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**Réf :....** 

Mémoire de Fin de Cycle En vue de l'obtention du diplôme

### MASTER

### Thème

## Valorisation de la figue de barbarie: séchage, formulation d'un yaourt et d'une boisson lactée

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#### List of abbreviation

OFI: Opuntia ficus indica TFC: Total Flavonoid compounds **TPC:** Total Phenolic Compounds UAE: Ultrasound assisted extraction **CV:** Conventional Extraction CM: Conventional Maceration CS : conventional Stirring MAE: Microwave Assisted Extraction MC: The moisture content MW: Microwave MWD: Microwave Drying °C: Celcus degrees °Bx : Degre brix DM: Dry Matter DPPH: 2, 2-Diphenyl-1-picrylhydrazyl GAE: Gallic Acid Equivalent CP: Centi poiseuille AW: Activity water RC: Reducing power RSA: Radical Scavenging Activity

3 BS: Biomathematics, Biochemistry, Biophysics and Scientometrics

### Summary

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Appendix

## Introduction

#### Introduction

The prickly pear cactus originates from Mexico, still witnessed the impressive genetic diversity of 400 species and a great number of varieties. The cactus pear due its agroecologic requirement has been for long time an important crop for the semi arid and arid zones of the planet, because of its special adaptive mechanism and capacity to produce biomass. For this reason, a greater interest has been observed in the last years for increasing their cultivation and diverse research has been carried out to enlarge the industrialization of fruit and the cladodes (**El Gharras, Hasib et** *al.* **2008**).

*Opuntia ficus indica* (OFI) is one of the widely cultivated species of the genus Opuntia in North Africa that is used for human consumption and animal food (Lefsih, Giacomazza et *al.* 2017). The Algerian cactus plantation is about 28,000 ha in 2015. Studies have found that one hectare of Opuntia above the age of five years is able to produce  $\approx 100$  t of fresh fruits each year in areas with little rainfall (150 mm or less) (Felkai-Haddache, Remini et *al.* 2016).

The first economic importance of this plant relies on the production of edible fruits. It is mainly consumed fresh or converted into drinks (nectars, juice), jams or marmelades. This food transformation generates a large amount of by-products (seeds and peels). Peels represent a large proportion of the whole fruit (from 40% to 50%) and constitute a source of bioactive compounds, notably phenolics, flavonoids and betalains. (Abou-Elella and Ali 2014). In a context of resource preservation, the determination of bioactive compounds from juicy pulp and by-products together with their re-valorization in the food, cosmetic or pharmaceutical industry give rise to an increasing social, economic and scientific interest.

In order to contribute to the valorization of this cultivation and to overcome the lack of knowledge about all aspects concerning this fruit in Algeria, current study has been designed to investigate indigenous OFI local variety, for their physico-chemical composition and their antioxidant activities, but also to assess drying effect on peels.

Drying is among the methods for the purpose to produce high quality dried products, which can be consumed directly or used as ingredient. Conventional air-drying has been widely used in industrial drying of food products, but this method is energy-intensive and time-consuming and often produces poor quality products. Hence, advanced drying methods is often recommended to reduce long drying times and poor product quality, namely Microwave drying (**Aydogdu, Sumnu et** *al.* **2015**). To the best of our knowledge no past research was conducted to investigate the drying kinetics of cactus pear peels. In the view of health promoting properties and high nutritional benefits of OFI peel, the present study was carried out to compare the effects of conventional oven drying and microwave drying on bioactive components and antioxidant activities.

Dairy products with added antioxidants from natural sources appear to be a convenient food format, to satisfy consumer interest with original beneficial effects, and health benefits of added antioxidants; creation of new high value-added products. Thus, a formulation of yoghurt with juicy pulp and milky juice with phenolic extract peels, at laboratory scale, was performed.

# Bibliography

#### I.1. Overview of prickly pear

Prickly pear: *Opuntia ficus indica* (OFI), is a cactus well adapted to arid and semiarid conditions. The central part of Mexico hosts the greatest diversity of this cactus in the world. This cactus produces an edible prickly pear that is consumed as a fresh fruit (**Jorge, De La Garza et al. 2013**). It was introduced into North Africa in the 16<sup>th</sup> century, and more than 1500 known species of cactus are in the genus *Opuntia* (**Salim, Abdelwaheb et al. 2009**).

In Algeria, Chile, Mexico and Brazil, large areas are used for the cultivation of cactus. It is regarded as an alternative or backup feedstock in periods of drought as cacti remain succulent and fresh for longer periods (Isaac 2016).

#### Vernacular names

- English : Prickly pear
- French : Figue de barbarie
- Arabic : Al-sebbar , Al-karmous
- Kabyle : Akermus, akermust

#### I.1.1. Morphological description

Cactus is an arborescent plant that can reach 3 to 5 meters of top her organization in cladodes, fluently called "rackets", is particular (**Fig.1**). The cladodes is stems modified of flattened shape, of 30 to 40-cm long out of 15 to 25-cm large and 1,5 to 3 cm thick (**Barbera**, **Carimi et al. 1992**). The fruit is a berry, varying in shape, size and colour and has a consistent number of hard seeds (**Drouet 2015**).

Kingdom: Plantae Under-kingdom: Tracheobionta Division: Magnoliophyta Class: Magnoliopsida Under-class: Caryophyllidae Order: Caryophyllales Family: Cactaceae Genus: Opuntia Species: Opuntia ficus indica



Figure 01: Taxonomic classification of cactus pear (Stintzing et al. 2002).

#### I.1.2. Compartments of the barbary fig fruit

#### a- The peels

The peels of the OFI constitute about 35% to 40% of the total weight of the fruit (**Habibi** 2004). It is delivered in a bow in sky of active color of the green, yellow, orange, red, purple and same to the brown (Feugang, Konarski et *al.* 2006). Peels are a new dessert of a dietary fiber and its content in galacturonic acid was superior to that of commercial cladode cactus raquet (El-Salid, Nagib et *al.* 2011).

#### **b-** Pulpy juice

Juice production is one of the most frequently utilized fruit and vegetable technology (**Tesoriere, Fazzari et** *a***l. 2005**). In some countries cactus pear juice is consumed at home, in vegetarian restaurants or in local health-food stores. Since technological problems are associated with its production, no commercial products are produced at industrial level. In addition, highly reactive molecules such as free radical-scavengers and antioxidants may be damaged during conventional operations, such as thermal sterilisation or evaporation, for juice preparation (**Cassano, Conidi et** *a***l. 2010**).

#### c- Seeds

Seeds contained in the pulp, accounts for 2 to 10%. Several research studies have been carried recently reported the chemical composition of seeds oil of *Oppuntia ficus indica*. According to literature data, oil processed from the seeds is characterized by a high degree of unsaturation where linoleic acid is the major fatty acid (56.1–77%). It is showed that cactus pear seed oil was rich in oleic (C18:1) acids (16.7%), which represented 87% of the total fatty acids. Vitamin E level accounted for only 0.04% of TL (**Ghazi, Ramdani et al. 2013**).

#### I.1.3. Chemical composition

Prickly pears were known to be a valuable source of vitamins, fibers, minerals, (**Table 1**) and antioxidant molecules (phenolics and betalains).

| Fraction             | Pulp      | Peels | References                 |
|----------------------|-----------|-------|----------------------------|
| Moisture content (%) | 94.4      | 90.33 |                            |
| Protein (%)          | 0.21-1.06 | -     |                            |
| Lipid (%)            | 0.7       | 1.06  |                            |
| Fiber (%)            | 0.02-3.15 | 40.8  | (Feugang, Konarski et al.  |
| Carbohydrates (%)    | 12-17     | -     | 2006; Salim, Abdelwaheb    |
| Vitamin K1 (mg)      | 53.2      | 109   | et al. 2009; Slimen, Najar |
| Vitamin C (mg)       | 1-48      | 59.82 | et al. 2016)               |
| Sodium (mg)          | 1.09      | 1.1   |                            |
| Magnesium (mg)       | 18.8      | 15.2  |                            |
| Potassium (mg)       | 199       | 98    |                            |
| Calcium (mg)         | 12.4      | 15.7  |                            |

Table 01: Chemical composition of OFI fruit

#### I.1.3.1. Bioactive compounds

"Polyphenols" (or phenolic compounds) is a generic term that refers to more than 8000 compounds widely dispersed throughout the plant kingdom. They can be defined as substances possessing an aromatic ring, carrying one or more hydroxyl groups, including their functional derivatives (**Yeddes, Chérif et al. 2013**). Flavonoids and phenolic acids are the main polyphenols of *Opuntia ficus-indica*. (**Abou-Elella and Ali 2014**).

