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CENTRO DE CIÊNCIAS AGRÁRIAS
Programa de Pós-Graduação em Ciência de Alimentos

**PROPRIEDADES TECNOFUNCIONAIS DE MASSA
ALIMENTÍCIA ENRIQUECIDA COM RESÍDUO DE PODA
DE ASPARGO**

DARIO SOUSA DA SILVA

Maringá

2023

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ALIMENTÍCIA ENRIQUECIDA COM RESÍDUO DE PODA
DE ASPARGO**

Dissertação apresentada ao programa de Pós-Graduação em Ciência de Alimentos da Universidade Estadual de Maringá, como parte dos requisitos para obtenção do título de Mestre em Ciência de Alimentos.

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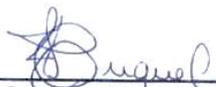
“PROPRIEDADES TECNOLÓGICAS DE MASSA ALIMENTÍCIA
ENRIQUECIDA COM RESÍDUO DE PODA DE ASPARGO”.

Dissertação apresentada à Universidade Estadual de Maringá, como parte das exigências do Programa de Pós-graduação em Ciência de Alimentos, para obtenção do grau de Mestre em Ciência de Alimentos.



Prof. Dra. Cássia Inês Lourenzi Franco

Rosa



Prof. Dra. Francine Lorena Cuquel



Prof. Dra. Paula Toshimi Matumoto Pinto

Orientadora

Maringá – 2023

Orientadora

Prof. Dra. Paula Toshimi Matumoto Pinto

BIOGRAFIA

Dario Sousa da Silva nascido em 17 de janeiro de 1994 na cidade de Lupionópolis, Paraná, Brasil. Filho de Delmiro da Silva e Maria Aparecida de Sousa. Possui formação técnica em Agropecuária e Secretariado e Assessoria pela Etec Deputado Francisco Franco – Rancharia – SP em 2011, graduação em Agronomia pela Universidade do Oeste Paulista, Unoeste – Campus II Presidente Prudente – SP em 2015. Pós-graduado em Segurança Alimentar pela Universidade Anhembi Morumbi – UAM em 2020 e Engenharia de Segurança do Trabalho com ênfase em Auditoria pela Faculdade de Minas – Facuminas em 2022. Atuou no setor Sucroenergético nas áreas de controle de qualidade agrícola e desenvolvimento agrônomo (2016-2019). Foi professor e coordenador para os cursos técnicos em agroindústria, agronegócio, agropecuária e açúcar e álcool (2019-2021). Ingressou no Programa de Pós-Graduação em Ciência de Alimentos em abril de 2021, com a defesa da dissertação em 27 de fevereiro de 2023. Tem experiência nas áreas de Ciência e Tecnologia de Alimentos atuando na área de ciência e tecnologia de produtos agropecuários.

Dedico

Tenho a certeza de que, sem a minha base familiar, este sonho não teria sido realizado. Dedico a finalização de minha pesquisa a toda minha família, especialmente a minha mãe. Também, em especial, dedico este trabalho aos meus avós por toda a orientação que me deram. Meus agradecimentos não serão suficientes para expressar minha gratidão.

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APRESENTAÇÃO

Esta dissertação de mestrado está apresentada na forma de artigo científico:

Dario Sousa da Silva; Bianka Rocha Saraiva; Anderson Lazzari; Andresa Carolina de Oliveira Cestario; Mariana Carla de Oliveira; Marcos Luciano Bruschi; Cássia Inês Lourenzi Franco Rosa; Paula Toshimi Matumoto Pinto. Technofunctional properties of pasta enriched with asparagus pruning residue. *International Journal of Food Sciences and Nutrition*.

GENERAL ABSTRACT

INTRODUCTION. The asparagus (*Asparagus officinalis*) pruning residue is obtained when the buds (or shoots) that give rise to the aerial part of the stem are not harvested at the ideal physiological maturation point, making the product rigid and unsuitable for commercialization, requiring pruning to give origin of new shoots. However, residues generated usually left in the field for decomposition, can be used due to functional and nutritional properties they present, and can be used as pharmaceutical excipients, food additives or included food matrices to obtain new products and functional foods. In addition to the concern with a healthier diet, with the increase in the consumption of more natural and nutritious foods, the concern with the sustainability of the food produced has also been growing. Agricultural and agro-industrial residues have been researched due to their composition rich in nutrients, the presence of bioactive compounds and technological properties. About 30 to 50% of asparagus residues are generated by pruning rods and are generally used as animal feed or left in the field as low-value products (fertilizers) and due to their functional characteristics, the residue can be used in pasta, a food that is widely consumed due to its low cost, easy preparation and storage, long shelf life, and source of carbohydrates with desirable sensory attributes. Although other nutrients, such as protein and fiber, and bioactive compounds are not found in significant amounts in the product, macaroni has a versatility that allows the incorporation of new ingredients in its composition.

AIMS. The objectives of this study were to produce and characterize the physicochemical properties of asparagus pruning residue (AF) flour and use it as a substitute for wheat flour in pasta production; and to evaluate the effect of this partial substitution and the use of different drying temperatures on the properties of product quality, texture, rheology and microstructure, and content of bioactive compounds and antioxidant activity

MATERIAL AND METHODS. Asparagus pruning residue was sanitized, dried, ground and standardized at 60 mesh, obtaining a flour, which was analyzed for chemical composition (moisture, crude fiber, crude protein, ash, total fat and total carbohydrates), content of total phenolic compounds (TPC), total chlorophyll (TC) and flavonoids (FC) and antioxidant activity by DPPH and ABTS methods. Pasta was produced with partial substitution of wheat flour in the proportion of 5% (P5) and 10% (P10) w/w, except for the control (PC), produced only with wheat flour and water. Pasta was submitted to different drying temperatures (60, 80 and 90 °C) and evaluated for the content of bioactive compounds (TPC, TFC and TC), antioxidant activity (DPPH and ABTS), quality properties, texture, rheology and microstructure.

RESULTS AND DISCUSSION. Asparagus pruning residue flour showed high protein and fiber content, high antioxidant activity, source of phenolic compounds and promising data as a possible natural dye due to chlorophyll presence. Pasta enriched with AF showed significant differences in color (luminosity) but did not differ when compared between temperatures. Treatment with 10% replacement of

asparagus pruning residue flour showed lower levels of total phenolic compounds and flavonoids, and antioxidant activity when dried at high temperatures (80 and 90 °C), the opposite occurred for total chlorophyll, which showed higher values compared to other treatments and temperature (80 and 90 °C). In terms of quality properties, the treatment enriched with 10% AF had a shorter optimal cooking time compared to the other treatments, and there were no significant differences between temperatures. Pasta enriched with 5% and 10% flour from asparagus pruning residue had lower water absorption when compared to PC, especially when dried at 80 and 90 °C. Pasta enriched with 5% and 10% flour from asparagus pruning residue dried at high temperatures showed significant differences in cooking loss compared to PC and between temperatures P10 showed significant differences, being dry at 90 °C the lowest loss. In the analysis of the resistance of the strands of pasta dried by breaking, the P10 dried at 90 °C obtained a higher value than the other treatments. Fresh pasta with partial substitution of wheat flour for asparagus pruning residue flour was more elastic than the control pasta, considering its viscoelastic behavior. The microstructure of pasta dried at 80 and 90 °C showed smaller and larger galleries compared to pasta dried at 60 °C.

CONCLUSIONS. The partial substitution of wheat flour by 10% AF flour improved several functional properties of the dough, such as total phenolic content, flavonoids, antioxidant capacity, excellent cooking time and texture. The optimal drying temperature found in this study was 90 °C, due to the drying properties and final quality of pasta enriched with asparagus pruning waste flour.

