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PhD Thesis summary

# **Research on obtaining and characterizing gluten free pasta enriched in bioactive compounds with a functional role**

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## INTRODUCTION

The quality of the food ingested is a determining factor for the physical and mental health of the consumer. In this regard, in recent years there has been a growing trend of re-educating the consumer by returning to the consumption of natural unprocessed foods that are rich in nutrients and bioactive compounds, all in order to compensate for the accumulated deficiencies. Pasta is one of the top preferences for most consumers, but unfortunately, celiac disease is one of the most common diseases in the world affecting consumer choices. The great advantage of gluten free pasta is that it can be eaten without restrictions by anyone, not affecting any category of consumers due to its ingredients, even if they suffer from different types of intolerances. Enriching cornmeal with vegetable powders (grape pomace, nettle and alucerne) rich in bioactive compounds with a functional role brings an important nutritional contribution, resulting in a significant increase in the biological and nutritional properties of gluten free pasta because grape pomace powder contains valuable bioactive compounds rich in fiber and polyphenols (BOJAN, 2020), nettle powder is a rich source of vitamin C, minerals and protein (PÂRVU, 2005), and lucerne powder has a high content of vitamin C and minerals (MARTA IGUAL, 2021). Due to the properties of the above-mentioned ingredients, pasta as a finished product can be included in the category of functional foods.

The scientific community believes that beyond the satiety provided by ingested nutrients, functional foods impart the food with other characteristics due to the bioactive compounds present that guarantee scientifically proven health benefits by preventing and treating various chronic diseases (CORINA MAXIM, 2019).

## STRUCTURE OF THE THESIS

The present thesis aims to obtain gluten free pasta made from corn flour and extruded corn flour enriched with vegetable powders: nettle, lucerne and grape pomace and the study of their bioactive potential. The biological material used to carry out the experiments consists of samples of maize flour, extruded maize flour, lucerne powder, nettle powder, grape pomace powder (consisting of the remains of seeds and husks of red grapes after pressing) and tapioca starch. The two types of corn flour were purchased from a factory specializing in milling corn products, and the rest of the ingredients were purchased from specialty stores in Cluj Napoca. The research for this paper was carried out in the laboratories of the Research Institute for Analytical Instrumentation (ICIA) Cluj-Napoca, in the laboratory of SC X SRL, in the laboratories

of the Faculty of Food Science and Technology: Food Quality Control Laboratory, Laboratory LICSA and the Bakery products Pilot Station , in the laboratories of the Institute of Life Sciences, within USAMV Cluj-Napoca.

The first part "**THE CURRENT STAGE OF KNOWLEDGE**, consists of 2 chapters. **Chapter 1. THE IMPORTANCE OF FOOD QUALITY** comprises 6 subchapters which include information summarized regarding the consumption of nutritious foods, general aspects on functional foods, general aspects on the use of by-products in the food industry and medicinal plants, bioactive compounds, celiac disease, general information about gluten free pasta, thus following the bibliographic study.

Chapter 2 **PRESENTATION AND CHARACTERIZATION OF RAW AND AUXILIARY MATERIALS USED IN THE MANUFACTURE OF AGLUTENIC FLOUR PASTE** includes 7 subchapters describing raw and auxiliary materials based on cumulative data from the literature.

The second part of the thesis, "**PERSONAL CONTRIBUTIONS**" consists of 6 chapters (Chapter 3-8).

Chapter 3 presented the purpose, objectives of the paper and experimental design.

Chapter 4 entitled **MATERIAL AND METHOD** describes the experimental material, equipment and chemicals, as well as methods used to perform the determinations. The following are mentioned in the subchapters: physical and sensory analysis of gluten free pastas, standardized methods used for physico-chemical determinations, methods of analysis using chromatographic and spectrophotometric techniques, quantitative determination of allergens and contaminants, calculation of energy value, sensory analysis, microbiological analysis, the general techniques for obtaining gluten free pasta, respectively the statistical-mathematical methods used.

Chapter 5 contains **RESULTS AND DISCUSSIONS** obtained from the determinations made for both raw and finished materials. In this chapter the discussions and interpretations were also noted, based on the results obtained.

Chapter 6 entitled **CONCLUSIONS AND RECOMMENDATIONS** summarizes the conclusions drawn from the interpretation of the results obtained from research conducted in this doctoral thesis.

