



Development of novel and advanced decontamination sustainable technologies for the production of high quality dried herbs and spices

Research for the benefit of specific groups Research for SME associations/groupings

Grant Agreement Number 285838 FP7-SME-2011

Attachment to the final Publishable Summary Report

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1 Quality requirements, product specifications.

Quality requirements have been collected for paprika, pepper and oregano. The objective was to get an overview of actual figures for these quality parameters, but also to make variations and similarities visible between different recommendations, countries and companies. The results are summarized in the following specification sheets.

Table 1. Specification sheet of Paprika.

| | Drodust | | Dar-:1 | ' | | | | | | | | |
|--|-------------|--|-----------------|-----------------|---------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|------------------|
| Specification Sheet | Product | | Papril | ка | | | | | | | | |
| L. Product description | | | | | | | | | | | | |
| Product name | Paprika –C | `apsicur | n annu | um | | | | | | | | |
| Origin | Hungary, S | gary, Spain, South America, United States, Asia | | | | | | | | | | |
| Application | Ingredient | in a bro | oad var | iety of | dishes, | to seas | son and | l color | rice, ste | ws, and | soups, in | the |
| Application | preparatio | n of sau | usages, | as a ga | rnish; | used in | cosme | tics; for | the pr | oduction | of oleon | esin. |
| 2. Quality requirements | | | | | | | | | | | | |
| 2.1 Physico chemical paramete | rs | | | | | | | | | | | |
| Reference | ESA^{1} | | IS | O ² | | | TS | SE ³ | | TF | C ⁴ | JJA ⁵ |
| Category | , | 1 | 11 | III | IV | <i>l</i> * | <i> *</i> | ** | <i> **</i> | * | ** | |
| Moisture (%), | | | | | | | | | | | | |
| by mass, max | 11 | 11 | 11 | 11 | 11 | 11 | 11 | 11 | 11 | 11 | 11 | |
| Water activity (a _w), | | | | | | | | | | | | |
| nax | 0,65 | | | | | | | | | | | 0,59 |
| Volatile oil (ml/100 g), | | | | | | | | | | | | |
| on dry basis, min | | | | | | 0,5 | 0,2 | 0,4 | 0,2 | 0.2 | 0.2 | |
| Fotal ash (%), | | | | | | | | | | | | |
| by mass on dry basis, max | 10 | 8 | 8 | 8,5 | 10 | 6,5 | 8 | 7 | 8 | 8 | 9 | |
| Acid insoluble ash (%), | | | | | | | | | | | | |
| by mass on dry basis, max | 2 | 0,6 | 0,7 | 0,9 | 1 | 0,5 | 1,0 | 1,0 | 1,5 | 1 | 1.5 | |
| Particle/Mesh size (µm) | | | | | | | | | | | | |
| nax | | | | | | | | | | 560 | 560 | 850 |
| Cellulose by mass on dry basis, | | | | | | | | | | | | |
| max (%, m/m) | | | | | | | | | | 25 | 25 | |
| Nonvolatile ether extract (%), | | | | | | | | | | | | |
| on dry basis, mass fraction, | | 17ª | 17 ^a | 20 ^a | 25ª | 12 ^b | |
| 'max value ^a , min value ^b) | | | | | | | | | | | | |
| Capsaicin content (μg/g), | | | | | | | | | | | | |
| пах | | 30 | 30 | 30 | 30 | | | | | | | |
| Cellulose (% m/m), | | | | | | | | | | | | |
| dry matter, max | | | | | | 25 | 25 | 25 | 25 | 25 | 25 | |
| *Sweet, **Hot | | | | | | | | | | | | <u> </u> |
| 2.2 Sensorical parameters | | | | | | | | | | | | - |
| , and a second part of the second | | | | | | | | | | | | |
| Reference | , | 1: | SO ² | | | | | | | | | |
| Appearance | - | | | | | Fine r | owder | | | | | |
| 11 - 1 - 1 - 1 | From vivid | brillian | t red th | nrough | | Reddi | | | | | | |
| Colour | yellowish a | | | | ale | | | | | | | |
| | reddish-br | | | r | | | | | | | | |
| | | | | | | Arom | atic, sli | ghtly si | oicy and | sweet | | |
| Aroma | Pungent o | r free fr | ינום mo | ngencv | | | , | J -1 °I | -, | | | |
| | pleasantly | | | J - · · - 1 | • | | | | | | | |
| Геxture | - | - Fine powder | | | | | , | | | | | |
| | | requirements depending on the product according to ASTA (American Spice Trade Association) | | | | | | | | | | |

| 3. Microbiological limits | | | | |
|---|------------------------|------------------------------|----------------------------|------------------|
| | | Мах соц | ınt (CFU/g) | |
| Reference | DGHM ⁶ | 2004/24/EC ⁷ | R.D.2242/1984 ⁸ | JJA ⁵ |
| Salmonella spp in 25 g | Not detected | Not detected | Not detected | |
| Yeasts and Moulds (all together) | 1,00E+05 | | | 1,00E+003 |
| Enterobacteriaceae | | 1,00E+02 | | 100 |
| E.coli | 1,00E+04 | | 10 | 10 |
| B. cereus | *1,00E+04 | 1,00E+04 | | *100 |
| Clostridium perfringens | | 1,00E+03 | | 100 |
| Clostridium sulf-red anaerobes | | | 1,00E+03 | 100 |
| Spores of sulphite reducing clostridium | 1,00E+04 | | | |
| Total plate count | | | | 1,00E+005 |
| Listeria monocytogenes | No reference limits ac | cording to ASTA ⁹ | · | |
| Staphylococcus aureus | No reference limits ac | cording to ASTA ⁹ | | |
| *Presumptive B.cereus | | | | |

4. Contaminants

4.1 Mycotoxins

| , | | | | | | | |
|--------------------------|-----------|--------------------------------|-------------------------------|-------------------|-------------------|-----------|------------------|
| | Reference | Reg 1881/2006 ¹⁰ | Reg 105/2010 ¹¹ | EC1 ¹² | EC2 ¹³ | TFC⁴ | JJA ⁵ |
| Aflatoxin B ₁ | | 5 ppb | | 5 ppb | | 5 ppb | |
| Aflatoxin Tot | | 10 ppb | | 10 ppb | | 10 ppb | |
| Ochratoxin A | | | 30 μg/kg | | *15 μg/kg | *15 μg/kg | **30 μg/kg |

^{*}From 1.7.2012 (30 μ g/kg from 1.7.2010 until 30.06.2012) **NOTE!** The limit 30 μ g/kg will be extended until 31.12.2014 (publication pending).