Key words: *Asparagus officinalis*, vegetable residues; new food; pasta; bioactive compounds; functional food;

RESUMO GERAL

INTRODUÇÃO. O resíduo da poda do aspargo (*Asparagus officinalis*) é obtido quando as gemas (ou brotos) que dão origem à parte aérea do caule não são colhidas no ponto maturação fisiológica ideal, tornando o produto rígido e impróprio para comercialização, necessitando de poda para dar origem a novos brotos. No entanto, os resíduos gerados, geralmente deixados no campo para decomposição, podem ser aproveitados devido às propriedades funcionais e nutricionais que apresentam, podendo ser utilizados como excipientes farmacêuticos, aditivos alimentares ou matrizes inclusas em alimentos para a obtenção de novos produtos e alimentos funcionais. Além da preocupação com uma alimentação mais saudável, com o aumento do consumo de alimentos mais naturais e nutritivos, também vem crescendo a preocupação com a sustentabilidade dos alimentos produzidos. Resíduos agrícolas e agroindustriais têm sido pesquisados devido a sua composição rica em nutrientes, presença de compostos bioativos e propriedades tecnológicas. Cerca de 30 a 40% dos resíduos de aspargos são gerados por hastes de poda e geralmente são usados como ração animal ou deixados no campo como produtos de baixo valor (fertilizantes) e devido às suas características funcionais, o resíduo pode ser utilizado em massas alimentícias, alimento amplamente consumido devido ao seu baixo custo, fácil preparo e armazenamento, longa vida de prateleira e fonte de carboidratos com atributos sensoriais desejáveis. Embora outros nutrientes, como proteínas e fibras, e compostos bioativos não sejam encontrados em quantidades significativas no produto, o macarrão possui uma versatilidade que permite a incorporação de novos ingredientes em sua composição.

OBJETIVOS. Os objetivos deste estudo foram produzir e caracterizar as propriedades físico-químicas da farinha do resíduo de poda do aspargo (AF) e utilizá-la como substituto de farinha de trigo na produção de macarrão; e avaliar o efeito dessa substituição parcial e do uso de diferentes temperaturas de secagem nas propriedades de qualidade do produto, de textura, reologia e microestrutura, e teor de compostos bioativos e atividade antioxidante.

MATERIAL E MÉTODOS. O resíduo da poda do aspargo foi higienizado, seco, moído e padronizado em 60 mesh, obtendo-se uma farinha, a qual foi analisada quanto à composição química (umidade, fibra bruta, proteína bruta, cinzas, gorduras totais e carboidratos totais), teor de compostos fenólicos totais (TPC), clorofila total (CT) e flavonoides (FC) e atividade antioxidante pelos métodos DPPH e ABTS. A massa foi produzida com substituição parcial da farinha de trigo na proporção de 5% (P5) e 10% (P10) m/m, exceto a testemunha (PC), produzida apenas com farinha de trigo e água. A massa foi submetida a diferentes temperaturas de secagem (60, 80 e 90 °C) e avaliada quanto ao teor de compostos bioativos (TPC, TFC e TC), atividade antioxidante (DPPH e ABTS), propriedades de qualidade, textura, reologia e microestrutura.

RESULTADOS E DISCUSSÃO. A farinha de resíduo de poda de aspargo apresentou alto teor de proteínas e fibras, alta atividade antioxidante, fonte de compostos fenólicos e dados promissores como um possível corante natural devido

à presença de clorofila. As massas enriquecidas com AF apresentaram diferenças significativas na cor (luminosidade), mas não diferiram quando comparados entre as temperaturas. O tratamento com 10% de substituição da farinha de resíduo de poda de aspargo apresentou menores teores de compostos fenólicos totais e flavonoides, e atividade antioxidante quando seco em altas temperaturas (80 e 90 °C), o contrário ocorreu para a clorofila total, que apresentou valores superiores em relação aos demais tratamentos. Tratamentos e temperatura (80 e 90 °C). Em termos de propriedades de qualidade, o tratamento enriquecido com 10% de AF apresentou menor tempo ótimo de cozimento em relação aos demais tratamentos, não havendo diferenças significativas entre as temperaturas. As massas enriquecidas com 5 e 10% da farinha do resíduo de poda do aspargo apresentaram menor absorção de água quando comparados ao PC, principalmente quando secos a 80 e 90 °C. Os tratamentos P5 e P10 secos em altas temperaturas apresentaram diferenças significativas nas perdas por cozimento em relação ao PC e entre as temperaturas P10 apresentaram diferenças significativas, sendo seco a 90 °C a menor perda. Na análise da resistência dos fios de massa seca por quebra, o P10 seco a 90 °C obteve valor superior aos demais tratamentos. A massa fresca com substituição parcial de farinha de trigo por farinha de resíduo de poda de aspargo foi mais elástica que a massa controle, considerando seu comportamento viscoelástico. A microestrutura do macarrão seco a 80 e 90 °C apresentou galerias menores e maiores em relação ao macarrão seco a 60 °C.

CONCLUSÕES. A substituição parcial da farinha de trigo por 10% de farinha de AF melhorou diversas propriedades funcionais da massa, como teor de fenólicos totais, flavonoides, capacidade antioxidante, excelente tempo de cozimento e textura. A temperatura ótima de secagem encontrada neste estudo foi de 90 °C, devido às propriedades de secagem e à qualidade final do macarrão enriquecido com farinha de resíduo de poda de aspargo.

Palavras chaves: *Asparagus officinalis*; resíduos vegetais; novo produto; massa; compostos bioativos; alimento funcional.

1 **Technofunctional properties of pasta enriched with asparagus pruning residue**

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3 Dario Sousa da Silva^a; Bianka Rocha Saraiva^a; Anderson Lazzari^a; Andresa Carolina
4 de Oliveira Cestario^b; Mariana Carolina de Oliveira^c, Marcos Luciano Bruschi^{cd}; Cássia
5 Inês Lourenzi Franco Rosa^e; Paula Toshimi Matumoto Pintro^{a,e*}

6

7

8 ^aPrograma de Pós-Graduação em Ciência de Alimentos, Universidade Estadual de
9 Maringá, CEP: 87020-900. Maringá, PR, Brasil.

10 ^bPrograma de Pós-Graduação em Agronomia, Universidade Estadual de Maringá,
11 CEP: 87020-900. Maringá, PR, Brasil.

12 ^cPrograma de Pós-Graduação em Ciências Farmacêuticas, Universidade Estadual de
13 Maringá, CEP: 87020-900. Maringá, PR, Brasil.

14 ^dDepartamento de Farmácia, Universidade Estadual de Maringá, CEP: 87020-900.
15 Maringá, PR, Brasil.

16 ^eDepartamento de Agronomia, Universidade Estadual de Maringá, CEP: 87020-900.
17 Maringá, PR, Brasil.

18

19 *Corresponding author: Departamento de Agronomia, Universidade Estadual de
20 Maringá, Av. Colombo, 5700, Jd. Universitário, 87020-900, Paraná, Brasil. E-mail:
21 ptmpintro@uem.br (Paula T. Matumoto-Pintro). Telephone number: +55 (44) 3011-
22 8946.

Abstract

Asparagus pruning residue (stem aerial part that are not harvested at the sale ideal point) are left in the field for decomposition. Due to its functional and nutritional properties, it can be used to enrich pasta, widely consumed product that can have its properties improved. Objective was used asparagus pruning residue flour (AF) as replace to wheat flour by up to 10% w/w in pasta production; and to evaluate different drying temperatures effects on their technofunctional properties. Pasta enrichment whit AF increased bioactive compounds content and antioxidant activity but were affected by drying temperature (except chlorophyll). Its quality properties were also improved, due enrichment and drying high temperatures used. Dried pasta strands resistance to breaking was less when enriched, while cooked pasta hardness is maintained when used high drying temperatures. AF and high temperature use improved pasta quality properties, showing that AF can be an alternative ingredient for foods enrichment.