Chapter 7 describes the **ORIGINALITY AND INNOVATIVE CONTRIBUTIONS OF THE PhD THESIS** entitled "Research on obtaining and characterizing gluten free pasta enriched in bioactive compounds with a functional role."

Chapter 8 outlines the possible **FUTURE RESEARCH PERSPECTIVES**.

## AIM AND OBJECTIVES

The research carried out in this doctoral thesis "Research on obtaining and characterizing gluten free pasta enriched in bioactive compounds with functional role" has as main purpose the testing of product variants in the category of gluten free pasta with a different content of functional ingredients.

## GENERAL AND SPECIFIC OBJECTIVES

In order to achieve the goal, the 3 directions of research were approached:

- Evaluation and characterization of raw materials
- Obtaining an innovative product with a functional role
- Characterization of variants of gluten free pasta enriched with vegetable powders and evaluation of the parameters for determining their functional role

## RESEARCH RESULTS

Ascorbic acid (Vitamin C) cannot be synthesized in the body, which is why it is considered an essential vitamin (CARMEN SOCACIU, 2017). The results on the Vitamin C content of pasta are shown in Figure 1.

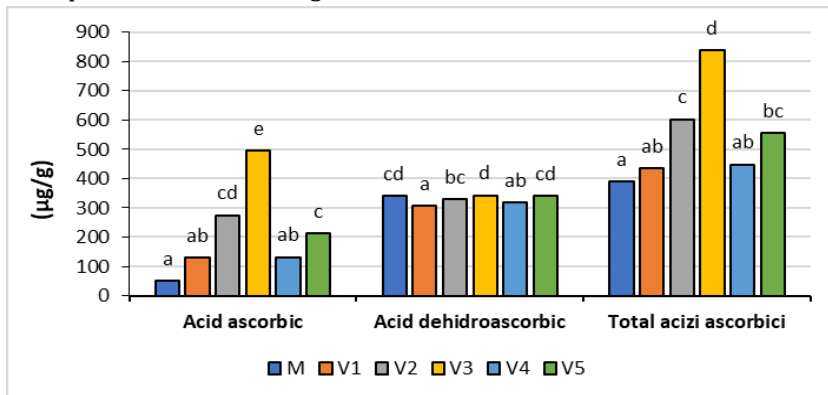


Fig. 1 Ascorbic acid content of gluten free pasta samples (µg/g)

Regarding the amount of ascorbic acid, no statistically significant differences were found between the control sample (49.41 µg / g), V1 (129.05 µg / g) and V4 (131.25 µg / g) and between V2 (275.11 µg / g) and V5 (213.67 µg / g). In contrast, the highest content of total ascorbic acids (838,059 µg / g) was determined in V3 (495.48

$\mu\text{g} / \text{g}$ ), a statistically significant content ( $p < 0.05$ ) higher than in the other variants analyzed. V3 ( $342.57 \mu\text{g} / \text{g}$ ) has a statistically significant higher dehydroascorbic acid content than V1 ( $307.68 \mu\text{g} / \text{g}$ ), V2 and V4 ( $317.66 \mu\text{g} / \text{g}$ ), but there are no statistically significant differences between the control sample ( $341.18 \mu\text{g} / \text{g}$ ) and V2 ( $328.32 \mu\text{g} / \text{g}$ ), V3 and V5 ( $341.3 \mu\text{g} / \text{g}$ ). As it was also observed after individual analyses (ascorbic acids and dehydroascorbic acids), the analyses of total ascorbic acids indicate the highest statistically significant content ( $p < 0.05$ ) in V3 ( $838.06 \mu\text{g} / \text{g}$ ) compared to the control ( $390.60 \mu\text{g} / \text{g}$ ) and other variants.

Another category of bioactive compounds analyzed were phenols. They are chemicals that have one or more aromatic rings with one or more hydroxyl groups as their structure (SHENG, 2018). The generic name of phenolic compounds includes as subclasses phenolic acids, flavonoids (flavonols, flavones, isoflavones, catechins). By LC-MS analysis, individual compounds from these classes were separated, identified and dosed, and the sum of the compounds determined for the pasta variants are shown in Figure 2.

