been suitably mixed and tested in a real production settings of ravioli stuffed with spinach and ricotta, with the purpose of assessing in time (for the duration of the commercial products) the moisture migration in stuffed pasta.

The food powder ingredients were evaluated individually and in combination with each other on the basis of their ability to absorb moisture, through the evaluation of the VARIATION OF THE MOISTURE CONTENT, through the construction of ADSORPTION ISOTHERMS, and their ability to retain moisture over time and under particular conditions, through ASSAY OF WATER RETENTION CAPACITY (WRC).

The analysis of adsorption isotherms of the individual components can be a valid screening, to select components based on their ability to be in equilibrium with the environment's humidity by absorbing water in function of their initial state.

The following table shows the initial values of "moisture content" (m_0) and water activity (a_{w0}) of the compounds examined in this part of the experiment (see "Materials and Methods").

The values are listed in ascending order by the value of m_0 (Table 1.3).

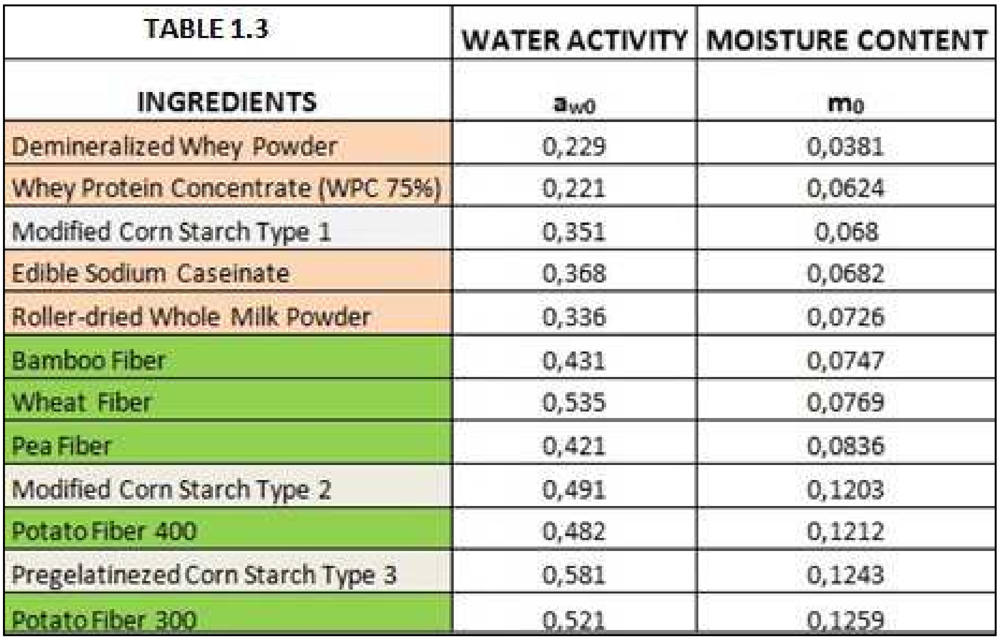


TABLE 1.3	WATER ACTIVITY	MOISTURE CONTENT
INGREDIENTS	a_{w0}	m_0
Demineralized Whey Powder	0,229	0,0381
Whey Protein Concentrate (WPC 75%)	0,221	0,0624
Modified Corn Starch Type 1	0,351	0,068
Edible Sodium Caseinate	0,368	0,0682
Roller-dried Whole Milk Powder	0,336	0,0726
Bamboo Fiber	0,431	0,0747
Wheat Fiber	0,535	0,0769
Pea Fiber	0,421	0,0836
Modified Corn Starch Type 2	0,491	0,1203
Potato Fiber 400	0,482	0,1212
Pregelatinezed Corn Starch Type 3	0,581	0,1243
Potato Fiber 300	0,521	0,1259

It is evident that the initial state of the examined components is quite different, not only among those of different origin, but also within more or less homologous categories: see, for example, the substantial difference between the two starches named "1" and "2". It was considered appropriate to focus on these values, since they can be useful keys to the results that will be presented later.

Figure 1.21 illustrates graphically the increases in moisture content of the individual components (delta "m" - red bar), compared to the initial value of their moisture content ("m" - green bar). The data are listed in increasing order of the value of "m_0". The figure of increase was calculated by subtracting the final value of moisture content obtained at the end of the construction of the adsorption isotherms of the initial one (Figure 1.21).

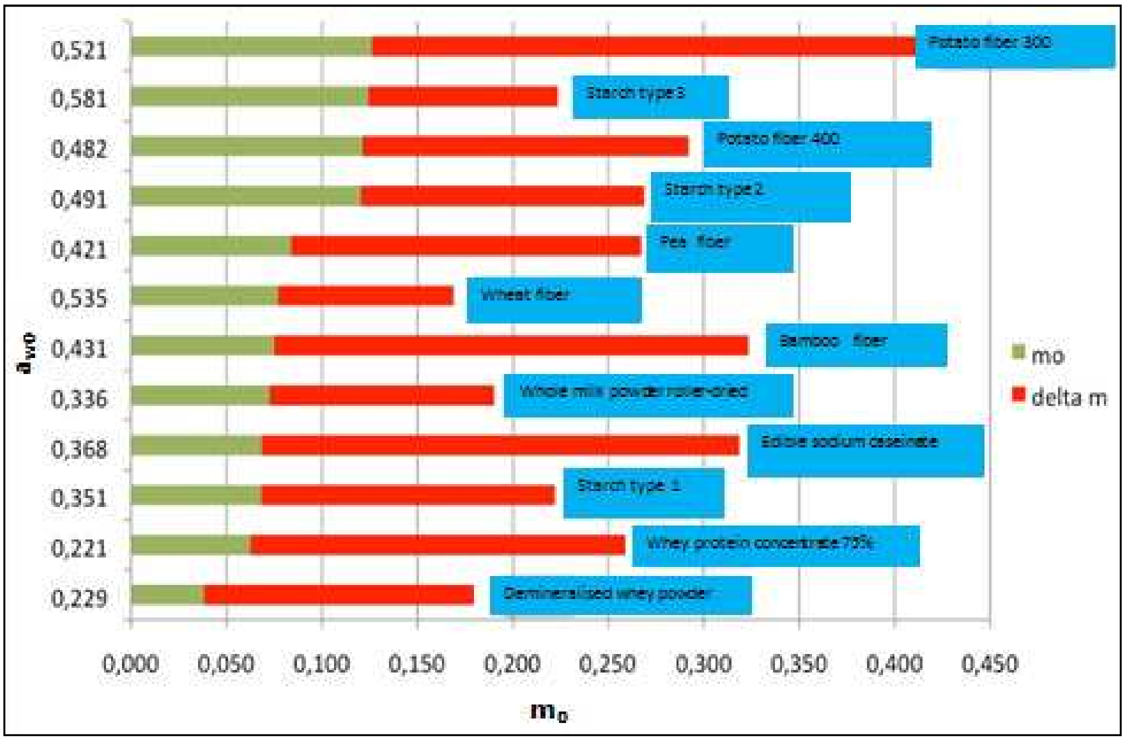


FIG 1.21 Variation of the moisture content in the ingredients selected.

It is evident that the initial state of the selected compounds does not automatically determine the capacity to adsorb water: one can notice, for example, the difference between the potato fiber 300 and starch 3; despite the fact that the value of m_0 is essentially the same, the potato fiber 300 triples almost three time its moisture content.. Clearly, the hygroscopic capacity of food ingredients, but also of food products, depends not only on the amount of water and the value of their "a_w", but is a function of the particular bindings that the water is able to establish with the physical state and chemical components.

For this reason, it becomes important to know the water adsorption isotherms of the food, or of individual ingredients and additives.

For each of the tested compounds, the respective isotherms were then built in laboratory (see "Materials and Methods"), and in the following figures the isotherms obtained are shown. In each figure, compounds were grouped according to their source analogy, or according to their similarity of composition (Figure 1.22, Figure 1.23 and Figure 1.24).

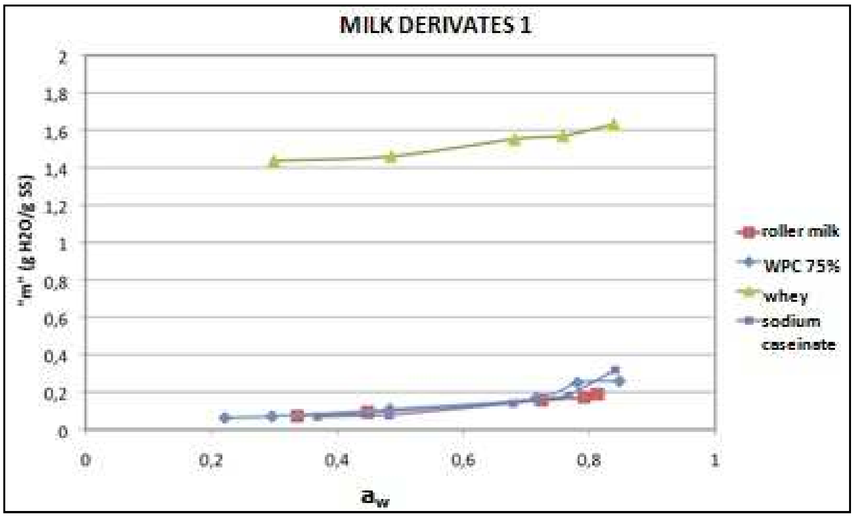


FIG 1.22 Moisture Adsorption Isotherm for MILK DERIVATES.

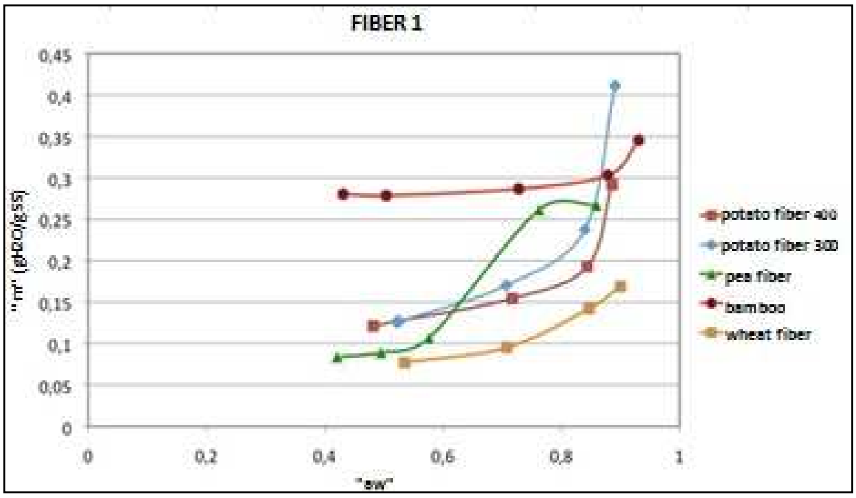


FIG 1.23 Moisture Adsorption Isotherm for FIBERS.

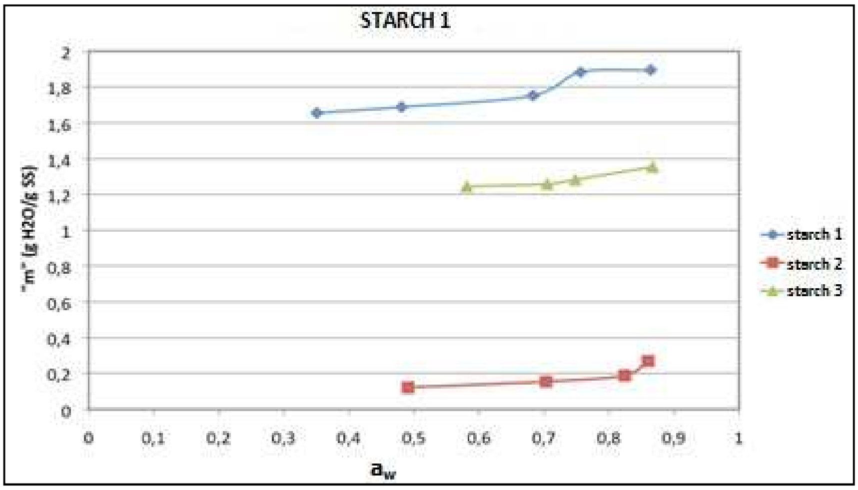


FIG 1.24 Moisture Adsorption Isotherm for STARCHES.

All tested compounds begin their isotherm at about half the value of the scale of Aw, thus information is missing on their behavior in the lower isothermal values of "a_w". However this was not the purpose of the experiment: the compounds are used in their native state, so they exercise their hygroscopic properties from their initial value of "a_w" and "m_0".

Comparing the three groups of isotherms, it is possible to highlight two different behaviors:

1. STARCHES and MILK DERIVATES: they are all in the "flat" area of a theoretical isotherm, where small changes in moisture content are associated to huge variations of "a_w"

2. FIBERS: for all fibers, except for those of bamboo and wheat, there is a steep change of slope (inflection point), around "a_w" values equal to 0.8 for potato fibers, and around "a_w" 0.6 for pea fibers. The behavior of bamboo and wheat fibers is the closest one to that of starches and dairy products; with the same initial "a_w" of other fibers, the bamboo has a much higher value of "m".

Since different starting conditions might upset some of the comments, in the isotherms charts the difference of "m_0" was eliminated by subtracting this value to the values of "m" calculated for each point in the isotherms (Figure 1.25, Figure 1.26 and Figure 1.27).

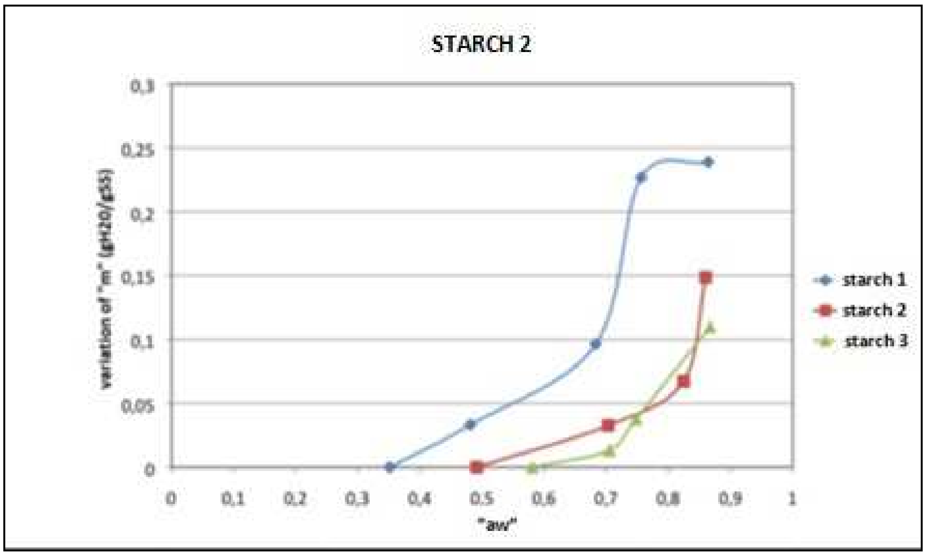


FIG 1.25 Moisture Adsorption for STARCHES as function of variation of "m".

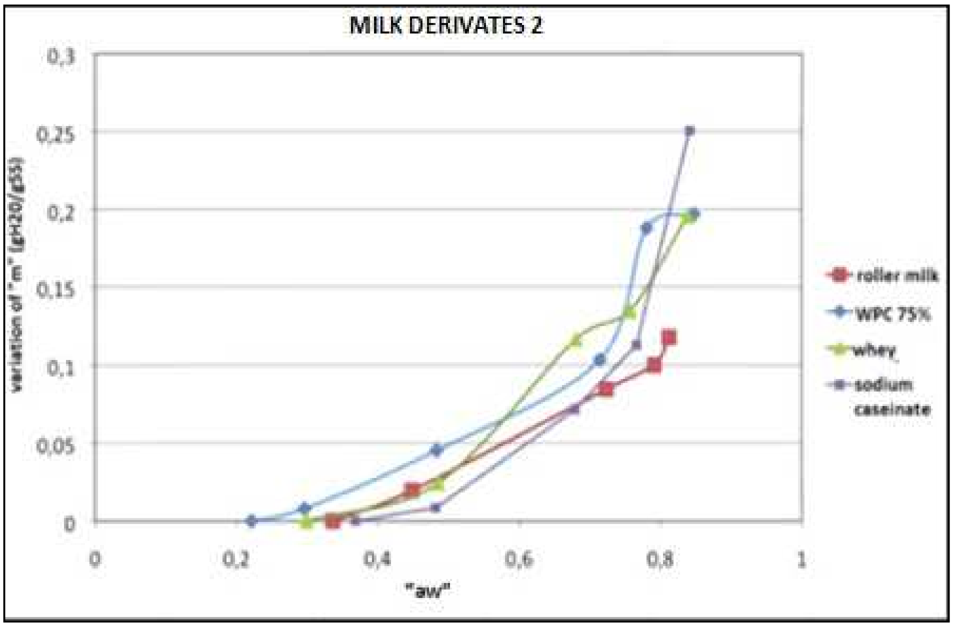


FIG 1.26 Moisture Adsorption for MILK DERIVATES as function of variation of "m".

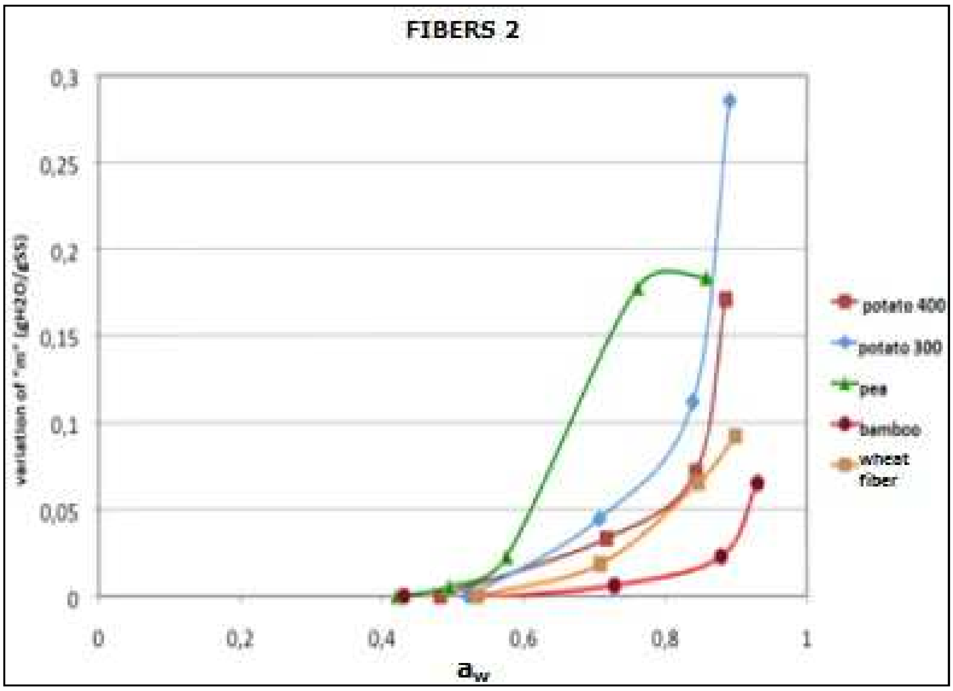


FIG 1.27 Moisture Adsorption for FIBERS as function of variation of "m".

Representing the isotherms data in this way, not usual indeed, it is possible to better emphasize the behavior of selected hygroscopic compounds. While in the case of STARCHES and MILK DERIVATES there were no significant differences, FIBERS (bamboo, pea and wheat) show a conduct similar to that of the two Potato fibers (300 and 400), able to absorb moderate amounts of water until the end of the isotherm (corresponding to an "a_w" value close to 0.90). The pea fibers reach rapidly the saturation, whereby for any appreciable increase in "m" there is a noticeable change in "a_w", while the bamboo fiber can be considered the worst in terms of hygroscopic capacity.

Water Retenction Capacity Of Selected Ingredients

The hygroscopic behavior of the compounds is not the only parameter to be taken into consideration. In fact, the way individual compounds equilibrate with the surrounding environment (be it the external environment or the aqueous phase of a food) is important, but, on the other hand, it is important to determine whether the water bound them is also advantageously retained, and for how long. For this reason, in parallel with the analysis of the isotherms, the Water Retention Capacity (WRC) of the different ingredients and subsequent preparations (see " Material and Methods") was also determined.
In Table 1.4 the data obtained are summarized: the WRC of the different food additives was determined over a period of 30 days (half of shelf-life of the food matrix in which the compounds carry on their function).