^{**}From 1.7.2010 until 31.12.2014 (publication pending)

| Refe | rences | |
|------|---------------|---|
| 1 | ESA | European Spice Association Quality Minima Document. Applies to "buisness to buisness" and not for direct sale |
| 1 | ESA | to consumers |
| 2 | ISO | ISO 7540-2006 Ground paprika (Capsicum annuum L.) –Specification |
| 3 | TSE | Turkish standard TS 2419 –Red pepper (Capsicum annuum L.) –Ground (powdered) |
| 4 | TFC | Turkish Food Codex |
| 5 | JJA | Juan José Albarracín, S.A. (SMEs) |
| 6 | DGHM | Deutsche Gesellschaft für Hygiene und Mikrobiologie (2010). Includes both warning and guideline values for |
| 0 | DGHIVI | products out to consumer. |
| 7 | 2004/24/EC | Commission Recommendation (Dec, 2003). Official control of foodstuffs for 2004. Production and retail |
| | 2004/24/10 | premises. |
| 8 | R.D.2242/1984 | R.D.2242/1984. Condimentos y especias: Reglamentación Técnico-Sanitaria para elaboración, circulación y |
| 0 | N.D.2242/1904 | comercio. (Repealed legislation, only as a technical reference) |
| 9 | ASTA | Guidance from the American Spice Trade Association |
| 10 | Reg 1881/2006 | Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in |
| 10 | Neg 1881/2000 | foodstuffs |
| 11 | Reg 105/2010 | Regulation (EU) No 105/2010 of 5 February 2010 amending Regulation (EC) No 1881/2006 setting maximum |
| 11 | Neg 103/2010 | levels for certain contaminants in foodstuffs as regards ochratoxin A |
| 12 | EC1 | COMMISSION REGULATION (EC) No 1881/2006 |
| 12 | LCI | Setting maximum levels for certain contaminants in foodstuffs |
| | | COMMISSION REGULATION (EC) No 105/2010 |
| 13 | EC2 | Amending Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs as |
| | | regards ochratoxin A |

Table 2. Specification sheet of Oregano.

| | | Table | z. specifi | cation sheet of (| Oregano |) . | | | | | | |
|--|---------------------|---|-------------------------|---|-----------|-------------|----------------------|---------------|------|--|--|--|
| Specification Sheet | P | roduct | | Oregano | | | | | | | | |
| 1. Product description | | | | | | | | | | | | |
| Product name | Mexican Lamiaced | Oregano -Commercially divided into Mediterranean Oregano (e. g. <i>Oregano vulgare</i>) and Mexican Oregano (e. g. <i>Lippiagraveolens, Lippiaberlandiori</i>). Totally 6 families in the market: amiaceae (Turkish and Greek types), <i>Verbenaceae</i> family, <i>Rubiaceae</i> , <i>Scrophulariaceae</i> , <i>Spiaceae</i> and <i>Asceraceae</i> . | | | | | | | | | | |
| Origin | Asia, Unit | ted State | s and Me | | | | | | | | | |
| Application | Used in p | rocessed ods, mea | I foods (e ats and m | ent dishes; toma .g. meat produc eat products, co ts and oils. | ts). Oreg | gano oil is | used in alcoho | lic beverages | , | | | |
| 2. Quality requirements | | | | | | | | | | | | |
| 2.1 Physico chemical paramete | | T | | 2 | | | 9 | 1 1 | | | | |
| Reference | ESA ¹ | | ISC | | | TSE | | AEGEAN⁴ | TFC⁵ | | | |
| | | P* | SP** | Ground (powdered) | Р* | SP** | Ground (powdered) | | | | | |
| Moisture (%), by mass, max | 12 | 12 | 12 | 12 | 12 | 12 | 12 | | 12 | | | |
| Water activity (a _w), max | 0,65 | - | - | | 1 | 1 | | | | | | |
| Volatile oil (ml/100 g), on dry basis, min | 1,5 | 1,8 | 1,5 | 1.5 | 1 | 1 | 1 | | 1 | | | |
| Total ash (%), by mass on dry basis, max | 10 | 10 | 12 | 12 | 12 | 14 | 12 | | 14 | | | |
| Acid insoluble ash (%), by mass on dry basis, max | 2 | 2 | 2 | 2 | 3 | 5 | 3 | | 5 | | | |
| Mesh size (mm) | | | | | | | | 1-4 | | | | |
| Extraneous matter (% m/m), max | | | | | | | | | 2 | | | |
| *Processed, **Semi-processed | | I | | | | | | 1 | | | | |
| 2.2 Sensorical parameters | | | | | | | | | | | | |
| Reference | ISO ² | | | | | | | | | | | |
| Appearance | - | | | | | | | | | | | |
| Colour | Greyish g | Greyish green to olive green for dried leaves | | | | | | | | | | |
| Aroma | Fragrant, | Fragrant, warm, pungent and bitter flavour | | | | | | | | | | |
| Texture | - | | | | | | | | | | | |

| 3. N | Aicrobiological limits | | | | | | | | |
|---|--------------------------|---|-------------------------------|------------------------------------|-----------------------------------|--|--|--|--|
| | Reference | DGF | <i>HM</i> ⁶ | 2004/24/EC ⁷ | TFMC ⁸ | | | | |
| | -3, | Warning | Guideline | , , - | - | | | | |
| Salı | monella spp in 25 g | Not detected | | Absent | Absent | | | | |
| | sts and Moulds | 110t detected | 1 x 10 ³ | 71030110 | 71000110 | | | | |
| | together) | | 1 / 10 | | | | | | |
| | erobacteriaceae | | | < 10 (Satisfactory) | | | | | |
| LIIL | erobacteriaceae | | | $\geq 10^2$ (Unsatisfactory) | | | | | |
| Г - | <u></u> | 1 x 10 ⁴ | 1 x 10 ³ | 2 10 (Ulisatisfactory) | | | | | |
| E.co | DII | 1 X 10 | 1 X 10 | | | | | | |
| В. с | ereus | *1 x 10 ⁴ | *1 x 10 ³ | < 10 ³ (Satisfactory) | < 10 ³ (Satisfactory) | | | | |
| | | | | ≥ 10 ⁴ (Unsatisfactory) | ≥ 10 ⁴ (Unsatisfactory | | | | |
| Clo | stridium perfringens | | | < 10 ² (Satisfactory) | | | | | |
| | | | | ≥ 10 ³ (Unsatisfactory) | | | | | |
| Spc | res of sulphite reducing | 1 x 10 ⁴ | 1 x 10 ³ | | | | | | |
| - | tridium | | | | | | | | |
| Coagulase positive <10 ³ (Satisfactory | | | | | | | | | |
| | ohylococcus | | | | ≥ 10 ⁴ (Unsatisfactory | | | | |
| *Pr | esumptive B.cereus | | | | | | | | |
| 4. 0 | ontaminants | | | | | | | | |
| 4.1 | Mycotoxins | | | | | | | | |
| | Reference | | | | | | | | |
| Afla | itoxin B ₁ | No maximum limit spec | ified for Oregano acc | cording to EC legislation | | | | | |
| | itoxin Tot | No maximum limit spec | | | | | | | |
| | ratoxin A | No maximum limit spec | | | | | | | |
| Ref | erences | | J | 0 0 | | | | | |
| 1 | ESA | European Spice Associatio | n Quality Minima Docu | ment. Applies to "buisness to b | ouisness" and not for | | | | |
| | | direct sale to consumers | · | | | | | | |
| 2 | ISO | ISO 7925: 1999 Dried Oreg | gano (<i>Origanum Vulgar</i> | e L.) –Whole and ground leaves | S –Specification | | | | |
| 3 | TSE | Turkish standard TS 3786: 2003 Tyme, oregano and marjoram | | | | | | | |
| 4 | AEGEAN | Aegean Exporters' Associations | | | | | | | |
| 5 | TFC | Turkish Food Codex | | | | | | | |
| 6 | DGHM | Deutschen Gesellschaft für Hygieneund Mikrobiologie. Includes both warning and guideline values for | | | | | | | |
| | | products out to consumer. | | | | | | | |
| 7 | 2004/24/EC | Commission Recommendation (Dec, 2003) 2004/24/EC. Official control of foodstuffs for 2004. Production | | | | | | | |
| | | and retail premises | | | | | | | |
| 8 | TFMC | Turkish Food Microbiological Codex | | | | | | | |

