Effet de la durée d'exposition aux micro-ondes sur la structure des graines de pois chiches

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Résumé

Ce travail visait à étudier les changements physiques, morphologiques et structurels des graines de pois chiches soumises à différents temps d'exposition aux micro-ondes. Les graines pré-hydratées ont été exposées aux ondes électromagnétiques, en supposant que les molécules d'eau (dipôles) absorbent l'énergie et changent d'état. Lorsque l'eau se vaporise, la pression interne augmente, et des pores, fissures et cavités peuvent se former. Les effets du traitement thermique ont été évalués par la caractérisation physique des graines et par des images microscopiques. Comparées aux graines pré-hydratées, les graines traitées avaient des dimensions moins importantes, un volume et une surface inférieurs, entraînant une diminution de la densité et du volume du lit granulaire. Les traitements ont rendu les graines plus fragiles que les graines natives, réduisant leur force de rupture. Les images ont révélé une modification de la porosité, la formation de pores, fissures et cavités, ainsi qu'un détachement du tégument des cotylédons. Selon le temps d'exposition, les graines ont été séchées et torréfiées, avec un changement de couleur et de teneur en eau finale. Ainsi, ces pré-traitements des graines peuvent faciliter les étapes de décorticage, broyage ou mouture, tout en améliorant la conservation et en développant de nouveaux arômes et goûts.

Effect of microwave exposure time on the structure of chickpea

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ABSTRACT

The aim of this work was to study the physical, morphological and structural changes in chickpea grains subjected to different microwave exposure times. Pre-hydrated grains were exposed to electromagnetic waves, on the assumption that water molecules (dipoles) absorb energy and change state. As the water vaporizes, internal pressure increases, and pores, cracks and cavities can form. The effects of heat treatment were assessed by physical characterization of the grains and by microscopic images. Compared with pre-hydrated grains, treated grains had smaller dimensions, volume and surface area, resulting in a reduction in granular bulk density and volume. The treatments made the grains more brittle than native grains, reducing their breaking strength. Images revealed a change in porosity, the formation of pores, cracks and cavities, as well as detachment of the cotyledon integument. Depending on the exposure time, the grains were dried and roasted, with a change in color and final moisture content. These pre-treatments of the grains can facilitate the hulling, grinding or milling stages, while improving preservation and developing new aromas and tastes.

Context

The microstructure of food contributes to the determination of its physical characteristics and its textural and sensory properties. Describing the relationships between physical behavior and the microstructure of food helps to understand the mechanisms induced by manufacturing processes. Describing modifications in the microstructure of cereal grains and legume grains during their transformation through unit operations such as grinding, dehulling, or storage contributes to determining their usage qualities, for Schoeman L. in 2016.

Drying and heat treatments (such as roasting) affect the biochemical characteristics and structure of legume grains. Depending on the type and intensity of the heat treatment applied, the modifications more or less impact the shape, volume, specific surface area, porosity, and/or density of the grains. The structural changes in the grains notably depend on their initial water content and water transport phenomena within the matrix. When the water contained in the grains is heated to high temperatures, it can cause an increase in internal pressure. If the pressure exceeds the mechanical resistance of the cellular matrix, it can vaporize abruptly and induce structural changes: "puffing" phenomena. During drying processes, free water in the pores can be eliminated without grain shrinkage. Conversely, bound water migrates to the intercellular spaces and induces cell shrinkage, the formation of pores, cavities, and cracks, and may cause cell collapse, for Alean, J. in 2020, Divekar, M. T. in 2017 and Han, Z. in 2020.

During microwave treatment, electromagnetic waves are converted into heat within the material, leading to heat transfer from the inside out. Under the influence of an alternating electromagnetic field, the movements of dipolar molecules such as water are accompanied by intense intermolecular friction, causing self-heating of the material and structural modifications of the constituents of the plant matrix, for Curet, S. in 2019. In this context, this study aims to describe the mechanisms induced by intermittent microwave treatment and to evaluate the effects of microwave exposure time on the structural changes of pre-hydrated chickpea grains.

Materials and methods

Materials

Chickpea grains (Kabuli variety) were purchased from a market in Montpellier (France). The grains were sorted using a separator-cleaner (LA/LST, Westrup) at a speed of 1700 rpm, retaining only those with a diameter between 7 and 9 mm. The grains were stored in an airtight plastic bag weighing one kilogram, kept at 5 °C.