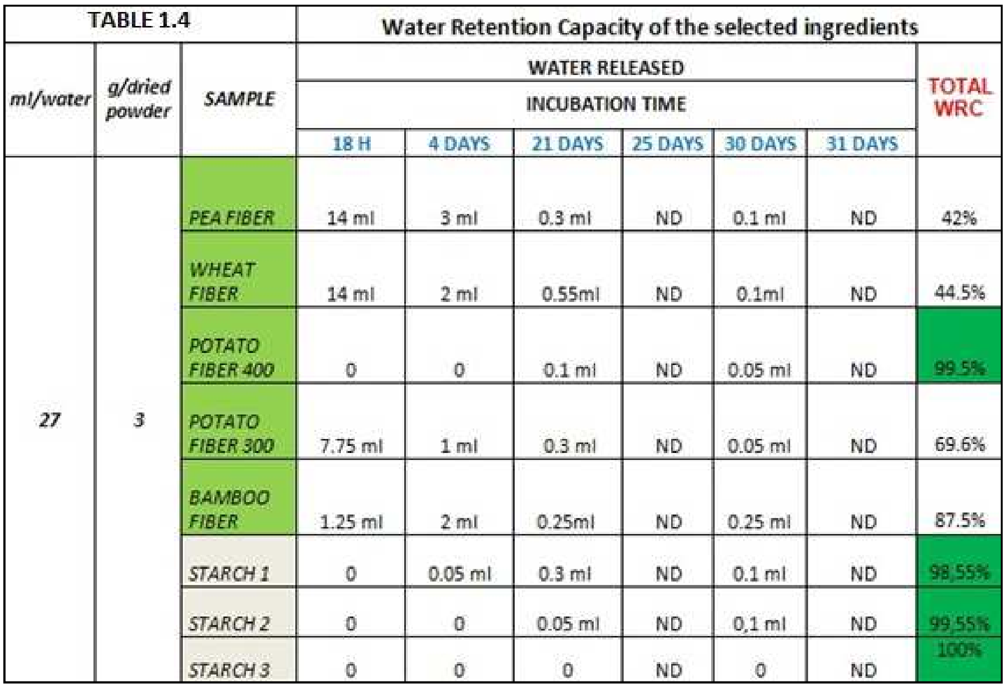


TABLE 1.4			Water Retention Capacity of the selected ingredients						
ml/water	*g/dried powder*	*SAMPLE*	WATER RELEASED						TOTAL WRC
			INCUBATION TIME						
			18 H	4 DAYS	21 DAYS	25 DAYS	30 DAYS	31 DAYS	
27	*3*	*PEA FIBER*	14 ml	3 ml	0.3 ml	ND	0.1 ml	ND	42%
		WHEAT FIBER	14 ml	2 ml	0.55ml	ND	0.1ml	ND	44.5%
		POTATO FIBER 400	0	0	0.1 ml	ND	0.05 ml	ND	99.5%
		POTATO FIBER 300	7.75 ml	1 ml	0.3 ml	ND	0.05 ml	ND	69.6%
		BAMBOO FIBER	1.25 ml	2 ml	0.25ml	ND	0.25 ml	ND	87.5%
		STARCH 1	0	0.05 ml	0.3 ml	ND	0.1 ml	ND	98,55%
		STARCH 2	0	0	0.05 ml	ND	0,1 ml	ND	99,55%
		STARCH 3	0	0	0	ND	0	ND	100%

The data collected in Table 1.4 are related to the compounds that show in this test the highest values of WRC. As can be seen, milk and its derivatives are not included, since, for this study, they are considered wetting agents, due to their strong hygroscopicity.

Technology Formulation

This first analysis has provided a lot of information about the behavior of the considered ingredients, and at the same time has allowed the selection of the most suitable one in terms of water retention. This proved to be an important starting point for the development of a formulation capable of slowing down the transfer of water in the tortelli, from vegetables filling to pasta sheet.

Then, the analysis continued on different combinations of these compounds, in order to study their interactions and to better understand how to associate them in a formulation (Table 1.5).

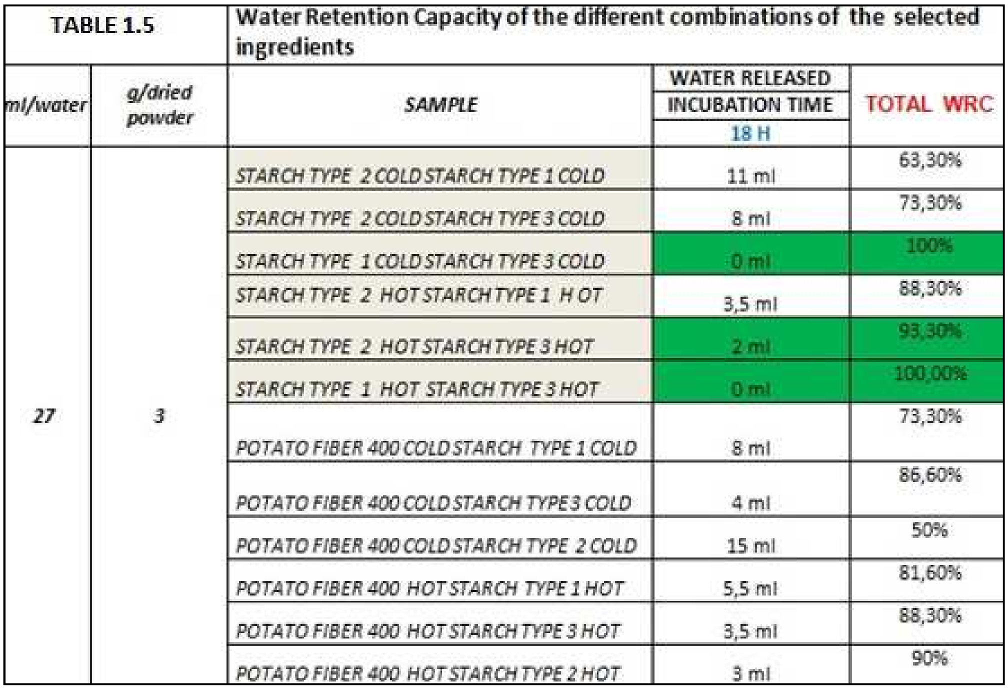


TABLE 1.5		**Water Retention Capacity of the different combinations of the selected ingredients**		
ml/water	*g/dried powder*	*SAMPLE*	WATER RELEASED INCUBATION TIME 18 H	TOTAL WRC
27	*3*	*STARCH TYPE 2 COLD STARCH TYPE 1 COLD*	11 ml	63,30%
		STARCH TYPE 2 COLD STARCH TYPE 3 COLD	8 ml	73,30%
		STARCH TYPE 1 COLD STARCH TYPE 3 COLD	0 ml	100%
		STARCH TYPE 2 HOT STARCH TYPE 1 H OT	3,5 ml	88,30%
		STARCH TYPE 2 HOT STARCH TYPE 3 HOT	2 ml	93,30%
		STARCH TYPE 1 HOT STARCH TYPE 3 HOT	0 ml	100,00%
		POTATO FIBER 400 COLD STARCH TYPE 1 COLD	8 ml	73,30%
		POTATO FIBER 400 COLD STARCH TYPE3 COLD	4 ml	86,60%
		POTATO FIBER 400 COLD STARCH TYPE 2 COLD	15 ml	50%
		POTATO FIBER 400 HOT STARCH TYPE 1 HOT	5,5 ml	81,60%
		POTATO FIBER 400 HOT STARCH TYPE 3 HOT	3,5 ml	88,30%
		POTATO FIBER 400 HOT STARCH TYPE 2 HOT	3 ml	90%

Table 1.5 shows the combinations of ingredients with the highest WRC values. The analysis of the behavior of these substances in combination has made a significant contribution in terms of data needed for the development of a formulation with a strong capacity to retain water in the vegetables-based filling of stuffed pasta. In particular, the analysis has better clarified the formulation features in terms of composition and functionality.

In this regard, it was decided to perform the test by making changes compared to the reference protocol (see "Materials and Methods"), in order to observe the behavior of the compounds under study in the supposed conditions of the formulations, namely the temperatures reached by the filling in the production phase of stuffed pasta.

The experiments described so far enabled the development of different formulations shown in Table 1.6.

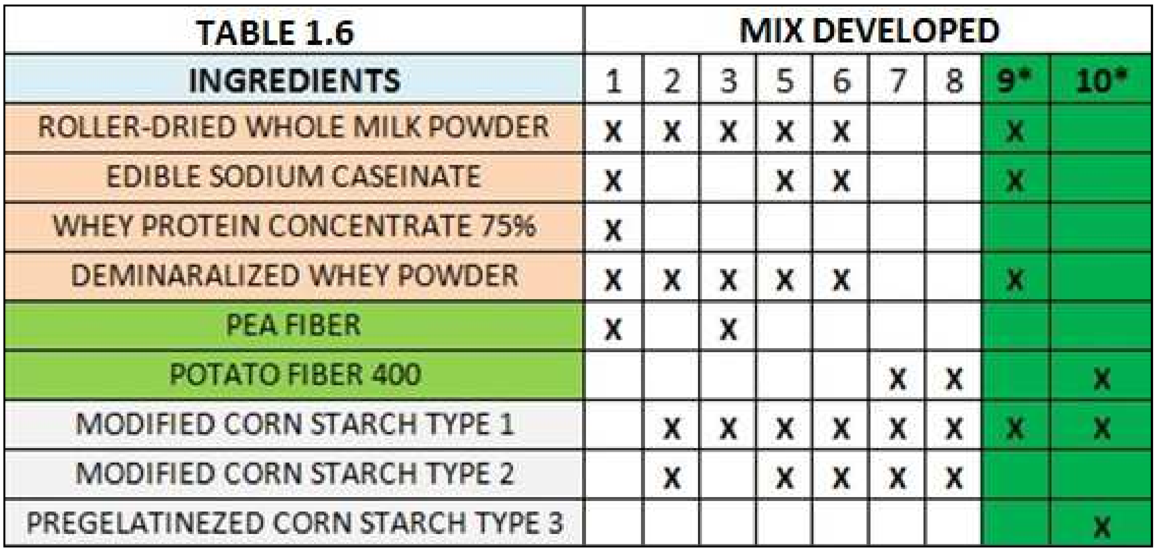


TABLE 1.6	MIX DEVELOPED								
INGREDIENTS	1	2	3	5	6	7	8	9*	10*
ROLLER-DRIED WHOLE MILK POWDER	X	X	X	X	X			X	
EDIBLE SODIUM CASEINATE	X			X	X			X	
WHEY PROTEIN CONCENTRATE 75%	X								
DEMINARALIZED WHEY POWDER	X	X	X	X	X			X	
PEA FIBER	X		X						
POTATO FIBER 400						X	X		X
MODIFIED CORN STARCH TYPE 1		X	X	X	X	X	X	X	X
MODIFIED CORN STARCH TYPE 2		X		X	X	X	X		
PREGELATINEZED CORN STARCH TYPE 3									X

Applied Research

The formulations developed in the previous phase of the experiment were examined for their ability of water retention according to the protocol described in "Materials and Methods". From the results obtained by these tests (data not shown) two formulations have been selected: formulation "9", based on milk powder and milk derivatives, and formulation "10", void of milk allergen.

Small Scale Prototype Built In A Laboratory Environment

The trial continued with a more in-depth evaluation of the effectiveness of formulations 9 and 10, through the development of two prototypes performed for additional laboratory tests. More in detail, a water retention capacity assay (WRC) was performed, on the rates of filling prepared as described in "Materials and Methods". Then the formulations were added, in three hypothetical doses, 2%, 4% and 6%; the above analysis was prolonged for 30 days, equal to about half the shelf life of the industrial filled pasta (Figure 1.28).

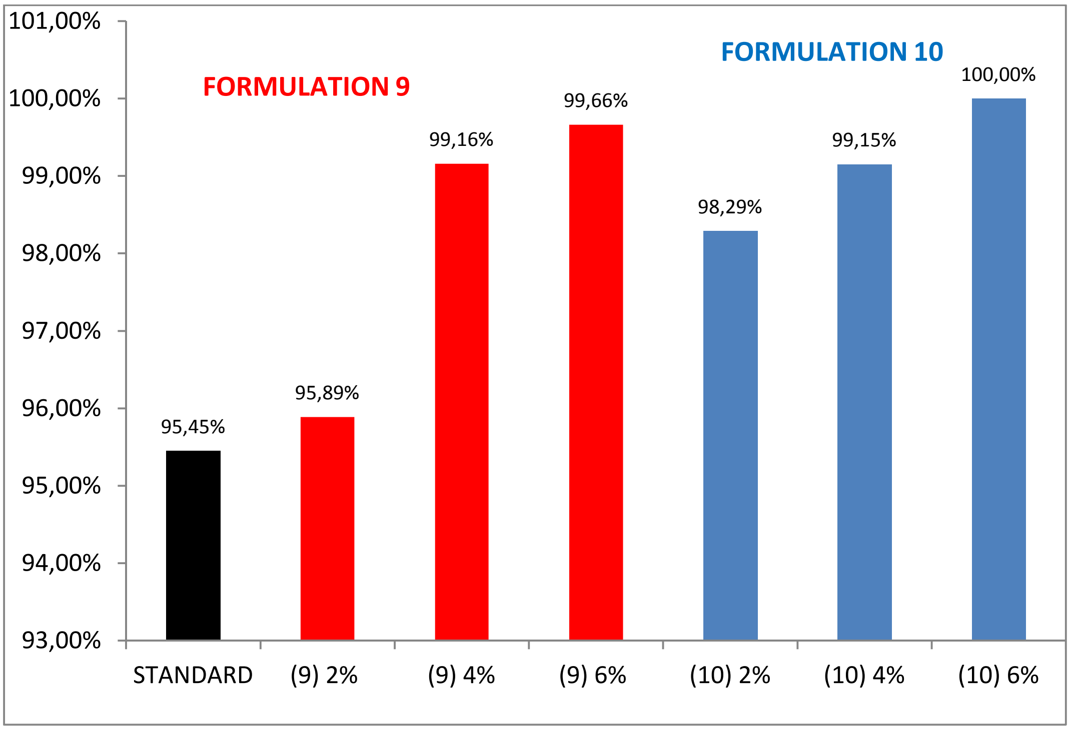


FIG 1.28 Water retention capacity of formulation 9 and 10 added to the filling.

As shown in Figure 1.28, the test on the two prototypes (formulation 9 and 10), carried out on a small scale, is satisfactory in terms of functionality and effectiveness of the two formulations developed, which are indeed already observable at an average dose (4%). In this regard it was decided to move to the more advanced stage of the trial, i.e. the proof on a large scale.

Large Scale Prototype Tested In Industrial Process

Taking into account the results obtained from previous tests, it was decided to assess the effectiveness of selected formulations in a real use setting, adding them to a filling of ricotta and spinach, according to the recipe provided by the industrial partner involved, used for the preparation of vegetables stuffed pasta.

The purpose of this experiment was to evaluate whether the formulations added to the filling, with an established dosing according to a small-scale test (4%),were able to slow down the moisture transfer from the filling to dough, a phenomenon that provoke in the short period an excessive humidification of the dough, resulting in sticking and rupture of the stuffed pasta, and, in the long run, in a retraction of the filling (for moisture loss) which reduces the appeal for the consumer due to the product bad appearance).

For the evaluation, it was decided to determine the moisture contents of filling and dough separately, since the measurement of water activity was not considered an index sensitive enough to assess the transfer of moisture (see "Materials and Methods").

The test lasted for a period of sixty days, during which, at fixed intervals, the absolute humidity of the filling and the pastry (after careful separation of the two phases) were assessed, on ravioli made with a filling standard (control) and with two fillings additives respectively with the formulation 9 (MIX MILK) and 10 (MIX STARCH).

The charts below show the calculated data of humidity and the respective standard deviations for the filling and the pastry of different experimental theses (Figure 1.29 and Figure 1.30).

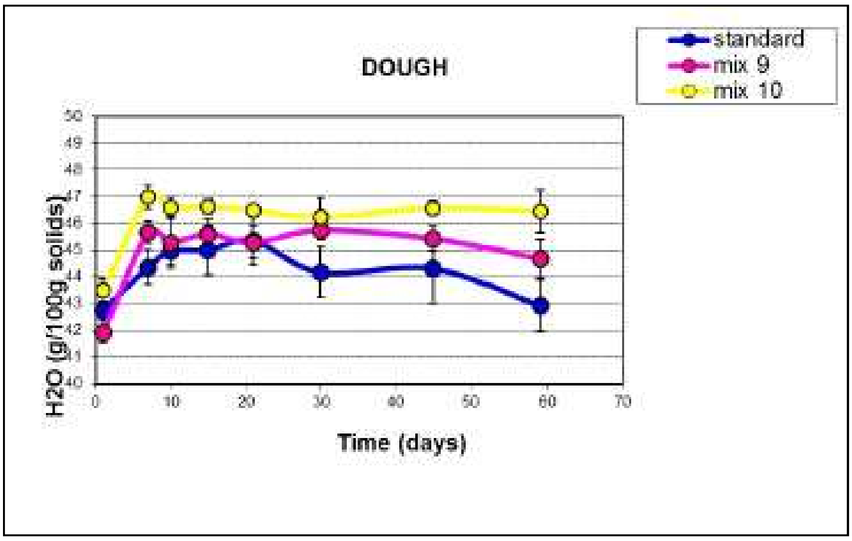


FIG 1.29 Variation of the moisture content in the dough.

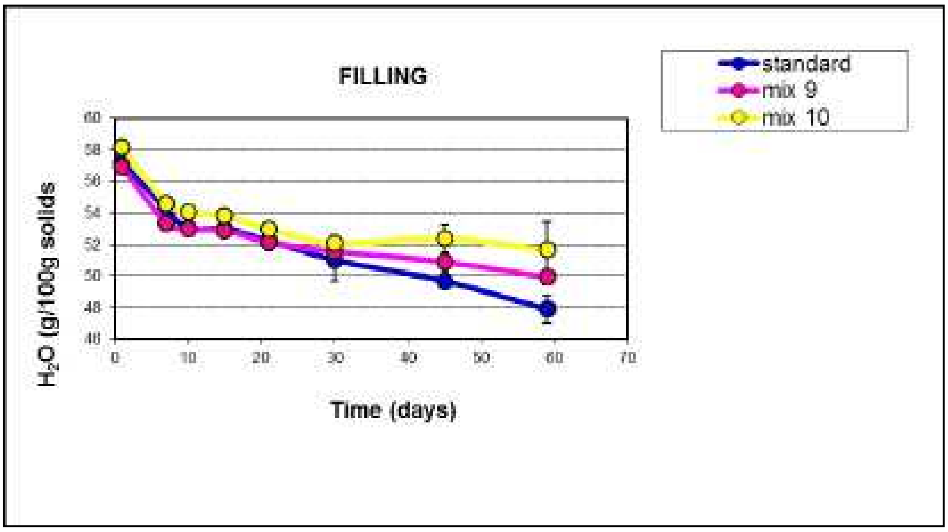


FIG 1.30 Variation of the moisture content in the filling.

The data reported in the above figures show an influence of the two formulations in modulating the transfer of moisture between the filling and dough, to a different extent in the MIX MILK and the MIX STARCH. In fact, compared to the standard, which consisted of stuffed pasta industrially produced, according to the recipe and the technology of the industrial partner, a greater retention of moisture by the filling for the whole time of the test can be inferred.

For a better understanding of the process, starting from the data shown graphically in the two previous figures, cumulative changes of humidity of the dough and the filling (expressed in percentage points) have been calculated. The two tables below show the results of this test (Table 1.7 and Table 1.8).

TABLE 1.7		**MOISTURE ABSORPTION FROM THE DOUGH**						
TIME (days)								
SAMPLE	**1**	**7**	**10**	**15**	**21**	**30**	**45**	**59**
STANDARD	0	1.6014	2.1913	2.2417	2.5933	1.4026	1.5531	0.1542
+ MIX MILK (9)	0	3.7278	3.3482	3.6646	3.3365	3.8099	3.4897	2.7374
+ MIX STARCH (10)	0	3.4794	3.0719	3.1197	2.9752	2.723	3.061	2.9453

What stands out from the data is the apparent minor transfer of moisture to the dough in stuffed pasta standard: the dough of this product humidifies in time, for transfer of moisture from the fillings, but to a lesser extent than the other two treatments. The comparison between MIX MILK and MIX STARCH brings out a different degree of humidification of the dough, increased for the first mix.

The global variation of moisture percentage of the filling is shown in the following table (Table 1.8); iin this case, the control sample shows the higher moisture loss, compared to those calculated for the fillings added with the two additives mix (Table 1.8).

TABLE 1.8		**LOSS OF MOISTURE OF THE FILLING**						
TIME (days)								
SAMPLE	**1**	**7**	**10**	**15**	**21**	**30**	**45**	**59**
STANDARD	0	- 2.9796	-4.1875	-4.2184	-4.9622	-6.2062	-7.5447	-9.3241
+ MIX MILK (9)	0	-3,5645	-3.9533	-4.0510	-4.7947	-5.3728	-6.0425	-7.0140
+ MIX STARCH (10)	0	-3.5591	-4.0901	-4.3355	-5.1719	-6.0768	-5.7897	-6.5032

Comparing the two tables, it is evident that the moisture gain of the dough does not compensate the moisture loss of the filling, in all the thesis considered. Such behavior is not surprising, since the transfer of moisture from the filling to pastry is definitely followed by a subsequent transfer of moisture to the confined environment of the pack, whose relative humidity is definitely lower than that of the product.

However, if the total loss of moisture from the tortelli is calculated (adding the data from the two tables above) interesting results are obtained (Table 1.9 and Figure 1.31).

TABLE 1.9		**TOTAL LOSS OF MOISTURE OF THE STUFFED PASTA**						
TIME (days)								
SAMPLE	**1**	**7**	**10**	**15**	**21**	**30**	**45**	**59**
CONTROL-	0	-1.3783	-1.9961	-1.9767	-2.3689	-4.8035	-5.9915	-9.1699
+ MIX MILK (9)	0	-0.1633	-0.6051	-0.3864	-1.4582	-1.5629	-2.5528	-4.2767
+ MIX STARCH (10)	0	-0.0797	-1.0182	-1.2158	-2.1967	-3.3539	-2.7287	-3.5580

The considerations that can be drawn from these calculations are the following:

- the two mixes added to the filling slow down effectively the transfer of moisture inside the stuffed pasta (or from the filling to the dough).
- The lower rate of water migration to the dough results in a smaller loss of water by evaporation from the same, and therefore the global variation of humidity of the product is much lower.
- The two formulations do not behave the same way, as d the one called MIX STARCH shows the best properties of the regulation of migration of water between two phases of a heterogeneous product, even if at the end of the experiment (about 60 days) the results obtained for the two experimental theses are similar.