Table 3. Specification sheet of pepper.

| | | | | i abic 3 | . specii | ication | SHEEL | oi pepi | Jei. | | | | | | | |
|---|---------|------------|-------------|------------------|----------|---------|------------------|---------|-------------|---------|---------|---------|--------|--------|-------|-----------------|
| Specification Sheet | | Produc | ct | Pep | per | | | | | | | | | | | |
| 1. Product description | | | | | | | | | | | | | | | | |
| Product name | Pepper | –Piper n | igrum | | | | | | | | | | | | | |
| Origin | Indone | sia, Vietr | am, Braz | il, Chin | a etc. | | | | | | | | | | | |
| Application | Sold fo | r consum | iers, used | by the | food i | ndustry | as a co | ondime | nt for fo | ood pro | ducts a | nd as a | food p | reserv | ative | |
| 2. Quality requirements | | | | | | | | | | | | | | | | |
| 2.1 Physico chemical paramete | | | | | | | | | | | | | | | | |
| Reference | ES | $5A^1$ | | ISO ² | | | ISO ³ | | | TSE⁴ | | | TSE⁵ | | | SE ⁶ |
| | Black | White | | Black | | | White | | | Black | | | White | | Black | White |
| | | | NP or SP | Р | G | SP | Р | G | NP or SP | Р | G | SP | Р | G | | |
| Moisture (%), by mass, max | 12 | 12 | 13 | 13 | 13 | 14 | 14 | 14 | 13 | 13 | 13 | 14 | 14 | 14 | 12 | 14 |
| Water activity (a _w), max | 0,65 | 0,65 | | | | | | | | | | | | | | |
| Volatile oil (ml/100 g), on dry basis, min | 2 | 1,5 | 2 | 2 | 1* | 1 | 1 | 0.7 | 2 | 2 | 1* | 1 | 1 | 0.7 | 2 | 1 |
| Total ash (%), by mass on dry basis, max | 7 | 3,5 | 7 | 6 | 6 | 3.5 | 3.5 | 3.5 | 7 | 6 | 6 | 2 | 2 | 2 | 8 | 2 |
| Acid insoluble ash (%), by mass on dry basis, max | 1,5 | 0,3 | | | 1.2 | | | 0.3 | | | 1.2 | 0.3 | 0.3 | 0.3 | 1 | 0.3 |
| Non-volatile ether extract % (m/m), min, on dry basis | | | 6 | 6 | 6 | 6.5 | 6.5 | 6.5 | 6 | 6 | 6 | 6.5 | 6.5 | 6.5 | 6 | 6.5 |
| Piperine content % (m/m), min, on dry basis | | | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | | |
| Crude fibre, insoluble index, | | | | | 17.5 | | | | | | 17.5 | | | | 18** | 6** |

| % (m/m), max, on dry basis | | | | | | | | | | |
|-----------------------------|-----|-----|-----|-----|-----|-----|-----|-----|----|-----|
| Extraneous matter, % (m/m), | 2.5 | 1.5 | 1 | 0.8 | 2.5 | 1.5 | 0.5 | 0.8 | 1 | 0.5 |
| max | 2.5 | 1.5 | 1 | 0.0 | 2.5 | 1.5 | 0.5 | 0.0 | 1 | 0.5 |
| Light berries, % (m/m) | 10 | 5 | | | 10 | 5 | | | 10 | |
| max | 10 | 3 | | | 10 | 3 | | | 10 | |
| Pinheads or broken berries, | 7 | 4 | | | 7 | 4 | | | | |
| % (m/m), <i>max</i> | ′ | 4 | | | , | 4 | | | | |
| Broken berries, % (m/m), | | | 4 | 3 | | | 3 | 3 | | 3 |
| max | | | 4 | 3 | | | 3 | 3 | | 3 |
| Black berries, % (m/m), | | | 15 | 10 | | | 15 | 10 | | 10 |
| max | | | 15 | 10 | | | 15 | 10 | | 10 |
| Bulk density, (g/l), | 450 | 490 | 600 | 600 | 450 | 490 | 600 | 600 | | |
| min | 430 | 490 | 000 | 000 | 430 | 490 | 000 | 000 | | |

NP: Unprocessed, SP: Semi-processed, P:Processed, G:Ground

2.2 Sensorical parameters

| Refe | rence McCor | mick ⁷ | |
|------------|-------------------|-------------------|--|
| | Black | White | |
| Appearance | Dried berry | Dried seed | |
| Colour | Brownish black | Light brown | |
| Aroma | Spicy hot, woody, | Spicy hot, musty | |
| | pungent | | |
| Texture | Whole, crushed or | Whole, ground to | |
| | ground to powder | powder | |

^{*}The volatile oil content should be determined immediately after grinding.

^{**}Cellulose, on dry basis, max (%m/m)

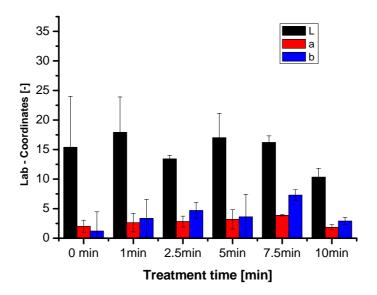
| Reference | DGHN | \mathcal{N}^8 | 2004/24/EC ⁹ | TFMC ¹⁰ |
|---|----------------------|----------------------|--|---|
| | Warning | Guideline | | |
| Salmonella spp in 25 g | Not detected | | Absent | Absent |
| Yeasts and Moulds (all together) | | 1 x 10 ³ | | |
| Enterobacteriaceae | | | < 10 (Satisfactory) ≥ 10² (Unsatisfactory) | |
| E.coli | 1 x 10 ⁴ | 1 x 10 ³ | | |
| B. cereus | *1 x 10 ⁴ | *1 x 10 ³ | < 10 ³ (Satisfactory) ≥ 10 ⁴ (Unsatisfactory) | <10³ (Satisfactory) ≥ 10⁴ (Unsatisfactory) |
| Clostridium perfringens | | | < 10 ² (Satisfactory) ≥ 10 ³ (Unsatisfactory) | |
| Spores of sulphite reducing clostridium | 1 x 10 ⁴ | 1 x 10 ³ | | |
| Coagulase positive Staphylococcus | | | | <10³ (Satisfactory) ≥ 10⁴ (Unsatisfactory) |
| *Presumptive B. cereus | | | | · |
| 4. Contaminants | | | | |
| 4.1 Mycotoxins | | | | |
| Reference | EC1 ¹¹ | | EC2 ¹² | TFC ¹² |
| Aflatoxin B ₁ | 5 ppb | | | 5 ppb |
| Aflatoxin Tot | 10 ppb | | | 10 ppb |
| Ochratoxin A | | | *15 µg/kg | *15 µg/kg |

| Refe | rences | |
|------|------------|---|
| 1 | ESA | European Spice Association Quality Minima Document. Applies to "buisness to buisness" and not for direct sale to consumers |
| 2 | ISO | ISO 959-1:1998 Pepper (Piper nigrum L.), whole or ground-Specification Part 1 Black pepper |
| 3 | ISO | ISO 959-2:1998 Pepper (Piper nigrum L.), whole or ground-Specification Part 2 White pepper |
| 4 | TSE | Turkish standard TS 2290: 2000 Pepper (Piper nigrum L.)- whole or ground-Specification Part 1 Black pepper |
| 5 | TSE | Turkish standard TS 5103: 2003 Pepper (Piper nigrum L.)- whole or ground-Specification Part 1 Black pepper |
| 6 | TFC | Turkish Food Codex |
| 7 | McCormick | US company that manufactures spices, herbs and flavouring for retail, commercial and industrial markets. |
| 8 | DGHM | Deutschen Gesellschaft für Hygiene und Mikrobiologie (2010). Includes both warning and guideline values for products out to consumer. |
| 9 | 2004/24/EC | Commission Recommendation (Dec, 2003) 2004/24/EC. Official control of foodstuffs for 2004. Production and retail premises |
| 10 | TFMC | Turkish Food Microbiological Codex |
| 11 | EC1 | COMMISSION REGULATION (EC) No 1881/2006 |
| 11 | ECI | Setting maximum levels for certain contaminants in foodstuffs |
| 12 | EC2 | COMMISSION REGULATION (EC) No 105/2010 |
| 12 | LCZ | Amending Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs as regards ochratoxin A |