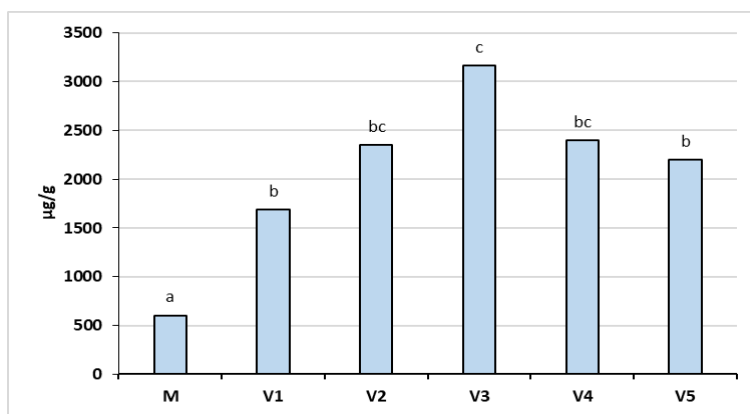


Fig. 2 Phenolic content of gluten free pasta samples ( $\mu\text{g/g}$ )

All variants analyzed have a statistically significant content ( $p < 0.05$ ) higher than the control ( $604.795 \mu\text{g} / \text{g}$ ) based on the data in table 2. But between variants V1 ( $1684.75 \mu\text{g} / \text{g}$ ), V2 ( $2349.74 \mu\text{g} / \text{g}$ ), V4 and V5 ( $2198.08 \mu\text{g} / \text{g}$ ) no significant differences were detected, as in the case of variants V2, V3 ( $3161.26 \mu\text{g} / \text{g}$ ) and V4 ( $2394.19 \mu\text{g} / \text{g}$ ).

The data obtained were compared with those from raw materials and the concentrations expected were calculated considering the percentages of raw materials in flour products.

In variant V1, 2% nettle, 3% lucerne and 2.5% grapepomace were used. If we consider their initial concentrations of  $54640 \text{ mg} / \text{kg}$ ,  $7047 \text{ mg} / \text{kg}$  and  $6772 \text{ mg} / \text{kg}$

respectively, the values obtained would be  $1092.8 \text{ mg / kg} + 211.41 \text{ mg / kg} + 169.39 \text{ mg / kg} = 1473.6 \text{ mg / kg}$

According to Fig. 2,  $1648,759 \text{ mg / kg}$  were obtained, a value that does not indicate loss by processing. In variant V2, 4% nettle, 6% lucerne and 5% grapepomace were used. If we consider their initial concentrations of  $54640 \text{ mg / kg}$ ,  $7047 \text{ mg / kg}$  and  $6772 \text{ mg / kg}$  respectively, the values obtained would be  $2185.6 \text{ mg / kg} + 422.82 \text{ mg / kg} + 338.6 \text{ mg / kg} = 2947.02 \text{ mg / kg}$ . According to Fig. 2  $2349,744 \text{ mg / kg}$  were obtained, meaning 25% loss by processing.

In variant V3, 6% nettle, 9% lucerne and 7.5% grape pomace were used. If we consider their initial concentrations of  $54640 \text{ mg / kg}$ ,  $7047 \text{ mg / kg}$  and  $6772 \text{ mg / kg}$  respectively, the values obtained would be  $3278.4 \text{ mg / kg} + 634.23 \text{ mg / kg} + 507.9 \text{ mg / kg} = 4420.3 \text{ mg / kg}$ . According to Fig. 2, a total of  $3161,267 \text{ mg / kg}$  were obtained, meaning 39% loss by processing.

These data indicate that processing losses are proportional to the percentage of powders added, and are due to the degradation of phenolic compounds in these raw materials.

On the other hand, the content of total polyphenols of gluten free pasta was analyzed using the Folin-Ciocalteu method, and the results are represented in Figure 3. The results of statistical analyses obtained based on the data in the figure show that the control sample had the lowest content of polyphenols ( $150 \text{ mg / kg GAE}$ ). A similar value was identified in V5 ( $255 \text{ mg / kg GAE}$ ), followed by V2 ( $303 \text{ mg / kg GAE}$ ), and in the case of V1 ( $440 \text{ mg / kg GAE}$ ), V3 ( $447 \text{ mg / kg GAE}$ ) and V4. ( $463 \text{ mg / kg GAE}$ ) statistically significantly higher values ( $p < 0.05$ ) were identified compared to the other variants.