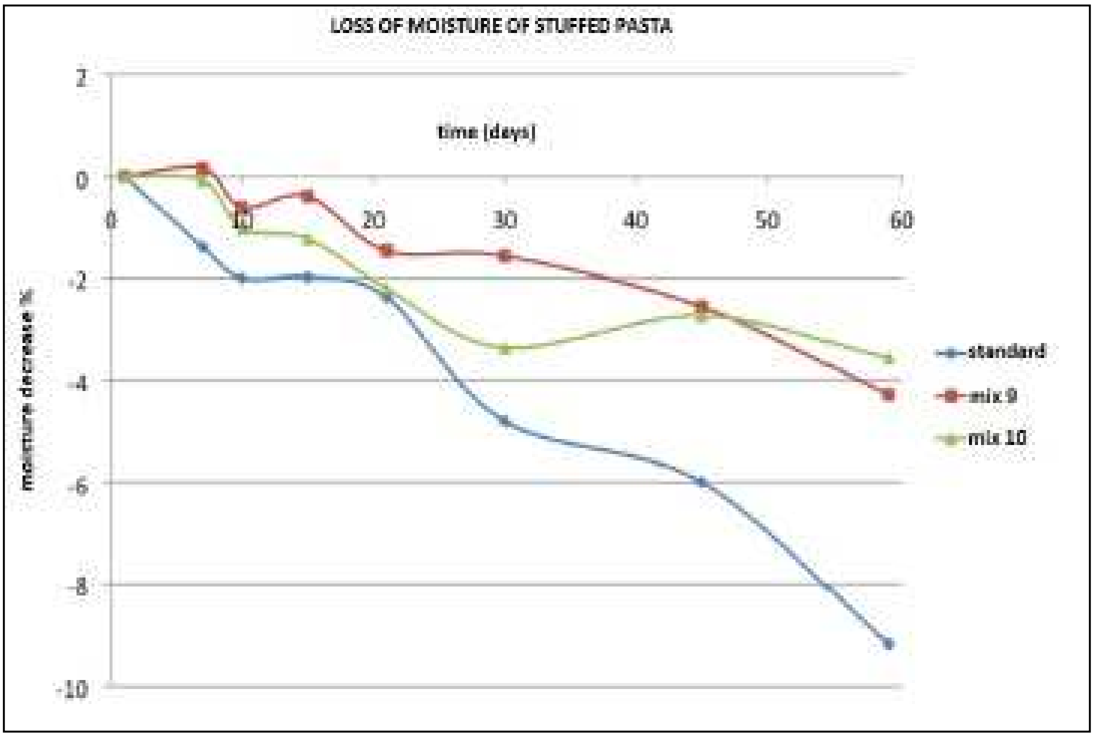


FIG 1.31 Evaluation of the moisture total loss of the industrial stuffed pasta.

It can therefore be concluded that, at the end of this experimental phase on a large scale, the two different prototypes exert the function for which they were designed and prepared, with a slight prevalence of the MIX STARCH. In particular, it is possible to observe the maintenance of a greater creaminess and softness of the filling, maintaining a turgid appearance of stuffed pasta and a right moisture level of the dough; on the contrary the ravioli control, in the same period of observation provided by the experimentation, show a marked wrinkling and their filling is more dry and hardened (FIgure 1.32 and Figure 1.33).

Appearance of the sample of stuffed pasta towards the end of their shelf-life
C= control; L= filling with MIX MILK; A= filling with MIX STARCH

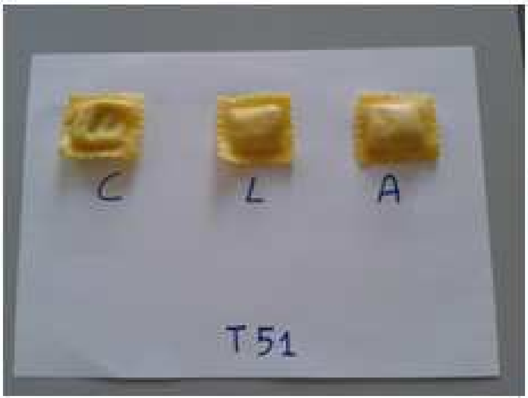


FIG 1.32 Frontal view of the three types of sample

FIG 1.33 Vision of a cross section of the three types of samples

In addition, the choice of the formulation could be related to the need to the presence of the milk allergen, or to other considerations not related to the effectiveness in slowing the transfer of moisture from the filling to the dough in stuffed pasta.

Demonstration System Operating In Operational Environment At Pre-Commercial Scale

Each of the two prototypes described above has been assigned a commercial brand, in this case the formulation 9, based on milk, was called LATOR, the formulation 10, based on starches, was named AMTOR.

In order to publicize the two new products, a flyer was developed, in collaboration between the research and development area, the commercial area and the marketing area. The flyer is visible on the company website.

The next phases of project development are not the subject of this thesis.

Conclusions

The goal of this part of this thesis was to identify methods and analytical procedures suitable for the evaluation of hygroscopic formulations aiming to regulate the moisture transfer in heterogeneous food products, or between the food and the surrounding environment.

The construction of the adsorption isotherms of water is the first step towards the evaluation of hygroscopic properties, both for ingredients and for complex food products. Knowing how to change the value of relative humidity (or water activity) of a food matrix according to the variation of its moisture contents is crucial for the evaluation of technological processes such as dehydration or partial water evaporation, with the aim of reaching an area of biological and chemical stability of the matrices themselves.

Isotherms of ingredients with potential hygroscopic activities allowed to obtain interesting information. At first, it has been found that apparently similar ingredients, with the same values of initial moisture content (m_0) and water activity (a_{w0}), have very different water adsorption capacities (see the comparison between starch type 3 and potato fibers). Evidently, the initial state of the products give not complete information about the complex interactions that water has with the functional components of the ingredient and with its physical structure. The examination of experimental isotherms enabled, therefore, the selection of two groups of potential strongly hygroscopic ingredients: milk derivatives and starches. However, the simple isotherms are not able to provide information on the potential reversibility of the adsorption phenomenon, so it was important to assess the ingredients according their Water retention capacity (WRC), or the ability to bind water and retain it.

Taking into account the results obtained, it was possible come up with the selection of ingredients and the formulation of homogeneous mixtures with a high degree of WRC, and subsequent use in an industrial trial. Certainly the selection of the various ingredients for the formulation to be use in the production phase of the stuffed pasta needed to take into consideration treatments with the high temperatures expected in the industrial pasteurization, assessing the effectiveness and characteristics of these preparations both hot and cold. The set of results obtained then led to the selection of two mixtures, called MIX MILK and MIX STARCH, which were then added to the stuffing for filled pasta, prepared according to the normal industrial process.

At the end of the trial on stuffed pasta, one can draw the following considerations:

- the two mixes added to the filling are able to slow down the transfer of moisture to the interior stuffed pasta (ie from the filling to dough).
- The lower water migration rate to the dough results in a smaller loss of water by evaporation from the dough, and therefore the global variation of humidity of the product is much lower.
- Furthermore, the two formulations do not behave the same way and the one called MIX STARCH seems to possess the best properties, with regards to the regulation of water migration between

two phases of a heterogeneous product, even if at the end of the experiment (about 60 days) it may be stated that both formulations (MIX MILK and MIX STARCH) display a satisfying performance, and that the choice of one over the other formulations could be related other parameters.

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Chapter 2

A New Leavening Powder For Bakery

ABSTRACT

"Baked products are foods manufactured from recipes largely based on or containing significant quantities of wheat or other cereal flours which are blended with other ingredients, are formed into distinctive shapes and undergo a heat-processing step which involves the removal of moisture in an oven located in a bakery".

Baked products have a long history of production, though the moment in time when humans first learnt how to bake with cereal grains to improve their palatability and digestibility is not known.

The world of bakery products does not consist of discretely-defined groups clearly separated from one another by rigid rules. In fact a large number of new products are successful because they break the conventional rulesets that have evolved to define particular product areas.

The most common ingredients used in baking are the following: **Flour, Yeast, Fat, Sugar, Baking soda, Baking powder, Egg, Salt.** In practice, bakers tend to group ingredients into three categories based on their level of usage in formulations: major, minor and micro.

In this work particular attention has been given to the study of a particular type of minor ingredients, the leavening agents.

Although these minor ingredients typically range from 5 to 10% (or sometimes less) on a formula weight basis, they can make or break product success.

Leavening acids are selected primarily on the basis of reactivity how fast they react and at what temperatures. Reactivity depends mostly on solubility, which in turn depends on chemical composition, particle size, and special treatments such as coating. The choice of which type of baking powder to use in a given mix will be based on when gas evolution is required and will depend on the end product and how it is prepared. Furthermore the selection of leavening acid and ratio of leavening acid to baking soda influences the taste of baked product, eg SAPP and GDL have a slightly bitter aftertaste in some applications, but the SAPP taste can be masked by using sufficient baking soda and by adding a source of calcium ions, sugar, or flavorings.

The aim of this work was to develop a new chemical leavening agent for cakes, with an improved leavening action, and able to endow the finished product with a more natural taste and less artifact than REIBAKING POWDER (old formulation).

In a preliminary phase, the new formulation was developed selecting the most suitable acidulant. Then the two products, the new formulation and REIBAKING POWDER were compared in their leavening action, by making sponge cakes.

Ultimately a paired comparison test was performed for determining whether a perceptible difference exists between the two products and which one is preferred by the panelists.

Introduction

Definition

The term "baked products" is applied to a wide range of food products, including breads, cakes, pastries, cookies and crackers and many other products, and it can be difficult to identify a common thread linking the members of such a diverse group. The most commonly identified link is that they all use recipes that are based on wheat flour. This definition, though, would need to be expanded to include baked goods such as gluten-free products, used by people with coeliac digestive disorders, or rye bread, which are still considered to be baked products even though they are based on cereals other than wheat. However, the same leniency of definition could hardly be extended to include meringues, which contain no cereal-based material at all, let alone wheat flour, their main components being sugar and egg white. It may be more appropriate to consider that baked products are those products which are manufactured in a bakery, that is the place of manufacture defines the product rather than some ingredient, recipe or process feature.

One view is that baked products should be defined as having undergone heat processing baking which causes changes in both form and structure.

The weakness of defining baked products as being those which have undergone a heat-processing step is that the same definition could be applied to any form of cooked product. Physical and chemical structures in all food are changed through heating, albeit in many cases adversely. In many people’s minds there is no distinction between the "baking" of bread and the "cooking" of bread, though bakers would be loath to accept that bread is "cooked". If we are to characterize or define baked products then it will have to be using a composite definition, perhaps something like:

"*Baked products* are foods manufactured from recipes largely based on or containing significant quantities of wheat or other cereal flours which are blended with other ingredients, are formed into distinctive shapes and undergo a heat-processing step which involves the removal of moisture in an oven located in a bakery".

History Of Baked Products

Baked products have a long history of production, though the moment in time when humans first learnt how to bake with cereal grains to improve their palatability and digestibility is not known. A flat, unleavened bread is most likely to have been the first baked product developed in the ancient Middle East, the accepted home of domesticated cereal-grain production. It is likely that the flat breads of antiquity were similar to those made by traditional means in the Middle East to this day. Baking would have been a craft practiced in most, if not all, households following its discovery. No doubt not all early bread production was based on wheat, with barley being a common ingredient, even in the peasant breads of the Middle Ages in Europe.

It is said that the Babylonians passed on the art of baking to the ancient Egyptians who in turn developed the first organized bakeries, that is, they made baking a specialist occupation. A painted panel of Rameses III at Thebes, dated c. 1200–1175 BC, depicts the court bakery making breads of different types (Pomeranz and Shellenberger, 1971).

It also shows the manufacture of cakes in different forms, including some baked in moulds or pans and others which were fried in hot oil.

In many cases the moulds or pans used to manufacture the breads and cakes took the shape of animals (some sacred to the Egyptians) and this suggests that the products were used in religious ceremonies or ritual feasting. No doubt the consumption of elaborate forms of breads, and certainly the more expensive cakes, was mainly restricted to the higher social classes, with bread consumption in the lower classes being confined to coarse, flat breads.

The ceremonial functions of bread The ceremonial functions of bread are recorded in many ancient texts. Fermentation and its role in bread aeration were known about at

this time. The ancient Hebrews distinguished between the leavened and unleavened forms of bread. Even today the unleavened bread is reserved for certain ceremonial occasions. Bread quickly took its place in the psyche of humankind in the ancient world, and the technology spread rapidly wherever wheat and other cereal grains could be grown.

Later, as wheat and other grains began to be imported and exported around the ancient world, the art of baking either spread with the grain or was discovered in different locations. No doubt three thousand years ago bakers were developing their own distinctive style of bread based on their cultural beliefs or just for the simple reason of wanting to be different from their competitors.

References to bread and baking begin to appear in Greek literature from the seventh century BC. Wheat became so important that at one time its export from Greece was prohibited, and bread was such a staple and important food that its weight and price were fixed in law.

The place of wheat and bread in religion remained pivotal and the Greeks built temples to the goddess Demeter, who has remained associated with agriculture since those ancient times.

The importance of bread was not lost on successive Roman emperors either, and the goddess Ceres was high on the list of important gods. So important was the provision of bread to the Romans, that it is considered that much of the expansion of their empire was driven by the need to acquire control of more wheat-growing areas to feed her armies and growing homeland population. Indeed, it is claimed by some that the Roman invasion of the British Isles was mostly about acquiring control of the large wheat and barley growing areas that existed at that time.

The status of the baker began to change during the years of the Roman Empire. It became a profession for men, and baking acquired a respectable and significant status as a trade. During this period the first guilds, or trade unions, of bakers began to form, reflecting the respectable nature of the trade. Government interference with the trade of baking was never far away. This was because of the political importance of bread and its use to manipulate popular opinion (popularized in the saying ‘bread and circuses’ when applied to pleasing the masses).

Control was ever present, with the weights of bread and its price being fixed on many occasions. Free bread was the Roman form of alms and if the Emperor could not provide everyone with bread he soon lost the Imperial Crown, if not his life!

While the manufacture of bread may largely have disappeared from the historical records of the so-called Dark Ages it certainly still persisted. There are occasional references to baking activities. For example, in England a legend has it that an Anglo-Saxon king, Alfred, burnt the cakes while thinking about the forthcoming struggle with the Vikings for control of England. Whether true or not such stories continue to reinforce the crucial position that bread and baking had in people’s minds. Control of the baking industry was ever present throughout history. In the UK, the Assize of Bread was introduced in 1226 to control weight and price, and remained in force for 450 years.

In the Middle Ages baking was well established as a profession throughout Europe and many of today’s bread forms were developed.

The basis of some of the change and development was the use of sifting to remove branny materials from the ground meal. White flour was used to make products for the richer elements of society with whole-meal and coarse, mixed grain breads being reserved for the lower orders. The diversification of baked products which accelerated in the medieval period in Europe gave us the basis of our modern cakes and pastries (Stanley P. et al., 2006).

Classification Of Baked Products

Given that baking has such a long history and so many traditions associated with it, how have the various groups of baked products come to be defined? Unlike botany or zoology there has never been an attempt to develop a specific taxonomy of baked products. In part this may be because of the long, local traditions associated with the manufacture of baked products and therefore the difficulties associated with translation from one tongue to another of the terms and descriptors used for the products and their associated baking processes.

To some extent, this nomenclature problem has persisted to the present day. For example, in English the term ‘biscuit’ is commonly used for describing a low moisture, hard-eating, sweetened, thin product with a long shelf-life, that is eaten as a snack. In the USA, however, it commonly refers to a sweetened product of intermediate moisture, commonly eaten at breakfast along with savoury foods. The UK biscuit is closer to the US cookie while the US biscuit is closer to a UK scone. To increase the confusion, the French biskuit refers to a low moisture, dry-eating, long-shelf-life, sponge-type cake with an aerated structure. The closest UK product to the French biskuit is indeed a sponge cake, though with higher moisture content.

We cannot blame differences in language and culture entirely, though, for the lack of a baking taxonomy – after all the same problems must have arisen (and probably still exist today) in botany and zoology.

However, scientists involved in such subjects did eventually agree a common taxonomy (largely) and a common descriptive language (Latin). One wonders whether the long traditions and more emotive nature of baking have prevented such a development. After all, get a handful of bakers together in a room and they seldom agree on anything to do with baked products. Despite (or because of) its long history, baking still has strong and deep roots in the craft and still struggles to develop its scientific credibility. Until it truly graduates to being a science a common taxonomy remains impossible.

Common English dictionary definitions for groups of baked products include:

- *Bread* – *n*. food made of flour or meal (and) baked
- *Cake* – *n*. baked, sweetened bread
- *Biscuit* – *n*. dry, small, thin variety of cake
- *Pastry* – *n*. article of food made chiefly of flour, fat and water

All of the above definitions illustrate the difficulties associated with defining the various groups of baked products.

As stated earlier, one of the purposes of the approaches that will be taken to considering the family of baked products is to provide the opportunity for greater innovation using underpinning knowledge of how baked products are characterized.

In Figure 2.1 the positions of examples of baked products are plotted using a 2-dimensional diagram in which the two axes are based on the ratio of sugar to flour in the recipe (X axis) and the ratio of fat to flour in the recipe (Y axis).

The world of bakery products does not consist of discretely-defined groups clearly separated from one another by rigid rules. In fact many successful new products are successful because they break the conventional rulesets that have evolved to define particular product areas (Stanley P. et al., 2006)

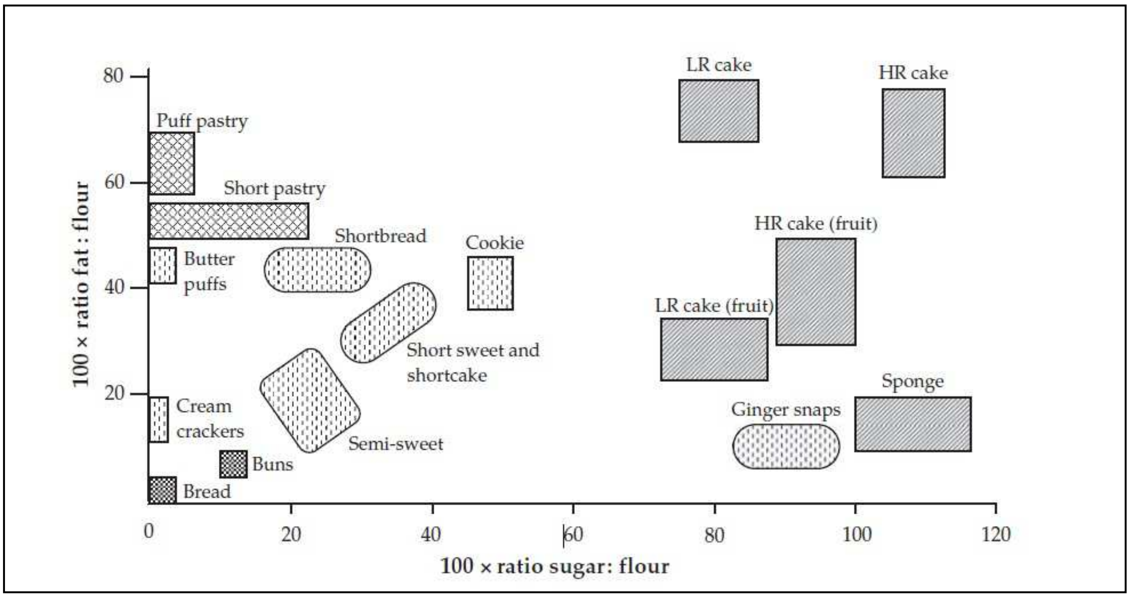


FIG 2.1. A two-dimensional representation of bakery products based on ratios of sugar and fat to flour in the recipe.

Bakery Ingredients

The most common ingredients used in baking are the following:

- **Flour:** this provides most of the bulk of the baked item. For bread baking, the flour should be a wheat flour which is high in gluten (protein) as this is the substance that gives bread its fine texture and supports the ingredients during rising.
- **Yeast**: Yeast is a plant that feeds on starch and sugars, releasing CO2, alcohol and sugar. The CO2 bubbles give the dough a light, airy texture.
- **Fat:** gives a softer texture and helps prevent the CO2 bubbles from escaping from the mixture too soon.
- **Sugar**: provides a direct food source for the yeast, improving its action.
- **Baking soda**: releases CO2 according to the equation

  $2NaHCO3 \rightarrow Na2CO3 + CO2 + H2O$

  However, as this also produces a strong base, Na2CO3, which has to be neutralized, baking powder is usually used instead.
- **Baking powder:** baking powder is baking soda with acid added. This neutralizes the base and produces more CO2 according to the following equation:

  $NaHCO3 + H+ \rightarrow Na+ + H2O + CO2$
- **Egg:** beaten egg white, like fat, helps to retain gas bubbles, while egg alone acts as a binder. egg white, like fat, helps to retain gas bubbles, while egg alone acts as a binder.

- **Salt:** salt adds flavour, and strengthens soft fat and sugar mixtures.

These ingredients are used in varying proportions to produce different products. As well as home baking, these include commercial cake mixes and breads (Pop G., 2007).

In practice, bakers tend to group ingredients into three categories based on their level of usage in formulations: major, minor and micro.

- **Major**, also termed "bulk," ingredients make up the majority of the formulation. Flour, for example, constitutes around 55 to 60% (formula weight) or more of bread's raw materials.
- **Minor** ingredients typically range from 5 to 10% (formula weight)
- **Micro** ingredients are those added at 5% or less.

This classification came about when bakeries started installing automated ingredient handling systems.

Manual scaling and hand-add delivery usually characterize the handling of minor and micro ingredients. A good number of large bakeries do automate their ingredient systems through the micro level, but it is far more common to find only the bulk materials dispensed through computerized systems (Pyler et al., 2008).

Focus On Minor Ingredients

Although minor ingredients typically range from 5 to 10% (or sometimes less) on a formula weight basis, they can make or break product success. Within this category, we include **leavening systems** — microbial cultures of yeast and/or bacteria, **chemical leavening**, air and steam. Other ingredients used at this level include dairy products and eggs, added starches and air enhancement ingredients (Pyler et al., 2008).

Leavening Agents

Leavening lightens doughs, enhancing the volume, texture, eating quality. The word "leaven" can be tracked through Middle English's "levain" to the Latin "levare," meaning "to raise." The function of leavening agents is to aerate the dough or batter and make it light and porous. When baked, the porosity translates into the crumb of the finished product. Leavening, thus, also tenderizes the crumb and contributes to the esthetic enjoyment of the final product by giving it uniform cell structure, bright crumb color, soft texture and enhanced palatability.