2 Prototype samples obtained during the project.

Here are presented some prototype samples obtained during the development of the project.



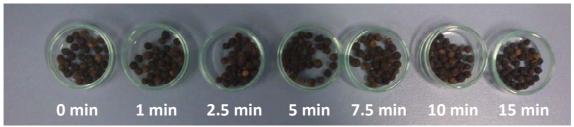


Figure 1. Impact of a direct plasma exposure with a plasma jet on the product colour of black pepper.



Figure 2. 3g of oregano treated for 10 min in the DBD plasma system.

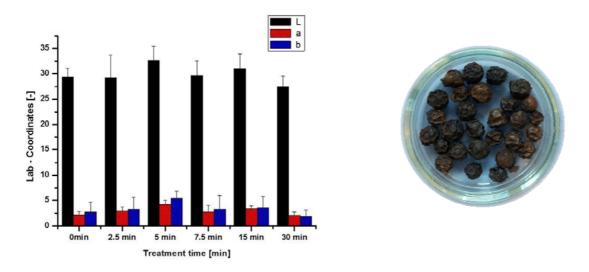


Figure 3. Impact of a direct DBD plasma exposure on the product colour of black pepper. The sample displayed, was treated for 30min.

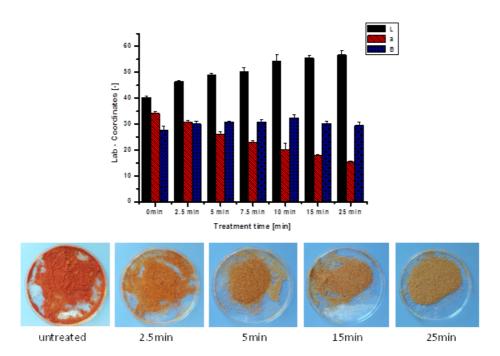


Figure 4. Impact of a direct DBD plasma exposure on the product colour of paprika powder.

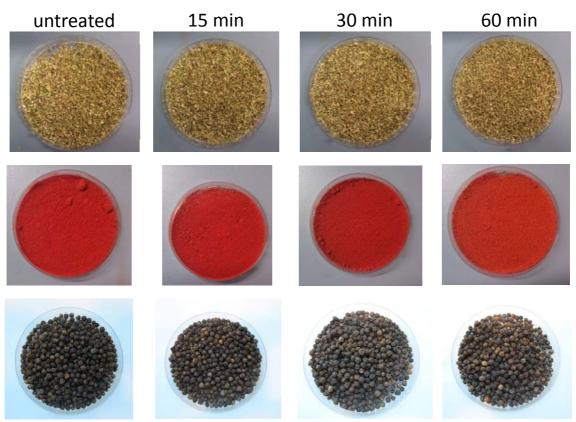


Figure 5. Oregano, paprika and black pepper seeds after various exposure times to indirect plasma.

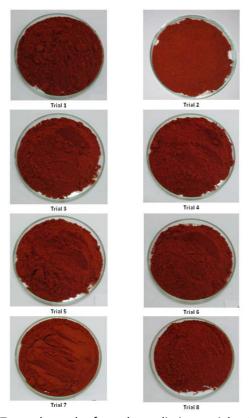


Figure 6.Treated samples from the preliminary trials with HPCD.

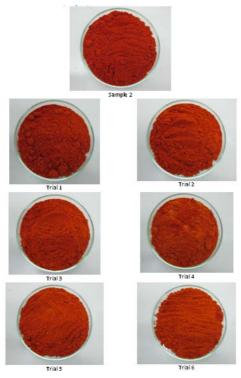


Figure 7. Treated samples from the trials performed with sample 2 with HPCD+US.

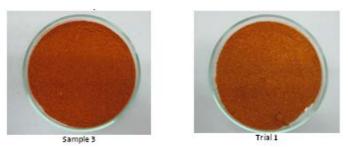


Figure 8. Sample 3 before and after treatment with HPCD+US.

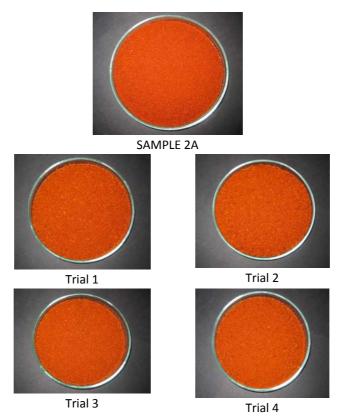


Figure 9. Sample 2A before and after treatment with HPCD+US.



Figure 10. Sample 2B before and after treatment with HPCD+US.

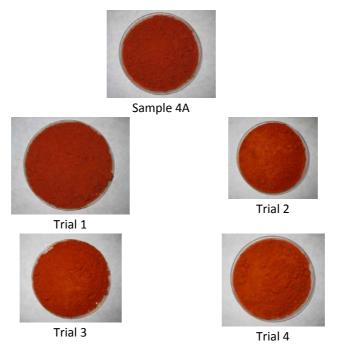


Figure 11. Sample 4A before and after treatment with HPCD+US.

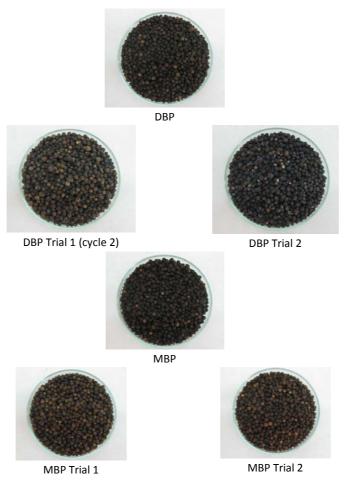


Figure 12. Samples of moistened and dry pepper before and after treatment with HPCD+US.

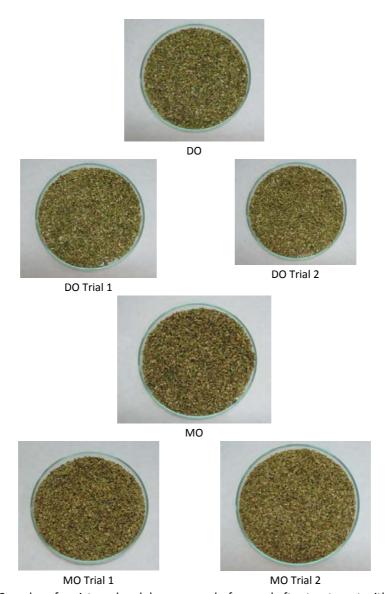


Figure 13. Samples of moistened and dry oregano before and after treatment with HPCD+US.