Keywords: Vegetable residues; valorization; drying temperature; pasta; bioactive compounds.

Introduction

Interest in residue reuse rich in bioactive compounds, carbohydrates, proteins meet increasing consumer demand for food made natural molecules, which promote people well-being and contribute for foods sustainable development. Agro-industrial residues from pruning and post-harvesting vegetables can become new alternative ingredients for food enrichment, such as carrot pomace (Gull et al. 2015), apple pomace (Hernández-Carranza et al. 2016), tomato peel (Paladino et al. 2017), spinach residues (Derrien et al. 2017), onionskin (Michalak-Majewska et al. 2020), banana peel (Rehman et al. 2013), olive pomace (Simonato et al. 2019), and watermelon rind (Ho and Dahri et al. 2016).

Asparagus (*Asparagus officinalis* L.) is a vegetable known for its unique flavor, texture and phytochemicals presence that perform desirable physiological functions. The edible part of the asparagus plant is the young stem, which emerges when the soil temperature rises above 50°F in spring and asparagus beds can be productive for 15, 20, sometimes even 30 years. Stems that have passed the point of physiological maturation, becoming a fibrous and hardened structure, rich in phytochemical compounds with biological activity (Zhang et al. 2019). They are unfeasible for commercialization, however, they can be reused.

Annual asparagus production is about 8.74 tons and an average of 3.49 tons of waste is generated after industrial processing. An alternative to add value to this material is to use it as raw material in products development to add phytochemicals and nutrients, such as phenolic compounds, saponins and dietary fibers (Fan et al. 2015).

Residues have been used as new functional ingredients (Marinelli et al. 2015, Galanakis 2012), mainly in breads (Osuna et al. 2018), cereal bars (Niu et al. 2020), ice creams (Durmaz et al. 2020), coloring drinks (Oliveira et al. 2017), confectionery products (Backes et al. 2020) and pasta (Koca et al. 2018).

Pasta is a very popular product because it is a staple food, versatile, with a long shelf life, in addition to representing a great carrier of health-promoting substances (Desai et al. 2018; Pasqualone et al. 2016) and can be enriched with different types of ingredients.

Pasta quality is defined by raw material properties, processing type, drying conditions and way of storing the product, in addition to good manufacturing practices (Ogawa et al. 2015). Pasta making process involves mixing, extruding, and drying. Drying may be the most critical step in determining pasta properties. Protein quality and starch thermal properties determine the product final quality as function of protein coagulation and starch gelatinization, which occur during the drying process (Zweifel et al. 2003).

Drying temperature falls into three categories: low temperature (about 50 °C), high temperature (about 70 °C), and very high temperature (above 80 °C). High temperature and very high temperature are currently the most used, as microbiological control is easy and production efficiency is increase due to shorter drying time compared to low temperature. Furthermore, it denatures the gluten protein, causing an increase in baked pasta hardness, while simultaneously reducing cooking loss, thus improving the pasta quality (Dexter et al. 1981; Stefanis and Sgrulletta 1990).

Physicochemical properties of pasta with asparagus pruning residue addition and dried at different temperatures have not yet been investigated. Thus, objective of this work was to evaluate the replacement of wheat flour for flour from asparagus pruning residue at different concentrations, and to study the changes in the techno functional properties under different drying temperatures.

Materials and Methods

Asparagus pruning residue

Asparagus leaves were collected after pruning in a small growing area in Marialva

(Parana, Brazil). Material was sanitized with water and hypochlorite (200 ppm; 5 min), washed and dried in an oven at 55 °C for 12 hours. Leaves was ground and standardized (60 mesh), obtaining the asparagus pruning residue flour (AF). It was stored was stored away from light at 8 °C for further analysis.

Chemical composition and color

AF chemical composition was determined by moisture, crude protein (Kjeldahl method), total fat (Soxhlet method), ash and crude fiber analyzes (AOCS 1996; AOAC 2005). Total carbohydrate was estimated by difference and all results were expressed on a dry matter basis. AF color was measured by CIELAB system using the parameters L* (100 = white; 0 = black), a* (+, red; -, green) and b* (+, yellow; -, blue) in a colorimeter CR-400 (Minolta, Mahwah, New Jersey, USA) with C illuminant.

Preparation of pasta

Control treatment (PC) was produced with wheat flour and distilled water (2:1 w/w) according to Saraiva et al. (2022). It was homogenized, pasta remained at rest for 30 minutes and was shaped and cut into strands (30 cm long and 0.6 cm wide) in a pasta machine. Pasta was pre-dried (12 min at 30 °C) and then dried at 60 °C (165 min), 80 °C (105 min) and 90 °C (90 min) to 12% final moisture. Drying times were determined when the moisture content reached a value less or equal than 12% (wet basis) by drying curve performed at 105 °C (Macedo et al. 2020; Vimercati et al. 2020). Pasta was stored in polyethylene bags for 36 h until analysis. AF enriched pastas were prepared by replacing wheat flour at concentrations of 5% (P5) and 10% (P10) w/w.

Bioactive compounds and antioxidant activity

Extraction of bioactive molecules

Compounds were extracted in 100% methanol for AF (1:200 w/v) and pasta (1:10 w/v). Samples and solvent were homogenized (15 min), centrifuged (10 min; 963 \times g) and the supernatant recovered for analysis.

Total phenolic compounds

Total phenolic compounds (TPC) were quantified by Folin-Ciocalteu method according to Singleton and Rossi (1965). An extract aliquot was mixed with Folin-Ciocalteu reagent and sodium carbonate, homogenized and left to rest in the dark (30 min). Samples were read in a spectrophotometer (EvolutionTM 300, Thermo Scientific) at 725 nm, and results were expressed in mg of Gallic acid equivalents (GAE) per g of AF and mg of GAE/100g of pasta.

Flavonoid content

Flavonoid contents (FC) were determined according to Buriol et al. (2009). An extract aliquot was mixed with aluminum chloride and 100% methanol. Reading was performed in a spectrophotometer at 425 nm and the results were expressed in mg of Quercetin equivalent (QE) per g of AF and mg QE/100g of pasta.

Total chlorophylls

Chlorophylls a, b and total were determined according to Nagata and Yamashita (1992). Reading was performed at 645 and 663 nm and results were calculated by equations:

$$\text{Chlorophyll a} = 0.999A_{663} - 0.0989A_{645}$$

$$\text{Chlorophyll b} = -0.328A_{663} + 1.77A_{645}$$

Total Chlorophyll = Chlorophyll a + Chlorophyll b

The results obtained were expressed in mg/g of AF and mg/100g of pasta.

Antioxidant potential

DPPH assay was performed according to Li et al. (2009) with modifications. AF and pasta extracts (150 μ L) were mixed with 2.85 mL of DPPH solution (60 μ M). After incubation in the dark for 30 min, absorbance was measured at 515 nm. ABTS free radicals were generated by ABTS reaction (7 mM) with potassium persulfate (2.45 mM). It was allowed to stand at room temperature for 16 h in the dark. ABTS⁺ solution was diluted with ethanol to absorbance of 0.70 ± 0.02 at 734 nm. ABTS assay was performed according to Re et al. (1999) with modifications. ABTS⁺ solution (1960 μ L) was mixed with AF and pasta extracts (40 μ L), and absorbance was measured at 734 nm after 6 min in the dark. Antioxidant activities were calculated using the following equation:

$$\text{Antioxidant activity (\%)} = (1 - (A_{\text{sample } t} / A_{\text{sample } t=0})) \times 100$$

Where:

$A_{\text{sample } t}$ is the absorbance of sample at 30 min (DPPH) and 6 min (ABTS), and $A_{\text{sample } t=0}$ is the absorbance of sample at time zero.