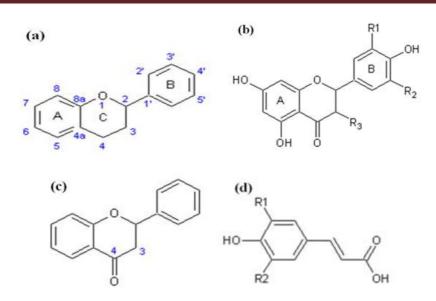
#### I.1.3.1.1. Flavonoids

Flavonoids have the C6–C3–C6 general structural backbone in which the two C6 units (Ring A and Ring B) are of phenolic nature. Due to the hydroxylation pattern and variations in the chromane ring (Ring C), flavonoids can be further divided into different subgroups such as flavones, flavonols and anthocyanins (**Tsao 2010**).

#### I.1.3.1.2. Phenolic acids

There are two main classes of phenolic acid; the derivatives of benzoic acid (C1-C6) and derivatives of cinnamic acid (C3-C6) (**Tsao 2010**). The concentration of the hydroxybenzoic acid is generally very low in edible vegetable. These derivatives are quite rare in the human diet by those against hydroxycinnamic acids which are very present (**Macheix, Fleuriet et al. 2005**).

- The basic structure of some *Opuntia* polyphenols is presented in figure 2.



**Figure 02:** Basic structures of some polyphenols from *Opuntia*: (a) Flavonoids, (b) Flavonols, (c) Flavones and (d) hydroxycinnamic acids. (**Slimen, Najar et** *al.* **2016**).

- These phenolic compounds are present in different *cactus* tissues at various concentrations, as detailed in table 2. (El-Mostafa, El Kharrassi et *al.* 2014).

| Plant tissue | Main component<br>identified | Content in mg/100g MF |
|--------------|------------------------------|-----------------------|
|              | Total phenolic acid          | 218.8                 |
|              | Quercetin                    | 9                     |
|              | Isorhamnetin                 | 4.94                  |
| Pulp         | Kaempferol                   | 0.78                  |
|              | Luteolin                     | 0.84                  |
|              | isorhamnetin glycosides      | 50.6                  |
|              | Kaempferol                   | 2.7                   |
|              | Total phenolic acid          | 48-89                 |
|              | Feruloyl-sucrose isomer 1    | 7.36–17.62            |
|              | Feruloyl-sucrose isomer 2    | 2.9-17.1              |
| Seeds        | Sinapoyl-diglucoside         | 12.6-23.4             |
|              | Total Flavonoids             | 1.5–2.6               |
|              | Total Tannins                | 4.1–6.6               |
|              | Total phenolic acid          | 45,700                |
|              | Total Flavonoid              | 6.95                  |
| Skin fruits  | Kaempferol                   | 0.22                  |
|              | Quercetin                    | 4.32                  |
|              | Isorhamnetin                 | 2.41–91               |

#### I.1.3.1.3. Betalains

Betalains are vacuolar pigments composed of a nitrogenous core structure, betalamic acid (chromoalcaloids). Betalamic acid condenses with imino compounds (*cyclo*-DOPA its glucosyl derivates) or amino acids derivates to form violet betacyanins and yellow, betaxanthins (**Fig.3**). Betalains are present in the pupl and the peel of *OFI*. Betacyanins and betaxanthins concentrations vary according to the color of the fruit. In addition to neobetanin, betanin, isobetanin, betanidin and indicaxanthin are present in the pulp of *Opuntia* fruits. Betanin and indicaxanthin were detected in the peel.

Betalains due their antioxidant activity to their phenolic hydroxy groups in addition to their imino and tetrahydropyridine groups. The common electronic resonance system supported between the two nitrogen atoms allows creating a stable carbocation upon an electron abstraction. (Slimen, Najar et *al.* 2016).

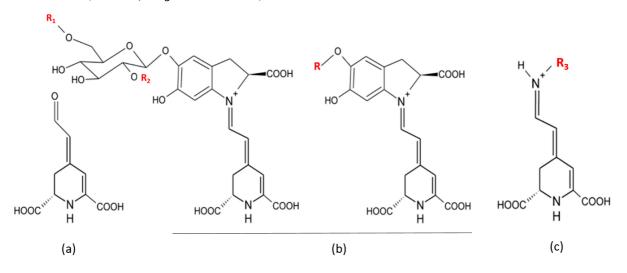


Figure 03: General structure of betalamic acid (a), betacyanins (b) and betaxanthins (c) (El-Mostafa, El Kharrassi et *al.* 2014).

#### I.1.4. Biological activities

Nopal cactus is employed in health, nutrition and cosmetics in the forms of tea, jam, juice and oil extracted from prickly pear seeds. It is used as a herbal remedy for diverse health problems in different countries.

Table 03: Major bioactive effects of cactus preparations in different experimental models.

| Biological<br>Activity | Source of Cactus<br>Products  | <i>In Vivo</i> and <i>in Vitro</i><br>Models  | References  |
|------------------------|---|---|---|
| Anti-diabetic          | Aqueous extract of the<br>cladode and fruit<br>and mixture<br>Cladode and fruit skin<br>extract capsule | Rats<br>Man   | (El-Mostafa, El<br>Kharrassi et <i>al</i> .<br>2014).     |
|                        | Indicaxanthin from Cactus<br>Pear Fruit   | Rat Pleurisy obtained by injection of 0.2 ml of $\lambda$ -carrageenin into the pleural cavity              |   |
| Anti-<br>Inflammatory  | Methanolic extracts of<br>prickly pear fruits (Betalain<br>Indicaxanthin)                               | In vitro study of the<br>interaction between<br>purified Betalains and<br>HOCL and human<br>myeloperoxidase |   |
|                        | Betalain a pigment purified<br>from fresh<br>pulp of cactus pear  | Endothelial cells human<br>umbilical vein<br>(HUVEC)  | (El-Mostafa, El<br>Kharrassi et <i>al</i> .<br>2014).     |
|                        | Betanin prickly pear fruit<br>Extracts  | Chemical and biological<br>(human RBC, LDL)<br>systems  |   |
| Antioxidant            | Flavonoid fraction of juice<br>of whole fruits<br>Glycoprotein (90 kDa)<br>isolated from                | Rats  | (El-Mostafa, El<br>Kharrassi et <i>al</i> .<br>2014).     |
|                        | Cactus pear fruit   | Healthy humans (10<br>women and 8 men)<br>supplemented with cactus<br>pear or Vit C                         |   |
|                        | Methanolic fruit extracts<br>(Betalain Indicaxanthin)   | healthy human   | (Butera, Tesoriere<br>et <i>al.</i> 2002)                 |
|                        | Juices of prickly pear fruits   | In vitro  | (BARBARIE 2006)   |
|                        | Cactus fruit  | Man   | (Osuna-Martínez,<br>Reyes-Esparza et <i>al</i> .<br>2014) |
| Diuretic<br>activity   | The cladodes, flowers and non commerciable fruits   | Rat   | (Galati, Tripodo et <i>al.</i> 2002)                      |

#### I.2. Drying techniques

Drying, or dehydration, is an important industrial process that involves the removal of moisture from a wet solid by means of facilitated heat and mass transfer (ElKhodiry, Suwaidi et *al.* 2015). Drying of materials having high moisture content is a complicated unit operation process involving simultaneous, coupled heat and mass transfer, particularly under transient conditions. It is one of the oldest methods of preservation and one of the most important in post-harvest processing of fruits, vegetables and other agricultural products (Harish, Rashmi et *al.* 2014). Thus, it prevents microbial contamination by reducing the water activity of the fresh agricultural commodities (ElKhodiry, Suwaidi et *al.* 2015).

- There are many different drying methods that have been applied to fruits and vegetables, from the most basic technique such as solar/sun drying to more expensive methods like freeze or microwave drying (McSweeney and Seetharaman 2015).

#### I.2.1. Conventional (air) drying

Conventional (air) drying is the most frequently used dehydratation operation in food and chemical industry, due to its controllable conditions and less dependency on climatic conditions (**Motri, Touil et** *al.* **2013**).

Air-drying is an ancient process used to preserve foods in which the material to be dried is exposed to a continuously flowing hot stream of air where moisture evaporates. The phenomenon underlying this process is a complex problem involving simultaneous mass and energy transport in a hygroscopic, shrinking system. Air-drying offers dehydrated products with their shelf life being extended by a year, but the quality of a conventionally-dried product is usually drastically reduced compared to that of the original foodstuff (Ciurzyńska and Lenart 2011).

#### I.2.2. Microwave drying

Microwave drying (MWD) has been considered as an alternative to oven drying by various researchers. The rise in its use for drying of agricultural commodities is borne out of the problems associated with hot air drying which includes long drying time involved coupled poor quality of final product. MWD has gained popularity, since it helps to cut down the time required for drying, homogeneous energy distribution and improves the final quality of the dried products. However it should be noted that drying process with the aid of microwave if not properly and carefully applied could result to low quality product (**Omolola, Jideani et** 

*al.* **2014**). Microwave heating is a result of dipolar interaction of water molecules inside the food materials (**TP**, **Harish et** *al.*).

