

VOL. 44, 2015



Guest Editors: Riccardo Guidetti, Luigi Bodria, Stanley Best Copyright © 2015, AIDIC Servizi S.r.I., ISBN 978-88-95608-35-8: ISSN 2283-9216

# Microwave Technology Applied in Post-Harvest Treatments of Cereals and Legumes

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For agricultural purposes microwave heating is an emerging technology today successfully applied in postharvest disinfestation treatments of many agricultural products, such as cereals and legumes, susceptible of degradation due to the presence of natural infesting fauna. Microwave irradiation is proposed as an effective alternative to chemical methods of disinfestation which can be characterized by severe side effects. Extensive studies focused on disinfestation treatments assisted by microwave have proved their effectiveness. Overall goal of the present research is to point out correlations between thermo-physic properties and irradiation conditions (power, time to exposure, load configuration) to stabilize cereals and legumes in short time without damages of structures and nutritive features. In this work, two kinds of agro-foods, weak wheat and beans, were undergone to microwave heating and several physical properties, devoted to control structural possible changes, were investigated on irradiated samples. The achieved results show that the applied irradiation protocol globally does not affect water uptake phenomena in swelling and cooking treatments, peel hardness and germination capability of seeds.

# 1. Introduction

Microwave heating processes are currently applied in many fields: from minerals treatments (Palma et al., 2011) and environmental remediation processes (Barba, d'Amore, 2012, Bientinesia et al., 2013); from food industry (Marra, 2012, Lyng et al., 2014) to pharmaceutical emerging technologies (Chandrasekaran et al., 2012). The reasons for this growing interest are due to the peculiar mechanism for energy transfer: during microwave heating, energy is delivered directly to materials through molecular interactions with electromagnetic field via conversion of electrical field energy into thermal energy. This allows unique benefits, such as high efficiency of energy conversion and short processing times, thus reductions in manufacturing costs thanks to energy saving, and selective heating (Acierno et al., 2004). According to (Barba, d'Amore, 2012) main features are briefly summarized in the following. Microwaves are electromagnetic radiation with frequency ranging from 0.3 GHz to 300 GHz; the most common frequencies dedicated to microwave power applications for industrial, scientific and medical (ISM) purposes are: 0.915 GHz and 2.45 GHz. Microwave heating of a material is strictly related to the dielectric properties (or permittivity) which express the energy coupling of a material with the electromagnetic microwave field. Dielectric properties are indicated as a relative complex number:  $\epsilon r = \epsilon/\epsilon_0 = \epsilon' - i \epsilon''$  where  $\epsilon_0$  is the vacuum permittivity ( $\epsilon_0 = 8.85 \times 10^{-12}$  F/m);  $\epsilon'$  is the part (Re) named dielectric constant and  $\varepsilon''$  is the imaginary part (Im) known as loss factor ( $j=\sqrt{-1}$  is the imaginary unit). The dielectric constant is a measure of how much energy from an external electric field is stored in the material; the loss factor accounts for the loss energy by dissipative mechanisms in the material. A material with a high loss factor, i.e. water and wet materials, is easily heated by microwaves; mixtures of different substances can be selectively heated on the bases of their capability to dissipate energy. Microwave energy dissipation is given by the common form of the average power loss density (power dissipation per unit volume, W/m<sup>3</sup>, P) drawn from the Poynting's theorem (Metaxas, Meredith, 1983):

$$P = \frac{1}{2} \omega \varepsilon_0 \varepsilon^{"} \left| E \right|^2 \tag{1}$$

where  $\omega$  is the angular frequency.  $2\pi$ f and E is the electrical field strength [V/m]. Bulk heating is achieved when penetration depth ( $D_p$ ), defined as the distance from the material surface at which the power drops to e<sup>-1</sup> of its initial value, is of the same order of magnitude of materials dimensions. Assuming electromagnetic field as a plane wave that travels along one axis, penetration depth is calculated as following:

$$D_p = \frac{c}{2\sqrt{2\pi}f\sqrt{\varepsilon'}\left[\sqrt{1+\tan^2\delta} - 1\right]^{\frac{1}{2}}}$$
(2)

where  $\tan \delta$  (= $\epsilon$ "/ $\epsilon$ ') is the loss tangent, a parameter frequently used in dielectric heating literature, providing indications of how the material can be penetrated by an electric field and how it dissipates the energy in heat. When bulk heating is not achievable, a temperature levelling effect can occur in thick layer depending from materials thermal diffusivity that can drive the heat distribution within the whole bulk.