Quality properties of pasta

Ideal cooking time

Pasta strands (70 mm length) were cooked in boiling water (1:50 w/v). During cooking, every 60 seconds, disappearance of white crumb was evaluated by squeezing each pasta strand between two transparent glass plates (AACC Approved Methods of Analysis, Method 66-50 2000). The time when the white core completely disappeared was considered the optimal cooking time (OCT).

Cooking loss

Solid lost content in the cooking water was determined according to AACC Approved Methods of Analysis, Method 66-50 (2000). Pasta was cooked in boiling water (1:50 w/v) in its respective OCT, and then washed with 100 mL of cold water. Cooking water (25 mL) was collected in an aluminum container, and dried at 105 °C until constant weight. Residue was weighed and expressed as percentage of starting material.

Water absorption capacity

Water absorption was determined by increase in weight of 10g of pasta after cooking process and expressed as a percentage.

Color analysis

Pasta color was measured after the drying and cooking processes using the CIELab system.

Texture analysis

Samples were analyzed using a Brookfield-CT III Texture Analyzer (Middleborough, USA). Hardness parameter (N) was evaluated in pasta breakage analysis using probe TA7/TA-JTPB, test speed 2.0 mm/s, distance of 7.0 mm and force of 0.1N. Shear force was evaluated by cutting five strands of cooked pasta (5 cm) by hardness parameter using probe TA52/TA-SBA, test speed 1.0 mm/s, distance 10.0 mm, force 0.09N. Results represent the average of 5 replicates per sample.

Rheological properties of pasta

For rheological analyses was used fresh pasta (item 2.2) after rest 30 min and before

being shaped and cut into strands. Oscillatory test was performed according to Liu et al. (2022) with modifications. Rheometer (MARS II, Hache Thermo Fisher Scientific Inc., Newington, Germany) was used with parallel plates geometry of 35 mm with a gap adjusted to 3 mm at 25 ± 0.1 °C. A solvent trap was used to prevent sample surface dehydration. Samples (3 g) were applied and remained for 1 min to equilibrate the temperature and reduce the induced stress before analysis. Viscoelastic region for each treatment was determined and frequency sweep analysis was performed at a constant amplitude strain of 1% from 0.1 to 20 Hz. Storage modulus (G') and loss modulus (G'') were calculated using RheoWin 4.10.0000 (HaakeR) software.

Scanning electron microscopy of pasta

For microstructure analysis was performed by scanning electron microscope (SEM) (QUANTA 250) at 20 kV. Samples were used after molding and cutting (fresh pasta) and after drying (dry pasta), were frozen by liquid nitrogen and lyophilized (Christ Alpha 1-4 LD plus, Marin Christ, Germany). Small sample fragments were mounted on aluminum stubs using carbon tape and coated with a layer of gold (sputter coater, Baltec SCD 050, Balzers, Liechtenstein).

Statistical analysis

Results were evaluated by analysis of variance (ANOVA) using general linear model procedure in SPSS (v. 19.0) (IBM SPSS Statistics, SPSS Inc., Chicago, USA). Data were presented as mean and standard deviation. Significance level was considered at $p < 0.05$; Tukey's test was performed. The experiment was repeated twice with two replicates per treatment.

Results and Discussion

Asparagus pruning residue flour properties

AF was evaluated for physicochemical properties, bioactive compounds, and antioxidant activity (Table 1). AF has a high protein concentration (19.26%) making it an ingredient with potential to be used as protein source for several products. This protein value found in asparagus residue can be higher than obtained in other vegetable residues, as cauliflower leaf (4.76%), broccoli leaf (6.16%), cabbage leaf (3.39%), beet leaf (5.36%) (Sedlar et al. 2021).

High crude fiber value founded can be justified by stage in which the raw material was collected, asparagus leaves are the result of commercial stems ripening when not harvested at correct point. This residue can be considered another important active ingredient that can be added to foods as a source of fiber (Fuentes-Alventosa et al. 2009); and can be used as an ingredient for the development of functional foods (Jaramillo-Carmona et al. 2019).

In addition to protein and fiber, ash content of AF can contribute to improve foods minerals composition (as calcium, magnesium, iron, zinc, among others) when residue is transformed and used as ingredient (Redondo-Cuenca et al. 2023).

FC and TPC of AF were high (Table 1). Phytochemical characterization of asparagus leaf powder showed contained significant of flavonoids, that was like that previously described by other authors (Fuentes-Alventosa et al. 2013; Santiago et al. 2021). AF demonstrated high antioxidant potential by scavenging the DPPH and ABTS radical. Antioxidant activity can be explained in relation to the high levels of phenolic compounds determined by this study, including flavonoids and chlorophyll. The results on the

chlorophyll content in the leaves are presented in Table 1. Chlorophylls are used as natural dyes responsible for giving green color to foods. Lower chlorophyll contents were reported in *Amaranthus hypochondriacus*, *Amaranthus tricolor* (Khanam and Oba 2013), *Chenopodium quinoa* (Rodríguez et al. 2016) and *Portulaca oleracea* (Youssef and Mokhtar 2014) compared to the present study. Beyond pigments green, who can be extracted from various plant sources, (Viera, et al. 2019; Sharif et al. 2020; Wojdylo et al. 2021), at chlorophylls have anti-inflammatory, antioxidant and antimutagenic effects, reducing oxidative effects induced by free radicals (Sampaio et al, 2021). Data showed that asparagus residue can be considered an interest product due to their nutritional content and phytochemicals, such as phenolic compounds.

Quality properties of pasta

The total chlorophyll, TPC, flavonoids content present in the AF were incorporated into the pasta as shown in Figure 1. High drying temperatures can degrade FC and TPC as increasing temperatures are applied in the process. Opposite happened with TC, which increased with high temperatures and became responsible for the pigment in the mass, remaining adhered without detaching, indicating good adhesion of AF. Bioactive compounds are not thermostable, therefore, during drying they can be degraded, resulting in the decrease of these compounds (Roy et al. 2007; Sultana and Anwar 2008). In general, it can be stated that the lower the processing temperature, the lower the degradation rate (Rocha et al. 1993).

As for the evaluation of the antioxidant activity of the paste, the sample of dry pasta made from P10 showed a better percentage of oxidation inhibition in DPPH and ABTS (Fig. 2), mainly due to the presence of phenolic compounds in the AF and interactions with other compounds of the pasta matrix contributing to reduce its loss. The addition of

fruit residue in the pasta strongly increases the antioxidant properties of the integrated pasta, being a powerful source of many active substances that inhibit and neutralize cellular oxidative stress as reported in previous studies (Sivam et al. 2013; Sun-Waterhouse et al. 2013; Bustos et al. 2019). Lower temperature (60 °C) had a positive influence on the antioxidative activity conservation of pasta, the opposite occurred for the drying temperature at 90 °C (Fig. 2). This behavior is correlated to the presence of phenolic compounds in the pasta that degrade when exposed to high temperatures. It is notable that the decrease in phenolic compounds did not negatively affect the antioxidant activity (DPPH and ABTS values) of pasta enriched with AF, which, in general, were significantly higher than the PC.

Figure 3 shows the color values for dry and cooked pastas. AF level increased from 5% to 10% led to a significant decreased product luminosity (a) compared to the control treatment. An increase in green coloration (b) and a decrease in yellowing (c) were observed as AF level was added. Comparing temperatures applied in pasta drying, no statistical differences were observed in pastas colors when analyzed after drying. Several studies have investigated pasta enrichment with dried, powdered or pureed fruits, vegetables or compounds extracted from these raw materials in recent years (Dziki, 2021). Currently, pasta is available in colors wide range, result of synthetic dyes or plant extracts addition (Oliviero et al. 2016).