MWD process consists of three drying phases. A heating up period is the first phase, in which the microwave energy is converted into thermal energy within the moist portions of the food. Gradually the temperature of the food increases and when the temperature is above that of the environment the food begins to lose moisture at a slow rate. The second phase then begins and is called the rapid drying period. The thermal energy that was created from the microwave energy is used to vapourize the moisture in foods. The reduced drying period is the last phase and if the energy needed for moisture vapourization is below the amount of thermal energy created, it can result in overheating or charring. (McSweeney and Seetharaman 2015).

#### I.3. Yoghurt

#### I.3.1. Definition and classification

Yogurt is a product made from heat treated milk that may be homogenized prior to the addition of lactic acid bacteria (LAB) cultures containing *Lactobacillus bulgaricus* and *Streptococcus thermophilus* (Andino 2011).Yoghurt is one of the most popular fermented milk products worldwide and has gained widespread consumer acceptance as a healthy food (Mckinley 2005). The general composition of yogurt (Table 4) is more or less similar to that of milk. Therefore, yogurt is a rich source of milk proteins, carbohydrate and minerals such as calcium and phosphorous (Weerathilake, Rasika et al. 2014)

| Constituent (per 100 g) | Full-fat yoghurt | Low-fat yoghurt | Fruit yoghurt |
|-------------------------|------------------|-----------------|---------------|
| Water (g)               | 81.9             | 84.9            | 77.0          |
| Total solids (g)        | 18.1             | 15.1            | 23.0          |
| Fat (g)                 | 3.0              | 0.8             | 0.7           |
| Protein (g)             | 5.7              | 5.1             | 4.1           |
| Lactose (g)             | 7.8              | 7.5             | -             |
| Calcium (mg)            | 200              | 190             | 150           |
| Phosphorus (mg)         | 170              | 160             | 120           |
| Sodium (mg)             | 80               | 83              | 64            |
| Potassium (mg)          | 280              | 250             | 210           |
| Zinc (mg)               | 0.7              | 0.6             | 0.5           |

Table 04: Chemical composition of typical yoghurt (Kumar and Mishra 2004).

- Industrially, yoghurts can be largely divided into two types. A set-style yogurt is made in retail containers giving a continuous undisturbed gel structure in the final product. On the other hand, stirred yogurt has a delicate protein gel structure that develops during fermentation .In stirred yogurt manufacture, the gel is disrupted by stirring before mixing with fruit and then it is packaged. Stirred yogurts should have a smooth and viscous texture. In terms of rheology, stirred yogurt is a viscoelastic and pseudoplastic product. Yoghurts come in a variety of textures (e.g. liquid, set, and smooth), fat contents (e.g. luxury, low-liquid, virtually fat-free) and flavors (e.g. natural, fruit, cereal), can be consumed as a snack or part of a meal, as a sweet or savory food, and are available all year round. This versatility, together with their acceptance as a healthy and nutritious food, has led to their widespread popularity across all population subgroups (Mckinley 2005).

#### I.3.2. Health benefits

In general, yogurt is considered as a nutrition-dense food due to its nutrient profile and is a rich source of calcium that provides significant amounts of calcium in bio-available form. In addition, it provides milk proteins with a higher biological value and provides almost all the essential amino acids necessary to maintain good health (Weerathilake, Rasika et *al.* 2014).Yogurt (and other dairy product) consumption has been shown to have various beneficial effects on many aspects of human health, including blood pressure (BP) and low density lipoprotein (LDL) cholesterol reduction, muscle building and prevention of various metabolic diseases. Concerning gastrointestinal health, yogurt contains cultures of lactic acid bacteria that improve digestive system function (Georgakouli, Mpesios et *al.* 2016).

# Material and methods

#### **II. Material and methods**

#### **II.1.** Chemicals

All solvents and reagents used were of analytical grade. Sodium carbonate ( $N_{a2}CO_3$ ), Folin ciocalteu's phenol reagent, aluminum chloride hexahydrate (AlCl<sub>3</sub>-6H<sub>2</sub>O) and 2,2diphenyl-1-picryl-hydrazil (DPPH), trichloroacetic acid extra pure (C<sub>2</sub>HCl<sub>3</sub>O<sub>2</sub>) were purchased from Sigma-Aldrich (Germany). Sodium acetate anhydrous (C<sub>2</sub>H<sub>3</sub>N<sub>a</sub>O<sub>2</sub> 3H<sub>2</sub>O) and potassium chloride (KCl) (UK) Gallic acid and quercetin were supplied from Biochemchemopharma (UK), potassium ferricyanide [K<sub>3</sub>F<sub>e</sub>(CN)<sub>6</sub>] was purchased from Biochemchemopharma (USA). Sodium hydroxide (NaOH) was purchased from Biochemchemopharma (Monterial, Quebec H2YOA).

#### **II.2. Plant material**

The fruits *of Opuntia ficus indica* L (**Fig.4**) were collected from Tibane, Bejaia (Algeria) in Novembre 2016, then washed by distilled water. Fruits were peeled and pressed to the juicy pulp, which preserved at -20°C. The whole peels were dried by toven and microwave methods.



Figure 04: Photography of *Opuntia ficus indica fruit*.

#### **II.3.** Drying

#### II.3.1. Oven drying

The OFI peels were air dried in a ventilated oven (ECOCELL), at different temperatures (40, 60, 80, 100 and 120 °C), until constant weight then ground using an electric coffee mill (KSW445 CB) and sieved to granulometry  $< 250 \mu m$  prior to extraction. The water activity (aw) of powders was determined by HygroPalm AW. The fine powders were stored in air tight containers until use.

#### **II.3.2.** Microwave drying

Microwave drying experiments were performed in a domestic microwave oven (NN-S674MF. Samsung. Malaysia) with cavity dimensions of 22.5 cm  $\times$  37.5 cm  $\times$  38.6 cm and 2450 kHz working frequency was used. The apparatus was equipped with a digital control system for irradiation time and microwave power (the latter linearly adjustable from 100 to1000 W). Different microwave power (100, 300, 500, 700 and 900W), were used in the drying of cactus peel, until constant weight, then ground and sieved to < 250 µm particle size. The water activity was also determined.

#### **II.3.3.** Evaluation of moisture content

Thermal drying method was used in the determination of humidity. 10 g of sample were placed in an oven « ECOCELL» to dryness at  $103\pm2^{\circ}$ C, until constant weight. The moisture content (MC) was calculated by expressing the weight loss upon drying as a fraction of the initial weight of sample used:  $MC\% = \frac{W0}{W1} \times 100$ . Where W<sub>0</sub> correspond to the loss in weight (g) on drying and W<sub>1</sub> correspond to the initial weight of sample (g).

#### **II.3.4.** Color assessment

Color of the samples was measured using a color reader (PCE-TCR 200, USA) under white light at 90° angle. The colorimetric coordinates of the powders of cactus peel, were computed in the CIELAB scale. In this scale, each color is numerically specified by a unique set of three cylindrical coordinates ( $L^*a^*b^*$ ):  $L^*$  indicates the luminance and changes from 0 for black to 100 for white,  $a^*$  changes from - 60 for green to + 60 for red,  $b^*$ changes from - 60 for blue to + 60 for yellow (**Achat, Tomao et al. 2012**). Data were the average of three measurements.

#### **II.4.** Extraction procedure of bioactive compounds

Extraction is the first key step to isolate natural bioactive substances from plant materials. A preliminary study was performed in order to select the extraction method for the rest of investigation. By fixing extraction time (15 min), solvent (Ethanol: 70 %), ratio (25 ml/g) and dried peel powders (40°C and 100W): samples were extracted with conventional, ultrasound and microwave techniques.

#### **II.4.1.** Conventional extraction (CV)

OFI peels were macerated in ethanol at room temperature under /without stirring, then the mixture was covered with parafilm and aluminum foil, to prevent light exposure.

#### II.4.2. Ultrasound assisted extraction (UAE)

The dried peel powder was extracted in an ultrasonic apparatus (SONICS Vibra cell, VCX 130 PB, Stepped microtips and probes, No. 630-0422) with working frequency fixed at 20 kHz (**Dahmoune, Boulekbache et** *al.* **2013).** 



Figure 05: Ultrasonic equipment used in UAE.

#### II.4.3. Microwave assisted extraction (MAE)

A domestic microwave, as reported in Section II.3.2, oven was used for the extraction of cactus peels. The oven was modified in order to condensate into the sample the vapors' generated during extraction of the sample (Fig.06) (Dahmoune, Nayak et *al.* 2013). The mixture was irradiated at 700 W according to oven operation.



Figure 06: Microwave equipment used in MAE.