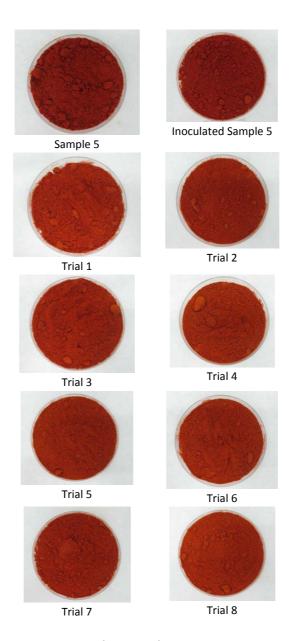


Figure 14. Sample 5 before and after treatment with HPCD+US.

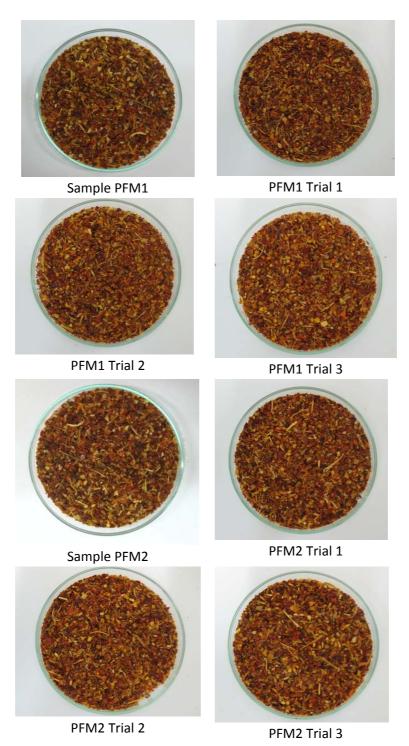


Figure 15. Paprika flakes before and after treatment with HPCD+US.

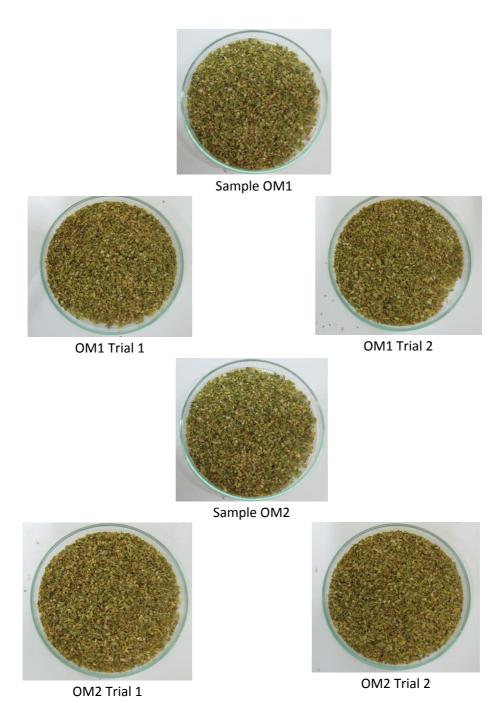


Figure 16. Moistened oregano before and after treatment with HPCD+US.

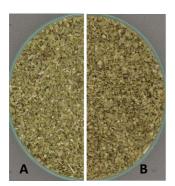


Figure 17. A) Untreated raw material vs. B) IR treated oregano at 90°C for 10 min followed by final drying.

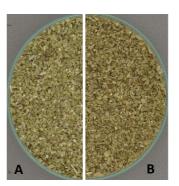


Figure 18. A) Untreated raw material vs. B) IR treated oreagano at 100°C for 10 min followed by final drying.

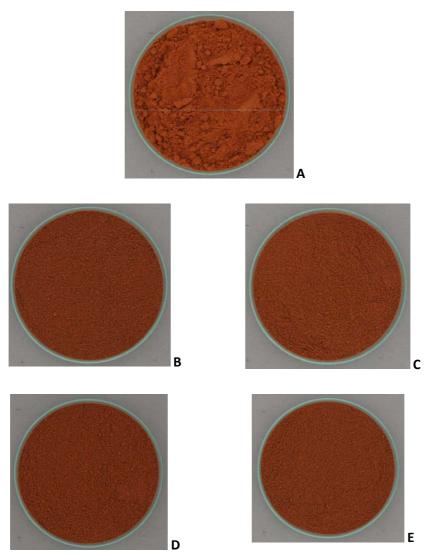


Figure 19. A) Untreated raw material, B) IR treated paprika for 10 min 98 $^{\circ}$ C, C) IR treated paprika for 20 min 98 $^{\circ}$ C, D) MW treated paprika for 10 min 98 $^{\circ}$ C and E) MW treated paprika for 20 min 98 $^{\circ}$ C. B) – E) have been subjected to a final drying step after the IR/MW treatment.



IR 98°C 10 min Dried to aw 0.28 Δ E 9.2



IR 98°C 10 min Dried to aw 0.54 ΔE 15.71

Figure 20. Example of two IR treated samples at 98° C for 10 min followed by drying to two different water activities (0.28 vs. 0.54). The sample with the higher water activity appeared to have a darker colour and showed clearly a higher Δ E compared to the undtreated raw material.

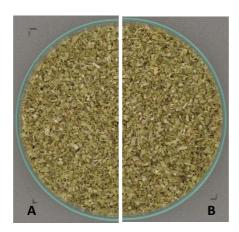


Figure 21. A) Untreated raw material, compared to B) the most affected IR treated sample (sample 6a) in terms of total colour change and water activity.

3 Cold atmospheric plasma - summary of product-process interactions

Paprika powder

Characterization of the raw material

| Natural flora | TPC | 1.1 x 10 ⁷ |
|----------------------|---------------------------|-----------------------|
| [CFU/g] | Aerobic mesophilic spores | 5.6 x 10 ⁶ |
| | Yeasts and Molds | 2.4×10^4 |
| Color / | L | 31.76 ± 1.346 |
| CIELab [-] | a | 23.16 ± 2.6 |
| | b | 53.7 ± 2.0 |
| Moisture content [%] | 6.2 ±0.2 | |

Sample properties after plasma treatment

| Treatment | Microorganism | Microbial | | Col | or | |
|------------------------------|--------------------------------------|--|------|-------|-------|------|
| conditions | | Reduction [log (N/N ₀)] | L | а | b | ΔΕ |
| DBD- System (25 min) | TPC | -0.43 ± 0.2 | 56.7 | 15.31 | 29.41 | 35.7 |
| PLEXc- system (60 min) | TPC Aerobic spores Yeasts and moulds | -3.1 ± 0.2 -1.3 ± 02 Below detection limit | 26.6 | 20.16 | 45.1 | 10.5 |

Oregano

Characterization of the raw material

| Natural flora | TPC | 1.7 x 10 ⁵ |
|----------------------|---------------------------|-----------------------|
| [CFU/g] | Aerobic mesophilic spores | 3.1×10^3 |
| | Yeasts and Molds | 9.7 x 10 ³ |
| Color / | L | 31.74 ± 3.15 |
| CIELab [-] | a | 4.25 ± 2.52 |
| [-] | b | 43.04 ± 6.85 |
| Moisture content [%] | 11.5 ± 0.6 | |