Hydration of the grains

The native grains, with a water content of 0.13 g water / g dry matter, were hydrated by soaking for 2 h in liquid water maintained at 5 °C in a beaker (grain:water mass ratio ; 1:3). After hydration, the grains were collected, wiped, and stored for 24 h at 5 °C in an airtight container to ensure an even distribution of water throughout the grain volume. The water content of the hydrated grains is 0.68 (±0.01) g water / g dry matter.

Microwave heat treatment

The hydrated grains were treated with microwaves using intermittent wave application. The treatments were carried out in a household appliance (LG® brand, model MH6535GDS), with a power of 1000 W at a frequency of 2.45 GHz. A sample of 150 ± 0.05 g of grains was placed to form a thin layer directly in the center of the rotating tray (Fig. 1). To homogenize the temperature, the samples were subjected to different treatments, combining 30 sec of microwave exposure with 10 sec without exposure. We tested the influence of the number of successive treatments, between 4 sequences (total microwave exposure time = 120 sec; total treatment time = 160 sec) and 20 sequences (total microwave exposure time = 600 sec; total treatment time = 800 sec). After the treatment, the grains were left on the rotating tray for 20 min at room temperature (about 20 ºC) for cooling, then stored in a plastic bag.

Figure 1. Grain placement on the rotating tray for microwave treatments.

Water content

The water content of the grains (expressed in g water / 100 g dry matter) was determined according to the ISO 24557:2009 - FR standard. A 3 ± 0.010 g sample of ground grains was dried in an oven at 130 °C for 2 h. The analyses were performed in triplicate.

Dimensions, shape, surface and volume

The grains were characterized by dimensional properties (height (H), width (L), and thickness (l)) measured using a digital caliper with a precision of 0.01 mm (Model: 500-181-30, Mitutoyo, Tokyo, Japan). Measurements were taken on 15 randomly selected grains. Assuming an approximately spherical geometry of the grains, we calculated the equivalent diameter (D; mm), surface area (S; mm²), volume (V; mm³), and sphericity (φ) of the grains according to the equations of Ortiz-Hernandez, A. A. in 2020.

$$D = (H \times L \times l)^{\frac{1}{3}} \quad (1)$$

$$\varphi = \frac{D}{H} \quad (2)$$

$$S = \pi \times D^2 \quad (3)$$

$$V = \frac{\pi D^3}{6} \quad (4)$$

The volumetric expansion index (Ψ; --) was used to describe the change in grain volume induced by the hydration treatment or the microwave treatment, with being the volume of the grains before treatment (V_{before}) and the volume of the grains after treatment (V_{After}).

$$\Psi = \frac{V_{after}}{V_{before}} \quad (5)$$

Apparent grain density and granular bulk density

The density of the grains was determined using the Hubbard pycnometer. Before each analysis, the pycnometer was cleaned, dried, and weighed empty (M_1). A sample of 5 grains was weighed (M_2) and placed in the pycnometer, which was then filled with paraffin oil (Gilbert®) with a density (ρ) of 0.862 g/mL, while removing air bubbles. The mass of the pycnometer with the grains and oil was measured (M_3). The pycnometer was emptied, dried, filled only with paraffin oil, and weighed (M_4). The measurements were performed in triplicate. The volume of oil displaced by the presence of the grains (V_{grains}; mL) and the apparent grain density (ρ_{grains}; g/mL) were calculated using the following equations.

$$V_{grains} = \frac{(M_4 - M_1) - (M_3 - M_1 - M_2)}{\rho_{paraffin}} \quad (6)$$

$$\rho_{grains} = \frac{M_2}{V_{grains}} \quad (7)$$

The apparent density of the grain bed was measured on a 50 g sample using a 250 mL graduated cylinder. A funnel consistently positioned at the same height, ensuring uniform distribution and repeatable filling, was used to pour the grains into the cylinder. Measuring the volume occupied by the grains in the cylinder allows for the calculation of the granular bulk density ($\rho_{granular\ bulk}$). The measurements were performed in triplicate.

$$\rho_{granular\ bulk} = \frac{grains\ mass}{Volume\ occupied} \quad (8)$$

Grain texture

The analysis of grain texture was performed using a texture analyzer (Model TA.XT2, Stable Micro Systems, Godalming, UK) equipped with a 3 mm diameter flat cylindrical metal probe. The texture measurement was carried out on a cotyledon. To separate the two cotyledons, a blade was used to cut the grains. The cotyledon was placed on the platform of the texture analyzer, and the probe was positioned directly above the center of the cotyledon. The maximum rupture force (N) was measured during a penetration test with a constant probe speed (1 mm/sec) and a maximum displacement of 90% of the initial height of the cotyledon. The measurements were repeated 20 times.