After filtration through a Whatman filter paper, all extracts, were stored at 4°C and subsequently used for the determination of total phenolic content (TPC), flavonoïds, betalains, total anthocyanins compounds (ANC) and antioxidant activity.

#### **II.5.** Determination of polyphenols

#### **II.5.1.** Total phenolic content

The amount of total phenolic (TPC) in the extracts was determined using Folin-Ciocalteu method. Oxidations of phenolic compounds with this reagent include reaction with the mixture of H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub> and H<sub>3</sub>PMo<sub>12</sub>O<sub>40</sub> acids in the alkaline medium. At this reaction a mix of blue oxides is formed (**Georgé, Brat et al. 2005**). Thus, a 2.5 mL sample of waterdiluted Folin-Ciolcateu reagent (1/10) was added to the different extracts of OFI. The mixture was incubated for 2 min at room temperature, and 2 mL of sodium carbonate (75 g/L) was added. The mixture was incubated for 15 min at 50 °C and finally cooled in a water-ice bath. The specific absorbance at 750 nm was immediately measured, using Uv-vis light spectrophotometer (SHIMADZU-UV-1800, Germany). TPC concentration was calculated from a calibration curve, using gallic acid as a standard and the results were expressed as mg gallic acid equivalents per 100 g of dry matter (mg GAE/ 100 g). All determinations were carried out in triplicate.

#### II.5.2. Flavonoïds content

The total flavonoid content (TFC) was determined using the method of (**Ghafar**, **Prasad et al. 2010**), based on the formation of aluminium- flavonoid complexes (**Fig.07**). Briefly, 1 mL of 2% (w/v) aluminium chloride (AlCl<sub>3</sub>) was added to 1 mL of diluted extracts or quercetin (positive control) and then mixed using vortex mixer (EV-102, tehtnica zelezniki, Germany). The mixture was allowed to stand for 15 min. Absorbance of the mixture was determined at 430 nm versus the prepared blank using Uv-vis light spectrophotometer (SHIMADZU-UV-1800, Germany). TFC was expressed as mg quercetinc equivalent per 100 g of dry matter (mg QE/ 100 g DM). Samples were measured in triplicate.

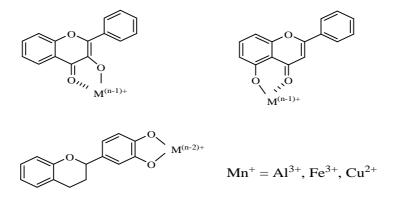


Figure 07: The chelation of metal ions by flavonoids (Dangles 2006).

#### **II.5.3.** Betalains content

Betacyanins and betaxanthins content were reported like mg equivalent betanin/L and mg equivalent indicaxanthin/L, respectively. Betacyanins were detected at 538 nm and betaxanthins at 480 nm (Sumaya-Martínez, Cruz-Jaime et *al.* 2011).

Betacyanins or betaxanthins content [mg/L] = [(A \* DF \* MW \* 100 / E \* 1)]

Where: A = absorbance at 535 or 480 nm; DF = dilution factor; MW = molecular weight;  $\mathcal{E}$  = extinction coefficient; l = width of the spectrophotometer cell (1 cm);

The  $\mathcal{E}$  of betacyanin is 60,000 L/ (mol.cm) and MW = 550 g/mol. For betaxanthins the  $\mathcal{E}$  is 48,000 L/ (mol.cm) and MW = 308 g/mol.

- Total betalains (betacyanins + betaxanthins) were expressed as mg/100g of dry weight (DW).

#### **II.5.4.** Total anthocyanins content

Total monomeric anthocyanins (ANC) content of prickly pear samples was monitored by the pH differential method as outlined by (Lee, Durst et al. 2005). Monomeric anthocyanin pigments reversibly change color with a change in pH; the colored oxonium form exists at pH 1.0, and the colorless hemiketal form predominates at pH 4.5. The difference in the absorbance of the pigments at 520 nm is proportional to the pigment concentration. After dilution of the extracts with potassium chloride buffer (0.025 M, pH = 1.0) and sodium acetate buffer (0.040 M; pH = 4.5) and allowed to equilibrate for 20 minutes. The absorbance of equilibrated samples was measured versus a blank cell for pH 1.0 and 4.5 at maximum absorbance wavelengths ( $\lambda_{visile max} = 520$  nm) and at 700 nm to correct for haze. Measurements were performed in triplicates. Results are expressed as cyanidin-3-glucoside basis, as follows:

Anthocyanin pigment (cyanidin – 3 – glucoside equivalents, mg/L) =  $\frac{A \times MW \times DF \times (1000)}{\varepsilon \times l}$ 

Where: A =  $(A_{520nm} - A_{700nm})$  pH 1.0 –  $(A_{520nm} - A_{700nm})$  pH 4.5; MW (molecular weight) = 449.2 g/mol for cyanidin-3-glucoside (cyd-3-glu); DF = dilution factor; l = pathlength in cm;  $\varepsilon$  = molar extinction coefficient = 29 000 L.mol<sup>-1</sup> .cm<sup>-1</sup> for cyd-3-glu;  $10^3$  = factor of conversion from g to mg.

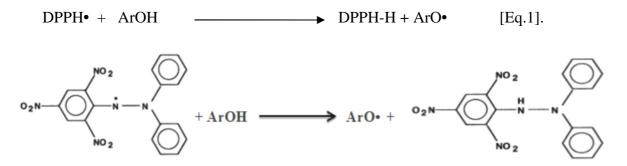
#### **II.6.** Antioxidant assays

#### **II.6.1. Radical-scavenging test**

The radical-scavenging activity of samples was evaluated by the DPPH• assay. DPPH (2,2-diphenyl-1-picrylhydrazyl) is a stable highly colored free radical that can abstract labile hydrogen atoms from phenolic antioxidant (ArOH) with concomitant formation of a colorless hydrazine (DPPH-H), according to equation 1 and figure 08 (**Molyneux 2004**). The free radical-scavenging activity (RSA) of an extract can be expressed as the percentage of DPPH reduced by a given amount of extract. The free radical-scavenging activity (RSA) was measured, following (**Achat, Tomao et al. 2012**) method. 1 ml of extract was added to 2 ml of DPPH solution (2.10<sup>-4</sup> M/L in methanol) and the mixture was left in the dark at room temperature for 20 min. The total RSA of each extract was expressed as the percentage of DPPH reduced and was calculated by the following equation:

$$RSA = \frac{A0 - A}{A0} \times 100$$

-  $A_0$ , absorbance of DPPH solution without any antioxidant; A, absorbance of DPPH solution after reaction with the extract. All experiments were performed in triplicate.



Diphenylpicrylhydrazyl (free radical)

Diphenylpicrylhydrazine (nonradical)

#### Figure 08: DPPH• radical reduction (Molyneux 2004)

-  $IC_{50}$  value is the concentration of sample required to scavenge 50% of DPPH free radical and was calculated from the plotted graph of radical scavenging activity against the concentration of extracts.

#### **II.6.2. Reducing power assay**

The reducing power of different extracts were measured according the method used by (**Rohman, Man et al. 2011**) 1 ml of extracts with different concentrations was mixed with 2.5 ml of phosphate buffer (200 mM; pH 6.6) and 2.5 ml of potassium ferricyanide (1%) and incubated at 50 °C for 20 min. The mixture was added with 2.5 ml of 10% trichloroacetic acid

and centrifuged at 3000 rpm for 10 min. A 2.5 ml of supernatant was mixed with 2.5 ml of distilled water and 0.5 ml of ferric chloride (0.1%) and the absorbance was measured spectrophotometrically at 700 nm. A higher absorbance indicates a higher reducing power. RC50 (mg/ml) is the effective concentration at with the absorbance was 0.5 for reducing power and was obtained by interpolation from linear regression analysis. All testes were carried out in triplicate.

#### **II.7.** Analysis of cactus juice

For the juicy pulp of OFI, degrees Brix (°Bx), pH and titrable acidity were determined by triplicate (**Tab.05**). TPC, TFC, betalains and antioxidant activity were also quantified in cactus juice.

| Measure               | Method  |  |  |  |
|-----------------------|---|--|--|--|
| рН                    | The pH value of cactus juice was measured at fixed temperature (20-25°C) with a calibrated pH electrode (HANNA HI 213). The pH reading was performed in triplicate. (Friedrich 2001).   |  |  |  |
| Titrable acidity (TA) | 10 g of centrifuged sample (2500 rpm, 20 minutes at room temperature) was added to 100 g distilled water, and then titrate with NaOH solution (0.1 N) to the endpoint of pH 8.2. Thus TA was calculated:<br>$TA(g/100ml) = \frac{(V)(N)(meq.wt)(100)}{(1000)(v)}$ |  |  |  |
|                       | Where: V, N were volume (ml) and normality of NaOH respectively; meq. wt. is milliequivalent weight of the standard, v is sample volume (ml). (Friedrich 2001).   |  |  |  |
| Brix degree           | The soluble solids content of the OFI juicy pulp was assessed by the refractometer, where sugar content value was given.  |  |  |  |

Table 05: Physico-chemical properties of OFI juice

#### **II.8.** Formulation of milk products at laboratory scale

#### II.8.1. Manufacture of yoghurts and milky juice

The preparation of yoghurt was made in the laboratory 3BS (University of Bejaia) respecting the diagram for making plain yoghurt with addition of prickly pear juice and peels. The adapted recipe is the one determined within the work which is included in our research laboratory project. Thus, four steamed yoghurts were manufactured; cow milk was homogenized and heated to 95 °C for 5 min then cooled to 40 °C. After then, traditional

starter culture was added and the mixture was incubated until the gel structure was formed. The gel was stirred and stored at refrigerator ( $6 \pm 2^{\circ}$ C), in this case a standard stirred yoghurt was obtained. The same experiment was done with the other yoghurts except that whereas dried cactus juice and peels were added.