The process of leavening involves creating and enlarging the gas cells in dough or batter, cells that expand under the influence of time and heat to increase the overall size of the dough piece before its starch-and-protein matrix gelatinizes and sets. Mixing incorporates air into the dough mass, thus nucleating the bubbles essential to every style of leavening. Batters cannot create their own cells, only mixing does. Without the bubble nuclei, any gas generated by biological or chemical means would merely dissolve in the free water of the dough (Pyler et al., 2008). The tiny air bubbles formed during mixing collect the gaseous products of leavening. While such air bubbles are enough to leaven angel food cakes, nearly every other formulation requires additional leavening gases. The ingredients that contribute leavening effects often provide other functional properties and add to, or detract from, the product's texture and appearance. Leavens such as baker's yeast, ferment or a portion of fermenting sponge consist of living microbes that generate carbon dioxide, ethanol and other volatile organic compounds that all and inflate the air cells created by mixing. Another category of ingredients leavens by chemical action. This process combines alkaline baking soda with an acid material such as buttermilk or leavening acids to generate carbon dioxide, which aerates and expands the batter's volume before the heat of the oven sets its structure. Not all leavening powders are alike in their gassing power, as noted in Table 2.1.

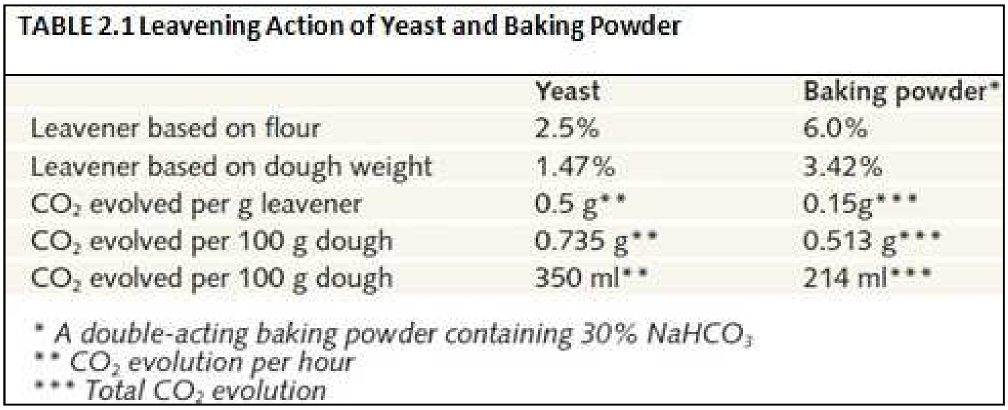


TABLE 2.1 Leavening Action of Yeast and Baking Powder

	Yeast	Baking powder*
Leavener based on flour	2.5%	6.0%
Leavener based on dough weight	1.47%	3.42%
CO_2 evolved per g leavener	0.5 g**	0.15g***
CO_2 evolved per 100 g dough	0.735 g**	0.513 g***
CO_2 evolved per 100 g dough	350 ml**	214 ml***

** A double-acting baking powder containing 30% $NaHCO_3$*
*** CO_2 evolution per hour*
**** Total CO_2 evolution*

While chemical leavening releases its gas relatively quickly, there is no further leavening action as with yeast. But yeast may not be efficient in all baked foods (Pyler et al., 2008).

Chemical Leavening

Chemical leaveners are used to give cookies, cakes, and other baked goods their characteristic textures. They produce gas from the reaction that takes place when a carbon dioxide source and an acid are mixed together and come into contact with water. The gas forms bubbles that are trapped in the batter or dough and then expand during baking to form the holes that are retained in the finished product.

The timing of the release of CO2 is critical in establishing uniform cell structure. Upon heating, the CO2 will release and expand, resulting in the increased volume and desirable texture characteristic of good tasting, high quality baked goods (Manthey, 2002).

Baking soda is the most common carbon dioxide source. It is low in cost, high in purity, easy to handle, and leaves no after taste. Flour and other ingredients are slightly acidic, so baking soda will release some carbon dioxide if added by it but will produce more when more acid is added.

Baking powder contains baking soda, one or more leavening acids, and a filler. The leavening acids are added in their powdered form as salts which do not react until they dissolve in water. The filler stabilizes the product by keeping the baking soda and leavening acid separate and standardizes it to the desired strength (Pop, 2007).

Leavening acids are selected primarily on the basis of reactivity how fast they react and at what temperatures. Reactivity depends mostly on solubility, which in turn depends on chemical composition, particle size, and special treatments such as coating (Pop, 2007).

Single-acting baking powders contain a single leavening acid and can be slow acting or fast acting. Slow-acting types are the most common and use a slow-acting acid like SALP (sodium aluminum phosphate) that reacts very little until heated in the oven. Fast-acting types are less common but use a fast-acting acid such as monopotassium tartrate (cream of tartar) to provide gas production at low temperatures immediately after addition (Brodie, 2006).

Double-acting baking powders contain a mixture of a fast-acting leavening acid like MCP (monocalcium phosphate monohydrate) and a slow-acting leavening acid like SAPP (sodium acid pyrophosphate). They react partially at low temperatures and partially at high temperatures to provide uniform leavening throughout processing (Brodie, 2006).

Taking all into consideration we could say that the key to superior chemical leavening is the selection of the correct type and grade of bicarbonate and acid for the baked good and the baking process being used (Table 2.2).

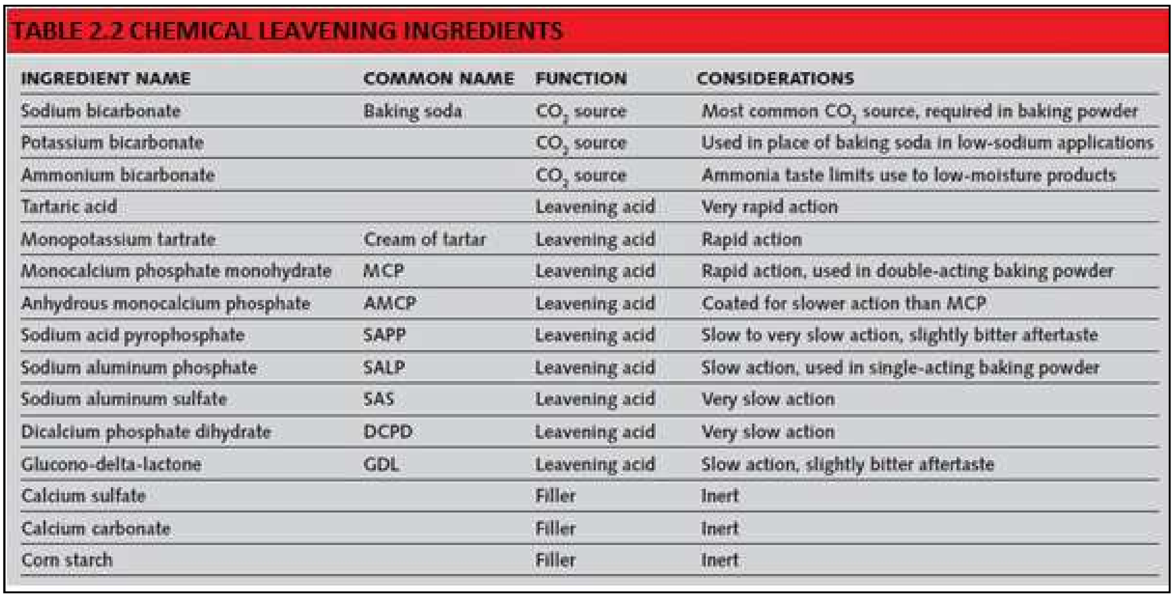


TABLE 2.2 CHEMICAL LEAVENING INGREDIENTS

INGREDIENT NAME	COMMON NAME	FUNCTION	CONSIDERATIONS
Sodium bicarbonate	Baking soda	CO_2 source	Most common CO_2 source, required in baking powder
Potassium bicarbonate		CO_2 source	Used in place of baking soda in low-sodium applications
Ammonium bicarbonate		CO_2 source	Ammonia taste limits use to low-moisture products
Tartaric acid		Leavening acid	Very rapid action
Monopotassium tartrate	Cream of tartar	Leavening acid	Rapid action
Monocalcium phosphate monohydrate	MCP	Leavening acid	Rapid action, used in double-acting baking powder
Anhydrous monocalcium phosphate	AMCP	Leavening acid	Coated for slower action than MCP
Sodium acid pyrophosphate	SAPP	Leavening acid	Slow to very slow action, slightly bitter aftertaste
Sodium aluminum phosphate	SALP	Leavening acid	Slow action, used in single-acting baking powder
Sodium aluminum sulfate	SAS	Leavening acid	Very slow action
Dicalcium phosphate dihydrate	DCPD	Leavening acid	Very slow action
Glucono-delta-lactone	GDL	Leavening acid	Slow action, slightly bitter aftertaste
Calcium sulfate		Filler	Inert
Calcium carbonate		Filler	Inert
Corn starch		Filler	Inert

All leavening acids react with carbon dioxide sources to produce gas. The potential gas production depends on the amount of the carbon dioxide source. The actual gas production depends on the ratio of total leavening acids to carbon dioxide source.

The neutralizing value (NV) is the parts by weight of baking soda from which all available carbon dioxide will be released by 100 parts by weight of a leavening acid:

NV = g NaHCO3 neutralized by 100 g acid

e.g. cream of tartar has an N.V. of 200 and tartaric acid an N.V. of 100. Tartaric acid is more efficient (because only half the weight of tartaric acid is required to neutralise a given amount of baking soda compared with cream of tartar), but it cannot be used alone since it reacts very quickly. This means that a lot of gas is formed initially, but because it does not continue to be formed the gas doesn't hold the cake long enough for it to have the bubbles baked into it and so the cake collapses.
Leavening balance is achieved by using the neutralizing value to match the amount of leavening acid to the amount of baking soda so that the maximum amount of carbon dioxide is produced (Table 2.3). If too little leavening acid is added, less gas is produced and the residual baking soda raises the pH of the finished product. If too much leavening acid is added, gas production remains the same but SAPP and GDL will leave a bitter aftertaste in the finished product (Lallemand Baking Update).

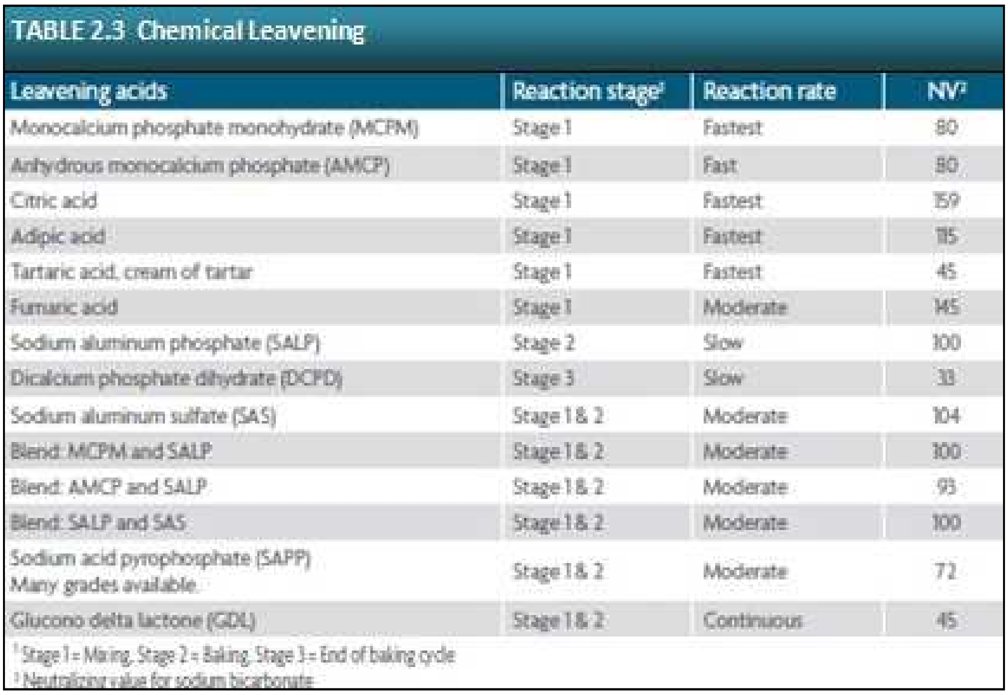


TABLE 2.3 Chemical Leavening

Leavening acids	Reaction stage[1]	Reaction rate	NV[2]
Monocalcium phosphate monohydrate (MCPM)	Stage 1	Fastest	80
Anhydrous monocalcium phosphate (AMCP)	Stage 1	Fast	80
Citric acid	Stage 1	Fastest	159
Adipic acid	Stage 1	Fastest	115
Tartaric acid, cream of tartar	Stage 1	Fastest	45
Fumaric acid	Stage 1	Moderate	145
Sodium aluminum phosphate (SALP)	Stage 2	Slow	100
Dicalcium phosphate dihydrate (DCPD)	Stage 3	Slow	33
Sodium aluminum sulfate (SAS)	Stage 1 & 2	Moderate	104
Blend: MCPM and SALP	Stage 1 & 2	Moderate	100
Blend: AMCP and SALP	Stage 1 & 2	Moderate	93
Blend: SALP and SAS	Stage 1 & 2	Moderate	100
Sodium acid pyrophosphate (SAPP) Many grades available	Stage 1 & 2	Moderate	72
Glucono delta lactone (GDL)	Stage 1 & 2	Continuous	45

[1] Stage 1 = Mixing, Stage 2 = Baking, Stage 3 = End of baking cycle
[2] Neutralizing value for sodium bicarbonate

The rate at which carbon dioxide is released is a key factor in the use of baking powder (see TABLE 2.3). The rate of carbon dioxide release depends on a number of factors, including the temperature of the batter. A significant factor in controlling the rate is the particle size of the acid and bicarbonate components and the choice of acidulant. It is important to ensure that the components of the baking powder are able to go to complete reaction. If the particle size of any of the components is too coarse then not only will there be a loss of carbon dioxide through an incomplete reaction but there may also be spots of unreacted material that lead to quality losses associated with product appearance and taste (Parks et al., 1960).

The nature of the acidulant is probably the most important factor in controlling the rate of carbon-dioxide evolution. In broad terms, baking powders may be classed as slow- or fast-acting, or, if a mixture of slow and fast-acting acids is used, as double-acting (Table 2.3). The choice of which type of baking powder to use in a given mix will be based on when gas evolution is required (Lallemand Baking Update).

The Choise Of Leavening Powder

The choice of leavening agents will depend on the end product and how it is prepared.

In particular choosing a leavening agent are taken into account the different variables which are examined below.

Potassium bicarbonate and **ammonium bicarbonate** are carbon dioxide sources that are sometimes used in place of baking soda. Potassium bicarbonate can be used for low sodium applications. Ammonium bicarbonate can be used for low-moisture finished products (such as cookies) where its ammonia taste dissipates after baking.

Taste is influenced by the choice of leavening acid and ratio of leavening acid to baking soda. SAPP and GDL have a slightly bitter aftertaste in some applications, but the SAPP taste can be masked by using sufficient baking soda and by adding a source of calcium ions, sugar, or flavorings.

Texture is also influenced by the choice of leavening acid; calcium and aluminum containing leavening acids allow good gluten development for volume and texture, while sulfates and phosphates interfere with gluten development and reduce gas retention.

GDL also allows good gluten development and gives acceptable crumb texture in breadlike products which normally are leavened with yeast.

Crumb color is influenced by the pH of the finished product; a low pH from low baking soda levels gives whiter crumb color.

A high pH from high baking soda levels gives a darker crumb color that is desirable in chocolate products (Lallemand Baking Update).

Applications

Cake donuts require little leavening during mixing, then some for buoyancy at the start of frying, and considerable fast leavening during frying. SAPP is commonly used as the leavening acid, sometimes with added MCP for initial activity or GDL for late activity.

Layer cakes require leavening during mixing to give good batter consistency for pan fill, and additional leavening during baking for optimum crumb texture and volume. Double-acting baking powder is commonly used with MCP as the fast leavening acid and SAPP or SALP as the slow-leavening acid.

Refrigerated doughs for biscuits require a leavening system which produces some gas in the can after mixing, but which also retains the major part of the activity until the biscuit can is opened and the biscuits are baked. MCP is used to have some gas produced in the can, while SALP or DCPD are used to produce gas in the oven after a long period of refrigerated storage.

Biscuits and **muffins** require fast leavening in the oven because of their short bake times. Double-acting baking powder is commonly used with MCP or coated MCP as the fast-leavening acid and SALP as the slow-leavening acid (Lallemand Baking Update).

Aim

In bakery the leavening systems are included in the category of minor ingredients, typically range from 5 to 10% (by weight); these ingredients lighten the dough, enhancing the volume, texture, eating quality.

The process of leavening involves creating and enlarging the gas cells in dough or batter, cells that expand under the influence of time and heat to increase the overall size of the dough piece before its starch-and-protein matrix gelatinizes and sets.

Leavens such as baker's yeast, ferment or a portion of fermenting sponge consist of living microbes that generate carbon dioxide. The ingredients that contribute leavening effects often provide other functional properties and add to, or detract from, the product’s texture and appearance. Leavens such as baker's yeast, ferment or a portion of fermenting sponge consist of living microbes that generate carbon dioxide, ethanol and other volatile organic compounds that all and in ate the air cells created by mixing. Another category of ingredients leavens by chemical action. This process combines alkaline baking soda with an acid material such as buttermilk or leavening acids to generate carbon dioxide, which aerates and expands the batter’s volume before the heat of the oven sets its structure.

While chemical leavening releases its gas relatively quickly, there is no further leavening action as with yeast.

Leavening acids are selected primarily on the basis of reactivity how fast they react and at what temperatures. Reactivity depends mostly on solubility, which in turn depends on chemical composition, particle size, and special treatments such as coating.

The choice of which type of baking powder to use in a given mix will be based on when gas evolution is required and will depend on the end product and how it is prepared.

Furthermore the selection of leavening acid and ratio of leavening acid to baking soda influences the taste of baked product, eg SAPP and GDL have a slightly bitter aftertaste in some applications, but the SAPP taste can be masked by using sufficient baking soda and by adding a source of calcium ions, sugar, or flavorings.

The aim of this work was to develop a new chemical leavening agent for cakes, with an improved leavening action, and able to endow the finished product with a more natural taste and less artifact than REIBAKING POWDER (old formulation).

The proposal to develop a new product stemmed from the company need to answer customers requirements, aiming to align the performance of the old product to that of market competitors.

Materials And Methods

BAKING SODA: also known as SODIUM BICARBONATE is the chemical compound with the formula $NaHCO_3$. Sodium bicarbonate is a white solid that is crystalline but often appears as a fine powder. It has a slightly salty, alkaline taste resembling that of washing soda (sodium carbonate). The natural mineral form is nahcolite. It is a component of the mineral natron and is found dissolved in many mineral springs. It is among the food additives encoded by European Union, identified by the initials E 500. Since it has long been known and is widely used, the salt has many related names such as *baking soda*, *bread soda*, *cooking soda*, and *bicarbonate of soda.* Sodium bicarbonate is the most common carbon dioxide source. It is low in cost, high in purity, easy to handle, and leaves no after taste (Smith et al., 2011)..

CREAM OF TARTAR: also known as POTASSIUM BITARTRATE, is a white powder; It is a natural product made from the natural crystalline acid deposited on the walls of wine-cellar vats. The production process includes strict and thorough quality control supervised by our engineers in order to ensure the ultimate quality of the product. Natural Cream of Tartar is included in the list of food additives under code number E-336(i) (Smith et al., 2011).

NATIVE CORN STARCH: is the starch of the corn grain obtained from the endosperm of the corn kernel and is not processed further after recovery from the source. In the food industry, starch has a wide range of applications ranging from being a thickener, gelling agent, to being a stabilizer for making snacks, meat products, fruit juices (Manek, et al., 2005). In baking powders native corn starch is used as an anti-caking agent, namely it stabilizes the product by keeping the baking soda and leavening acid separate and standardizes it to the desired strength (Manek, et al., 2005).

REIBAKING POWDER: old formulation of the baking powder producted by the company. It is composed of Sodium Acid Pyrophosfate (SAPP), Sodium Bicarbonate and native corn starch.

NEW LEAVENING POWDER: is a chemical leavening composed of Potassium Bitartrate, Sodium Bicarbonate and Native Corn Starch.

Evaluation Of Leavening Action

The leavening action of Reibaking powder and the new leavening powder are tested by making sponge cakes.

The sponge cake recipe is following.

Sponge Cake Recipe

Ingredients

200 g flour

180 g sugar

250 ml milk

20 ml water

3 tablespoon corn oil

6 g baking powder

Descriptions

1. Preheat the oven to 180°C.
2. Weigh all ingredients.
3. Put flour, sugar and baking powder in a medium bowl and mix well.
4. Add 3 tablespoons of corn oil and milk slowly, stirring constantly. Mix the ingredients together without leaving lumps.
5. Transfer the mixture into a baking dish and bake in the preheated oven for 30 to 45 minutes or until the cake tests done in the centre and the top is golden brown. Remove the cake from the oven and allow to cool completely.

The Paired Comparison Test

The method is applicable whether a difference exists in a single sensory attribute or in several, which means that it enables determination of whether there exists a perceptible difference concerning a given attribute, and the specification of the direction of difference, but it does not give any indication of the extent of that difference. The absence of difference for the attribute under study does not signify that there does not exist any difference between the two products.

This method is only applicable if the products are relatively homogeneous.