Grain color

The color of the whole grains was determined using a digital colorimeter (CR-400, Konica Minolta Optics, Inc., Tokyo, Japan) with the CIE-Lab color scale, considering the parameters: L* (lightness), a* (redness index), and b* (yellowness index). A 50 g sample of grains was placed in a black box to form a thick layer. The analyses were performed in triplicate.

Image acquisition with a Stereomicroscope

The microstructure of the grains was described using a stereomicroscope (model SMZ 18, Nikon). The surface of the grains was described from images of 18 whole grains randomly selected. The internal structure of the grains was described from a sample of 6 grains, which were sectioned perpendicular to the junction of the two cotyledons. The stereomicroscope was set to a magnification of 0.75x for surface description and 4x for internal structure description.

Scanning Electron Microscopy (SEM)

The internal microstructure of the grains was described using a scanning electron microscope (model Phenom ProX, Thermo Fisher Scientific) with an acceleration voltage of 10 kV. Images were captured at a magnification of 300x without pre-treatment. The grains were sectioned perpendicular to the junction of the two cotyledons. SEM observation was conducted on 6 randomly selected grains.

Statistical analyses

The data were expressed as means and standard deviations. Analysis of variance (ANOVA) and the Tukey test were used to test for significant differences ($p < 0.05$) with a 95% confidence interval.

Results

Effect on grain water content

Microwave treatment results in a decrease in the water content of hydrated grains (Figure 2). An increase in treatment duration leads to a non-linear decrease in water content, converging towards a value close to 0.02 g water / g dry matter for a 10 min microwave exposure time. The final water content is significantly lower than the water content of native grains (0.13 g water / g dry matter). Park, H. W. in 2018 studied the drying of soybean grains using a conventional method, which reached a water content of 0.3 g water / g dry matter after 5 h at 45 °C. During microwave treatment, hydrated grains undergo drying mechanisms. The absorption of electromagnetic waves by water molecules causes volumetric heating of the grains, with temperatures (not measured) that can exceed the vaporization temperature of water, for Ojwang, D. in 2021. Our results

demonstrate the effectiveness of microwave treatment in reducing grain water content with significantly shorter treatment durations (in this case, 10 min) compared to conventional drying processes, which require several hours of drying with hot air circulation.

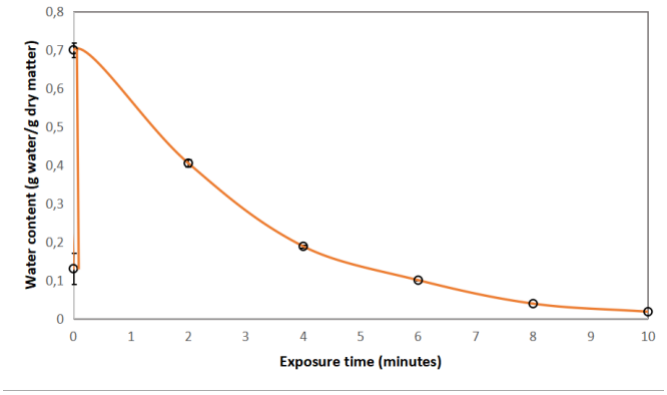

Figure 2. Variation in grain water content as a function of microwave exposure time.

Effect on grain dimensional characteristics

The volume of native grains (169 ± 10 cm³) is significantly lower than the volume of hydrated grains (227 ± 10 cm³). Water penetrates the grain coats, rehydrates the fibers, the cellular matrix, and the starch granules, causing the grains to swell, for Miano, A. C. in 2015. The effect of microwave treatment on the characteristic dimensions of the grains was evaluated based on the relative changes in values $((y_{(n-1)} - y_0) / y_0)$ as a function of microwave exposure time, with reference to the hydrated grain (y_0) (Figure 3).