**Table 06:** Recipe of stirred, flavored yoghurts with the prickly pear juices and enriched milky juice with polyphénols of cactus peels.

| Recipe                  | Milk | Sugar (g) | Cactus                 | Lactic Ferment | Water   |
|-------------------------|------|-----------|------------------------|----------------|---------|
|                         | (L)  |           |                        | (%)            | (g)     |
| Plain yoghurt           | 1    | 80-100    | 0                      | 0.02           | 0       |
| Flavored yoghurt        | 1    | 80-100    | Juice ()               | 0.02           | 0       |
| Stirred yoghurt         | 1    | 80-100    | Juice ()               | 0.02           | 0       |
| Plain milky Juice       | 1    | 70-100    | 0                      | 0              | 600-900 |
| Enriched milky<br>Juice | 1    | 70-100    | Phenolic<br>extract () | 0              | 600-900 |

#### **II.8.2.** Physico-chemical properties

Physico-chemical properties of the manufactured milk products (yoghurts and milky juices) were determined namely, pH, dornic acidity, viscosity, the dry extract and fat contents (**Tab.07**).

Table 07: Physico-chemical properties of prepared dairy products

| Measure                                 | Method  |  |  |  |
|---|---|--|--|--|
| pH                                      | The pH value of yoghurt was measured at fixed temperature (9.5-       |  |  |  |
|   | 10.5°C) with a calibrated pH electrode (HANNA HI 2210).               |  |  |  |
| Viscosity (g)                           | Apparent viscosity of yoghurt was expressed using a viscometer        |  |  |  |
|   | "TAXT EXPRESS" during 45 S.   |  |  |  |
| Dornic acidity (°D)                     | 10 g of sample (adjusted with distilled water up to 60 g), was put in |  |  |  |
|   | acidometer apparatus then the result was directly displayed.          |  |  |  |
| Brix degree                             | The soluble solids content of the filtered yoghurt (whey) and was     |  |  |  |
|   | assessed by the refractometer, where sugar content value was given.   |  |  |  |
| Total dry extract (%)                   | 50 g of yoghurt was placed in "Food scan" apparatus which give the    |  |  |  |
| Protein content (%)<br>Fat contents (%) | values of total dry extract, protein and fat contents.                |  |  |  |

These tests were carried out at the laboratory of the dairy industry "DANONE DJURDJURA".

#### **II.8.3.** Microbiological analysis

Microbiological quality of prepared yoghurts and lacted juices was evaluated by enumerating total viable organisms. The organisms enumerated include total flora, yeast, moulds, total coliforms and specific bacteria of yoghurt (**Tab.08**).

| Micro-organisms               | Selective me- | Incubation  | Incubation | Method  |  |  |
|-------------------------------|---------------|-------------|------------|---|--|--|
|                               | diums         | temperature | time       |   |  |  |
| Total Coliforms               | VRBL          | 30°C        | 24h        |   |  |  |
| Total Flora                   | PCA           | 30°C        | 72 h       |   |  |  |
| Yeasts, moulds                | YGC           | 25°C        | 5 days     |   |  |  |
| Streptococcus<br>thermophilus | M17           | 37°C        | 48h        | 3g of the Yoghurt<br>samples was spread<br>plated in triplicates into<br>prepared and dried |  |  |
| Lactobacillus<br>bulgaricus   | MRS           | 37°C        | 72h        | petri-plates of suitable<br>media for the counts of<br>different organisms.                 |  |  |
| Staphilaucocus<br>aureus      | Baird-parkeur | 37°C        | 48h        |   |  |  |
| Salmonelles                   | SS            | 37°C        | 24h        |   |  |  |
| VRBL: Violet Red Bile Agar    |               |             |            |   |  |  |

Table 08: Microbiological analysis of manufactured yoghourts.

VRBL: Violet Red Bile Agar
YGC: Yeast extract glucose chloramphenicol agar
M17: M17 agar
MRS: Rogoza and Sharpe agar
SS: salmonella-shigella

#### **II.8.4.** Antioxidant acitivity

The Radical scavenging capacity was measured in manufactured yogurts and lacted juice by the DPPH<sup>•</sup> assay (Section II.6.1). The TPC and betalains content in prepared milk products were also determined by using colorimetric methods (Section II.5.1 and Section II.5.3 respectively).

#### Sample preparation

#### - Yogurt water extract

Yogurt sample (10g) was mixed with 2.5ml distilled water and the yogurt pH was adjusted to 4.0 using 1M HCl. The yogurt was then incubated at 45°C for 10 minutes, followed by

centrifugation (10000 rpm, 20 minutes, 4°C). The supernatant was harvested and the pH was adjusted to 7.0 using NaOH. The neutralized supernatant was recentrifuged (10000 rpm, 20 minutes, 4°C) and the supernatant was used in analysis (**Zainoldin and Baba 2009**).

#### Statistical analysis

All experiments were conducted in triplicate and results are expressed as mean  $\pm$  standard deviation (SD). The analysis of variance (*ANOVA*) was performed using XLSTAT Release 10 (Addinsoft, Paris, France). Tukey's multiple range test (HSD) was used to compare means of the determined parameters. Evaluations were based on the *p* < 0.05 significance level.

# Results and discussion

#### III. Results and discussions III.1. Peels III.1.1. Drying of OFI peels

In this study, conventional drying (oven) and the innovative drying (microwave) methods were adopted, because of their ability to keep bioactive compounds of the OFI peels and compare their performance.

#### III.1.1.1 Oven drying kinetic

The weight loss depending on the time temperature of ventilated oven drying of cactus peels was shown in figure 9.

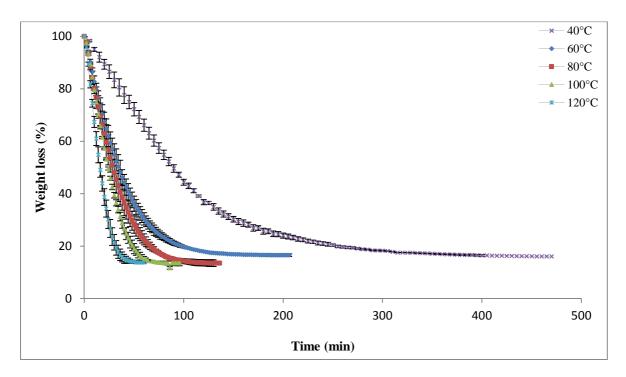


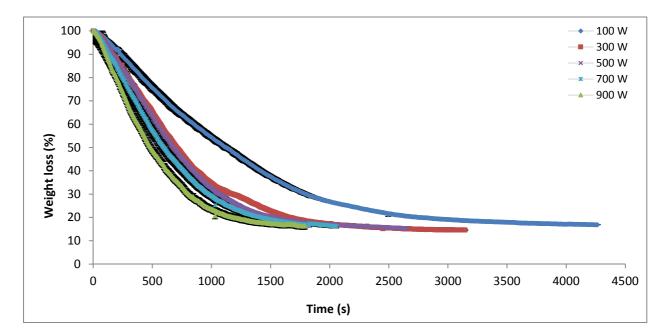
Figure 09: Weight loss evolution as a function of time oven drying of OFI peels.

- The graph showed that the weight of the sample decreases with time progression. The longer drying time was obtained at 40 °C (8  $\pm$  0.766 hours), however the shortest one was attributed to 120 °C after 1.2  $\pm$  0.07 hours. The obtained results revealed that the drying time is inversely proportional to the applied temperature, where high temperatures (100, 120°C) accelerated the water loss. Indeed temperatures below 60°C do not promote sufficient displacement of the water vapor from the material to reach the desired humidity and above 80°C volatilization of product components starts (**Correia, Loro et al. 2015**).

- The same results were reported by (ElKhodiry, Suwaidi et *al.* 2015; Kumar 2015) in drying kinetics of eggplants peels in a fluidized bed dryer, and banana peels with tray dryer respectively.