The method is effective

a) for determining

— whether a perceptible difference exists (paired difference test), or

— whether no perceptible difference exists (paired similarity test) when, for example, modifications are made to ingredients, processing, packaging, handling or storage operations, or

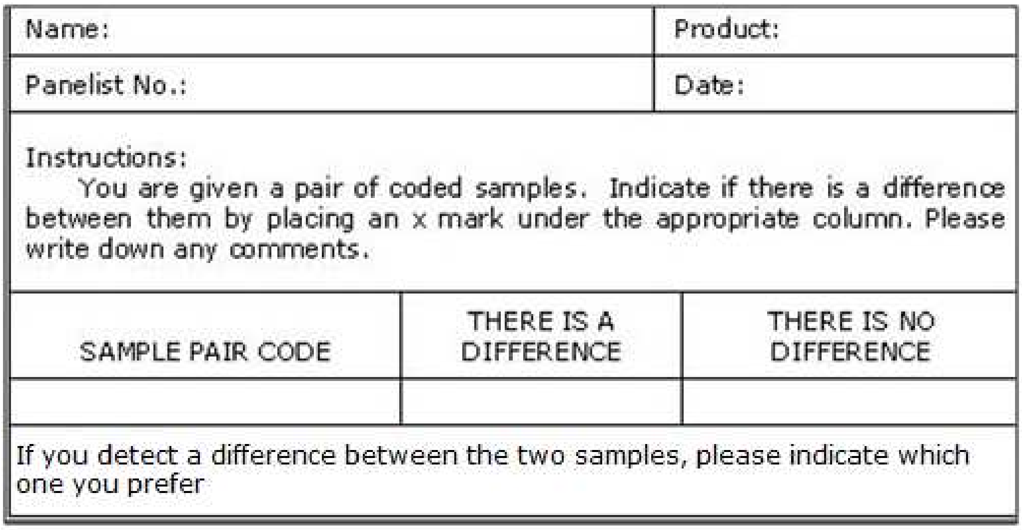
b) for selecting, training and monitoring assessors.

The paired test can also be used in order to compare two products in terms of preference (UNI ISO 5495:2005).

In this case, a paired comparison test was run to evaluate the effect of two different leavening agent on the flavour and texture of sponge cake. A sample (A), sponge cake contained REIBAKING POWDER, was compared with a sample(B), which contained the NEW LEAVENING AGENT DEVELOPED.

1. Sponge cake with two different leavening powder are prepared and then divided in many samples.
2. The samples were coded with three digit random numbers and presented to a panel of 30.
3. The panelists are presented simoultaneously with two samples and asked whether they perceive the samples to be the same or different; If they detected a difference between two sample, ask which one they prefer(Figure 2.2).

4. The collected data are processed using the tables of significance (Table 2.4).



Name:

Product:

Panelist No.:

Date:

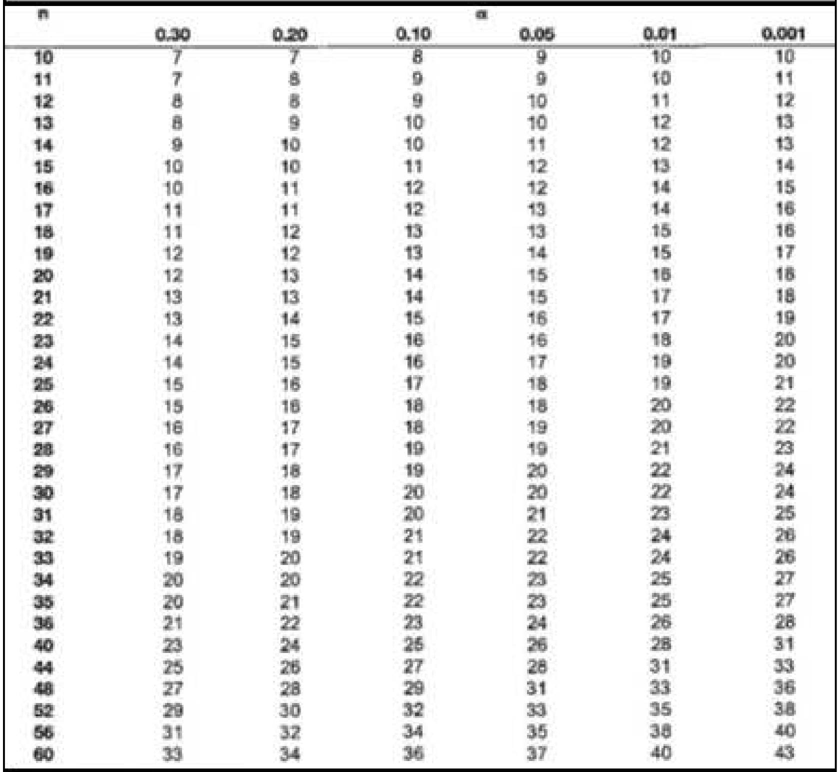
Instructions:
You are given a pair of coded samples. Indicate if there is a difference between them by placing an x mark under the appropriate column. Please write down any comments.

SAMPLE PAIR CODE	THERE IS A DIFFERENCE	THERE IS NO DIFFERENCE

If you detect a difference between the two samples, please indicate which one you prefer

FIG 2.2. Score sheet.

Table 2.4 Table of significance for the Paired Comparison Test



n	α					
	0.30	0.20	0.10	0.05	0.01	0.001
10	7	7	8	9	10	10
11	7	8	9	9	10	11
12	8	8	9	10	11	12
13	8	9	10	10	12	13
14	9	10	10	11	12	13
15	10	10	11	12	13	14
16	10	11	12	12	14	15
17	11	11	12	13	14	16
18	11	12	13	13	15	16
19	12	12	13	14	15	17
20	12	13	14	15	16	18
21	13	13	14	15	17	18
22	13	14	15	16	17	19
23	14	15	16	16	18	20
24	14	15	16	17	19	20
25	15	16	17	18	19	21
26	15	16	18	18	20	22
27	16	17	18	19	20	22
28	16	17	19	19	21	23
29	17	18	19	20	22	24
30	17	18	20	20	22	24
31	18	19	20	21	23	25
32	18	19	21	22	24	26
33	19	20	21	22	24	26
34	20	20	22	23	25	27
35	20	21	22	23	25	27
36	21	22	23	24	26	28
40	23	24	25	26	28	31
44	25	26	27	28	31	33
48	27	28	29	31	33	36
52	29	30	32	33	35	38
56	31	32	34	35	38	40
60	33	34	36	37	40	43

n= number of panelists
α= significance index

Results and Discussion

As well as in the previous chapter, the following paragraphs have been organized according to the sequence of Technology Readiness Levels (TRL), adopted by the European Commission in the 2014-2020 Horizon 2020 Programme for Research and Innovation (www.ec.europa/research/horizon2020), and it is reproduced below:

- TRL 0: Idea.
- TRL 1: Basic research.
- TRL 2: Technology formulation.
- TRL 3: Applied research.
- TRL 4: Small scale prototype built in a laboratory environment.
- TRL 5: Large scale prototype tested in intended environment.
- TRL 6: Prototype system tested in intended environment close to expected performance.
- TRL 7: Demonstration system operating in operational environment at pre-commercial scale.
- TRL 8: First of a kind commercial system.
- TRL 9: Full commercial application, technology available for consumers.

Idea

In this work the common chemical leavening ingredients were studied in order to develop a new baking powder for bakery, with an improved leavening action, and able to endow the finished product with a more natural taste and less artifact than REIBAKING POWDER (old formulation).

Basic Research

The preliminary phase has been oriented to the study of the common chemical leavening agents (listed in Table 2.2) in order to identify the ingredients which complies to the requirements for the new product.

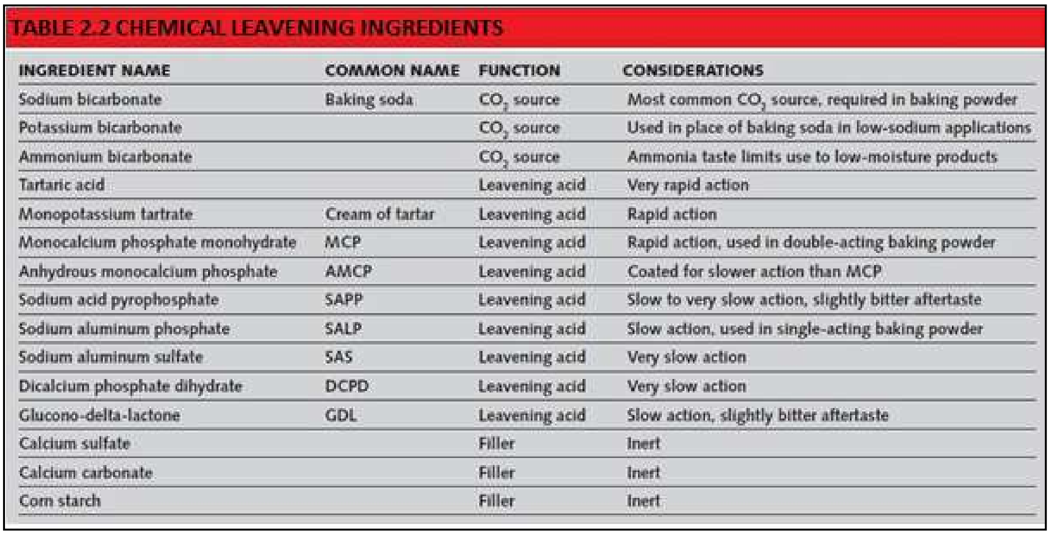


TABLE 2.2 CHEMICAL LEAVENING INGREDIENTS

INGREDIENT NAME	COMMON NAME	FUNCTION	CONSIDERATIONS
Sodium bicarbonate	Baking soda	CO_2 source	Most common CO_2 source, required in baking powder
Potassium bicarbonate		CO_2 source	Used in place of baking soda in low-sodium applications
Ammonium bicarbonate		CO_2 source	Ammonia taste limits use to low-moisture products
Tartaric acid		Leavening acid	Very rapid action
Monopotassium tartrate	Cream of tartar	Leavening acid	Rapid action
Monocalcium phosphate monohydrate	MCP	Leavening acid	Rapid action, used in double-acting baking powder
Anhydrous monocalcium phosphate	AMCP	Leavening acid	Coated for slower action than MCP
Sodium acid pyrophosphate	SAPP	Leavening acid	Slow to very slow action, slightly bitter aftertaste
Sodium aluminum phosphate	SALP	Leavening acid	Slow action, used in single-acting baking powder
Sodium aluminum sulfate	SAS	Leavening acid	Very slow action
Dicalcium phosphate dihydrate	DCPD	Leavening acid	Very slow action
Glucono-delta-lactone	GDL	Leavening acid	Slow action, slightly bitter aftertaste
Calcium sulfate		Filler	Inert
Calcium carbonate		Filler	Inert
Corn starch		Filler	Inert

Baking powder contains a CO_2 source, one or more leavening acids, and a filler. The leavening acids are added in their powdered form as salts which do not react until they dissolve in water. The filler stabilizes the product by keeping the baking soda and leavening acid separate and standardizes it to the desired strength (Pop, 2007).

As shown in the Table 2.2 each ingredient in addition to having a specific role in the baking powder in turn has the specific characteristics for which reason you choose one rather than the other. All leavening acids react with carbon dioxide sources to produce gas. The potential gas production depends on the amount of the carbon dioxide source. The actual gas production depends on the ratio of total leavening acids to carbon dioxide source (Parks et al., 1960).

Moreover the rate at which carbon dioxide is released is a key factor in the use of baking powder (see TABLE 2.3). The rate of carbon dioxide release depends on a number of factors, including the temperature of the batter. A significant factor in controlling the rate is the particle size of the acid and bicarbonate components and the choice of acidulant.

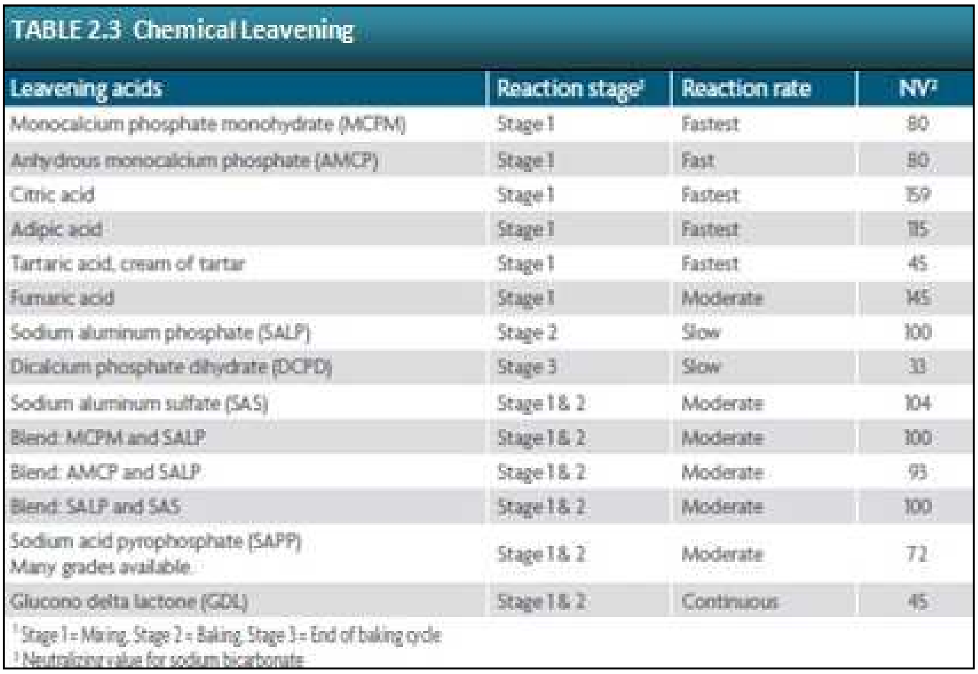


TABLE 2.3 Chemical Leavening

Leavening acids	Reaction stage[1]	Reaction rate	NV[2]
Monocalcium phosphate monohydrate (MCPM)	Stage 1	Fastest	80
Anhydrous monocalcium phosphate (AMCP)	Stage 1	Fast	80
Citric acid	Stage 1	Fastest	159
Adipic acid	Stage 1	Fastest	115
Tartaric acid, cream of tartar	Stage 1	Fastest	45
Fumaric acid	Stage 1	Moderate	145
Sodium aluminum phosphate (SALP)	Stage 2	Slow	100
Dicalcium phosphate dihydrate (DCPD)	Stage 3	Slow	33
Sodium aluminum sulfate (SAS)	Stage 1& 2	Moderate	104
Blend: MCPM and SALP	Stage 1& 2	Moderate	100
Blend: AMCP and SALP	Stage 1& 2	Moderate	93
Blend: SALP and SAS	Stage 1& 2	Moderate	100
Sodium acid pyrophosphate (SAPP) Many grades available	Stage 1& 2	Moderate	72
Glucono delta lactone (GDL)	Stage 1& 2	Continuous	45

[1] Stage 1 = Mixing, Stage 2 = Baking, Stage 3 = End of baking cycle

[2] Neutralizing value for sodium bicarbonate

Baking soda is the most common carbon dioxide source. It is low in cost, high in purity, easy to handle, and leaves no after taste. Flour and other ingredients are slightly acidic, so baking soda will release some carbon dioxide if added by it but will produce more when more acid is added (Pop, 2007).

The selection of leavening acid and ratio of leavening acid to baking soda influences the taste of baked product, eg SAPP and GDL have a slightly bitter aftertaste in some applications, but the SAPP taste can be masked by using sufficient baking soda and by adding a source of calcium ions, sugar, or flavorings (Lallemand Baking Update, 2001).

The old formulation, named REIBAKING POWDER, is composed of SAPP, baking soda and native corn starch; it showed little performance for leavening action and gave the finished product a metallic aftertaste.

It was decided to develop a new product looking for a acidulant, that put in the right proportion compared with sodium bicarbonate, conferring on baking powder these features:

- rapid leavening action
- natural taste, without aftertaste

Knowledge the reactivities of chemical leavenings allowed us to select Cream of Tartar as leavening acid able to give the new leavening agent requirements expected.

The Cream of Tartar is attributed to both a rapid action leavening that the ability to give the finished product a natural taste (see "Materials and Methods").

Technology formulation

All leavening acids react with carbon dioxide sources to produce gas. The potential gas production depends on the amount of the carbon dioxide source. The actual gas production depends on the ratio of total leavening acids to carbon dioxide source.

The neutralizing value (NV) is the parts by weight of baking soda from which all available carbon dioxide will be released by 100 parts by weight of a leavening acid:

$$\mathbf{NV} = \text{g } NaHCO_3 \text{ neutralized by 100 g acid}$$

Leavening balance is achieved by using the neutralizing value to match the amount of leavening acid to the amount of baking soda so that the maximum amount of carbon dioxide is produced.

Taking all into consideration a prototype of the new leavening powder was developed, composed of Cream of Tartar, Baking Soda and NativeCorn Starch, as filler.

Applied Research

The new formulation of baking powder, developed in preview phase, was tested by making sponge cakes (see Material and Methods), and it was compared to REIBAKING POWDER, in order to evaluation their reactivity.

Each of the chemical leavening agents has been added to the batter, using the same dosage, to measure their action leavening.

(a) **(b)**

FIG 2.3 Test of leavening action

(a) cake with REIBAKING POWDER **(b)** cake with the New Leavening Agent

As shown in Figure 2.3 both samples with chemical leavening agents had a good final volume, but the cake made with the New Leavening powder is more soft and less compact, and has more open porosity.

In these conditions we could say that although both leavening agents have the same influence on the final volume of the cake, the new formulation gives the finished product a better texture.

Small scale Prototype in Lab

In this phase of development it was evalueted the influence of each type of leavening powder on sponge cake taste, through an sensory exam.

In particular a Paired Comparison Test was performed in order to detect any differences or similarities in taste determined by the two leavening powders.

After baking, the cakes were put on the rack for cooling, and after 15-20 minutes the cakes were removed 30 panelists were chosen within the company, who were unaware of the research project, and each were presented two samples to exam.

After the sensory exam, the data processing was performed through a table of significance (see "Materials and Methods").

22 judges on 30 have detected the difference in taste between the two samples; corresponding to a significance of the test of 99%. Moreover all 22 panelists preferred the cake made with the new leavening powder because has a more natural taste without aftertaste.

Taking into consideration all the results so far, it could be concluded that the new formulation is responding to the expected requirements.

The later stages of development of this product are not subject of this thesis.

Conclusions

Analyzing the obtained results from the study about the development of the New Leavening powder we can drop the following conclusions:

- Knowledge the reactivities of chemical leavenings allowed us to select Cream of Tartar as leavening acid able to give the new leavening agent requirements expected.
- The New Leavening Powder, composed of Cream of Tartar , Baking soda e Native Corn Starch , tested by making sponge cake, gives the finished product a good volume and improved texture.
- The cakes made with the New Leavening Powder are preferred to those made with REIBAKING POWDER for their natural taste and to be free of aftertaste.

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UNI ISO 5495:2005 Sensory analysis -- Methodology -- Paired comparison test

Link

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- www.tartaric.com
- www.classofoods.com
- www.papillevagabonde.blogspot.it

Chapter 3

A New Cocoa powder for sprinkling use

ABSTRACT

Today, people around the world enjoy chocolate in thousands of different forms, consuming more than 3 million tons of cocoa beans annually. Each country and region has unique preferences and distinctive blends for candy and desserts.

The cocoa, chocolate, and confectionery industry employs hundreds of thousands of people around the world and is a key user of other agricultural commodities such as sugar, dairy, nuts, and fruits.

The genus Theobroma (The Latin name for cocoa) originated millions of years ago in South America, to the east of the Andes. Theobroma has been divided into twenty-two species of which T. cacao is the most widely known.

Cocoa is produced in countries in a belt between 10ºN and 10ºS of the Equator, where the climate is appropriate for growing cocoa trees. The largest producing countries are Côte d'Ivoire, Ghana and Indonesia.

The natural habitat of the cocoa tree is in the lower storey of the evergreen rainforest, and climatic factors, particularly temperature and rainfall, are important in encouraging optimum growth.

The world’s stock of cocoa trees tends to be grouped, by tradition, into three main varieties: Criollo, Forastero and Trinitario. Typically, cocoa is produced by smallholder or family subsistence farming**.**

Two products are solely derived from cocoa beans: chocolate and cocoa powder. These products are interrelated, and two intermediate products play an important role – cocoa mass and cocoa butter.

The chocolate industry needs a certain quantity of cocoa butter and this implies that a fixed quantity of cocoa powder is also produced.

Global market share for processing has remained stable, even as grindings increase to meet demand. Average year-over-year demand growth has been just over 3% since 2008.

Cocoa powder is used as a natural color and flavor in a wide variety of sweet foodstuffs. These may be solid, semisolid, or liquid, and consumed frozen or hot.

Because of its nature highly hygroscopic, cocoa powder has an important effect on rheology and water absorption in many of the products in which it is used.

Traditional cocoa powders absorb very quickly moisture from the dessert or from condensation in packaging, thus compromising the fresh, attractive presentation. The color of the cocoa powder becomes darker and the powder partly dissolves.

The aim of this work was therefore to develop a new cocoa for sprinkling use with a non hygroscopic character, that makes it perfect for decorating or topping desserts that are kept for instance in moist environment.

In the first phase of development of the new cocoa powder, in order to reach a good dry mix, that a balance in water activity will be established between the various components, different ingredients were evaluated as follows:

1. Evaluation of their Wettability behavior, as the potential for a powder to wet and absorb water at a given temperature

2. Measurements and drawings of water adsorption isotherms for selected ingredients, which represent their Water Activity (a_w) versus a moisture content (g H2O / g d.m.)

The new formulations of cocoa powder, developed in the first phase of study, are tested by sprinkling on tiramisù, and only two have been selected as the starting point for the next study, which involved partnership with a supplier.

Introduction

Definition

“Cocoa Powder” and “Fat-reduced Cocoa Powder” and "Highly Fat-reduced Cocoa Powder" are the products obtained from cocoa cake (Cf. Standard for Cocoa (Cacao) Mass (Cocoa/Chocolate Liquor) and Cocoa Cake) transformed into powder (CODEX STAN 105-1981, Revised 2001).

Origin

The genus Theobroma (The Latin name for cocoa) originated millions of years ago in South America, to the east of the Andes. Theobroma has been divided into twenty-two species of which T. cacao is the most widely known. It was the Maya who provided tangible evidence of cacao as a domesticated crop. Archaeological evidence in Costa Rica indicates that cacao was drunk by Maya traders as early as 400 BC. The Aztec culture, dominant in Mesoamerica from the fourteenth century to the Conquest, placed much emphasis on the sanctity of cacao.

The first outsider to drink chocolate was Christopher Columbus, who reached Nicaragua in 1502 searching for a sea route to the spices of the East. But it was Hernan Cortés, leader of an expedition in 1519 to the Aztec empire, who returned to Spain in 1528 bearing the Aztec recipe for *xocoatl* (chocolate drink) with him. The drink was initially received unenthusiastically and it was not until sugar was added that it became a popular drink in the Spanish courts (International Cocoa Organization Online Archive).