For agricultural purposes microwave heating is an emerging technology today successfully applied in postharvest disinfestation treatments of many agricultural products, such as rice and beans, susceptible of degradation due to the presence of natural infesting fauna (Yadav et al., 2012, Mohapatra et al., 2014). Microwave irradiation is proposed as an effective alternative to chemical methods of disinfestation (fundamentally based on fumigation) which are characterized by several side effects, mainly undesirable residues in treated matrices, long treatment times and noncompliance with the international rules on protecting the earth's atmosphere established in the Montreal Protocol. Microwave technology for disinfestation application is based on the selectivity principles of heating driven by the dielectric loss mechanisms: when a mixture of dry materials (such as legumes and cereals at post-harvest conditions) and pests is irradiated, the insects are more quickly warmed up to lethal temperature due to their higher water content. Dry matrices can be unaffected or slightly heated by microwaves. Despite extensive researches and applications of microwave heating in post-harvest treatments (Wang et al., 2003, Zhao et al., 2007, Yadav et al., 2012, Mohapatra et al., 2014), a comprehensive study on structural properties and heat transport in starchy and protein structures, with low moisture content, is still lacking. Overall goal of the present research is to point out correlations between thermo-physic properties (evaluated by thermal analysis using thermogravimetry, TGA, and differential scanning calorimetry, DSC) and irradiation conditions (power, time of exposure, load configuration) to stabilize cereals and legumes in short time without damages of structures and nutrient contents. For this purpose a wide campaign of investigation on several legumes and cereals are being performed in the frame of a project focused on post-harvest microwave treatments.

Aim of this study is to characterize physical properties of two kinds of agro-foods in form of solid granules: weak wheat and beans. These were chosen as model matrices for cereals and legumes, respectively, to be processed by microwave irradiation cycles for drying and disinfesting purposes (post-harvest treatments). In particular, attention here is focused on physical characterization in terms of dielectric properties, water content, texture analysis and miscellaneous granular mass properties. These characterizations are finalized to show if microwave irradiation affects some features which contribute to define the overall quality after microwave treatments of beans and weak wheat.

#### 2. Materials and Methods

## 2.1 Materials

Beans (*cv. Borlotto*) and weak wheat (*cv. Risciola*) matrices (seeds) were kindly supplied by the Azienda Agricola Sperimentale Regionale *Improsta*, Eboli (SA) Italy. The matrices were stored at room conditions and used as received. For several characterizations (dielectric properties, moisture content) a milling process was applied, followed by a sieving step. Raw seeds constitute the control for all the determinations.

### 2.2 Methods

*Dielectric properties*. Dielectric properties were assayed using the coaxial protocol measurement (with three calibration standard) network analyzer Agilent Technologies mod ES 8753 and coaxial probe Agilent Technologies mod 85070D. The measurements were performed on milled raw granules, in triplicate. Results were reported as average values.

*Microwave treatment.* The microwave assisted treatments were performed by a multimodal microwave cavity (LBP 210/50 Microwave Oven 2300 W, InLand, USA; operative frequency: 2.45 GHz ) equipped by the True-To-Power<sup>TM</sup> system to continuously vary the power supply and by two integrated mode stirrers. During microwave irradiation temperature measurements were carried out by the infrared pyrometer Simpson mod. IR-10. A layer of 4 cm in thickness of beans and weak wheat were placed in a Pirex sample-holder and exposed to microwave irradiation. After several irradiating tests finalized to achieve a temperature of 70°C, temperature over the lethal temperature of typical infestants of beans and wheat (most species will not survive more than 24 h at 40 °C, 12 h at 45 °C, 5 min at 50 °C, 1 min at 55 °C, and 30 s at 60 °C (Fields, 1992)), in the solids bulk, a power of 1000 W and exposure time of 1'15" were chosen as operative protocol parameters. The seeds (beans and wheat granules) exposed to this microwave protocol were indicated in the following as "treated" matrices.

*Germination test.* Germination was determined on both raw (the control) and irradiated seeds using several tens of beans and wheat granules placed in Petri dishes on moist paper towels, left at room conditions, and moistened twice a day during the 5 days of observation.

*Moisture content.* Moisture contents of raw seeds, treated and not treated by microwaves, before and after cooking treatments, were performed using the Ohaus moisture balance mod MB45. All the measurements were performed in triplicate and the results were reported as average values.