Addition of AF also modified pasta quality properties (Table 2). A higher OCT value indicates the extended time required for cook pasta. Quality, physical, and textural properties of pasta can be affected when different residues are included in its treatment (Desai et al. 2018; Rachman et al. 2020). However, the replacement wheat flour to AF from 5 to 10% reduced OCT regardless of applied drying temperature (Table 2), while a non-significant variation was observed when comparing OCT between temperatures

regardless of applied treatment.

Water absorption of PC and AF enriched pastas are shown in Table 2. Starch and protein networks are main factors that affect the water absorption tendency of pasta, which ranged from 34.98 to 14.44% with AF addition from 0 to 10%. Decrease in water absorption is attributed to the changes made in starch structure by AF addition and temperature used in drying process. Structure has become porous and increased starch hydrophilic groups, absorbing less water compared to PC, this can be attributed to shorter cooking times, which can reduce the pastas water absorption (Michalak-Majewska et al. 2020).

In CP, the greatest loss of solids occurred in the dry pasta at 90 °C (Table 2). The incorporation of AF provided a decrease in the loss of solids with an increase in replacement from 5 to 10%, ensuring a good interaction with the other ingredients. The continuous network that retains the starch granules in the pasta can be guaranteed through the enrichment process, influencing the low leaching by cooking, and reducing the ideal cooking time, obtaining a good technological quality of the pasta (Bustos et al. 2015).

Using temperatures above 60 °C, the thermal treatment contributes significantly to stabilize the quality of the enriched product in relation to nutrient losses and obtained better results when the pasta was dried at 90 °C. (table 2). The loss of nutrients by cooking the pasta can be improved in the drying step where stabilization occurs in the formation of the network, which can be carried out at high temperature (70–90 °C) or low (40 °C–55 °C). temperatures (Hong et al. 2020; Martin et al. 2019).

Physical properties of pasta

A continuous increase in G' and G'' was noted with increasing frequency (Fig. 4). All pasta dough samples were considered more elastic than viscous across the frequency

range studied, based on G' values greater than G'' values pasta viscoelastic behavior is influenced by structural interactions between the starch-starch, starch-gluten association, as well as gluten polymers aggregations that play a key role (Jia et al. 2019).

Dynamic modules (G' and G'') for AF enriched pasta were higher than control pasta, showing AF addition increased the viscoelastic behavior of pasta samples. The increase in elastic (G') and viscous (G'') behavior with 10% AF in the pasta treatment compared to the control can be attributed to strengthened protein network induced by proteins and fibers presence in the inserted material, producing a firmer and more extensible pasta.

For breakage analysis, analyzed in dried pasta strands, significant differences were observed between PC and P10 treatments (Table 3). As percentage of AF in the pasta increased, it promoted a decrease in hardness for the dried pastas at 60 and 80 °C. For dry pasta at 90 °C, the P10 treatment showed greater hardness when compared to the other treatments. Pasta strands with 10% AF dried at 60 and 80°C were the pasta with the lowest breakage value analyzed dry, presenting a weak network and easily brittle at the time of handling.

As there were significant differences between treatments and especially in samples dried at 90 degrees, the hardness of pasta analyzed by breaking was influenced by the incorporation of AF and can be mainly attributed to alterations in the gluten content and in the porosity of the structure.

The dissolution in the gluten network and the porous formation of the structure due to the addition of AF in the pasta treatment may be responsible for softer pasta strands. Furthermore, the structure of the fibers tends to be more porous compared to wheat flour, which leads to pasta with a higher moisture content, another factor responsible for lower hardness (Naknaen et al. 2016).

Higher gluten strength expresses a tougher texture in the pastas; texture results

indicated a potential strong and well-developed gluten network in AF enriched pasta samples.

Gluten protein matrix mainly consists of wheat gluten, which dips the starch granules into a well-developed gluten network (Han et al. 2020). When wheat flour is replaced by residues rich in dietary fiber in this type of treatment leads to a reduction in starch concentration, in the protein content forming the gluten network, which may result in less formation of the gluten network (Xiao et al. 2021).

The firmness parameter, when analyzed in cooked pasta, showed a statistical difference between treatments (Table 3). Increase the replacement of wheat flour by AF applying a drying temperature of 60 and 80 °C, reduced pasta firmness. When comparing the drying temperatures and their effect on the mass when it is subjected to high temperatures (80 and 90°C), the firmness of the threads increases. As there is no significant difference in the behavior of the texture of the enriched pasta compared to the control, the present work shows that the addition of AF in the pasta may be a good combination for the isolated properties.

Overall cooking quality of final product (firmness and low cooking loss) is the result of several simultaneous phenomena within the pasta, its extent depends both on the raw material characteristics and temperature and moisture conditions applied during drying (Bresciani et al. 2022).

Regarding pasta properties, high temperature drying cycles (>65°C) are effective in improving their quality characteristics (Padalino et al. 2016). Pasta drying at temperatures above 60 °C can partially compensate for the weakening of the pasta structure due to the reinforcing effect provided by protein coagulation (D'Egidio et al. 1993).

In relation to microstructure of treatments (Fig. 5), control showed a relatively weak gluten network structure, with the gluten network and starch granules mixed in disorder

for fresh pasta (Fig. 5a) and for dry pasta at temperatures of 60, 80 and 90 °C (Fig. 5b, c and d). Compared with control, the gluten network of macaroni enriched with 10% AF was better developed for fresh pasta (Fig. 5e) when for dry pasta (Fig. 5f, g and h), presenting mostly starch granules incorporated into the formed network. In addition, in pasta with AF, grouped gluten network structures, well organized and more resistant were observed (Table 3) forming a dense mesh that covered almost all the starch granules.

AF contains protein, cellulose, and polysaccharides, which can better fill the gluten protein network structure, while the polyphenols it contains can interact with gluten proteins to promote the formation of the gluten network (Sim et al. 2021). The strengthening ability of gluten in AF enriched pasta could be due to its convergent action on proteins caused by interactions between polyphenols and gluten proteins (Wang et al. 2015).

The temperature applied in the drying process influenced the structure of the pasta, observing smaller galleries and in greater quantities for the enriched pasta (Fig. 5) when using high temperatures (80 and 90 °C). This more porous structure together with the AF had a positive influence on reducing the OTC and consequently decreasing the WA. Gluten expands and recombines during gelation, forming a three-dimensional network at temperatures above 60°C (Wang et al. 2017). Meanwhile, the behavior of starch during heating is also determined by temperature changes (Malumba et al. 2013).

The formation of a compact protein network is an effect caused by the insolubilization of proteins that occurs due to the high relative humidity of the environment combined with the high temperature applied (West, 2012). Furthermore, the rapid evaporation of water in the drying process generates pores in the internal structure of the pasta as the drying temperature increases (Maache-Rezzoug and Allaf 2009); favoring water absorption during cooking, resulting in reduced cooking time (Petitot et al. 2009).

When processed under different temperature conditions, the protein can change conformation and molecular size. The main reason is that, as the temperature increases, gluten releases more chemical groups and forms new chemical bonds between them, resulting in new surface properties and increased protein aggregates (Stănciuce et al. 2018).

Conclusions

Asparagus pruning residue flour partial substitution 10% to wheat flour allowed an improvement of pasta technofunctional properties, as total phenolic content, flavonoids, antioxidant capacity, optimal cooking time and texture. Optimal drying temperature condition found in this study was 90 °C, considering the drying characteristics and the final quality of the pasta enriched with flour from the asparagus pruning residue.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Figure legend

Fig. 1. Total phenolic compounds (TPC), total chlorophyll (TC) and flavonoids content (FC) of dried pasta: PC (a), P5 (b) and P10 (c). PC: pasta control, without asparagus pruning residue flour (AF); P5: pasta with replacement of wheat flour by 5% AF; P10: pasta with wheat flour replacement with 10% AF. Different lowercase letters indicate statistical differences ($p < 0.05$) between treatments and different uppercase letters indicate statistical differences ($p < 0.05$) between temperatures.