Sample properties after plasma treatment

| Microorganism | Microbial | | Co | lor | |
|----------------|--|--|---|---|---|
| | Reduction [log (N/N ₀)] | L | а | b | ΔΕ |
| TPC | -1.89 ± 0.46 | 29.47 | 4.72 | 27.52 | 15.7 |
| Aerobic spores | -0.59 ± 0.17 | | | | |
| TPC | -1.2 ± 0.1 | 33.59 | 5.63 | 29.1 | 14.1 |
| Aerobic spores | -1.3 ± 0.3 | | | | |
| Yeasts and | -1.0 ± 0.4 | | | | |
| | TPC Aerobic spores TPC Aerobic spores | $ \begin{array}{c} \text{Reduction} \\ [\log{(\text{N/N}_0)}] \\ \\ \text{TPC} \\ \text{Aerobic spores} \\ \end{array} \begin{array}{c} -1.89 \pm 0.46 \\ -0.59 \pm 0.17 \\ \\ \\ \text{TPC} \\ \text{Aerobic spores} \\ \text{Aerobic spores} \\ \text{Yeasts and} \\ \end{array} \begin{array}{c} -1.2 \pm 0.1 \\ -1.3 \pm 0.3 \\ -1.0 \pm 0.4 \\ \\ \end{array} $ | Reduction [log (N/N ₀)] L TPC -1.89 ± 0.46 29.47 Aerobic spores -0.59 ± 0.17 33.59 Aerobic spores Yeasts and -1.3 ± 0.3 $+0.3$ $+0.4$ | $ \begin{array}{cccccccccccccccccccccccccccccccccccc$ | $ \begin{array}{cccccccccccccccccccccccccccccccccccc$ |

Whole Black pepper

Characterization of the raw material

| Natural flora | TPC | 1.0 x 10 ⁸ |
|--------------------------|---------------------------|-----------------------|
| [CFU/g] | Aerobic mesophilic spores | 5.6 x 10 ⁷ |
| | Yeasts and Molds | 6.0×10^3 |
| Color / | L | 30.74 ± 1.7 |
| CIELab [-] | а | 1.78 ± 0.7 |
| 1.1 | b | 0.93 ± 1.9 |
| Moisture content [%] | 10.4 ± 0.4 | |
| Piperine [g/L] | $0.47 \pm 8.1^*10^5$ | |
| Essential oils [mL/100g] | 2.9 ± 0.2 | |

Sample properties after plasma treatment

| Treatment | Microorganism | Microbial | | Col | or | | Piperine | Essential oils |
|--------------------|------------------------------|---------------------------|------|------|------|-----|----------------------|----------------|
| conditions | | Reduction [log (N/N₀)] | L | а | b | ΔΕ | [g/L] | [mL/100g] |
| Plasma jet | TPC | | 28.5 | 2.75 | 1.85 | 2.6 | 0.44 ± | 2.7 ± 0.2 |
| (15 min) | Aerobic spores | | | | | | 1.5*10 ⁻³ | |
| | E. coli | -4.11 ± 0.13 | | | | | | |
| | Salmonella enterica | -2.93 ± 0.33 | | | | | | |
| | B. subtilis spores | | | | | | | |
| | B. atrophaeus (dry) | | | | | | | |
| | B. atrophaeus (moistened) | | | | | | | |
| DBD- | TPC | -1.3 ± 0.8 | 27.4 | 2.2 | 2.1 | 3.5 | - | - |
| System (10 min) | Aerobic spores | -0.72 ± 0.4 | | | | | | |

| Treatment | Microorganism | Microbial | | Col | or | | Piperine | Essential oils |
|--------------------|----------------------------------|-----------------------------|-------|------|------|-----|------------------------|----------------|
| conditions | | Reduction | L | а | b | ΔΕ | [g/L] | [mL/100g] |
| | | [log (N/N ₀)] | | | | | | |
| PLEXc- | TPC (dry) | -3.2 ± 0.1 | 27.39 | 2.2 | 2.0 | 3.5 | $0.44 \pm 0.44 = 0.44$ | 2.8 ± 0.2 |
| system (30 min) | TPC (moistened) | -3.44 ± 0.6 | | | | | 1.2*10 ⁻³ | |
| (/ | Aerobic spores (dry) | -2.83 ± 0.7 | | | | | | |
| | Aerobic spores (moistened) | -3.3 ±0.7 | | | | | | |
| | B. subtilis spores (dry) | -2.15 ±0.2 | | | | | | |
| | B. subtilis spores (moistened) | -2.59 ± 0.3 | | | | | | |
| | B. atrophaeus spores (dry) | -2.88 ± 0.1 | | | | | | |
| | B. atrophaeus spores (moistened) | -2.1 ± 0.7 | | | | | | |
| PLEXc- | TPC | 4.10 ± 0.1 | 29.15 | 2.27 | 1.11 | 1.6 | 0.45 ± | 2.8 ± 0.2 |
| system (60 min) | Aerobic spores | 3.16 ± 0.3 | | | | | 1.4*10 ⁻³ | |
| (oo miii) | Yeast and Moulds | Below detection limit | | | | | | |
| | Salmonella enterica | -4.10 ± 0.87 | | | | | | |

4 Ultra sound assisted high pressure carbon dioxide treatment - summary of product-process interactions

| Groups of trials | Work conditions | Microbial Results | Other quality parameters. |
|----------------------|---|---|--|
| 1 PAPRIKA (SAMPLE 1) | 100, 200, 250, 300 bar 60/120 min | No significant reductions of total aerobic mesophilic cells and yeast and moulds. | Slight reductions in the moisture content. Colour: |
| PAPNIKA (SAIVIPLE 1) | 40/50°C | and yeast and modius. | ASTA: No significant |
| | 180 g of <u>paprika</u> | Slight reductions with longer | changes |
| | Flow of CO ₂ was 10 kg/h | treatment times (120 min) and high pressures (250-300 bar) | CieLab: Slight differences (ΔE<5) |
| | No ultrasounds | • | |
| 2 | 250/300 bar 60/120 min | Slight reductions (max 1log ₁₀) of total viable counts, yeasts | Reductions of the moisture content |
| PAPRIKA (SAMPLE 2) | 40/50°C | and moulds and sulfite | Colour: Slight |
| naturally | 230 g of <u>paprika</u> | reducing anaerobes. | differences, $(5<\Delta E<6)$ |
| contaminated with | Flow of CO ₂ : 10kg/h | | |
| Salmonella | Power of | Inactivation of Salmonella at | |
| | ultrasounds: 75V | 250 bar, 50ºC and 60 min | |
| 3 | 300 bar | None significant reductions | No mycotoxins |
| | 120 min | of yeast and moulds, total | reduction. |
| CHILLIE (SAMPLE 3) | 70ºC | viable counts and sulfite- | |
| | 230 g of <u>chillie</u> | reducing anaerobes. | Reduction of the |
| | Flow of CO ₂ : 10kg/h | | moisture content from |
| | Power of | | 4,7 % to 2,6% (Higher |
| | ultrasounds: 75V | | processing temperature 70°C). |
| | Analysis of | | 1 |
| | micotoxins | | No significant |
| | | | differences of colour. |