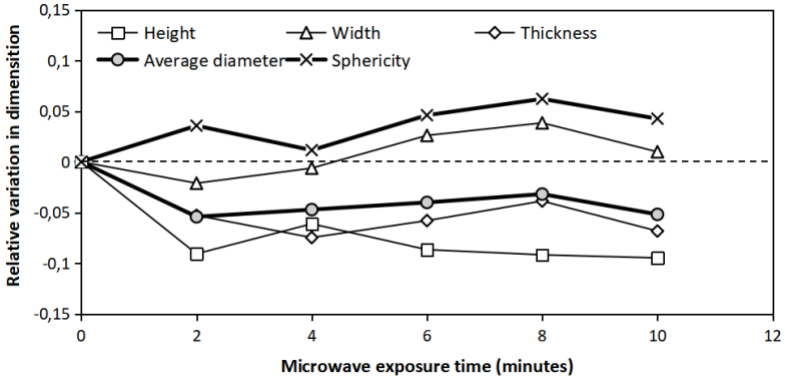

Figure 3. Variation in hydrated grain dimensional characteristics as a function of microwave exposure time.

Microwave treatment has no effect on grain width. In contrast, microwave treatment results in a monotonic decrease in height and thickness. These variations lead to a monotonic decrease in the calculated median diameter (Figure 3), volume, and surface area (data not shown) of the grains. Conversely, the results show a slight monotonic increase in grain sphericity, which can be associated with the loss of surface irregularities such as "wrinkles" and the appearance of a beak due to the hydration of grains, for Datta, A. K. in 2021. Anisotropic deformations may be related to the specific structure of chickpea grains.

Variations in grain size only partially describe the drying mechanisms during microwave treatment. The results after microwave treatment should be compared with the values of the characteristics of native grains (Table 1). The hydration step results in a significant increase in grain dimensional characteristics, which can be associated with water absorption by the grains. Regardless of the duration of microwave treatment, the values of grain dimensional characteristics remain significantly higher than those of native grains. The drying mechanisms induced by microwave treatment result in substantial water evaporation and a very significant

reduction in water content, reaching values well below those of native grains. Therefore, the reduction in water content is not associated with a proportional decrease in grain dimensional characteristics, as other mechanisms occur inside the grains during treatment, including expansion phenomena.

Table 1. Data on the characterization of native chickpea grains, after hydration, and after microwave treatment.

	Native grains	Grains after hydration	Grains after hydration and a 10 min treatment
Water content (g water / g dry matter)	0.130 ±0.010 ^{d}	0.677 ± 0.006 ^{a}	0.020 ±0.010^{g}
Height (mm)	8.12 ±0.27 ^{c}	9.81 ±0.27 ^{a}	8.88 ±0.33 ^{b}
Width (mm)	7.14 ±0.31 ^{c}	8.06 ±0.20 ^{ab}	8.14 ±0.48 ^{ab}
Thickness (mm)	6.80 ±0.25 ^{c}	7.76 ±0.16 ^{a}	7.23 ±0.39 ^{b}
Average diameter (mm)	7.33 ±0.22 ^{c}	8.49 ±0.18 ^{a}	8.05 ±0.31 ^{b}
Sphericity	0.903 ±0.028 ^{ab}	0.869 ±0.010 ^{c}	0.906 ±0.032 ^{ab}
Surface area (mm²)	169 ±10 ^{c}	227 ±10 ^{a}	204 ±16 ^{b}
Volume (mm³)	207 ±18 ^{c}	323 ± 20 ^{a}	274 ±32 ^{b}

The increase in water content induced by the hydration step results in a decrease in grain density (1.18 g/ml; hydrated grains) compared to native grains (1.29 g/ml). Conversely, microwave treatment, which induces a decrease in water content, results in a decrease in grain density (and granular bed density), with a monotonic decrease as a function of treatment duration (Figure 4). This phenomenon was observed by Jogihalli, P. in 2017, with a 34% reduction in the granular bed density of chickpeas after a 5 min microwave exposure time at 900 W. Microwave treatment induces other mechanisms that surpass the effects of water content reduction on grain dimensional characteristics.