There were attempts to satisfy Spanish domestic demand by planting cacao in Spanish territories like the Dominican Republic, Trinidad and Haiti but these initially came to nothing. More successful were the Spanish Capuchin friars, who grew criollo cacao in Ecuador in about 1635. The rush by European mercantile nations to claim land to cultivate cacao began in earnest in the late seventeenth century. France introduced cacao to Martinique and St Lucia (1660), the Dominican Republic (1665), Brazil (1677), the Guianas (1684) and Grenada (1714); England had cacao growing in Jamaica by 1670; and, prior to this, the Dutch had taken over plantations in Curaçao when they seized the island in 1620.

The explosion in demand brought about by chocolate's affordability required yet more cacao to be cultivated. Amelonado cacao from Brazil was planted in Principe in 1822, Sao Tomé in 1830 and Fernando Po in 1854, then in Nigeria in 1874 and Ghana in 1879. There was already a small plantation in Bonny, eastern Nigeria established by Chief Iboningi in 1847, as well as other plantations run by the Coker family and established by the Christian missions. The seeds planted in Ghana were brought from Fernando Po by Tetteh Quarshie or his apprentice Adjah, after previous attempts by the Dutch (1815) and the Swiss (1843) to introduce cocoa in Ghana had failed. In Cameroon, cocoa was introduced during the colonial period of 1925 to 1939 (International Cocoa Organization Online Archive).

Cocoa growing

The cocoa tree grows best in tropical regions, on a belt between approximately 10° north and 10° south of the equator. Most cocoa is grown at an altitude of less than 400 metres (1,200 feet) above sea level.

Cocoa trees can grow to a height of around 10 metres when shaded by large forest trees. The fruit, or pod, is between 15 and 25 cm (6 to 10 inches) long, and contains 30 to 40 seeds, which become cocoa beans when fermented and dried. The pods grow both along the main stem of the tree and throughout the canopy. A cocoa tree becomes productive four to five years after planting and can remain productive for several decades (International Cocoa Organization Online Archive).

Climate Conditions

The natural habitat of the cocoa tree is in the lower storey of the evergreen rainforest, and climatic factors, particularly temperature and rainfall, are important in encouraging optimum growth.

Cocoa plants respond well to relatively high temperatures, with a maximum annual average of 30 - 32ºC and a minimum average of 18 - 21ºC.

Variations in the yield of cocoa trees from year to year are affected more by rainfall than by any other climatic factor. Trees are very sensitive to a soil water deficiency. Rainfall should be plentiful and well distributed through the year. An annual rainfall level of between 1,500mm and 2,000mm is generally preferred. Dry spells, where rainfall is less than 100mm per month, should not exceed three months.

A hot and humid atmosphere is essential for the optimum development of cocoa trees. In cocoa producing countries, relative humidity is generally high: often as much as 100% during the day, falling to 70-80% during the night.

The cocoa tree will make optimum use of any light available and traditionally has been grown under shade. Its natural environment is the Amazonian forest which provides natural shade trees. Shading is indispensable in a cocoa tree's early years (International Cocoa Organization online archive).

Soil conditions

Cocoa is grown in a wide variety of soil types. Cocoa needs a soil containing coarse particles and with a reasonable quantity of nutrients, to a depth of 1.5m to allow the development of a good root system. Below that level it is desirable not to have impermeable material, so that excess water can drain away. Cocoa will withstand waterlogging for short periods, but excess water should not linger. The cocoa tree is sensitive to a lack of water, so the soil must have both water retention properties and good drainage. The chemical properties of the topsoil are most important, as the plant has a large number of roots for absorbing nutrients. Cocoa can grow in soils with a pH in the range of 5.0-7.5. It can therefore cope with both acid and alkaline soil, but excessive acidity (pH 4.0 and below) or alkalinity (pH 8.0 and above) must be avoided. Cocoa is tolerant of acid soils, provided the nutrient content is high enough. The soil should also have a high content of organic matter: 3.5% in the top 15 centimetres of soil. Soils for cocoa must have certain anionic and cationic balances. Exchangeable bases in the soil should amount to at least 35% of the total cation exchange capacity (CEC), otherwise nutritional problems are likely. The optimum total nitrogen / total phosphorus ratio should be around 1.5 (International Cocoa Organization online archive).

Cocoa varieties

The world's stock of cocoa trees tends to be grouped, by tradition, into three main varieties – **Criollo**, **Forastero** and **Trinitario** – from which many different hybrids have been, and continue to be, developed (Table 3.1).

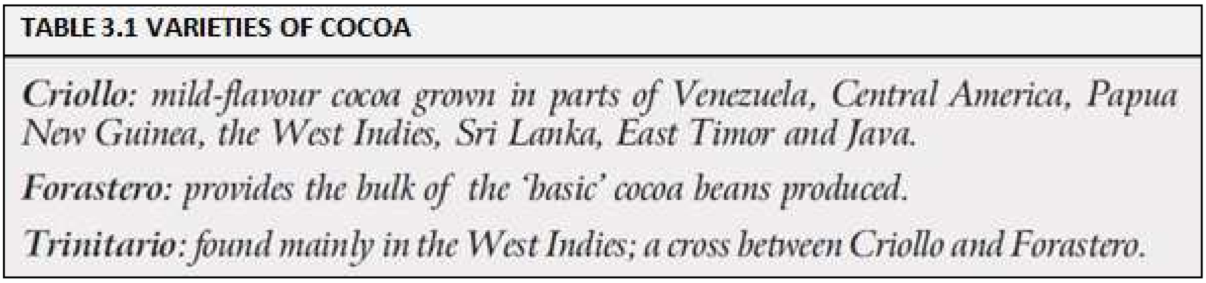

TABLE 3.1 VARIETIES OF COCOA

Criollo: mild-flavour cocoa grown in parts of Venezuela, Central America, Papua New Guinea, the West Indies, Sri Lanka, East Timor and Java.

Forastero: provides the bulk of the 'basic' cocoa beans produced.

Trinitario: found mainly in the West Indies; a cross between Criollo and Forastero.

Criollo-based varieties trace their origins back to Central America, including Mexico. **Forastero** varieties, accounting for by far the greatest tonnage of cocoa beans harvested, are believed to have originated further south, in the Amazon regions. The main producing countries rely for the majority of their production on the Forastero-based cocoa varieties and hybrids, the beans of which are usually referred to as basic.

Criollo and Trinitario beans, with some of their hybrids and the Ecuadorian Naçional, have produced what are known as fine or flavour cocoas. The world cocoa market distinguishes between two broad categories of cocoa beans: "fine or flavour" cocoa beans, and "bulk" or "ordinary" cocoa beans. As a generalisation, fine or flavour cocoa beans are produced from Criollo or Trinitario cocoa-tree varieties, while bulk cocoa beans come from Forastero trees. There are, however, known exceptions to this generalization. Nacional trees in Ecuador, considered to be Forastero-type trees, produce fine or flavour cocoa. On the other hand, Cameroon cocoa beans, produced by Trinitario-type trees and whose cocoa powder has a distinct and sought-after red colour, are classified as bulk cocoa beans (International Trade Centre Online Database).

The share of fine or flavour cocoa in the total world production of cocoa beans is just under 5% per annum. Virtually all major activity over the past five decades has involved bulk cocoa.

Being used almost exclusively for the making of speciality or ‘gourmet’ chocolate, fine or flavour cocoas have lost much of their commercial significance over the last century, even to the extent that the major trading houses have now ceased to deal in them (International Cocoa Organization Online Archive).

Cocoa Production

Two factors, above all, influence the supply of cocoa in the short to medium term. One is the area planted with cocoa; the other is the yield from the trees available for cropping.

Cocoa trees are capable of producing acceptable yields for several decades. Depending on the variety, it can take between eight and ten years for yields to peak, with the newer hybrids reaching their peak more quickly. The pace of decline thereafter is determined mainly by cultivation practices (International Cocoa Organization Online Archive).

The majority of Forastero cocoa trees planted today are hybrids either of several Forastero varieties or of a mix of Criollo and Forastero. Hybrids are preferred because they are more resistant to known diseases and they yield a higher tonnage per hectare. For example, where a cocoa farm cultivated under the traditional system of minimal maintenance may typically yield 300 to 500 kg per ha annually, newer hybrids cultivated commercially under ideal farming conditions have been known to yield as much as 2,500 kg per ha. Historically, the pattern of global cocoa production has been marked by periods of contraction, followed by periods of stabilization and then of rapid expansion (International Trade Centre Online Database).

Within these trends, however, there have been factors which have provided some balance to the overall supply picture. For example, at the time production levels were declining in certain traditional growing areas such as Brazil, Ghana, Nigeria and Cameroon – mainly owing to a combination of unfavourable economic conditions and crop disease – strong expansion was taking place in other countries such as Côte d’Ivoire and Indonesia (International Trade Centre Online Database).

Smallholder farming

Typically, cocoa is produced by smallholder or family subsistence farming. In West Africa, for example, many farms will have less than a hectare under cocoa. The same is generally true of South-East Asia, and of Indonesia in particular. There are exceptions, however. Some large cocoa farms can be found in Brazil and Ecuador, and plantations exist in Malaysia. Cocoa growing is not an especially labour-intensive activity, but at the same time it is ill-suited to mechanization. A large plantation, therefore, while probably yielding more per hectare than the subsistence farm, tends to operate on higher costs and finds profitability difficult to achieve when FOB (free on board) prices are below, say, US$ 1,000 per ton. Other plantation crops, such as rubber or oil palm, may then represent a more attractive proposition for these particular farmers (International Cocoa Organization Annual Report 2012-2013).

Harvesting cocoa consists of plucking the ripe pods from the trees, breaking them open, extracting the seeds from the pods, allowing them to ferment, and setting them out to dry – preferably in the open air and in sunshine. The dried seeds are bagged and brought to market as ‘cocoa beans’. On some larger plantations this natural drying process may be assisted or even totally replaced by the use of artificial heat. When the cocoa is dried artificially, however, without the proper aeration, the acetic acid present in the beans does not have the chance to escape fully, rendering them more acidic and thus less acceptable (International Cocoa Organization Online Archive).

Weather and disease

The main problems encountered by cocoa crops are adverse weather conditions and disease, which are often interrelated and capable of seriously restricting production. Generally speaking, the successful production of cocoa beans requires an evenly spread rainy season and plenty of sunshine (though the plant itself, having originated in the depths of the tropical rain forest, actually prefers shade). Where such ideal conditions exist, as in Costa Rica, for example, there is a harvest practically every month. Countries with pronounced dry and wet seasons normally show two harvests a year, a main crop and a mid-crop. The relative sizes of these crops depend on how long the wet seasons last. A pronounced drought, or a long cool, rainy season, will have a major impact on the total tonnage produced – and on prices (International Cocoa Organization Annual Report 2012-2013).

Among the most damaging diseases are witches’ broom (*Crinipellis perniciosa*), black pod (*Phytophthora palmivora, P. megakarya*), monilia (*Moniliophthora roreri*), the cocoa swollen shoot virus (CSSV) and, in the Pacific area, vascular streak dieback. Except for CSSV, these diseases are caused by fungi and can be treated to some extent by fungicides (Ploetz, R. C. 2007).

Crops in a number of producing areas also suffer damage from pests. In West Africa and, to a lesser degree, in Latin America, mirids (capsid) cause crop losses. In the Pacific area, including Indonesia and Malaysia, the cocoa pod borer (the larva of a small moth) is a major enemy.

Sufficient scientific knowledge exists today to find solutions to most of these problems, but there are obstacles standing in the way of their application. The social and economic infrastructure in a particular cocoa-producing area often presents the greatest barrier. Moreover, some measures, such as saturation spraying, are no longer ecologically acceptable, while others are simply too expensive to be practical. In cocoa-producing areas, the extension services, which advise farmers on these matters and which are usually the responsibility of local government, typically suffer from shortages of both staff and funds. A further limiting factor is a lack of proper husbandry and farm sanitation (Hebbar, P. K. 2007).

Scientists therefore continue to search for alternative methods. The most generally acceptable solution at present appears to be the replanting of affected areas with disease-resistant and pest-resistant hybrids. Many cocoa-producing regions have set up breeding and propagation programmes devoted to the distribution of the new plant varieties. Other measures now being employed include biological controls, such as the release of predatory insects which feed on pests, or the encouragement of fungi to inhibit the growth of disease. Another approach being closely studied is that of combining different methods into integrated disease and pest management regimes (CFC/ICCO/Biodiversity International Project 2004-2010-Final Report).

Sustainable production

There is considerable disagreement within the scientific, economic and policy communities on the precise meaning of 'sustainable development' and many definitions are used. As far back as 1987, the United Nations World Commission on Environment and Development came up with a definition that is commonly accepted and widely used today. It was included in what became known as the Brundtland Report and states that: '...sustainable development is development that meets the needs of the present without compromising the ability of future generations to meet their own needs' (International Cocoa Organization Annual Report 2012-2013).

During the late 1990s, much interest was generated in the concept of sustainability in the cocoa economy, focusing particularly on sustainable methods of cocoa production. This led to the signing of a declaration of intent in March 1999, in Paris, by a number of bodies involved in the cocoa market.

To a large extent, the declaration is based on the Sustainable Cocoa Program (SCP) developed by the industry-sponsored American Cocoa Research Institute (ACRI) in collaboration with other parties. The vision is to secure a sustainable supply of cocoa within 10 years, with emphasis on economics, and social and environmental improvements (CFC/ICCO/Biodiversity International Project 2004-2010-Final Report).

The International Cocoa Organization (ICCO) is also developing a sustainable cocoa economy programme, in association with its members. This includes work on both the production and the consumption side of the market. ICCO has set up a mechanism for coordination and exchange of information among the many parties involved in the research, in the hope that as much as possible will find its way through to practical field application (International Cocoa Organization Annual Report 2012-2013).

Cocoa Processing

After the cocoa beans have been received at the processing location, they are inspected and thoroughly cleaned of all extraneous matter, such as sticks, stones, metal fragments, as well as broken beans. This process involves blowers, which remove items that are lighter or heavier than cocoa beans, and sieves which eliminate items that are too small or too big.

Once the beans are cleaned, the processor has the option of roasting them before the shell is removed, or of removing the shell before roasting. Cocoa beans which have had their shells removed are the nib.

Generally speaking, chocolate manufacturers prefer to roast the beans before shelling them, while cocoa processors favour the nib-roasting process (Biehl et al., 2003).

Bean roasting allows for more variety in the degree of roast and development of flavour, but requires beans of a uniform size, while nib roasting is more even and does not require uniform bean size. Removing the shell before roasting prevents migration of cocoa butter from the bean into the shell during the roasting process (International Cocoa Organization online archive).

Once the beans have been shelled and roasted (or roasted and shelled, as the case may be), the nib is ground into a paste. The heat generated by this process causes the cocoa butter in the nib to melt, hence the name 'cocoa liquor'. It is also known as 'cocoa paste', 'chocolate paste', 'cocoa mass' or, as for example in the United States food standards of identity, simply as 'chocolate'. Once further refined, it is also called 'unsweetened baking chocolate'. Cocoa liquor destined for processing into cocoa butter and cake is refined to a very small particle size, as it is easier to reduce the particle size earlier, when the butter is still present, rather than later, when most of the butter has been pressed out. A smaller particle size makes butter extraction easier (Biehl et al., 2003).

Cocoa liquor destined for chocolate production need not be as finely ground. Indeed, a larger particle size is preferable because it requires less cocoa butter than finely ground liquor to give the same mouth feel to the finished chocolate. This is an important economic consideration.

In the cocoa-processing operation, the liquor is now fed into hydraulic presses that remove a predetermined percentage of the cocoa butter, leaving behind a cake which, according to the processor's requirements, may contain anything from 6% to 24% of cocoa butter. The cocoa butter so extracted is then filtered and stored in tanks in liquid form until ready to be turned over to the chocolate operation, if at the same location. Otherwise it is shipped to its final destination either in liquid form in tank trucks or in moulded form in cartons. It is sold as 'pure, prime pressed, natural' cocoa butter, usually considered to be best quality, particularly if made from all-African cocoa beans (International Cocoa Organization online archive).

The cocoa cake is either broken into smaller pieces (kibbled) and sold into the generic cocoa cake market, or it is ground into a fine powder. It is called natural because it has not been treated with alkali.

The cocoa processing is summarized in Figure 3.1.

Natural cocoa powder is primarily used in the baking and confectionery industry and often forms the flavour base for compound coatings (Biehl et al., 2003).

The cocoa processor has the option of treating the nib or the liquor with an alkali solution (alkalizing), which will reduce the acidity by increasing the normal pH factor from about 5.0 up to 8.0. This treatment is als Houten, who developed the cocoa butter pressing operation as well.

Alkalizing cocoa nib or cocoa liquor renders the powder darker, gives it a milder, but more chocolaty flavour, and allows it to stay in suspension longer in liquids such as milk. It generally commands a premium over natural cocoa powder (Biehl et al., 2003).

Although dubbed 'soluble', cocoa powders are not truly soluble in liquids – they are 'wettable'.

On the other hand, the cocoa butter extracted from alkalized liquor does have a more pungent and less desirable odour and flavour, and must be deodorized (normally by steaming it), as well as refined. It is then carefully blended with other cocoa butters, so that the resulting final butter for sale is consistent in its bland flavour, colour and viscosity. Typical of these butters are those marketed by the Netherlands cocoa-processing industry and known by their brand names.

Virtually all the cocoa butter produced by the international cocoa processing industry is used in the manufacture of chocolate, where it must be added to the liquor to achieve the desired result. The pharmaceutical and cosmetics industries, which also use cocoa butter, may obtain their requirements from sources using solvent extraction or methods other than pressing cocoa butter from cocoa shell. Some may use cocoa beans that are not suitable as a food item (International Cocoa Organization online archive).

Focus On Alkalizing Process

The alkalizing process can be used to create many types of cocoa powder with differing colors.

The alkalizing process was invented in the first half of the 19th century in the Netherlands.

It improves the quality of cocoa powder in two ways:

1. It takes away the slightly acid taste of cocoa.
2. It makes the color darker.

This is due to condensation reactions forming high-molecular-weight colored products. Careful control of the reactions can lead to different shades of color: orange, red, brown, and even black is possible.

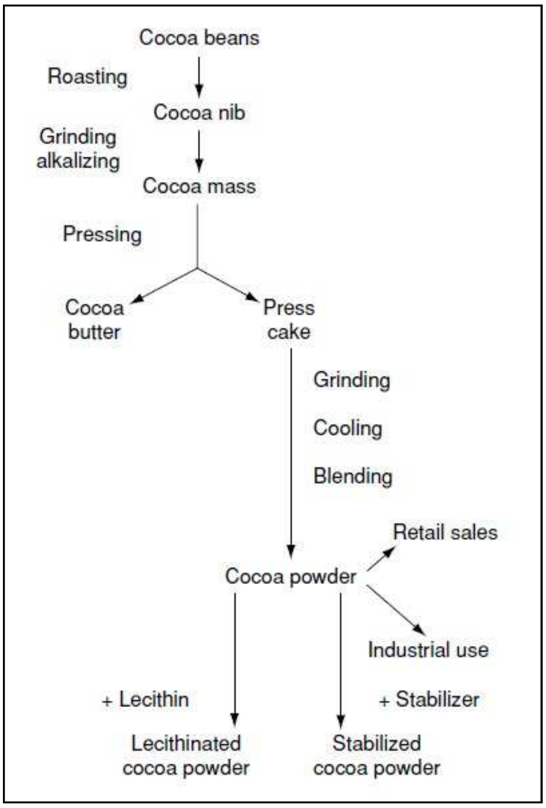

FIG 3.1 The production of cocoa powder.

Alkalizing consists of treating the cocoa with a solution of an alkali, mostly potash. Legally, the maximum amount of potash which may be used is 3%, calculated on the nib. The treatment can be performed with the nib, mass, press cake, or powder. The specific color is reached by choice of the reaction conditions: temperature, time, amount and concentration of the alkaline solution, and other factors (Biehl et al., 2003).

Grinding, cooling, and blending of cocoa cake

The press cakes are broken and then ground to powder in pin or other mills. The cocoa powder leaves the mill hot. It should be completely cooled before packing, otherwise the setting fat will turn it into hard lumps inside the package.

Blending of pieces of broken cake before grinding allows standardization of the color, or the preparation of blends with intermediate colors (Biehl et al., 2003).

Types

Variations in the two main processes lead to the formation of hundreds of different types of cocoa powder. Cocoa butter constitutes about half the weight of the cocoa nib. This fat is partially removed from the cocoa liquor by means of mechanical pressure as high as 450 kg/cm2.

The pressing can be performed to a fat content in the cake of 20% (giving cocoa powder) or to 10% (giving low-fat cocoa powder), while the alkalizing process creates many different colors Fat also masks both the bitter element of cocoa as well as the sour element, rendering a more chocolate-like, softer flavor.

Cocoa powder with 20% fat is the common household type. The food industry uses mostly low-fat cocoa powder (10%), in many different colors (International Cocoa Organization online archive).

Low-fat powders are recommended for use in compound coatings that contain lauric fats, as a higher cocoa butter content has a negative influence on the gloss retention of these coatings.

Due to the lower fat content, the 10-12% fat powders are less susceptible to lumping and are more free flowing. These powders are therefore better suited for products like vending mixes (Meursing, 2009).

For use in certain cocoa-flavored products, some special types of cocoa powder have been developed.

Lecithinated cocoa powder contains 5% of soya lecithin. Owing to its fat content, cocoa powder is difficult to wet and to disperse in water. Even in hot water or milk, lumps will be formed easily. Lecithin can improve these properties by its action as a wetting agent. Five percent of soya lecithin is intensively mixed with cocoa powder and the resulting lecithinated powder is agglomerated with sugar. This gives so-called instant cocoa, which can be put directly into cold milk.

Stabilized cocoa powder contains about 2% carrageenan. This is a polysaccharide derived from seaweed. It prevents the cocoa powder from settling in sterilized chocolate milk (Biehl et al., 2003).