### **III.1.1.2.** Microwave drying kinetic

The variation of the weight loss versus time and drying power in microwave (**fig.10**) showed that moisture contents of prickly pears decreased continuously throughout the drying time. The weight stability of the sample dried at 100 W was reached after 1 hours and 18 min  $\pm 0.11$  drying power while dried sample at 900 W, stabilizes after 50  $\pm 8$  min drying power.





The result of drying methods, oven and microwave reveal a remarkable dependency on temperature and power used to intensify the mass transfer.

The oven drying time is about  $8 \pm 0.76$  hours (40 °C) which is significantly higher than that of microwave power at 100 W (1 hours and 18 min  $\pm 0.11$ ).

Longer drying time was attributed to oven drying can be explained by (**Feng, Yin et al. 2012**) that microwave drying arises from the volumetric heating and internal vapor generation. Heating from the interior of a food product leads to the buildup of an internal vapor pressure that drives the moisture out of the product. This results in a significant reduction in drying time, leading to significantly improved product quality, this explains why microwave drying causes shorter time.

(Maskan 2000) confirms that mass transfer within a sample is rapid during microwave heating because heat is generated within the sample, creating a large vapour pressure differential between the centre and the surface of products.

### **III.1.2.** Moistures content

Thermal processing is one of the important method of food preservation, primarily intented to inactivate enzymes, deteriorate of microorganisms and reduce water activity by dehydratation (**Maskan 2001**). The drying efficiency was evaluated in terms of water loss; moisture and water activity for the various powders obtained (after drying and grinding) in the various conditions applied. The moisture content (MC) and water activity of cactus peels powder were shown in table 09.

| Temperature (°C) | $a_{w}$         | MC (%)           |
|------------------|-----------------|------------------|
| 40               | $0.41\pm0.00$   | $83.40\pm0.74$   |
| 60               | $0.34 \pm 0.04$ | $83.79\pm0.5$    |
| 80               | $0.26\pm0.00$   | $85.59 \pm 0.9$  |
| 100              | $0.25\pm0.00$   | $87.19 \pm 0.97$ |
| 120              | $0.22 \pm 0.02$ | $86.07 \pm 0.18$ |
| Power (W)        |                 |                  |
| 100              | $0.44\pm0.03$   | $83.09\pm0.02$   |
| 300              | $0.38\pm0.07$   | $85.93 \pm 0.73$ |
| 500              | $0.43\pm0.06$   | $84.54 \pm 0.1$  |
| 700              | $0.30\pm0.04$   | $85.54 \pm 0.03$ |
| 900              | $0.31\pm0.03$   | $83.57 \pm 0.1$  |

Table 09: The water activity of dried OFI peels.

- High water activity indicates more free water available for biochemical reactions and hence shorter shelf life. Generally food with  $a_w < 0.6$  is considered as microbiologically stable and itself is any spoilage occur. it is induced by chemical reactions rather than by micro-organism (Quek, Chok et al. 2007). From results, the water activities of OFI powders were in the range of 0.22 - 0.44. This means that the OFI peels powders were relatively stable microbiologically. The water content of cactus peels was  $87.19 \pm 0.97\%$ .

### **III.1.3.** Color assessment

Color of the dried prickly pear peels was investigated by CIE scale: lightness (L\*) and redness (a\*) and yellowness (b\*) values (**Tab.10**).

| Temperature (°C) | L* value         | a* value         | b* value         |
|------------------|------------------|------------------|------------------|
| 40               | 31.62±4.88       | 8.39±1.04        | 18.81±4.47       |
| 60               | $28.8 \pm 2.34$  | $8.06 \pm 3.67$  | $18.73 \pm 3.59$ |
| 80               | 25±3.44          | $6.87 \pm 0.48$  | 15.43±0.61       |
| 100              | 23.6±1.91        | $8.03 \pm 0.88$  | $16.71 \pm 1.30$ |
| 120              | $23.26\pm3.66$   | $9.13 \pm 1.11$  | $12.6\pm2.75$    |
| Power (watt)     |                  |                  |                  |
| 100              | $27.03 \pm 2.89$ | $10.2 \pm 1.64$  | $14.86 \pm 1.29$ |
| 300              | $21.76 \pm 5.68$ | $10.61 \pm 2.89$ | 15.45±6.69       |
| 500              | $21.83 \pm 1.58$ | 11.21±4.2        | $17.3 \pm 1.22$  |
| 700              | $20.02 \pm 1.49$ | 8.91±1.16        | $13.48 \pm 2.47$ |
| 900              | $21.73 \pm 3.66$ | 8.98±0.91        | 33.91±35.07      |

| <b>Table 10:</b> The color assessment of dried OFI peel |
|---|
|---|

OFI peels dried with microwave had lower L\*, b\* values and higher a\* values than prikly pears dried with hot air. This could be explained by Maillard reactions taking place in microwave drying. (Roncero-Ramos, Delgado-Andrade et *al.* 2013) showed that Maillard reaction was accelerated at temperatures over 50 °C. Therefore in microwave drying Maillard reaction rate was higher due to higher temperature.

It has been reported that many reactions can affect colour during thermal processing of fruits and their derivatives. Among them, the most common are pigment degradation, especially carotenoids and chlorophyll, and browning reactions such as Maillard condensation of hexoses and amino components, and oxidation of ascorbic acid (Maskan 2000; Aydogdu, Sumnu et *al.* 2015). However (Aydogdu, Sumnu et *al.* 2015) showed that microwave heating did not result in any significant color change in cakes.

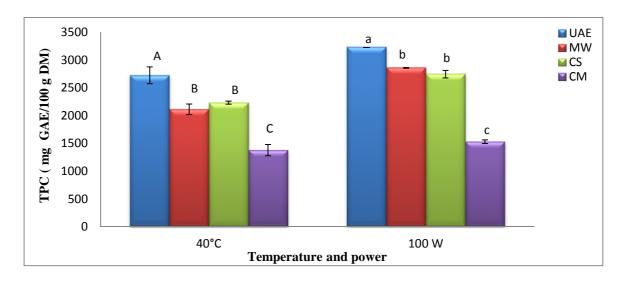
### **III.1.4.** Extraction of bioactive compounds

### **III.1.4.1.** Preliminary study

The effect of the extraction method on the rate of TPC, TFC, betalains and DPPH°, was evaluated with conventional, ultrasound and microwave techniques

### a. Total polyphenols content

Figure 11 depicted the amount of TPC of cactus peels, using different extraction methods.



**Figure 11:** TPC of OFI peels (UAE: ultrasound assisted extraction, MW: microwave, CS: conventional stirring, CM: conventional maceration).

Polyphenolic content presented significant differences according to extraction method used. The result obtained by UAE was the richest in phenolics (2725.82  $\pm$  151.63; 3230.17  $\pm$  0 mg GAE / 100 g DM) for dried cactus peels at 40°C and 100 W respectively. However, the lowest one was attributed to CM (1379.59  $\pm$  101.09; 1530.49  $\pm$  33.69 mg GAE/100 g DM) for 40°C and 100 W respectively.

The same results were obtained for some fruits like orange peels, blackberry residues and *Melastoma sanguineum* (Khan, Abert-Vian et *al.* 2010; Zhou, Xu et *al.* 2017), in which UAE showed the highest TPC compared to conventional extractions.

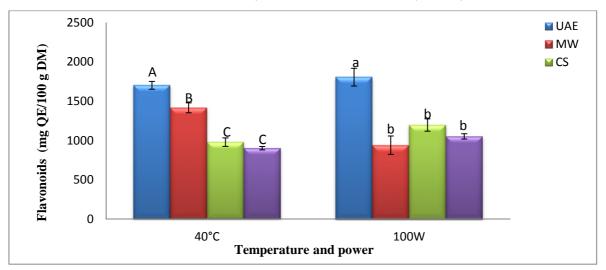
The results indicated that UAE was the most effective method among the three extraction methods. The mechanical effect and cavitation induced by ultrasound, disrupt the cell wall and increase mass transfer (Achat, Tomao et *al.* 2012), which can explain the high efficiency of UAE.

### b. Flavonoïds

Figure 12 shows that UAE yielded higher values of flavonoïd (1702.39  $\pm$  50.07; 1805.66  $\pm$  112.65 mg EQ/100 g DM) for 40 °C and 100 W respectively, in comparison with the use of MW, CS, CM at (p < 0.05).