Fig. 2. Free radical scavenging DPPH and free radical scavenging ABTS (%) for dried pasta treatments. PC: pasta control, without asparagus pruning residue flour (AF); P5: pasta with replacement of wheat flour by 5% AF; P10: pasta with wheat flour replacement with 10% AF. Different lowercase letters indicate statistical differences ($p < 0.05$) between treatments and different uppercase letters indicate statistical differences ($p < 0.05$) between temperatures.

Fig. 3. L^* (brightness) (a), a^* (redness) (b) and b^* (yellowing) (c) of pasta dried at different temperatures (60, 80 and 90 °C) and cooked pasta enriched with asparagus pruning residue flour (AF). PC: pasta control, without AF; P5: pasta with replacement of wheat flour by 5% AF; P10: pasta with wheat flour replacement with 10% AF. Different lowercase letters indicate statistical differences ($p < 0.05$) between treatments and different uppercase letters indicate statistical differences ($p < 0.05$) between temperatures.

Fig. 4. Oscillatory rheology of fresh pasta enriched with asparagus powder residue flour (AF). PC: pasta control, without AF; P5: pasta with wheat flour replacement for 5% AF; P10: pasta with wheat flour replacement for 10% AF. G' : storage modulus; G'' : loss modulus.

Fig. 5. Scanning Electron Microscopy (SEM) of fresh control pasta (a) and (e) fresh pasta enriched with 10% asparagus pruning residue flour (AF); control pasta (b) and pasta enriched with 10% AF (f) dried at 60°C; control pasta (c) and pasta enriched with 10% AF (g) dried at 80°C; control pasta (d) and pasta enriched with 10% AF (h) dried at 90°C with magnification x1500.

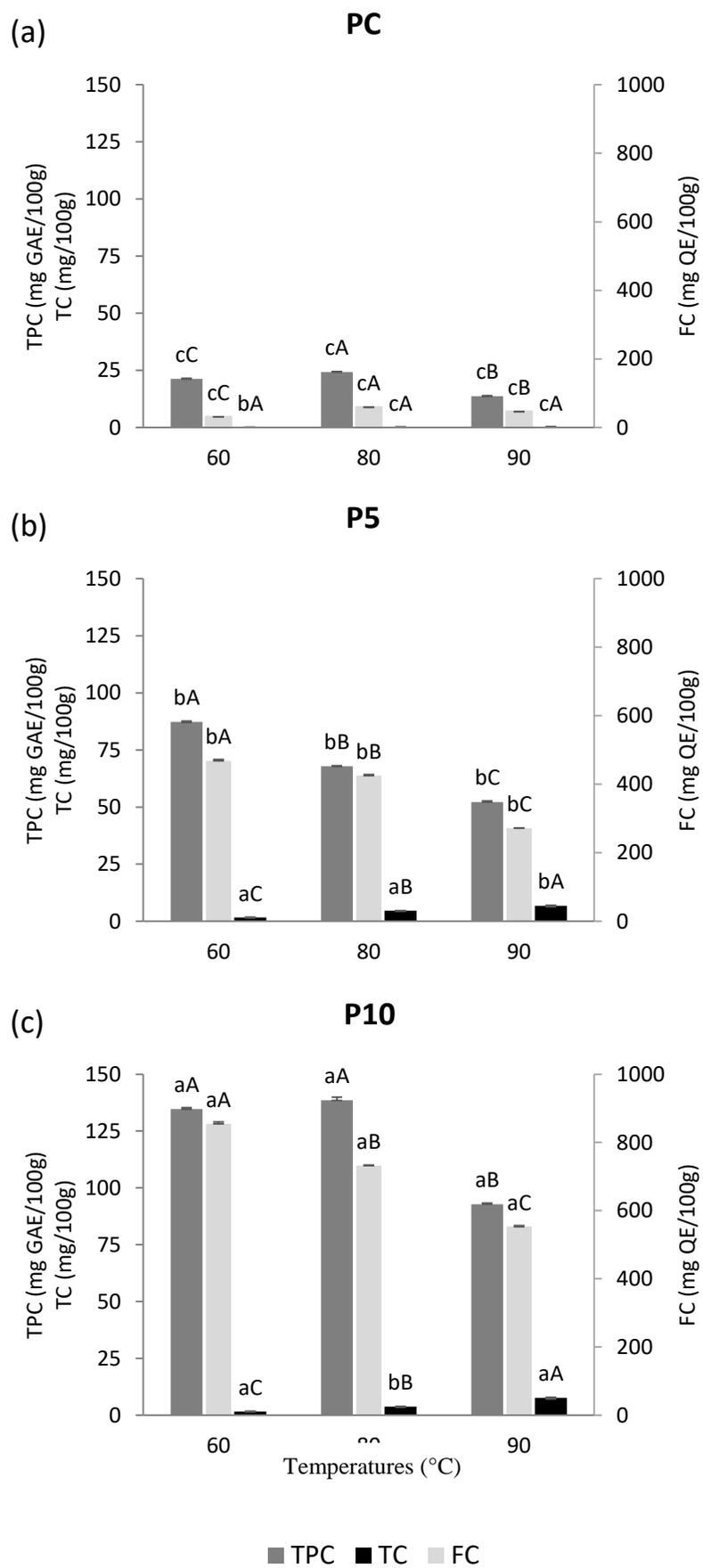


Fig. 1.

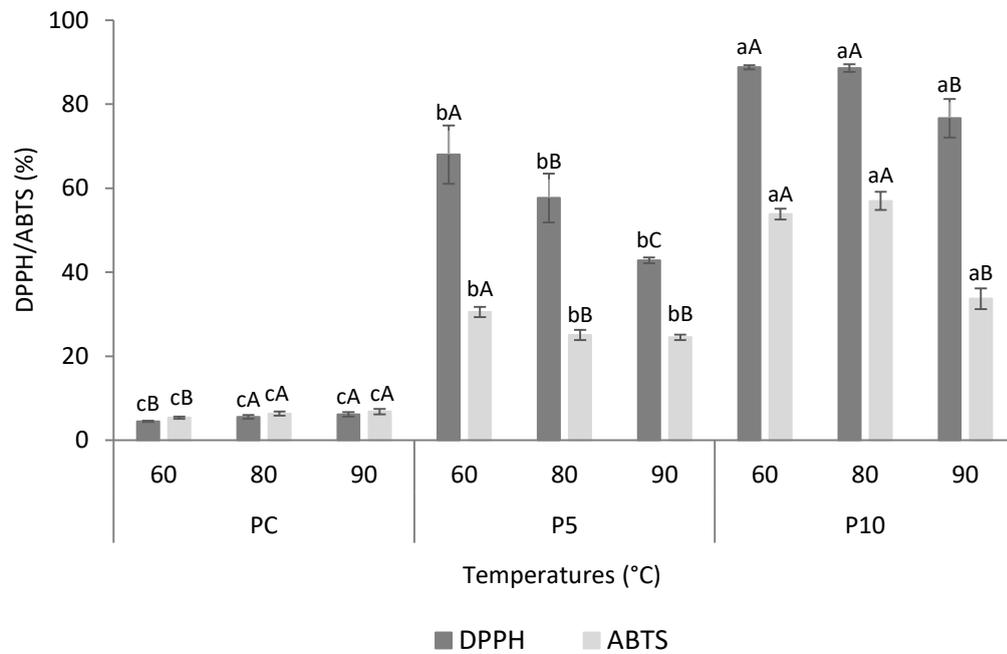


Fig. 2.