Practice of Fermentation

Handling and care during processing significantly affect raw cocoa quality and flavor. To improve raw cocoa quality, repeated attempts have been made to modify the methods.

Cocoa fermentation is a microbial succession of a wide range of yeasts, lactic-acid, and acetic-acid bacteria during which high temperatures of up to 50°C and microbial products, such as ethanol, lactic acid, and acetic acid, kill the beans and cause production of flavor precursors

The different methods of cocoa fermentation are mostly developed from local traditions under particular conditions in the various cocoa-growing countries.

Standardized methods, such as the heap and box fermentations, are used at well-established farms or estates but they are not in common use elsewhere (Biehl et al., 2003).

Origin of Flavor Differences

Some experts assume flavor differences to be due to edaphic, climatic, or plant genetic particularities in the cocoa-growing countries. However, these differences may partially or even predominantly be due to traditional processing practices which are not uniform in the various producing countries.

Soil nutrients and climate generally affect the physiology of plants. Climatic changes have influence on pod growth, seed size, the amount of seed constituents, and thus on the starting material used for cocoa processing. Apart from the variable response of cocoa cultivars to climate, there is inadequate knowledge about the significance of genetic diversity of the planted cocoa trees and harvested seeds with respect to its influence on flavor quality. Criollo (fine) and Forastero (bulk) raw cocoas reveal flavor differences which are attributed to genetic influences (Biehl et al., 2003).

Cocoa products

Two products are derived solely from cocoa beans: chocolate and cocoa powder. These products are interrelated, and two intermediate products play an important role – cocoa mass and cocoa butter. This relationship is made clear in Figure 3.2 (Macrae et al., 1993).

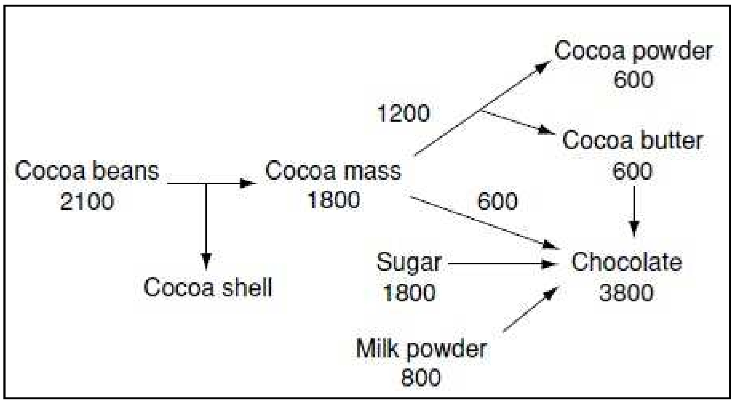

FIG 3.2 Products derived from cocoa beans. The figures are approximate and in 1000 tonnes.

The chocolate industry needs a certain quantity of cocoa butter and this implies that a fixed quantity of cocoa powder is also produced. However, the demand for cocoa powder does not always meet this volume. As a result, the price of cocoa butter is usually substantially higher than that of cocoa powder. The relatively high price of cocoa butter has made it attractive to lipid chemists to look for cheaper fats which could replace cocoa butter. This is not easy, as cocoa butter has unique properties, especially hardness and melting behavior.

Cocoa Market

Global Cocoa Production

Major producing countries in each region include:

- **Africa** Côte d’Ivoire, Ghana, Nigeria, Cameroon
- **Asia/Oceania** Indonesia, Malaysia, Papua New Guinea

- **Americas** Brazil, Ecuador, Colombia (Figure 3.3) (Cocoa Market Update-2014).

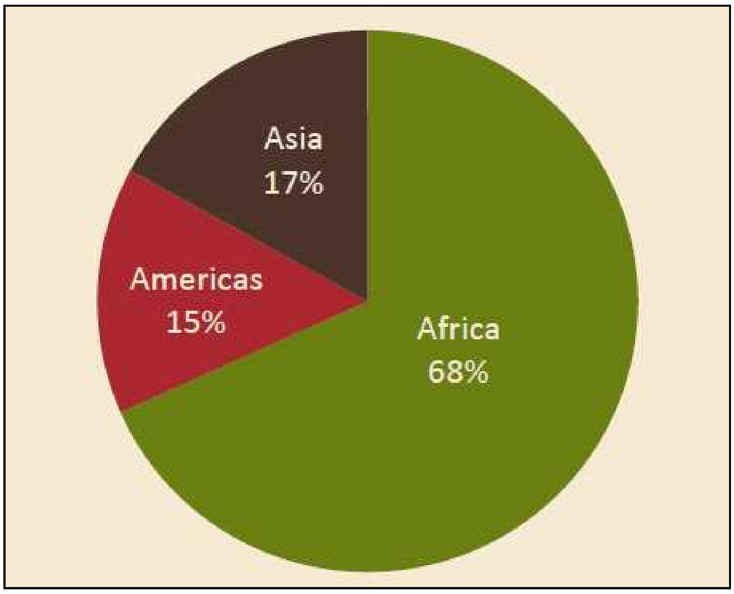

FIG 3.3 Global Cocoa Production
Source: World Cocoa Foundation

Unlike large, industrialized crops, 80% to 90% of cocoa comes from small, family-run farms, with approximately five to six million cocoa farmers worldwide. In Africa and Asia, the typical farm covers two to four hectares (five to ten acres). Each hectare produces 300 to 400 kilograms of cocoa beans in Africa and about 500 kilograms in Asia. Cocoa farms in the Americas tend to be slightly larger and produce 500 to 600 kilograms of cocoa beans per hectare. Yield per hectare varies not only by region, but also by country and by type of cocoa (Cocoa Market Update-2014).

Trade balances, pricing, and futures contracts depend on accurate supply estimates, so cocoa production is monitored throughout the supply chain, as well as by governments and international organizations.

As shown below, total production has increased by 13%, from 4.3 million metric tons in 2008 to 4.8 million metric tons in 2012. This represents an average year-over-year production increase of 3.1% (Figure 3.4).

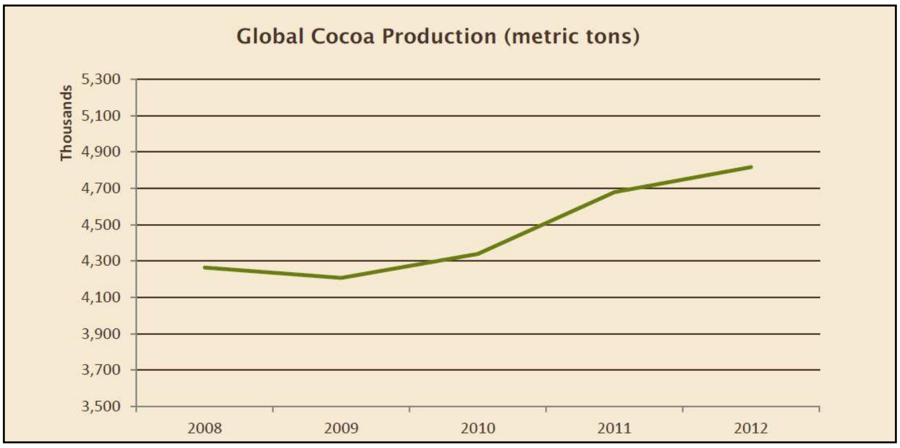

FIG 3.4 Global Cocoa Production (metric tons)
Source: World Cocoa Foundation

This rate of increase may slow in the coming years, as cocoa trees are sensitive to changing weather patterns. Periods of drought and of excessive rain or wind can negatively impact yield, and will continue to fluctuate as climate change intensifies (Cocoa Market Update-2014).

Consumption

Once cocoa beans have been harvested, fermented, dried, and transported, they are processed into separate components for commercial consumption. Processor grindings of cocoa beans serve as the key metric for market analysts, for an overall view of historical and anticipated demand. Processing is the last phase in which demand for cocoa beans can be equitably compared to supply—after this step, the individual components of the bean are sold across many industries to manufacture confections, soaps, and cosmetic items (Cocoa Market Update-2014).

Global market share for processing has remained stable, even as grindings increase to meet demand. The Netherlands is the largest processing country by volume, handling about 13% of global grindings. Though unsuitable for growing cocoa, Europe as a whole comprises nearly 40% of the processing market. The remaining 60% is divided almost evenly between Africa, Asia, and the Americas (Figure 3.5).

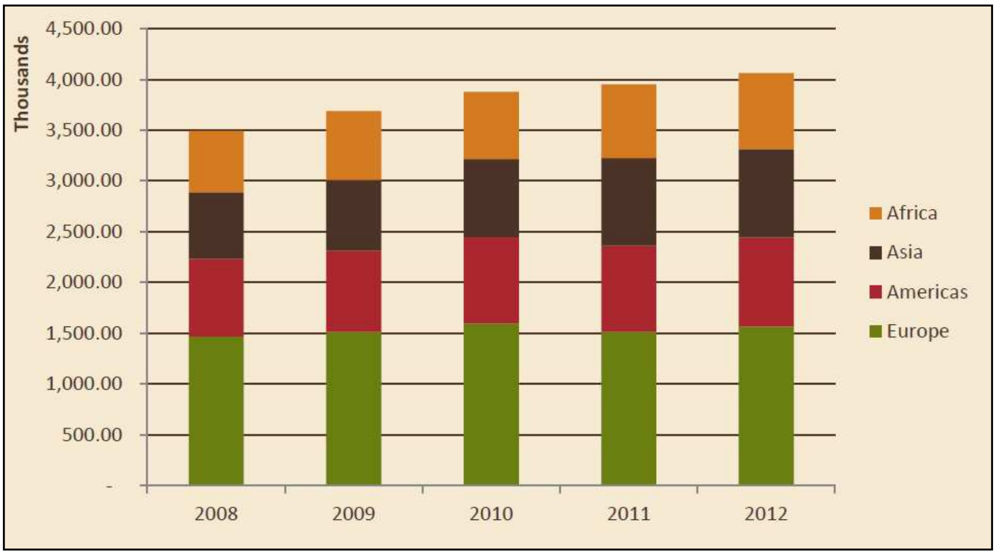

FIG 3.5 Market Share: Global Cocoa Grindings

Source: World Cocoa Foundation

Average year-over-year demand growth has been just over 3% since 2008. One of the primary drivers of this increase is the growing middle class in China, India, and Brazil. While Europe and North America are relatively mature markets, increasing discretionary household income in developing countries are a major factor in stable demand growth. As demand is projected to continue its stable growth, supply growth may slow due to changing weather patterns in the largest cocoa-producing areas. This combination of changes in supply and demand may put upward pressure on cocoa and chocolate prices, depending on how quickly these changes occur (Cocoa Market Update-2014).

Confectionery, Coatings And Cocoa products

This product category comprises applications based on fat-sugar, water-sugar, and water-fat-sugar systems. Fat-sugar systems are those in which the main ingredients are fat, sugar, and cocoa powder, such as compound coatings and fillings. Depending on the amount and type of fat, a product will be soft or hard at room temperature. For compound coatings made from different vegetable fats, both alkalized and non-alkalized cocoa powder can be used. This is a matter of flavor and color appreciation and of costs. The flavor/color impact of a lower level of alkalized cocoa powder may be stronger than that of a higher level of natural cocoa powder. Non-alkalized cocoa powders are lighter in color than alkalized powders. The whitish milk powder functions as a background that will emphasize the color and its brilliance. Cocoa powders with higher cocoa butter contents can have an adverse effect on the gloss stability of compound coatings made with lauric fats (Meursing, 2009).

For ice cream coatings, alkalized cocoa powders are often used. The reason for this is that the detection of the chocolate flavor is dulled by the low temperature of ice cream. The stronger flavor and darker color of the alkalized powders render their full impact in this application (Kattenberg, 1995).

Water-sugar and water-fat-sugar systems include products such as syrups, fudges, toppings, and frostings, where water forms the continuous phase. This has the effect of altering the rheology and mouth-feel of the product to respond to the specific demands of the application. Improved preservation is partially obtained by the addition of sugar and other preservative ingredients. This means that the quantity of sugar in these products is often higher than in products of fat systems. In these applications, in addition to non-alkalized cocoa powders, strongly alkalized powders are often used. This is balanced by the high sugar content present and its ability to mellow the sometimes pronounced flavor of the strongly alkalized cocoa powders.

When using water in relatively high viscosity products, consideration must be given to the total carbohydrate percentage of the cocoa components. The starches, sugar, and dietary fiber establish a bond with the water, as a result of which a thickening effect may occur over time, such as in syrups.

It is important that with sugar syrups, the correct proportions of the various types of sugar are chosen. An incorrect choice of sugars may lead to crystallization, which in turn produces a change in viscosity. In many cases, the cocoa powder is seen as the cause of this, while it is more often caused by problems in the area of the sugars used (Meursing, 2009).

Wettability And Hygroscopicity Of Cocoa Powder

Wettability and Hygroscopicity are two important physical properties of cocoa powder that influence their application in various sectors.

Wettability defines the potential for a powder to wet and absorb water at a given temperature.

Wettability = the time in seconds necessary to achieve complete wetting

During wetting, the void space inside a powder is replaced by water. Composition, particle size and shape or the presence of surface free-fat, an inhibitory factor, affect the powder wettability. Wettability is also determined by the temperature of the water used so water temperature should always be specified in wettability tests (Kyaw Hla et al., 1999).

When cocoa powder is added to cold water or cold milk, the powder tends to float on the surface because of its poor wettability. By its nature, cocoa powder is not inclined to disperse but to float on the surface of a liquid. This is primarily due to the cocoa butter present in the powder, which repels water and prevents the wetting of the powder particles (Hla et al., 1999). Wettability and dispersibility can be significantly improved by blending the cocoa powder with lecithin. As an emulsifying agent, lecithin is a mixture of phosphatides that is surface active. The lipophilic (fat-affinity) part of the molecule attaches to the cocoa butter present in the cocoa powder, and the hydrophilic (wateraffinity) part of the molecule attracts the water in the solution (Meursing, 2009)

Hygroscopicity is the capacity of a product (e.g. cargo, packaging material) to react to the moisture content of the air by absorbing or releasing water vapor. Of decisive significance for the absorption or release of water vapor is the water content of a product. A water sorption isotherm illustrates the capacity of a powder for uptake or release of water when placed in atmospheres at temperature. The presence of water in a powder can significantly affect its flowability and caking properties (Teunow et al., 1999). Reference data report that notwithstanding the fact that cocoa powder has poor wettability, it is very hygroscopic (Meursing, 2009).

Cocoa powder has an important effect on rheology and water absorption in many of the products in which it is used. A distinction can be made in food systems where water is the continuous phase (dough for bakery products, desserts, toppings, and chocolate beverages) or in products where fat forms the continuous phase (compound coatings, chocolate, and fillings on fat basis).

Traditional cocoa powders absorb very quickly moisture from the dessert or from condensation in packaging, thus compromising the fresh, attractive presentation. The color of the cocoa powder becomes darker and the powder partly dissolves (Meursing, 2009). It is known that whenever moisture is available, cocoa powder will compete with other ingredients to absorb it. It can take in moisture up to 100% of its own weight (Meursing, 2009). This means that in dry mixes, a balance in water activity will be established between the various ingredients.

Aim

Because of its highly hygroscopic nature, cocoa powder has an important effect on rheology and water absorption in many of the products in which it is used.

Traditional cocoa powders absorb very quickly moisture from the dessert or from condensation in packaging, thus compromising the fresh, attractive presentation. The color of the cocoa powder becomes darker and the powder partly dissolves.

The aim of this work was therefore to develop a new cocoa for sprinkling use with a non hygroscopic character, that makes it perfect for decorating or topping desserts that are kept for instance in moist environment.

In the first phase of development of the new cocoa powder in order to reach a good dry mix, that a balance in water activity will be established between the various components, different ingredients were evaluated as follows

1. Evaluation of their Wettability Behavior, as the potential for a powder to wet and absorb water at a given temperature

2. Measurements and drawings of water adsorption isotherms for selected ingredients, which represent their Water Activity (a_w) versus a moisture content (g H2O / g d.m.)

The new formulations of cocoa powder, developed in the first phase of study, are tested by sprinkling on tiramisù, and only two have been selected as the starting point for the next study, which involved partnership with a supplier.

Materials and Methods

POWDERED SUGAR: this sugar is a granulated sugar ground to a smooth powder and then sifted. It contains about 3% cornstarch to prevent caking. Powdered sugar is ground into three different degrees of fineness. The confectioners sugar available in supermarkets – 10X – is the finest of the three and is used in icings, confections and whipping cream. The other two types of powdered sugar are used by industrial bakers (Faravelli Group Datasheet).

NATURAL COCOA: is made from cocoa beans that are simply roasted, then pulverized into a fine powder. Natural cocoa powder has a light brown color and a pH level of 5.6. It has an acidic, somewhat astringent flavor with a typical chocolate note. It contains about 11% fat (Gerkens Datasheet).

ALKALIZED COCOA POWDER: all derived from the extraction of fat (cocoa butter) from cocoa beans. Roasted cocoa bean material is pressed at high intensity to extract most of the cocoa butter. The residual material is treated with a mild alkali solution (a process called "Dutching") to improve certain characteristics; the processed (alkalized) cocoa powder is darker in color, ranging from brownish red to nearly black, with a pH from 6.8 to 8.1 (Gerkens Datasheet). The alkalization process reduces bitterness and improves solubility.

All types of alkalized cocoa powder used in this study are listed below:

- ALKALIZED COCOA DB21: a dark brown powder, it has a chocolate-like flavour without being bitter. It contains about 21% fat and it has a pH level of 8.
- ALKALIZED COCOA PABN11: a black powder, it has a chocolate-like flavour without being bitter. It contains about 11% fat, and it has a pH level of 8.
- ALKALIZED COCOA RR21: a red powder, it has a chocolate-like flavour without being bitter. It contains about 21% fat, and it has a pH level of 8.
- ALKALIZED COCOA DARK RED (Type 1): a fine powder dark strong red , it has a chocolate-like flavour without being bitter. It contains about 22% fat and it has a pH level of 7.5.
- ALKALIZED COCOA DARK RED (Type 2): a fine powder dark strong red , it has a chocolate-like flavour without being bitter. It contains about 24% fat and it has a pH level of 8.

COCONUT OIL POWDER: a creamy /white powder, it contains with 80% of refined coconut oil. Refined coconut oil said to be the “mother of all oils”, is extracted from fresh coconut meat without any chemical processing. Coconut kernels are first disintegrated into small pieces in a Disintegrator and then fed into a screw press for milk extraction. The oil is then separated from the water using a mechanical centrifuge. The result is a clear coconut oil that retains the distinct scent and taste of coconuts. The oil is then homogenised and mixed with Glucose syrup, Caseinate, Pentasodium triphosphate (E451i),Silicon dioxide (E551). The resultant mixture is then spray dried into a fine coconut oil powder using a spray drier (Friesland Campina-Kievit Datasheet).

All formulations developed are summarized in Table 3.1

TABLE 3.1	FORMULATION										
TYPE OF COCOA POWDER	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**
POWDERED SUGAR	X	X	X	X	X	X	X	X	X	X	X
NATURAL COCOA	X	X	X	X	X	X	X	X	X	X	X
ALKALIZED COCOA Red RR21		X		X		X	X		X	X	X
ALKALIZED COCOA Dark Brown DB21			X	X	X		X	X	X	X	X
ALKALIZED COCOA Black PABN11	X	X	X			X	X	X			X
ALKALIZED COCOA Dark Red Type 1			X		X	X			X	X	
ALKALIZED COCOA Dark Red Type 2	X	X	X	X	X	X	X	X	X	X	
COCONUT OIL POWDER	X	X	X	X	X	X	X	X	X	X	X

Wetting Test

The wetting tester used for the measurements (Hla et al., 1997) consists of a 80 cm3 container for the testing liquid and a powder sample section which rests above the liquid container (see Figure 3.6). A slide, which is connected to a spring, forms the bottom of the powder sample section.

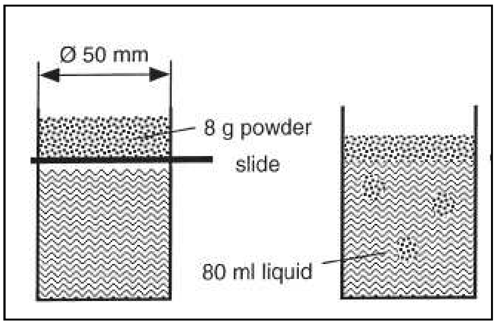

FIG 3.6 Schematic view of the wetting time testing device.

Protocol

1. Before each measurement, a powder sample of approximately 8 g was prepared from bulk material (usually a 500 g or 800 g package).
2. The liquid (e.g. milk at 7 °C) was poured into the liquid container and the powder sample section was placed on top of the container.
3. Unlocking the slide then let the spring quickly pull the slide out sideways, so that the powder contacted the liquid after falling 2–3 mm, started to wet and sink down into the liquid time, i.e. the time necessary for all the powder particles to disappear, was recorded using a stopwatch.
4. As it is generally difficult to determine exactly the wetting time for slowly-wetting powders, each experiment was repeated at least three times and the results were averaged.

Sorption Isotherm Curve Determination

Sorption behavior describes the ability of a hygroscopic product to absorb or release water vapor from or into the air until a state of equilibrium is reached. The relationship between moisture content and water activity is the Sorption Isotherm Curve, which is product and temperature specific.

It is very important an absolutely constant temperature during all measurements.

For such purpose it is used a set in combination with a LabMASTER/PARTNER-a_w (LabMASTER/PARTNER a_w Operating Instructions)

Protocol

In order to start, the substance must be dry, so first it must be totally desiccated in a oven for 2 h on 102°C.

1. Close the glass container before taking it out from the drying oven.
2. Weigh the probe afterwards with the closed weighing glass on a precision balance and define the net weigh of the probe.
3. Put first the SAL-T 6 in the deeper measurement chamber and afterwards the metallic sample cup containing the cooled sample over it and close the chamber.
4. After about one hour write the actual a_w value. It is not necessary to wait for the equilibrium inside the chamber.
5. Take our the probe and put it immediately in the glass container.
6. Weigh the probe again and write the net weigh a table and calculate the difference between the weights before and after humidification.
7. Put the next SAL-T into the measurement chamber and perform the same operation at point 2,3 ect. up to the SAL-T 97 humidity standard.