The flavanone concentrations (70.3 mg of naringin and 205.2 mg of hesperidin/100 g FW) obtained in orange peels from UAE, proved its efficiency when compared with the conventional method (Khan, Abert-Vian et al. 2010; Zhou, Xu et *al.* 2017). TFC of

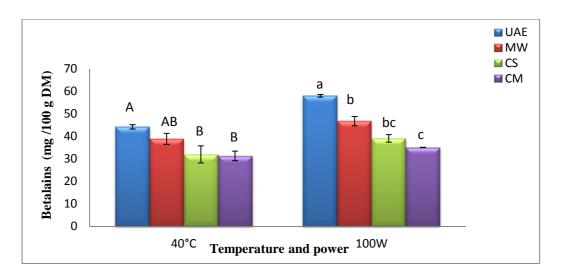
*Melastoma sanguineum*, using UAE, were also significantly higher than those extracted by maceration and Soxhlet method. (Khan, Abert-Vian et al. 2010; Zhou, Xu et *al.* 2017).



**Figure 12:** Total flavonoïd of OFI peels by different methods (UAE: ultrasound assisted extraction, MW: microwave, CS: conventional stirring, CM: conventional maceration).

### c. Betalains

Betalains are the characteristic pigments of the prickly pear. Spectrophotometric quantitation of these substances has been reported for the different extraction methods (Fig.13)



**Figure 13:** Total betalains of OFI peels by different extraction methods (UAE: ultrasound assisted extraction, MW: microwave, CS: conventional stirring, CM: conventional maceration).

Statistical analysis revealed significant differences (p < 0.05) between the extraction methods (UAE, MW, CS, CM). UAE exhibited the highest betalains contents:  $23 \pm 0.96 \text{ mg}/100 \text{ g DM}$  ( $40^{\circ}$ C);  $58.06 \pm 0.59 \text{ mg}/100 \text{ g DM}$  (100 W). Whereas the amount obtained with conventional extraction was the lowest for the samples dried at  $40^{\circ}$ C and 100 W ( $31.31 \pm 2.12$ ;  $35.08 \pm 0.05 \text{ mg}/100 \text{ g DM}$ ) respectively.

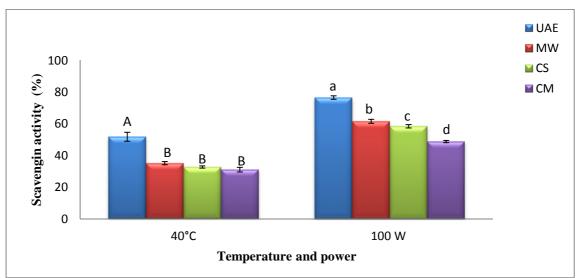
Betalains such as betacyanins and betaxanthins occur in a number of natural sources; however, prickly pears, beets, and the fruit from vine cactus are the only foods containing this class of compounds. Consistent with the results of others, we found that betanin and indicaxantin are the main betalain pigments of the prickly pear (**Butera, Tesoriere et** *al.* **2002**).

### d. Anthocyanins

Anthocyanins are water-soluble glycosides of anthocyanidins, normally found in the skin and responsible for the blue, red, purple and black colors of fruits. After juice extraction, many phenolic compounds, particularly anthocyanins, are still present in the solid residues and are suitable for extraction. (Khan, Abert-Vian et *al.* 2010; Zhou, Xu et *al.* 2017). Our results revealed that these compounds are undetectable in cactus peels extracts.

### e. Antioxidant capacity

The effect of extraction methods on the radical scavenging activity of ethanolic extracts of cactus peels was shown in figure 14.



**Figure 14**: Antiradical activity (DPPH°) of OFI peels by different extraction methods (UAE: ultrasound assisted extraction, MW: microwave, CS: conventional stirring, CM: conventional maceration).

- The best antioxidant activities were shown by UAE using both drying techniques oven and microwave:  $76.6 \pm 1.13 \% (100 \text{ W})$ ,  $51.91 \pm 2.84 \% (40 ^{\circ}\text{C})$  at (p < 0.05). However the lowest level of RSA was assigned to conventional maceration ( $28.08 \pm 5.90$  and  $52.42 \pm 5.01 \%$ ) for 40°C and 100 W respectively. These results are in accordance with the amount of bioactive components (TPC, TFC and betalains) quantified in cactus peels extracts. The extraction yield increases the scavenging activity for the peel of prickly pear fruit. UAE produces ultrasonic waves that attack the integrity of plant cellular walls. This resulted in increased permeability of cytoplasmic membranes and more solvent can enter into the plant cell while causing the release of more compounds into the solvent (**Ramli, Ismail et al. 2014**).

To evaluate the effect of drying, oven and microwave, on quality cactus pear peels, the obtained ultrasonic extracts were analyzed for bioactive components (TPC, TFC and betalains) and antioxidant activities (radical scavenging test and reducing power).

### III.1.5.1. Total phenolic content

The determination of total phenolic compounds for peel of OFI, dried with oven and microwave power are given in figure 15. The results obtained were significantly different (p < 0.05).

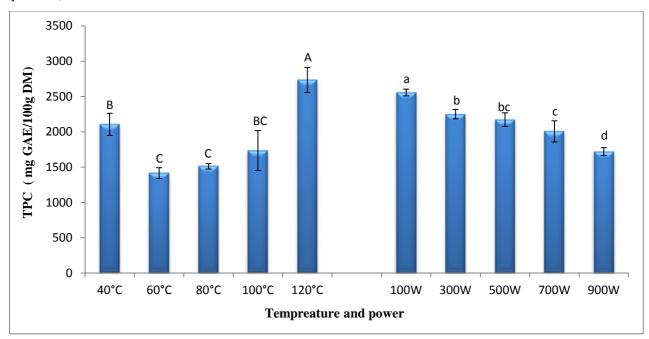


Figure 15: Contents of total phenolic of the OFI peels extracts dried in oven and microwave.

The drying oven indicated that the highest TPC was attributed to120°C and the lowest to 60°C respectively (2734.28  $\pm$  178.15, 1414.89  $\pm$  76.24 mg GAE/100g DM) at (p < 0.05), this probably due to generation of different antioxidant compounds having a varying degree of antioxidant activity. Ambiguous connections between the content of particular antioxidants and antioxidant activity are difficult to explain only on the basis of quantitative analysis. Some authors suggested that not only the evel of antioxidants but also a synergy occurring between them and the other fruit constituents might influence the differences in the antioxidant ability of food extracts (López, Uribe et al. 2010).

For microwave drying, the highest TPC was obtained with 100W and the lowest with 900W (2556.38  $\pm$  47.68, 1719.85  $\pm$  55.38 mg GAE/100g DM) respectively. This decrease can be explained by the degradation of these phenolic compounds by the strong radiation (**Orphanides, GOulAs et al. 2013**), some other studies have also reported that the phenolic content was decreased after heat or radiation treatment of the plant materials (**Galati, Tripodo et al. 2002; Hayat, Zhang et al. 2010**). TPC obtained in this study was so much higher than those obtained by (**Chougui, Djerroud et al. 2015**) (1512.58  $\pm$  31.5 mg GAE/100 g DM).

It was also noticed that the TPC was very preserved using drying oven at 120 °C (2734.28  $\pm$  178.15 mg GAE/100g DM) after 72 min, while time is significantly reduced (15.83 min) when microwave drying is applied but with a lower amount of TPC at 100 W (2556.38  $\pm$  47.68 mg GAE/100g DM).

### III.1.5.2. Flavonoids content

The total flavonoids contents, expressed as milligram quercetine equivalent per 100 gram of OFI peel dried using oven and microwave were represented in figure 16.

According to this results (**Fig.16**) at (p < 0.05), in oven drying we notices that the flavonoids content increases from 40°C to 80°C to reach a peak value of 815.30 mg EQ/100 g DM, on the other hand a tendency of reduction is observed 100°C and 120°C to reach a minimal value of 510.42 ± 28.55 mg/100g DM to 100°C. The same results was obtained with microwave, that the flavonoids content increases from 100W to 500W to reach a peak value for 984.46 ± 34.32 mg/100g DM then we have a reduction at 700W and 900W to reach a minimal value of 616.64 ± 73.93mg/100g DM.

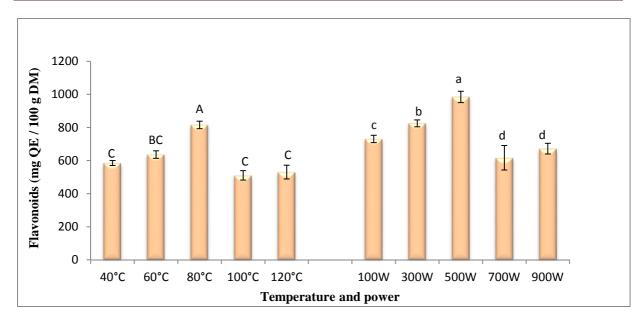


Figure 16: Total flavonoïds content of OFI peels dried in oven and microwave.

This reduction could be explained by the deterioration of the flavonoids by the strong powers and temperature of drying, and to see the type of the flavonoids present even in the fruit. The gotten results show that the content in flavonoids is influenced extensively by the high powers and temperature.