*Seed size*. Bean and wheat seeds size (main dimension) were measured by a manual gauge on several tens of seeds selected at random; the results were reported as average values.

T test was used to compare values of moisture and size for treated and untreated samples: p-value is the probability that the difference between treated and untread samples is casual, so if p < 0.05, there is difference between treated and untreated samples, on the contrary, if p > 0.05, the two samples are similar.

*Swelling test.* Swelling investigation were developed to test water absorption and seeds electrolytes leached into the soaking water (Berrios et al., 1999). To these aims several tens of seeds were soaked in 100 mL distilled water at room conditions for up to 48 h. The soaked seeds were blotted with a paper towel after given times to remove excess water, weighed and placed back into the soaking water. Water uptake absorption value was expressed as a percentage of water absorption:

Swelling ratio % = Percentage of adsorbed water = 
$$\frac{\text{Soaked seeds weight-Dried seeds weight}}{\text{Dried seeds weight}} \cdot 100$$
 (3)

Electrolytes leached into the soaking water were assayed in terms of minerals enrichment by the conductivitymeter Crison basic mod. GLP 31. Mineral losses were reported as increase of (water) solution conductivity. Soaking and conductivity measurements were performed in triplicate; the results were reported as average values.

*Cooking treatment.* To evaluate *seed peel hardness* after cooking treatments, beans and wheat were first soaked in water for 20 h and then boiled in water for 30 min.

Penetration test (seed peel hardness). To measure the seeds shell hardness of partially swollen and cooked seeds the texture analyzer TA.XTplus from Stable Macro System with the P2N needle probe and a 5 kg loading cell, was used. The penetration force on a single seed of untreated and treated beans and weak wheat, after swelling for 20 h and after cooking, was determined setting a needle speed of 0.05 mm/s and strain percentage of 20 % for beans (weaker than wheat) and 70 % for wheat. The penetration force (peel hardness) was evaluated as the peak of the curve "force vs strain" (Lee, Chung, 1989) (the strain at the penetration force was an index of peel elasticity, i.e. how much the peel resists before rupture). Moreover after the peak, the curve can be considered as a straight line with a slope that represents the compactness of internal structure: a higher slope is index of higher compactness (more resistance to penetration).

## 3. Results and Discussion

Dielectric properties measurements of untreated (or raw) grains of beans and wheat (Figure 1) have shown, as expected, low values mainly due to the reduced residual moisture content in post-harvest phase (12.2  $\pm$  0.24 % and 12.9  $\pm$  0.3 % wb - wet bases - for beans and wheat, respectively -Table 1). These allow to achieve high temperature in the seeds only in relatively long time, depending on power irradiation condition, well beyond the lethal conditions required to kill infestants. Moreover these values can guarantee a volumetric heating due to a large penetration achievable (for both the examined seeds the D<sub>p</sub> calculated by eq. 2, at 2.45 GHz, is roughly 7 cm).

After the irradiating treatment (following the protocol previously reported) seeds of beans and wheat were undergone to germination tests. These have shown that, for both beans and wheat, the germination capability has not been compromised (Figure 2).

In Table 1 moisture content on wet basis (%) and size (main dimension, mm) with related SD, of treated and untreated, before and after cooking, beans and weak wheat seeds were summarized. Seeds showed the following trends: microwave treatment reduced the moisture content (p < 0.05 for both beans and wheat) but not the size of seeds (p > 0.05 for both beans and wheat); cooking treatment allowed similar water uptake and size enlargement for both treated and untreated beans (p > 0.05), instead treated weak wheat absorbed more water and had a lower size increase during cooking with respect to untreated wheat (p < 0.05).



Figure 1: Dielectric properties (dielectric constant and loss factor) of beans and weak wheat (on milled raw materials)



Figure 2: Tests of germination for seeds of bean and wheat treated (on right) an not treated (on left) by microwave

Table 1: Moisture content on wet basis (%) and size (main) with related SD of treated and untreated, before and after cooking, beans and weak wheat seeds. There are also p values from comparisons done.