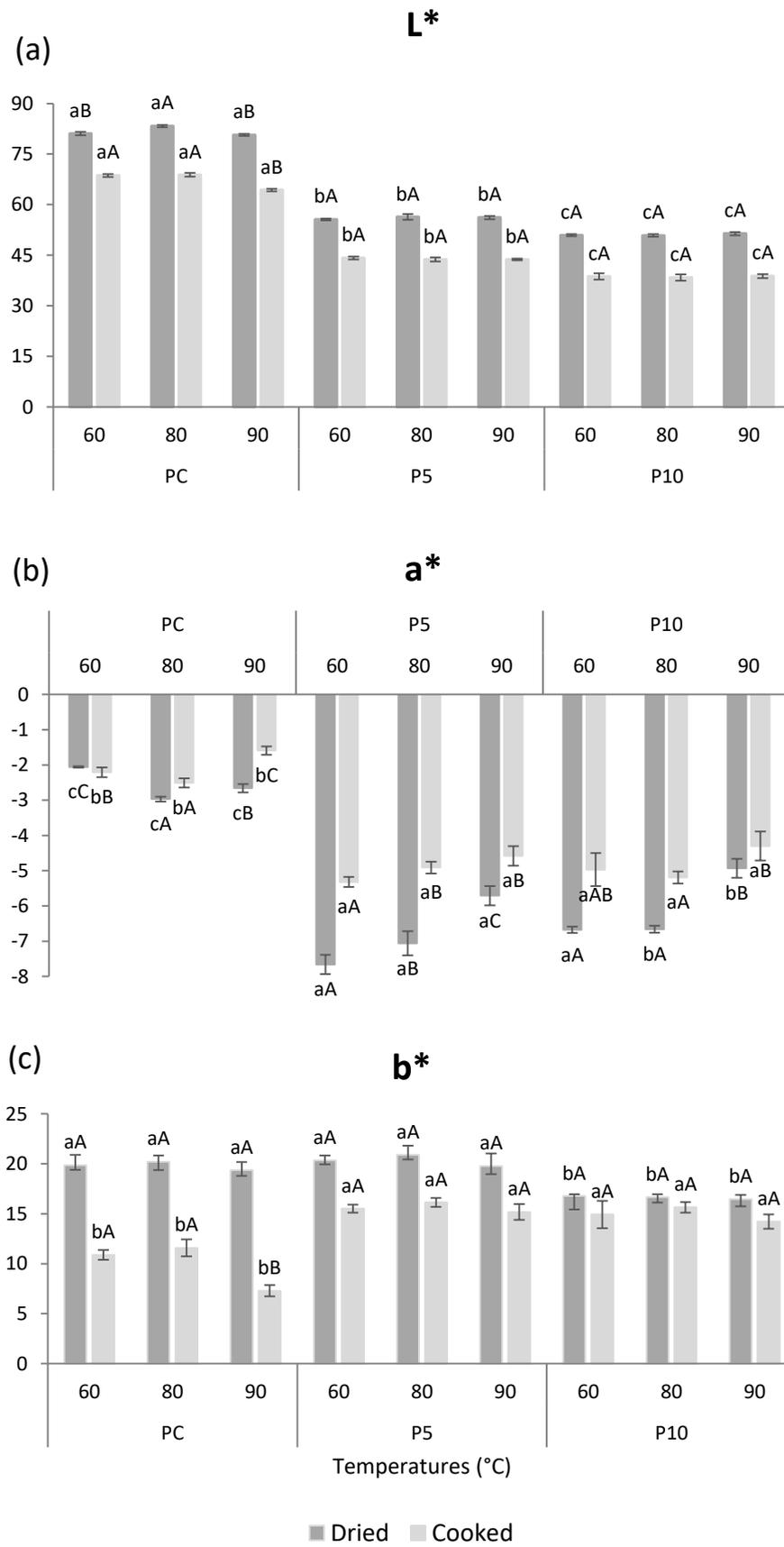
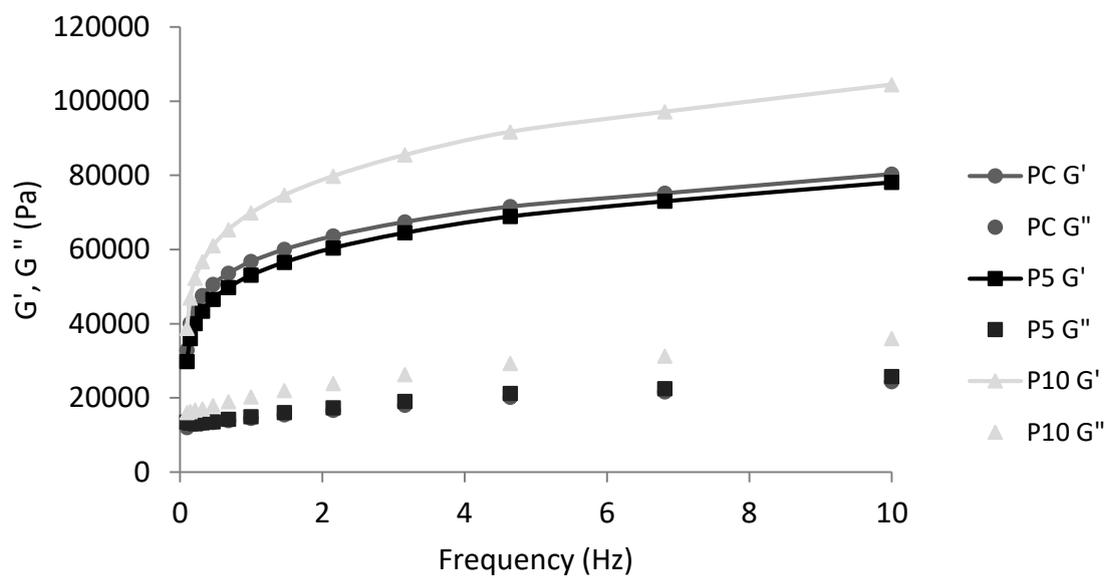


Fig. 3.