| Groups of trials | Work conditions | Microbial Results | Other quality parameters. |
|--|--|--|--|
| PAPRIKA (SAMPLE 2), naturally contaminated with Salmonella | 250bar 60/90min 50/70ºC 180g of <u>paprika</u> (stored 24-48h in refrigeration after wetting) Flow of CO₂: 10kg/h | Slight destruction of the total aerobes (0.5-0.6 log ₁₀ of reduction), but none effectiveness against spores. Total destruction of moulds | Decrease in the moisture content of up to 6,1% and in the aw of up to 0,12, higher at higher temperature and longer treatment times—samples closer to their features for |
| Moistened up to 19-20% | Power of ultrasounds: 75V | and yeasts (4 log ₁₀) in 3 treatments. | commercial use. Slight differences of colour |
| The longer storage time before | | In all trials Salmonella was eliminated. | $(\Delta E=5,52-7,21)$. |
| treatment and after wetting don't have an impact on the microbial reductions. | 250bar 60-90min 70ºC 180g of paprika (stored 7 days in refrigeration after wetting) Flow of CO ₂ : 10kg/h Power of ultrasounds: 75V | None effectiveness against spores of aerobes and a very low reduction of total viable counts (<0.5 log ₁₀) was achieved. Total destruction of moulds and yeasts (>3 log ₁₀) in all treatments | and no reductions of mycotoxins were observed. |
| | | In all trials Salmonella was eliminated. | |
| 5 PAPRIKA (SAMPLE | 250/300bar 30-60min 50ºC | Reduction of spores < 0.5log ₁₀ | |
| Inoculated with <i>E.coli</i> . 24 h before treatment Final approximated amount of 10 ⁴ of | 180g of paprika Moisture content of the inoculated material: 4,4% Flow of CO ₂ : 10kg/h Power of ultrasounds: 75V | No treatment reaches a total decontamination of the <i>E. coli</i> . Around 50% of reduction. | No significant changes of colour in 4A Δ E=4,38-5,31 and moderated changes of colour in 4B Δ E=4,54-6,40. |
| E.coli | 250/300bar 30-60min 50ºC | Reduction of spores < 0.5log ₁₀ | moisture content and a_w at longer treatment times (60 min) and higher |
| | 180g of paprika Moisture content of the inoculated material: 7,4% Flow of CO ₂ : 10kg/h Power of ultrasounds: 75V | Total destruction of <i>E. coli</i> in all treatments. Higher reduction of the microbial loads linked to the higher moisture content of the samples | pressures $(300 \text{ bar}) \rightarrow$ decrease in the moisture content of up to 2,1% and decrease of the a_w of up to 0,1. |

| Groups of trials | Work conditions | Microbial Results | Other quality parameters. |
|--|---|---|---|
| 6 PAPRIKA (SAMPLE | 150/250 bar 20/60 min 40/60°C | No effect on the spores content. | A decrease in the moisture content and the a _w was observed in all trials up to |
| 5) Inoculated with | 180g of <u>paprika</u> Moisture content of the inoculated | Reduction of <i>Enterococcus</i> faecalis up to 4 log ₁₀ . | features of commercial use. |
| Enterococcus faecalis | material: 7,1% Flow of CO ₂ : 10kg/h Power of | Total inactivation of moulds and yeasts (3 log ₁₀) | The higher differences have been found in trial 8 (60min; 250bar and 60°C). |
| Final concentration of 10 ⁷ cells/g of <i>E. faecalis</i> paprika | ultrasounds: 75V | | In all trials Δ E<5, except for trials 6 and 8 were Δ E is 5,39 and 5,07 respectively |
| | | | Slight reduction in the capsaicinoids concentration with increasing pressure of treatment (250 bar) |
| 9 Paprika flakes | 150bar 15, 30 and 60 min 80ºC | Nearly 1 log ₁₀ of aerobes reduction is observed after 60 min. The most moistened | Higher reduction of the moisture content and a _w at longer treatment times |
| Inoculated with | 180g of <u>paprika</u> Moisture content of | samples achieved 0,5 log ₁₀ of spores reduction after 15 | (60 min). Moisture and aw closer to features of |
| Enterococus faecium | the inoculated material: 13,30 and | min. | commercial use were achieved. |
| | 14%. | Total inactivation of natural | |
| Final concentration of 10 ⁶ CFU/g | a _w of the inoculated material: 0,67 and 0,73. Flow of CO ₂ : 10kg/h | content of mould and yeasts (4,5 log ₁₀ after 30 min). | No significant changes of colour in M1 (Δ E=0,90-2,42) and slight differences of colour in |
| | Power of ultrasounds: 75V | Total inactivation of natural content of Enterobacteriaceae (4log ₁₀ | M2 (ΔE=3,36-7,80). |
| | | of reduction) and of the inoculated <i>Enterococcus</i> faecium (5log ₁₀ of reduction) after 15 min. | |

| Groups of trials | Work conditions | Microbial Results | Other quality parameters. |
|---|--|--|--|
| 7 | 2 cycles trials | No effect of the moisture | No significant changes of |
| Black pepper | (treatment 1): | content on the spores | colour have been found |
| | 250bar | decontamination. | after treatment, in all |
| Application of | 30 min | | cases ΔE <5. |
| consecutive cycles | 70ºC | Moistened samples | |
| Effect of increased moisture content (from 10% to 20%) | 1cycle trials(treatment 2): 250bar 60 min 50°C | achieved the total inactivation of natural content of mould and yeasts (nearly 5 log ₁₀ of reduction) under treatment 1 and 2. | A decrease in the moisture content and the a_w was observed. up to features of commercial use. |
| | Flow of CO ₂ : 10kg/h Power of ultrasounds: 75V | | After two cycles the reduction of the moisture content and the a_w was higher. |
| 8 (with oregano) | 250 bar | No effect on the spores | No significant changes of |
| | 60 min 50/70ºC | decontamination. | colour have been found (ΔE <5) |
| Effect of increased | 180 g of <u>oregano</u> | Moistened samples | (== 5) |
| moisture content (from 6% to 19,5%) | Flow of CO ₂ : 10kg/h Power of ultrasounds: 75V | achieved the total inactivation of natural content of mould and yeasts (nearly 4,5 log ₁₀ of reduction) at 70°C, 60min and 250 bar. | A decrease in the moisture content and the a_w was observed |
| 10 Oregano | 150bar | Nearly 2 log ₁₀ of aerobes | Higher reduction of the |
| | 15/30 min | reduction was achieved | moisture content and $\boldsymbol{a}_{\boldsymbol{w}}$ |
| Inoculated with | 80ºC | after 15 min and 1 log ₁₀ | at longer treatment times |
| Enterococus | 120g of <u>oregano</u> | reduction of spores after | (60 min): |
| faecium | Moisture content of | 30 min. | Naciativia and a place to |
| Final | the inoculated material: 12,7 and | Total inactivation of | Moisture and a _w closer to features of commercial use |
| concentration of 10 ⁶ CFU/g | 14,5%. a _w of the inoculated | natural content of mould and yeasts (> 3 log ₁₀) abd | were achieved. |
| , | material: 0,66 and | of the inoculated | In all cases, Δ <5 \rightarrow no |
| Two different moisture levels were studied (M1 and M2) | 0,74. Flow of CO ₂ : 10kg/h Power of ultrasounds: 75V | Enterococcus faecium (5log ₁₀ of reduction) after 15 min. | influence in the colour of the samples. |

5 Infrared and microwave heating - summary of product-process interactions

Characterization of the raw material

Oregano

The results below are performed on the batch delivered to SIK in April 2013. The microbial characterization is a mean value of four replicates. More details are found in D2.1 C.