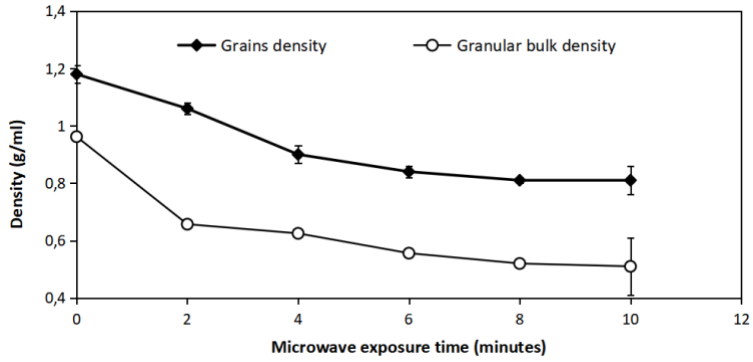

Figure 4. Variation in hydrated grains density as a function of microwave exposure time.

Effect on grain structure and microstructure

Microwave treatment alters the structure of the grains (Figures 5 and 6). Native grains have a spherical shape, with a compact and glassy structure. The grain coat adheres to the cotyledons, and the junction between the cotyledons is imperceptible (A). Hydrated grains have swollen, and the junction between the two cotyledons is visible as a line (B). Regardless of the duration of microwave treatment (C-G), the grains show a central cavity between the two cotyledons. The detachment of the grain coat becomes more pronounced after 6 min of treatment.

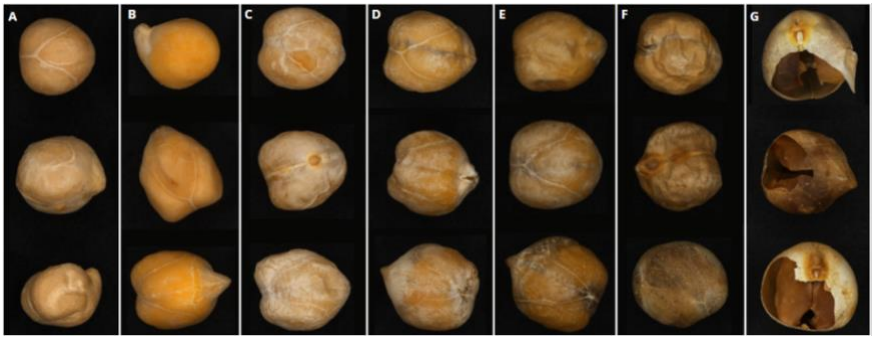

Figure 5. Native grains (A), hydrated grains (B), grains treated with microwaves for 2 min (C), 4 min (D), 6 min (E), 8 min (F), or 10 min (G).

The description of the internal microstructure of the grains as a function of microwave treatment duration (Figure 6) highlights the formation of an internal cavity between the two cotyledons, starting from 4 min of treatment. For longer treatment durations (Figure 6-G), such as 10 min, additional smaller cavities form within the grain structure at the cotyledon level or between the cotyledon and the outer grain coat. In this case, the internal temperature of the grain rises, allowing the water to transform into vapor. This phase change leads to an increase in volume, which in turn causes a rise in internal pressure within the grain, contained by its outer layers. At this stage, the grain's outer layers also dehydrate, losing their plasticity, which results in detachments and ruptures observed in the seed coat.

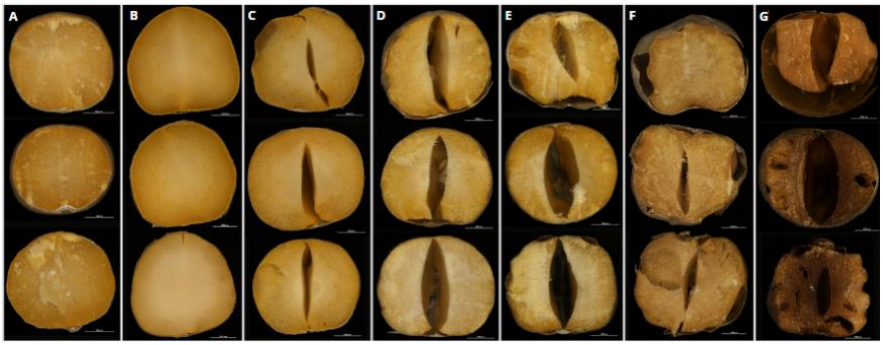

Figure 6. Native grains (A), hydrated grains (B), and grains treated with microwaves for 2 min (C), 4 min (D), 6 min (E), 8 min (F), or 10 min (G).

SEM images (Figure 7) show that in native grains, starch granules appear round, small, and well-organized, with a diameter of about 30 µm, for Wani, I. A. in 2016. Microwave treatment results in the formation of small pores between the starch granules. After 4 minutes of microwave treatment, swelling of the starch granules is observed, with them becoming more elongated and larger. For grains treated with microwaves exposure time for 6 to 10 min, the formation of pores between the starch granules is visible, with pore diameters estimated at around 15 µm. The pores appear to be evenly distributed within the cotyledon structure.