Comparing the results with those from the raw materials, similar to those discussed in the LC-MS determination of phenolic compounds, it was found:

For variant V1 where 2% nettle, 3% lucerne and 2.5% grape pomace were used, if we consider their initial concentrations of  $2805 \text{ mg / kg}$ ,  $2445 \text{ mg / kg}$ , and  $2888 \text{ mg / kg}$ , respectively, the theoretical values would be  $56.1 \text{ mg / kg} + 73.35 \text{ mg / kg} + 72.2 \text{ mg / kg} = 201.65 \text{ mg / kg}$ . According to Fig. 3  $440.0 \text{ mg / kg}$  were obtained, meaning an approximately double overestimated value.

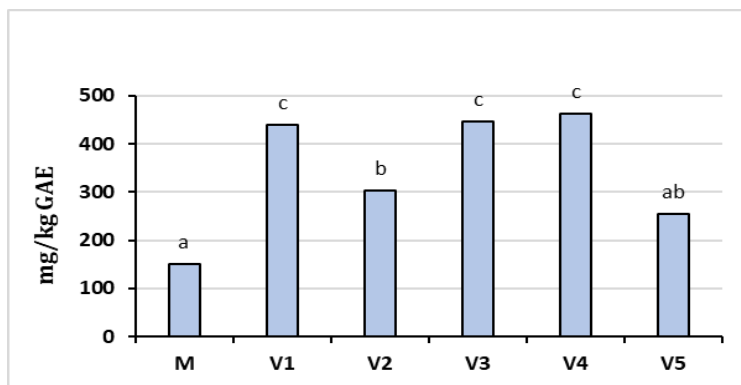


Fig. 3. Polyphenol content of gluten free pasta samples (mg/kg GAE)

In variant V2 where 4% nettle, 6% lucerne and 5% grape pomace were used, if their initial concentrations of 2805 mg / kg, 2445 mg / kg and 2888 mg / kg respectively are considered, the theoretical values would be  $112.2 \text{ mg / kg} + 146.7 \text{ mg / kg} + 144.4 \text{ mg / kg} = 403.3 \text{ mg / kg}$ . According to Fig. 3 a total of 303.0 mg / kg were obtained, a value indicating a loss of 25%.

In variant V3 where 6% nettle, 9% lucerne and 7.5% grape pomace were used, if we consider their initial concentrations of 2805 mg / kg, 2445 mg / kg, and 2888 mg / kg, respectively, the theoretical values would be  $168.3 \text{ mg / kg} + 220.05 \text{ mg / kg} + 216.6 \text{ mg / kg} = 604.9 \text{ mg / kg}$ . According to Fig. 3 447.0 mg / kg were obtained, a value indicating a loss of 27%. In this case, it was observed that the losses are proportional to the increase of the share of powders in the final product.

Compared to the LC-MS method the results are approximately similar, indicating that both methods are reproducible. In addition, we consider that the spectrophotometric method is more practical, less laborious and cheaper for routine determinations.

## GENERAL CONCLUSIONS AND RECOMANDATIONS

- Based on researches, it has been shown that by-products in the food industry as well as medicinal and fodder plants have bioactive potential and can be used in food
- Evaluations performed on gluten free pasta variants showed that V3 is the variant where the highest concentrations of bioactive compounds were detected. The big disadvantage of this variant is that the taste of pasta has not been sensorially assessed by consumers. Thus, further studies to improve sensory characteristics are recommended.



- Regarding the content of phenolic compounds, the data obtained were compared with those from raw materials and the concentrations were calculated considering the percentages of raw materials in flour products. These data indicate that processing losses are proportional to the percentage of powders added and are due to the degradation of phenolic compounds in these raw materials.

## **ORIGINALITY AND INNOVATIVE CONTRIBUTIONS**

The elements of originality of the present doctoral thesis consist in:

- Identification of bioactive compounds applicable in the food industry from corn flour, extruded corn flour, nettle powder, lucerne powder and grape pomace in order to obtain a product with an enriched nutritional quality intended for special nutrition;
- Study on the influence of the addition of nettle powder, lucerne and grape pomace on the nutritional quality of the pasta obtained;
- Use of advanced techniques to determine the detailed chemical composition of raw materials and finished product variants.

## **FUTURE RESEARCH PERSPECTIVES**

Based on the results presented in the thesis, research can be directed towards new directions:

- Development of new functional foods for special nutrition based on studied raw materials rich in biologically active compounds;
- Carrying out more detailed studies on the product variants obtained and testing them for their special purpose and to classes of consumers with special nutritional requirements (diabetics);
- Identification of flavour compounds in vegetable powders;
- Research on the antimicrobial potential of the studied vegetable powders.

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