|                       | Untreated       | Treated         | Untreated - cooked | Treated - cooked |  |
|-----------------------|-----------------|-----------------|--------------------|------------------|--|
| Moisture content % wb |                 |                 |                    |                  |  |
| Beans                 | $12.2 \pm 0.24$ | 11.0 ± 0.18     | 68.4 ± 0.89        | 70.2 ± 0.97      |  |
| p (t test)            | 0.0             | 028             | 0.0734             |                  |  |
| Weak wheat            | $12.9 \pm 0.30$ | 11.1 ± 0.52     | 60.4 ± 0.27        | 61.5 ± 0.15      |  |
| p (t test)            | 0.0             | 066             | 0.0038             |                  |  |
| Size, mm              |                 |                 |                    |                  |  |
| Beans                 | 14.55 ± 1.53    | 14.50 ± 1.13    | 18.30 ± 2.87       | 17.78 ± 1.12     |  |
| p (t test)            | 0.0907          |                 | 0.6065             |                  |  |
| Weak wheat            | $6.63 \pm 0.45$ | $6.36 \pm 0.48$ | 6.87 ± 0.81        | $5.92 \pm 0.58$  |  |
| p (t test)            | 0.1             | 934             | 0.0075             |                  |  |

Swelling tests results, in terms of swelling ratio and increase of water solution conductivity profiles were reported in Figure 3 and Figure 4. Within about 24 h the swelling ratio of both treated and untreated beans increases until to reach 100 % (Figure 3), as already seen in literature (Berrios et al., 1999). This means that microwave irradiation did not affect the water uptake capability of beans. A similar trend is recognizable in swelling ratio profiles of wheat grains (Figure 4), even if with a different magnitude (50 %) (Kashiri et al., 2010), essentially due to the higher strength of wheat peel, as shown in the following. Losses in minerals, evaluated in 48 h and by conductivity measurements in swelling bulk, were very high for the treated beans (Figure 3), almost the same for untreated and treated wheat grains.

Penetration tests are usually done on fruits to assess skin strength, flesh texture for ripeness and storage changes (www.zwick.com, www.dastecsrl.com). The same test was done on beans (Figure 5) and weak wheat (Figure 6) in order to assess the possibility of modifications in peel strength and in the internal structure due to the microwave treatment, after swelling and after cooking. The results about swollen beans revealed that the microwave treatment did not cause changes in peel hardness and elasticity (the penetration force and the percentage of strain at the peak are similar for not treated and treated beans, 0.144 kg and 4.46 %; 0.167

kg and 4.58 %, respectively). Also when beans were cooked, the penetration force was similar for the treated and untreated beans, but in the treated beans a larger deformation occurred before peel rupture (peel resistance: 10.81 % vs 6.54 %). After peel rupture, the resistance to penetration in cooked beans increased slowly with the strain % and in the same manner for both treated and untreated beans (the slope is the same) revealing a similar internal structure. In swollen beans, the curve force vs strain after rupture had a higher slope for the untreated systems, confirming a more compact structure.



Figure 3: Swelling ratio (on left) and solution conductivity of swelling bulk (on right) of treated and untreated beans



Figure 5: Penetration tests on cooked and swollen beans untreated and treated by microwave



Figure 4: Swelling ratio (on left) and solution conductivity of swelling bulk (on right) of treated and untreated weak wheat grains



Figure 6: Penetration tests on cooked and swollen weak wheat untreated and treated by microwave

Table 2: Values of penetration force, Kg, and peel resistance to rupture, expressed as the % of strain, for both untreated and treated matrices, after swelling for 20 h and after cooking

|            | Untreated - swollen   |                                 | Treated - swollen     |                                 | Untreated - cooked    |                                 | Treated - cooked      |                                 |
|------------|-----------------------|---------------------------------|-----------------------|---------------------------------|-----------------------|---------------------------------|-----------------------|---------------------------------|
|            | Penetration force, kg | Peel<br>resistance,<br>strain % |
| Beans      | 0.144 ± 0.02          | 4.46 ± 0.32                     | 0.167 ± 0.01          | 4.58 ± 0.39                     | $0.080 \pm 0.00$      | 6.54 ± 0.82                     | 0.082 ± 0.01          | 10.81 ± 0.61                    |
| Weak wheat | 0.136 ± 0.04          | 24.6 ± 5.23                     | 0.111 ± 0.01          | 31.0 ± 1.93                     | 0.115 ± 0.01          | 39.3 ± 1.38                     | 0.114 ± 0.01          | 29.8 ± 2.39                     |

For the weak wheat, the values of penetration force for both swollen and cooked, untreated and treated seeds, were similar. The answer to the effect of only water (swelling) was a larger elasticity for treated weak (31 %); instead the answer to the effect of combined water and heat (cooking) was a larger elasticity for untreated weak (39 %). The slope after peel rupture was the same for all the samples, showing that the microwave treatment had no effect on wheat internal structure.