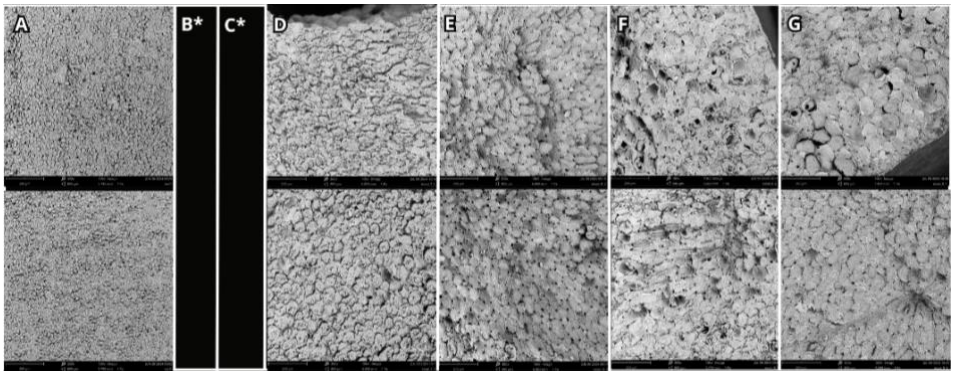

Figure 7. Native grains (A), grains treated with microwaves for 4 min (D), 6 min (E), 8 min (F), or 10 min (G). SEM images for hydrated grains (B) and grains treated for 2 min (C) could not be obtained due to their too high water content.

Effect on grain texture

The breaking force of native grains (210 ± 51 N) is significantly higher than that measured for hydrated grains (28.3 ± 2.3 N). Microwave treatment of hydrated grains results in an increase in the breaking force between 0 and 6 min of microwave exposure time, followed by a decrease beyond 6 min of treatment (Figure 8), due to the effect of two opposing mechanisms. Drying mechanisms are expressed as a progressive hardening of the grains with an increase in hardness for short treatment times, for Klamczynska, B. in 2001. When the thermal treatment becomes intense, the decrease in grain hardness may be associated with expansion mechanisms leading to the formation of cracks, cavities, and pores.

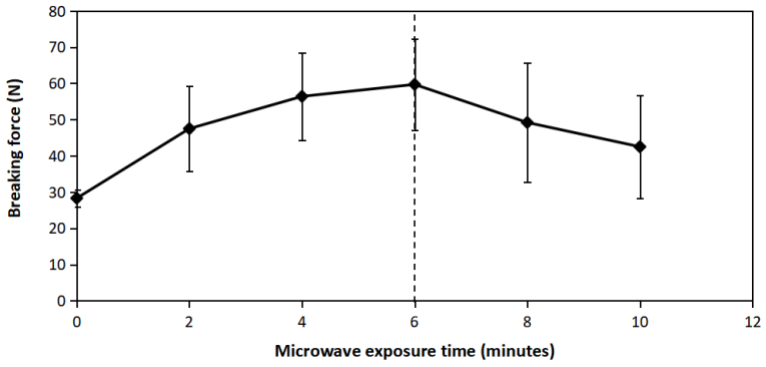

Figure 8. Variation of the breaking force of the hydrated grains as a function of microwave exposure time.

Effect on the appearance and color of grains

Microwave treatment has a significant impact on the color of the grains (Figure 9), highlighting two phases. Between 0 and 6 min of microwave exposure time, lightness increases slightly, while the red and yellow indices decrease. Beyond 6 min of treatment, lightness (L*) decreases, and the effects on the yellow and red indices are less pronounced. The decrease in the red index (a*) may be associated with the formation of brown pigments due to Maillard reactions, for Gujral, H. S. in 2011. Jogihalli, P. in 2017 observed that different exposure times affected the color of flours.