**Fig. 4.**

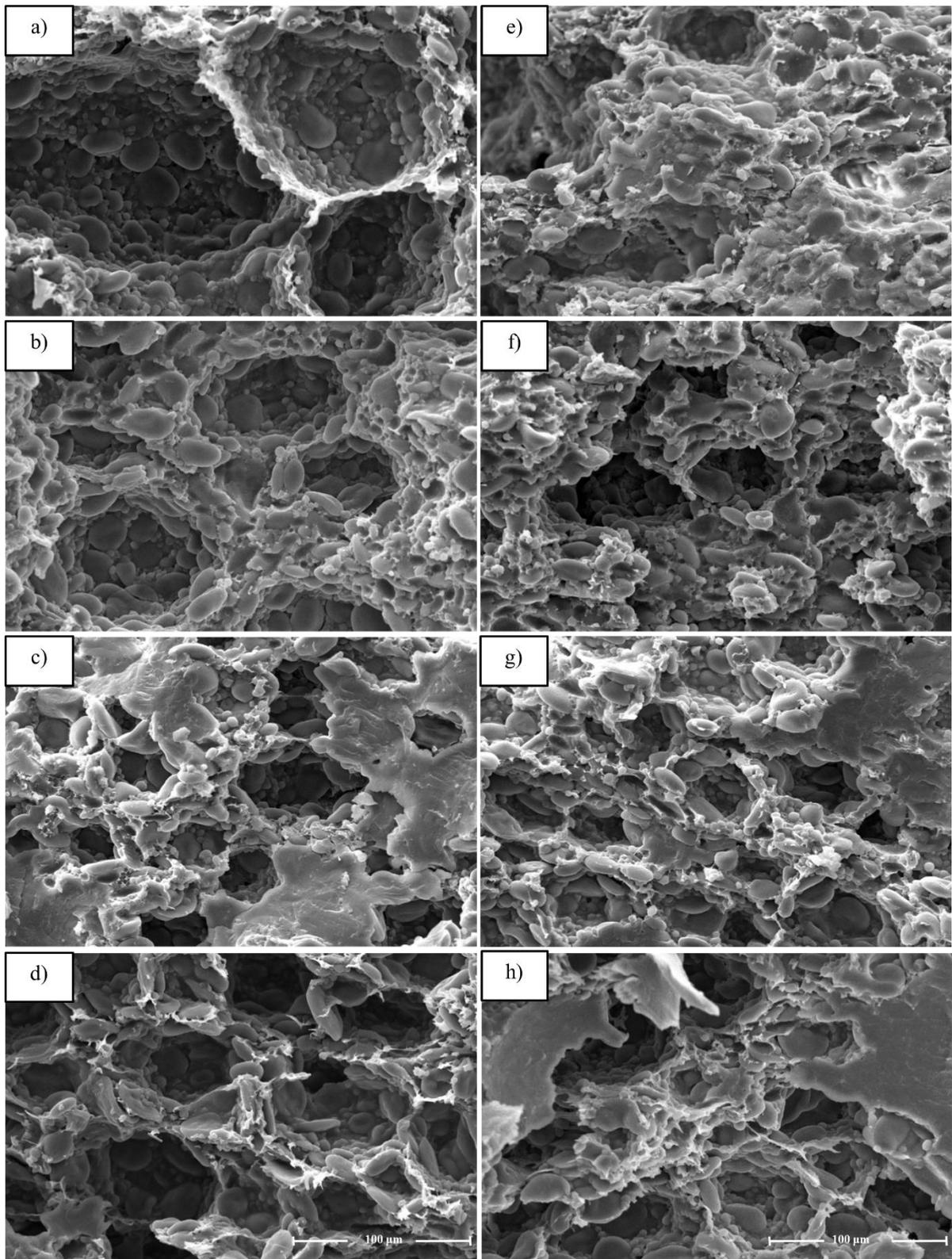


Fig. 5.

Tables

Table 1. Physical-chemical properties, bioactive compounds and antioxidant activity of asparagus pruning residue flour (AF).

| Physical-chemical properties | Results |
|------------------------------|---------------|
| Moisture (%) | 9.55 ± 0.23 |
| Crude protein (%) | 19.26 ± 0.15 |
| Fat (%) | 5.13 ± 0.18 |
| Crude Fiber (%) | 17.62 ± 0.17 |
| Ashes (%) | 8.13 ± 0.03 |
| Total carbohydrates (%) | 40.32 ± 0.11 |
| TPC (mg GAE/g) | 24.53 ± 0.13 |
| FC (mg QE/g) | 145.72 ± 4.68 |
| TC (mg/g) | 248.50 ± 4.34 |
| DPPH (%) | 83.47 ± 1.00 |
| ABTS (%) | 33.01 ± 1.05 |

Results are expressed as mean ± standard deviation. TPC: Total polyphenols content.

FC: Flavonoids content. TC: Total chlorophyll. GAE: Gallic acid equivalent. QE:

Quercetin equivalent. DPPH: free radical scavenging DPPH, ABTS: free radical scavenging ABTS.

Table 2. Quality properties of pasta enriched with asparagus pruning residue flour (AF) in different drying temperatures (DT). Optimal cooking time (OCT); Water absorption (WA); Cooking loss (CL).

| DT (°C) | Properties | PC | P5 | P10 | <i>P valor</i> |
|----------------|------------|-----------------------------|-----------------------------|-----------------------------|----------------|
| 60 | OCT (min) | 22.50 ± 1.73 ^{aB} | 15.50 ± 0.57 ^{bA} | 12.00 ± 0.81 ^{cB} | < 0.001 |
| 80 | OCT (min) | 26.50 ± 1.29 ^{aA} | 16.50 ± 1.00 ^{bA} | 12.25 ± 0.50 ^{cAB} | < 0.001 |
| 90 | OCT (min) | 24.00 ± 2.30 ^{aAB} | 17.50 ± 1.73 ^{bA} | 13.25 ± 0.50 ^{cA} | < 0.001 |
| <i>P valor</i> | | 0.035 | 0.116 | 0.044 | |
| 60 | WA (%) | 34.98 ± 2.80 ^{aA} | 20.64 ± 0.59 ^{bA} | 19.62 ± 1.29 ^{bA} | < 0.001 |
| 80 | WA (%) | 25.66 ± 1.14 ^{aB} | 18.10 ± 1.14 ^{bA} | 14.40 ± 1.10 ^{cB} | < 0.001 |
| 90 | WA (%) | 19.26 ± 1.60 ^{aC} | 16.85 ± 1.08 ^{abB} | 14.44 ± 1.36 ^{bB} | 0.003 |
| <i>P valor</i> | | < 0.001 | 0.002 | < 0.001 | |
| 60 | CL (%) | 22.97 ± 1.73 ^{aA} | 15.42 ± 0.41 ^{bA} | 13.14 ± 1.12 ^{bA} | < 0.001 |
| 80 | CL (%) | 23.93 ± 2.07 ^{aA} | 14.03 ± 1.31 ^{bB} | 11.34 ± 0.34 ^{bB} | < 0.001 |
| 90 | CL (%) | 26.45 ± 2.30 ^{aA} | 12.87 ± 0.73 ^{bB} | 7.51 ± 0.26 ^{cC} | < 0.001 |
| <i>P valor</i> | | 0.097 | 0.010 | < 0.001 | |

Results are presented as mean and standard deviation. Different lowercase letters indicate statistical differences ($p < 0.05$) between treatments and different uppercase letters indicate statistical differences ($p < 0.05$) between temperatures. PC: control, without AF; P5: pasta with replacement of wheat flour by 5% AF; P10: pasta with wheat flour replacement with 10% AF.

Table 3. Hardness of pasta enriched with asparagus pruning residue flour (AF) after drying in different temperatures (DT; breaking) and cooking process (cutting).

| DT (°C) | Hardness (N) | PC | P5 | P10 | <i>P valor</i> |
|----------------|--------------|----------------------------|----------------------------|---------------------------|----------------|
| 60 | Breaking | 3.83 ± 0.20 ^{aB} | 3.89 ± 0.28 ^{aA} | 2.66 ± 0.17 ^{bC} | < 0.001 |
| 80 | Breaking | 4.16 ± 0.22 ^{aB} | 4.02 ± 0.34 ^{abA} | 3.65 ± 0.12 ^{bB} | 0.045 |
| 90 | Breaking | 4.75 ± 0.28 ^{bA} | 3.99 ± 0.12 ^{cA} | 6.03 ± 0.22 ^{aA} | < 0.001 |
| <i>P valor</i> | | 0.002 | 0.797 | <0.001 | |
| 60 | Cut | 9.65 ± 0.54 ^{aB} | 7.90 ± 0.58 ^{bB} | 6.61 ± 0.49 ^{cB} | < 0.001 |
| 80 | Cut | 10.34 ± 0.49 ^{aA} | 8.36 ± 0.57 ^{bB} | 8.82 ± 0.57 ^{bA} | 0.002 |
| 90 | Cut | 9.34 ± 0.39 ^{aB} | 9.58 ± 0.64 ^{aA} | 8.66 ± 0.36 ^{aA} | 0.063 |
| <i>P valor</i> | | 0.044 | 0.009 | <0.001 | |

Results are presented as mean and standard deviation. Different lowercase letters indicate statistical differences ($p < 0.05$) between treatments and different uppercase letters indicate statistical differences ($p < 0.05$) between temperatures. PC: pasta control, without AF; P5: pasta with wheat flour replacement for 5% AF; P10: pasta with wheat flour replacement for 10% AF.