## 4. Conclusions

In this work the effects of microwave irradiation on two agro-foods (beans and weak wheat) have been quantified. The irradiation has been carried out at a level selected in order to inactivate the infesting fauna, without damaging the foodstuffs. To the purpose of the treatment's assessment, several characteristics of the two foods have been measured on both the untreated and the treated samples: the germination capability, the size and the moisture content, the capability of swelling and the electrolytes' cession rate, the peel resistance and the penetration force. All the samples' features remained essentially unchanged, confirming the feasibility of the treatment and fostering the adoption of the proposed microwave protocol for an effective and safe disinfestation, able to preserve the main physical characteristics.

#### Acknowledgements

This work has been supported by TECNAGRI-PSR Campania 2007-2013 Misura 124. Annalisa Dalmoro's research grant was supported by "POLIFARMA" - PON R&S 2007-13 02\_00029\_3203241.

#### References

- ACIERNO, D., BARBA, A. A. & D'AMORE, M., 2004, Heat transfer phenomena during processing materials with microwave energy, Heat and Mass Transfer, 40, 413-420.
- BARBA, A. A. & D'AMORE, M., 2012, Relevance of dielectric properties in microwave assisted processes, Microwave Materials Characterization, Intech Europe, Rijeka, Croatia.
- BERRIOS, J. D. J., SWANSON, B. G. & CHEONG, W. A., 1999, Physico-chemical characterization of stored black beans (Phaseolus vulgaris L.), Food Research International, 32, 669-676.
- BIENTINESIA, M., PETARCAB, L., CERUTTIC, A., BANDINELLIC, M., DE SIMONID, M., MANOTTID, M. & MADDINELLID, G., 2013, A New Technique for Heavy Oil Recovery Based on Electromagnetic Heating: Pilot Scale Experimental Validation, Chemical Engineering Transactions, 32, 2287-2292.
- CHANDRASEKARAN, S., RAMANATHAN, S. & BASAK, T., 2012, Microwave material processing—a review, AIChE Journal, 58, 330-363.
- FIELDS, P. G., 1992, The control of stored-product insects and mites with extreme temperatures, Journal of Stored Products Research, 28, 89-118.
- KASHIRI, M., KASHANINEJAD, M. & AGHAJANI, N., 2010, Modeling water absorption of sorghum during soaking, Latin American applied research, 40, 383-388.
- LEE, C. M. & CHUNG, K. H., 1989, Analysis of surimi gel properties by compression and penetration tests, Journal of Texture Studies, 20, 363-377.
- LYNG, J. G., ARIMI, J. M., SCULLY, M. & MARRA, F., 2014, The influence of compositional changes in reconstituted potato flakes on thermal and dielectric properties and temperatures following microwave heating, Journal of Food Engineering, 124, 133-142.
- MARRA, F., 2012, Microwave and Radio-Frequency Heating Processes for Food, Handbook of Food Process Design, 1031-1056.
- METAXAS, A. A. & MEREDITH, R. J. 1983. Industrial microwave heating, IET.
- MOHAPATRA, D., KAR, A. & GIRI, S., 2014, Insect Pest Management in Stored Pulses: an Overview, Food and Bioprocess Technology, 1-27.
- PALMA, V., CIAMBELLI, P. & MELONI, E., 2011, Influence of operative parameters on microwave regeneration of catalytic soot wff for diesel engines, Chemical Engineering Transactions, 25, 1001-1006.
- WANG, S., TANG, J., CAVALIERI, R. & DAVIS, D., 2003, Differential heating of insects in dried nuts and fruits associated with radio frequency and microwave treatments, TRANSACTIONS-AMERICAN SOCIETY OF AGRICULTURAL ENGINEERS, 46, 1175-1184.
- YADAV, D. N., ANAND, T., SHARMA, M. & GUPTA, R., 2012, Microwave technology for disinfestation of cereals and pulses: An overview, Journal of Food Science and Technology, 1-9.
- ZHAO, S., QIU, C., XIONG, S. & CHENG, X., 2007, A thermal lethal model of rice weevils subjected to microwave irradiation, Journal of Stored Products Research, 43, 430-434.