| Natural flora | TPC | 2.9 x 10 ⁴ |
|--|---|-----------------------|
| [CFU/g] | Bacterial spores | 8.4×10^3 |
| | Yeasts and Molds | 1.6 x 10 ⁴ |
| Color / | L | 50.6 ± 1.75 |
| CIELab | a | 2.7 ± 0.14 |
| | b | 25.3 ±0.54 |
| Moisture content [%] | 10.3 ± 0.40 | |
| Water activity | 0.61 ±0.01 | |
| Essential oils [%] | 2.8 | |
| Head space volatile compound | Composition (% fraction of total | ıl FID area) |
| lpha-Phellandrene | 1.9 | |
| α-Pinene | 1.3 | |
| Camphene | | |
| Campricine | 0.6 | |
| α-Myrecene | 0.6 3.1 | |
| • | | |
| α-Myrecene | 3.1 | |
| α-Myrecene α-Terpinen | 3.1 1.2 | |
| α-Myrecene α-Terpinen o-Cymene | 3.1 1.2 7.0 | |
| α-Myrecene α-Terpinen o-Cymene γ-Terpinene | 3.1 1.2 7.0 5.2 | |
| α-Myrecene α-Terpinen o-Cymene γ-Terpinene Terpinolene | 3.1 1.2 7.0 5.2 2.0 | |
| α-Myrecene α-Terpinen o-Cymene γ-Terpinene Terpinolene α-Linalool | 3.1 1.2 7.0 5.2 2.0 11.1 | |
| α-Myrecene α-Terpinen ο-Cymene γ-Terpinene Terpinolene α-Linalool Isoborneol | 3.1 1.2 7.0 5.2 2.0 11.1 1.4 | |
| α-Myrecene α-Terpinen ο-Cymene γ-Terpinene Terpinolene α-Linalool Isoborneol Thymol | 3.1 1.2 7.0 5.2 2.0 11.1 1.4 | |
| α-Myrecene α-Terpinen ο-Cymene γ-Terpinene Terpinolene α-Linalool Isoborneol Thymol Carvacrol | 3.1 1.2 7.0 5.2 2.0 11.1 1.4 0.4 39.2 | |

Paprika

The results below are performed on the paprika batch delivered to SIK in October 2013. The microbial characterization is a mean value of five samples.

| Natural flora | TPC | 7.2 x 10 ⁷ |
|----------------------|------------------|-----------------------|
| [CFU/g] | Bacterial spores | 5.6 x 10 ⁶ |
| Color / | L | 34.9 ±1.01 |
| CIELab | a | 36.8 ±0.18 |
| | b | 38.2 ±0.24 |
| Moisture content [%] | 5.6 ±0.09 | |
| Water activity | 0.38 ± 0.00 | |

Pepper

The results below are performed on the batch delivered to SIK in May 2013. The microbial characterization is a mean value of duplicates.

| Natural flora | TPC | 2.4 x 10 ⁷ |
|----------------------|------------------|-----------------------|
| [CFU/g] | Bacterial spores | 1.5 x 10 ⁷ |
| Color / | L | 20.5 ± 0.51 |
| CIELab | а | 1.85 ± 0.07 |
| | b | 4.80 ± 0.21 |
| Moisture content [%] | 7.4 ± 0.1 | |
| Water activity | 0.49 ± 0.00 | |

Summary of the sample properties after IR and MW treatment

Infrared treatment of oregano

| Conditions | Microorganism | Microbial Reduction [log CFU/g] | | Water activity | | Colour | | Volatile oil | GC- Head Space | |
|------------------------|------------------------------|---------------------------------|--------|----------------|-----------------|--------|------|--------------|---|--|
| | | | | After IR | After Drying | a* | ΔΕ | content (%) | Volatile compounds | |
| 90°C 2 min | Inoculated B. cereus spores | 2.71 ± 0.54 | | 0.87 | 0.65 | 4.99 | 1.91 | 2.4 | _ The total | |
| 90°C 10 min | | 5.55 ± 0.30 | | 0.87 | 0.63 | 5.43 | 2.47 | 2.4 | composition was - altered, but the | |
| 100°C 2 min | _ | 2.43 ± 0.27 | | 0.83 | 0.65 | 5.34 | 2.46 | 2.0 | key aroma | |
| 100°C 10 min | _ | 4.73 ± 0.18 | | 0.76 | 0.59 | 5.98 | 3.15 | 2.0 | compounds (carvacrol and thymol) were not | |
| 90°C 2 min 90°C 10 min | Natural flora | TPC | > 2.26 | | | | | | affected to any high extent (See | |
| | | Bact. Spores | > 1.15 | _ | | | | | D2.1C) | |
| | | Moulds | > 2.20 | _ | | | | | _ | |
| | | TPC | 2.26 | | | | | | | |
| | | Bact. Spores | 0.85 | _ | | | | | | |
| | | Moulds | > 2.20 | _ | | | | | | |

Infrared treatment of paprika and pepper

| | Conditions | Microorganism | Microbial Reduction | Water | activity | Colour | |
|---------|-------------|---------------|---|----------|-----------------|--------|------|
| Spice | | | [log CFU/g] | After IR | After drying | ΔΕ** | ΔΕ |
| Paprika | 90°C 2 min | Natural Flora | 0.16 ± 0.19 (TPC) 0.22 ± 0.21 (Bact. spores) | 0.88 | | 3.72 | |
| | 90°C 10 min | Natural Flora | 0.48 ± 0.02 (TPC) 0.48 (Bact. spores) | | | 3.82 | 7.7 |
| | 98°C 10 min | Natural Flora | 1.76 ± 0.12 (TPC) 1.62 ± 0.01 (Bact. spores) | 0.87 | 0.28 | | 9.2 |
| | 98°C 20 min | Natural Flora | 3.66 ± 0.14 (TPC) 2.28 ± 0.02 (Bact. spores) | 0.87 | 0.28 | | 11.7 |
| Pepper | 90°C 2 min | Natural Flora | 0.65 ± 0.07 (TPC) 0.55 ± 0.16 (Bact. spores) | 0.87 | | 1.39 | |
| | 90°C 10 min | Natural Flora | 0.38 ± 0.09 (TPC) 0.49 ± 0.10 (Bact. spores) | 0.86 | | 2.82 | 4.2 |

^{**} Compared to conditioned raw material

Microwave treatment of oregano and paprika

| Herb/Spice | Conditions | Microorganism | Microbial Reduction [log CFU/g] | Water activity | | | GC- Head Space Volatile |
|------------|-------------|-----------------------------------|---|----------------|-----------------|------|---|
| | | | | After MW | After drying | ΔΕ | compounds |
| Oregano | 90°C 2 min | Inoculated <i>B.cereus</i> spores | 0.83 ± 0.51 | 0.88 | | | The total composition was altered, but the key aroma compounds (carvacrol and |
| | 90°C 10 min | Inoculated <i>B.cereus</i> spores | 1.76 ± 0.12 | 0.86 | 0.62 | 2.9 | thymols) were not affected to any high extent (See D2.1C). |
| Paprika | 98°C 10 min | Natural Flora | 2.43 ± 0.57 (TPC) 2.23 ± 0.17 (Bact. spores) | 0.84 | 0.38 | 8.1 | |
| | 98°C 20 min | Natural Flora | 4.79 ± 0.00 (TPC) 3.25 ± 0.09 (Bact. spores) | 0.83 | 0.38 | 11.0 | |