Moisture content calculation

Each sample should be treated individually to calculate moisture content (Bell et al., 2000).

$$m = \frac{g\ H_2O}{g\ solids} = \frac{(w_f - w_{i,d})}{w_{i,d}}$$

where w_f is the mass of the product after moisture gain and $w_{i,d}$ is the initial mass of the dried product. For an initial sample of mass w_i having some initial moisture content (%H_2O) on a wet basis,

$$M = \frac{[wf - wi] + \frac{\%H20}{100} * wi}{wi * \left[\frac{100 - \%H20}{100}\right]} = \frac{gH20}{gSS}$$

Based on the acquired data it is possible define the Adsorption curve; dry basis moisture is plotted on the Y axis as function of a_w, on the X axis.

Sprinkling On Tiramisù

1. Sprinkle the new formulations of cocoa powder on samples of tiramisù.

2. Samples so prepared are covered with plastic wrap and stored in the fridge.
3. Check samples every 30 minutes up to 2 hours of preparation and assess the degree of humidity of the cocoa powder sprinkled.

Tiramisù Recipe

Ingredients

2 cups strong black coffee
1/2 cup marsala (see note)
3 eggs, separated
1/3 cup caster sugar
250g mascarpone
300ml thickened cream, lightly whipped

1 large packet of sponge fingers (savoiardi)

Descriptions

1. Pour coffee and marsala into a shallow dish. Set aside.
2. Beat egg yolks and sugar in a large bowl with electric beaters until pale and thick. Add the mascarpone and whipped cream, mixing gently until just combined.
3. Beat egg whites in a medium bowl with electric beaters until soft peaks form. Using a large metal spoon, gently fold egg whites into the mascarpone mixture.
4. Dip enough biscuits into the coffee mixture to cover the base of a 19cm square ceramic dish. Cover the biscuits with one-third of the mascarpone mixture. Repeat layers 2 times, ending with the cream.
5. Cover with plastic wrap and refrigerate for at least 2 hours.
6. Sprinkle cocoa and serve.

Instrumental Color Evaluation

Definition

The instrumental color evaluation of cocoa powder as such or as a slurry in water is expressed in L*-, C*-, and h-values measured with a color meter (KONICA Minolta).

The L*-, a*-, and b*-values are calculated from the CIE X-, Y-, and Z-values using the CIE 1976 equations. C*- and h-values are calculated from the a*- and b*-values according to the following:

- **L*** value – the lightness/darkness coordinate; a low value indicates a dark color, a high value indicates a light color;
- **a*** value – the red/green coordinate, with +a* indicating red and a* indicating green;
- **b*** value – the yellow/blue coordinate, with +b* indicating yellow and b* indicating blue;
- **C*** value – the chroma coordinate, indicating brightness; a higher value indicates a brighter color;
- **h** value – the hue angle; a lower value indicates more redness, a higher value indicates more yellowness.

Procedure

1. Fill a cuvette 3/4 full with the cocoa powder sample and tamp the powder down carefully.
2. Then add cocoa powder until it is heaped above the rim.
3. Level the powder evenly by using the edge of a spatula with tapping movements.
4. Remove the surplus powder carefully with the spatula to produce a flat surface in line with the rim (*note:* If the cocoa powder is lumpy, the surface will be irregular when evaluating It is then advisable to sieve the cocoa powder through a 500µm sieve and carefully break down the lumps; Mix the powder thoroughly).
5. Place the cuvette carefully against the illuminated window of the calibrated meter.
6. Read and record the L*-, C*-, and h-values.

7. Compare the values found with those of a standard sample

(Clydesdale, 1969; CIE, 1978; Instruction manual: Chroma Meter CR-400 KONICA MINOLTA).

Results and Discussion

As well as in the previous chapter, the following paragraphs have been organized according to the sequence of Technology Readiness Levels (TRL), is the one adopted by the European Commission in the 2014-2020 Horizon 2020 Programme for Research and Innovation (for details see the previous chapter).

Idea

In this work the different types of cocoa powder, alone and in combination with other ingredients were studied, in order to develop a new cocoa powder for sprinkling use. This cocoa powder is a innovative cocoa powder conceived with a non-hygroscopic character, that makes it perfect for decorating or topping desserts that are kept for instance in moist environment.

Basic Research

In a preliminary phase of development of the new cocoa powder it was studied the physics features of all ingredients considered in this project, or different types of cocoa powder, powdered sugar and coconut oil powder.

The wetting and the hygroscopic behaviour of all ingredients were determined, so as to define a formulation in which there was a balance in water activity between all the components in a humid environment.

Wettability Evaluation

The wetting behavior of these cocoa powder was characterized in a standardized test (see Materials and Methods).

All different types of cocoa powder (see Material and Methods) were tested for their wetting behaviour.

To get an impression of how different testing liquids might affect the wetting time, the wetting time of a powder sample was tested using water and milk of varied fat content.

The wetting time of different types of cocoa powder was tested with three different types of UHT milk (i.e., 0.3%, 1.5%, 3.5% fat content), to see how the different fat content could affect that behavior, and with deionized water (conductivity < 0.5 mS). It was found that a higher fat content in the milk leads to longer wetting times; With water the wetting time is much shorter as it contains no fat droplets, dissolved protein etc.

Sorption Isotherm Determination

It was decided to study the behavior of hygroscopic all the ingredients that presumably would be part of the mix in order to balance them properly.

Moisture sorption isotherms of all types of cocoa powder, powdered sugar and coconut oil powder were determined (see "Materials And Methods"). A sorption isotherm is the graphic representation of the sorptive behavior of a product. It represents the relationship between the water content of a product and the relative humidity of the ambient air (equilibrium) at a particular temperature.

The profile of a sorption isotherm is characteristic of the hygroscopicity of a product, the sorption behavior of a product being dependent on temperature. Highly hygroscopic products exhibit a steep sorption isotherm, while sparingly hygroscopic products exhibit flat sorption isotherms.

The moisture adsorption isotherm for cocoa powders are shown in Figure 3.7 and Figure 3.8.

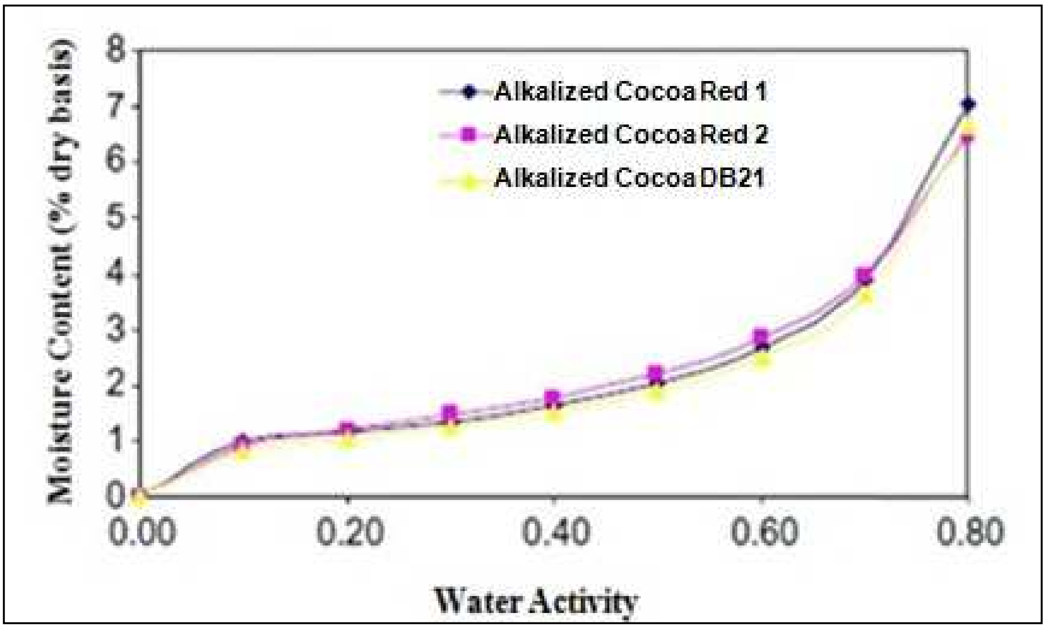

FIG 3.7 Moisture Sorption Isotherms for three types of alkalized cocoa powder.

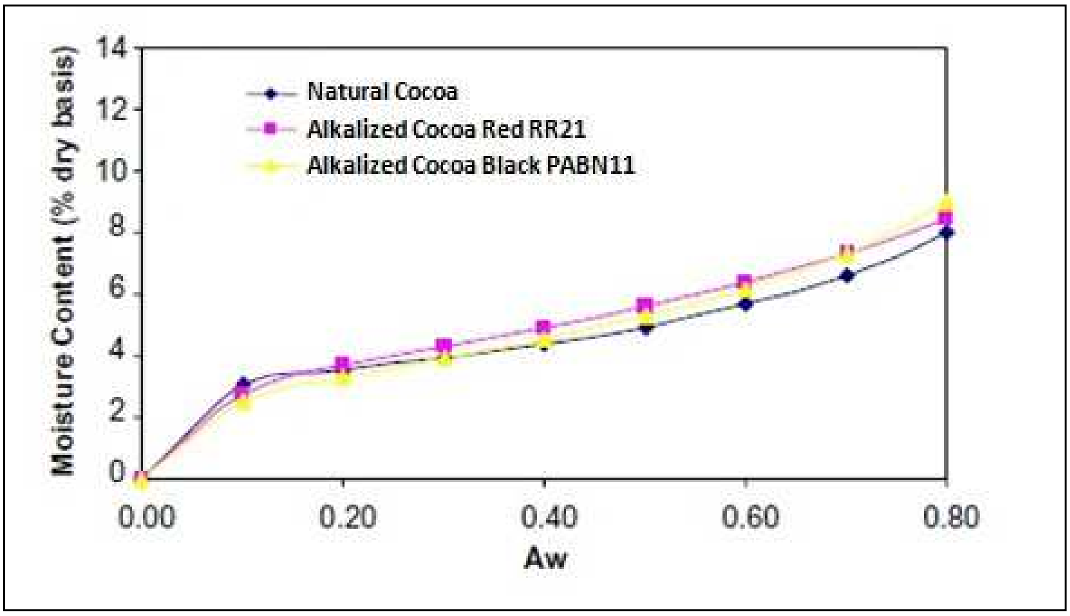

FIG 3.8 Moisture Sorption Isotherms for three types of alkalized cocoa powder.

The sorption isotherms of all different types of cocoa powder have a sigmoidal shape, which is typical of most food materials (Iglesias and Chirife, 1982).

The moisture adsorption isotherm of coconut oil powder and powdered sugar is shown in Figure 3.9.

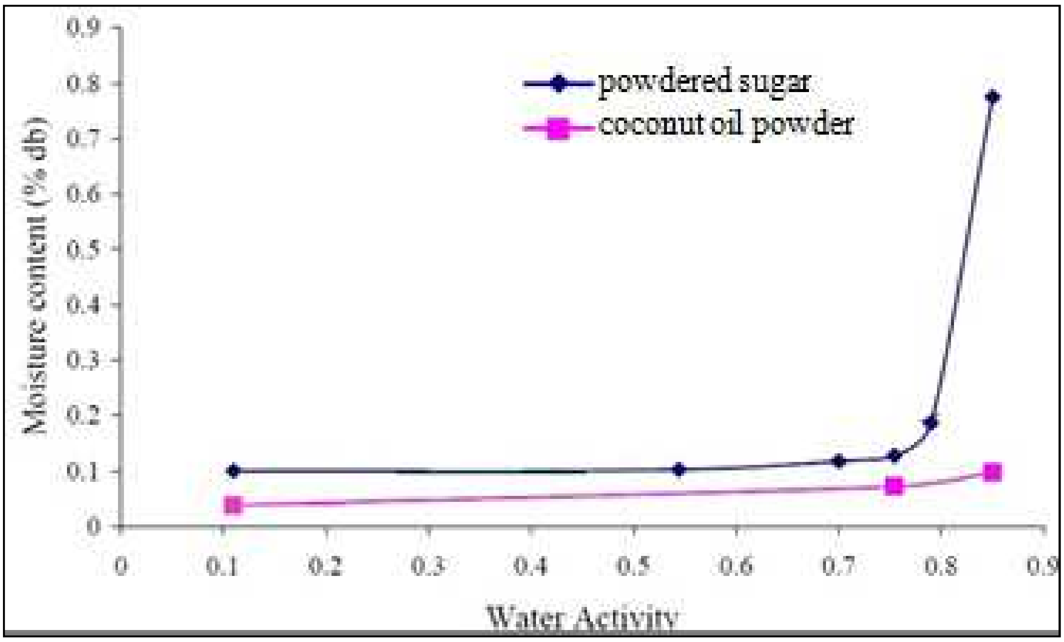

FIG 3.9 Moisture sorption isotherm of Powdered Sugar and Coconut Oil Powder.

Oils are hydrophobic materials and therefore the moisture adsorbed by the oils at any water activity is very low (<0.1%).

Also you can see in Figure 3.7, 3.8 and 3.9 as cocoa absorbs more moisture than powdered sugar at water activity levels below 0.85.

Technology Formulation

This first analysis has provided a lot of information about the behavior of the considered ingredients (see "Materials and Methods") in terms of wettability and hygroscopicity.

This proved to be an important starting point for the development of many formulations shown in Table 3.2.

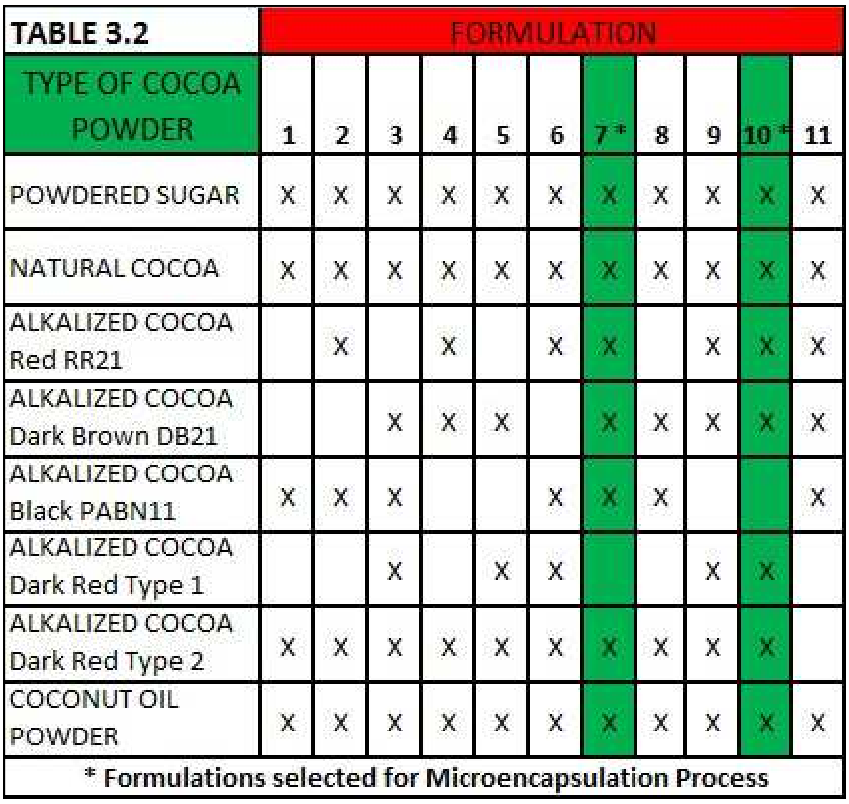

TABLE 3.2	FORMULATION										
TYPE OF COCOA POWDER	1	2	3	4	5	6	7 *	8	9	10 *	11
POWDERED SUGAR	X	X	X	X	X	X	X	X	X	X	X
NATURAL COCOA	X	X	X	X	X	X	X	X	X	X	X
ALKALIZED COCOA Red RR21		X		X		X	X		X	X	X
ALKALIZED COCOA Dark Brown DB21			X	X	X		X	X	X	X	X
ALKALIZED COCOA Black PABN11	X	X	X			X	X	X			X
ALKALIZED COCOA Dark Red Type 1			X		X	X			X	X	
ALKALIZED COCOA Dark Red Type 2	X	X	X	X	X	X	X	X	X	X	
COCONUT OIL POWDER	X	X	X	X	X	X	X	X	X	X	X

* Formulations selected for Microencapsulation Process

Applied Research

First phase: Test Of The New Product Formulations.

The formulations developed in the previous phase of the experiment were tested by sprinkling on tiramisù (see "Materials and Methods").

All formulations absorb moisture from the tiramisù and the cocoa powder becomes darker very quickly.

Second Phase: Study Of Innovative Technologies In Food Process.

The results of the previous stage of the new product development have led us to the need to find an innovative process that makes the whole mixture less hygroscopic, rather than continue to change the composition by varying the ingredients and their percentage.

The development of new cocoa for sprinkling use is continued with the search for a new technology to improve the water repellency.

References on innovation process indicate microencapsulation as innovative technology for improving food performance (Pegg et al., 2007).

The food industry applies encapsulation for a number of reasons (Andres, 1977; DeZarn, 1995):

1. Encapsulation/entrapment can protect the core material from degradation by reducing its reactivity toward the outside environment (e.g., heat, moisture, air, and light).
2. Evaporation or transfer rate of the core material to the outside environment is decreased/retarded.
3. The physical characteristics of the original material can be modified and made easier to handle.
   For example : hygroscopicity can be reduced.
4. The product can be designed to either release slowly over time or at a certain point (i.e., to control the release of the core material so as to achieve the proper delay until the right stimulus).
5. The taste of the core material can be masked.
6. The core material can be diluted when it is needed only in very small amounts, and to achieve uniform dispersion in the host material.
7. It can be used to separate reactive components within a mixture which would otherwise react with one another.

Encapsulation is a topic of interest in a wilde range of scientific and industrial areas, varying from pharmaceutics to agriculture, and pesticides to enzymes.

The development of these technologies is characterized by strong fundamental research and several industrial applications, demonstrated by the growing number of scientific papers and patent application (Ré et al., 2010).

Encapsulation involves the coating or entrapment of a desired component (active or core material) within a secondary material (encapsulating, carrier, coating, shell, or wall material) to prevent or delay the release of the active or core ingredient until a certain time or a set of conditions is achieved (Ré et al., 2010).

Encapsulation and microencapsulation are often used interchangeably when discussing the process technology. Microencapsulation is encapsulation at the microscale, producing delivery devices ranging from 1 to 1000 µm in size, generally less than 200 µm (Ré et al., 2010).

Microencapsulation is defined as a process in which tiny particles or droplets are surrounded by a coating, or embedded in a homogeneous or heterogeneous matrix, to give small capsules with many useful properties. Microencapsulation can provide a physical barrier between the core compound and the other components of the product (Gharsallaoui et al., 2007).

Study of spray-dried powders with 50% butteroil encapsulated in sucrose and double encapsulated in a matrix of vegetable waxes shows that double encapsulation method could improve capsule resistance to moisture sorption, but also decreases powder flow properties (Onwulata, Konstance,& Holsinger, 1998).

Therefore the next phase of the research project involved a partnership with a supplier in new product development, that agreed to cooperate with us in the development of the new product, by providing their production technologies.

Two formulations, including those developed in the previous phase of study, are selected on the basis of color (see "Materials And Methods"), which is another requirement from the market in addition to the hygroscopic character.

For each one was made an assessment of the color with the colorimeter (see "Materials and Methods"), comparing these formulations with a sample of cocoa competition.

The two formulations selected in this step, see table 3.2, were considered the starting point for the next phase of development, which for reasons of business secrecy is not the subject of this thesis.

Conclusions

Analyzing the obtained results from the initial phase of development of the New Cocoa Powder for Sprinkling use we can drop the following conclusions:

1. The evaluation of the Wettability and the Hygroscopicity of the different types of cocoa on the one hand provide us with information on the ability to absorb moisture on the other side is not enough to a new cocoa powder with not Hygroscopic character.

2. Application of innovative technologies as Microencapsulation is revealed decisive for the development of the new product.

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Conclusions

Although no guarantees can ever be offered for a new food product's success, the implementation of a carefully orchestrated plan significantly increases that probability. Planning is a series of well considered steps to be taken from gap analysis through concept generation and evaluation to prototyping and assessment, positioning through mapping, optimization, market testing, and scale-up for launch. Translating concepts into prototype products that can be viewed, sniffed, tasted, and savored is a choreographed series of events. Development is just that, a process of initiation and advance, error, iteration, adaptation, and reiteration directed toward an elusive goal of a nearly perfect manifestation of the product concept.

The Food Product Development Process in this work followed a systematic approach of Technology Readiness Levels (TRLs), which has allowed us to outline clearly all stages of the study of the product.

This measurement system, developed in Horizon 2020, provides a common understanding of technology status and addresses the entire innovation chain.

The idea of developing a new product is always born from the knowledge of the state of the 'art of food alteration processes and the insight to be able to improve performance through product or process innovation.

In the first project from the need to control water transfer in the stuffed pasta, we studied the hygroscopic behavior different ingredients (taken individually and diversely mixed), through measurements of initial Water Activity (Aw), determination of Sorption Isotherms and water retention capacity (WRC) .

Based on results obtained from this study we developed two mix, MIX STARCHES and MIX MILK; at the end of the experimental phase on a large scale both prototypes added to the filling slow down effectively the transfer of moisture inside the stuffed pasta.

In the second project from the demand to improve the performance of the old formulation, REIBAKING POWDER, in leavening action and taste, the reactivity of the common leavening agents was studied; at the end of this study a new baking powder for bakery was developed, with an improved leavening action, and able to endow the finished product with a more natural taste and less artifact than REIBAKING POWDER.

Finally the development of the new cocoa powder for sprinkling use provided in a first phase the study of hygroscopic behavior of all the ingredients of the new product, through determination of Sorption Isotherms. Secondly, the search for an innovative technology, as Microencapsulation, was decisive for improving the final performance of the new product.