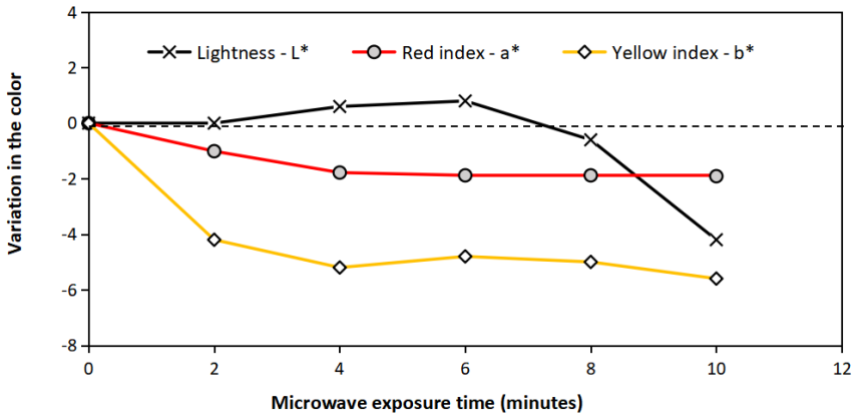

Figure 9. Variation in the color of the hydrated grains as a function of microwave exposure time.

Discussion

Microwave treatment of pre-hydrated chickpea grains leads to significant changes in their dimensional characteristics, density, and structure, which affect their color and texture. Microwave treatment induces several antagonistic mechanisms. The conversion of microwaves into heat within the grain results in an increase in the grain temperature, which causes dehydration phenomena. Rapid evaporation of water at the surface of the grains generates a moisture gradient within the grain, with higher moisture content at the center. This gradient favors the development of a temperature gradient, with a higher temperature at the center of the grain, as the conversion of microwaves into heat will be more pronounced at the center of the grain. The dual gradient of moisture content and temperature between the core and the surface of the grain induces water transfer mechanisms within the grain and rapid evaporation mechanisms. An increase in temperature beyond 100 °C at the center of the grain may also lead to an increase in water vapor pressure within the grain. When pressure values exceed the mechanical strength of the matrix, expansion phenomena may occur. Schoeman, J. in 2016 demonstrated by X-ray tomography that wheat grains roasted in a rotary drum at 180 °C for 140 sec exhibited separation of the cotyledons, cavities, and cracks.

At the molecular level, the conversion of microwaves into heat within the hydrated grain can also result in several reaction mechanisms. When the temperature exceeds 70-80 °C, hydrated starch granules may undergo gelatinization processes with the "melting" of crystalline regions between the amylose and amylopectin chains. The moisture content is not sufficient for gelatinization mechanisms to induce the breakdown of the granular form of starch granules, which remain visible in the grains. According to Lefevre, C. in 2021, partial gelatinization of chickpea starch occurs at temperatures between 100 and 110 °C when the moisture content is close to 0.5 g water/g dry matter. The increase in temperature in the grains also allows for the expression of the Maillard reaction, leading to the formation of colored compounds that affect the color of the grains, especially at relatively low moisture contents, for Mishra, G. in 2014. The increase in grain temperature also induces protein denaturation mechanisms.

The experimental approach implemented did not allow us to measure the temperature generated in the grains by microwave treatment as a function of microwave exposure time. Further work is planned to describe the reaction mechanisms at the molecular level, particularly those involving proteins and starch.

Conclusion

This study demonstrated that the microwave exposure time of pre-hydrated chickpea grains affects their morphology and structure. Microwave heat treatment reduces water content, leading to a decrease in the mass of the grains. After treatment, the grains exhibit expansion and a change in porosity compared to native grains. Another interesting aspect revealed by this study is the decrease in the rupture resistance of treated grains compared to native ones. This increased fragility could facilitate milling steps, likely requiring less mechanical effort. The images show that the treatments cause a detachment of the seed coat from the cotyledons, which could simplify the dehulling steps. The 10 min microwave exposure time induces color changes (and also aromatic profile changes) in the grains, altering their potential applications. Further studies are needed to assess the impact on other parameters, understand the mechanisms behind crack and cavity formation at both macro and microstructural levels, and to establish an energy balance of these treatments in grain processing.

Acknowledgements

The authors sincerely thank *Institut Agro Montpellier* for the grant provided under the program of the Research and Innovation Commission (CRI). The authors also express their gratitude to the PLANET platform (https://doi.org/10.15454/1.5572338990609338E12), managed by UMR IATE, for its experimental support in this work. Special thanks go to technician Aurélie Putois (INRAE) for her assistance with image acquisition. This work was partially supported by the *Coordenação de Aperfeiçoamento de Pessoal de Nível Superior* (CAPES), Brazil, through a scholarship for trainees – Funding Code 001.

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