The flavonoids content obtained in this study was near than those obtained by (Cai, Gu et *al.* 2010) which was 555 mg/100g DM and those obtained by (Abou-Elella and *Ali* 2014)  $427.33 \pm 4,67$  mg/100g DM.

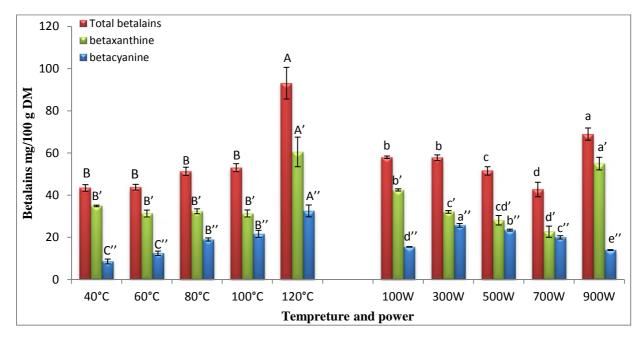
### **III.1.5.3.** Betalains content

The total betalains contents, expressed as milligram per 100 gram of OFI peel dried using oven and microwave were represented in figure 17.

The OFI peels dried in oven had a highest value of betalains at 120°C with 93.10  $\pm$  7.49 mg/100g DM, the other temperatures (40°C, 60°C, 80°C and 100°C) shown that there are not a significantly differences at (*p*<0.05) and the lowest value was attributed to sample dried at 40°C with 43.44  $\pm$  1.53 mg/100g DM.

However in microwave drying the power 900W give the greatest content of betalains (p < 0.05) (68.95 ± 2.89 mg/100g DM) which is close with betalains content at 120°C that obtained from dried OFI peels during 0.49 ± 0.08 hand 1.2 ± 0.07 h respectively. Thus, it is interesting to not that microwave drying reduces drastically time of drying, this is also illustrated in the case of drying at 40°C and 700W that resulted in the same betalains contents

 $(43.44 \pm 1.53 \text{ and } 42.67 \pm 3.44 \text{ mg/100g DM})$  respectively, but only at  $0.81 \pm 0.003$  h for 700W and at 7.83  $\pm$  0.76 h for 40°C. (Albano, Negro et *al.* 2015) have reported the same Total betalains in the fruit of OFI 39.3  $\pm$  5.2 mg/100g DM of betacyanins, compared to our result  $32.57 \pm 2.80$ mg/100g DM for the sample dried at 120°C.



**Figure 17:** Total betalains, betaxanthins and betacyanins content of OFI peel dried in oven and microwave.

### III.1.5.4. Antioxidant assays

Several methods have been developed to measure the efficiency of dietary antioxidants. These methods are based on different kinds of difference systems: scavenging reactive oxygen species (ROS), hydroxyl radicals, reduction of lipid peroxyl radicals, inhibition of the lipid peroxidation and chelating of the metal ions (Achat, Tomao et *al.* 2012).

### III.1.5.4.1. Radical scavenging

The DPPH radical is usually used as substrate to evaluate the antioxidative action of antioxidants by determining the free radical-scavaenging ability of various samples (**Achat**, **Tomao et** *al.* **2012**). Figure 18 shows the DPPH<sup>o</sup> radical scavenging activity of different extract of dried OFI peels.

The radical scavenging activity was investigated based on oven-drying and microwave (p < 0.05) as observed in (Fig.18). For the oven drying, where dehydration at high temperatures 100°C and 120°C whith 31.97 ± 0.23% and 70.08 ± 1.20% respectively shows

higher antioxidant activity rather than at low temperatures 40, 60 and 80°C with 24.33  $\pm$  0.49%, 26.48  $\pm$  0.38 % and 29.08  $\pm$  0.75% respectively.

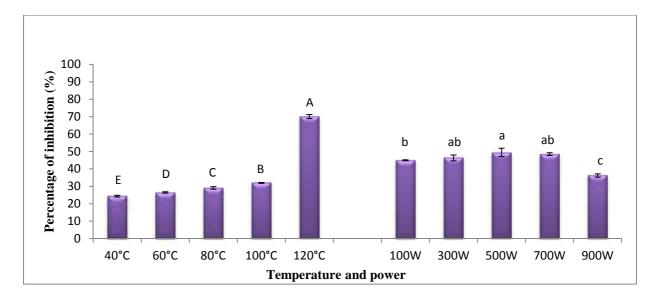


Figure 18: Antiradical activity (DPPH°) of OFI peel dried in oven and microwave.

For the oven drying, the correlation coefficient between TPC and DPPH scavenging activity was found to be weak ( $R^2$ =0.691), indicating that perhaps other phenolic or non-phenolic compounds might be also contributors to the antioxidant activity (**López, Uribe et al. 2010**). For microwave drying, the highest activity was obtained at 500W with 49.46 ± 2.38%, whereas the lowest value was assigned to 900W with 36.19 ± 1%.

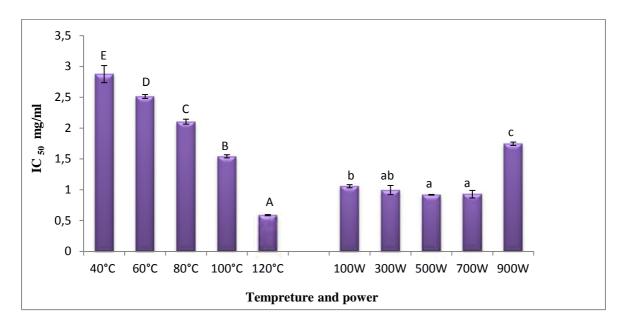


Figure 19: Antiradical activity (IC<sub>50</sub>) of OFI peel dried in oven and microwave.

The IC<sub>50</sub> values (the concentration reducing 50% DPPH) obtained for scavenging activities on DPPH° radical, were evaluated in (**Fig.19**), the lower IC<sub>50</sub> value was the greater free radical-scavenging activity. Thus the strongest activity (p < 0.05) was obtained in case of drying method at 120°C and 500W with (0.58 ± 0.009; 0.91 ± 0.004 mg/ml) respectively. However the sample at 40°C and 900W possessed weaker antioxidant effects (p < 0.05) with (2.88 ± 0.14; 1.74 ± 0.03 mg/ml) respectively.

For the oven drying, this behavior could be related to drying process at low temperatures which implies long drying times that may cause a decrease of antioxidant activity (Garau, Simal et *al.* 2007)

## For the microwave drying did not showed any significant differences at (p < 0.05) between 300W, 500W and 700W ( $0.99 \pm 0.07$ , $0.92 \pm 0.004$ and $0.93 \pm 0.06$ mg/ml) respectively, but we can see that when the power drying increased (100W, 300W and 500W) the antioxidant activity increased too, only at 900W it was decreasing.

This behaviour could be related to the microwave treatment of OFI peel that cleaved and liberated phenolic compounds, hence resulting in the increase of free phenolic compounds and enhancement of antioxidant capacity of the extracts (**Hayat, Zhang et** *al.* **2010**), the decreasing could be explained that at height power the phenolic compounds was distracted than the antioxidants activity was decreased.

The DPPH antioxidant scavenging capacity (IC<sub>50</sub>) of two Tunisian *O.F.I* forms and *O. stricta* fruit extract are ( $0.54 \pm 0.04$ ,  $0.57 \pm 0.02$  mg/ml). (Yeddes, Chérif et *al.* 2013) show the same value than results obtained in this study which is at  $120^{\circ}$ C ( $0.58 \pm 0.01$  mg/ml).

### III.1.5.4.2. Reducing power

The reducing power assay is often used to evaluate the ability of an antioxidant to donate an electron. In reducing power assay, antioxidants cause the reduction of the Fe3+ into Fe2+, thereby changing the solution into various shades from green to blue, depending on the reducing power of the compounds (**Abou-Elella and Ali 2014**).

According to the results obtained from the figure 20, reducing power (RC<sub>50</sub>) of the extracts dried in oven ranged from  $2.75 \pm 0.04$  to  $12.57 \pm 0.70$ mg/ml with a significantly differences at (p < 0.05). In fact, sample dried at 120°C exhibited better activity in reducing the ferric iron (RC<sub>50</sub>:  $2.75 \pm 0.04$  mg/ml) followed by 100°C with  $5.07 \pm 0.05$  mg/ml, 80°C with  $6.30 \pm 0.06$  mg/ml ,60°C with 10.04 ± 0.34 mg/ml and finally the lowest was for the temperature of 40°C with 12.57 ± 0.70 mg/ml. These findings indicate that the antioxidant activity is well correlated with the amount of phenolics constituent found in the extract. Therefore, phenolic

compounds of OFI peels are good electron donors and could terminate the radical chain reaction by converting free radical to more stable products (**Abou-Elella and Ali 2014**).

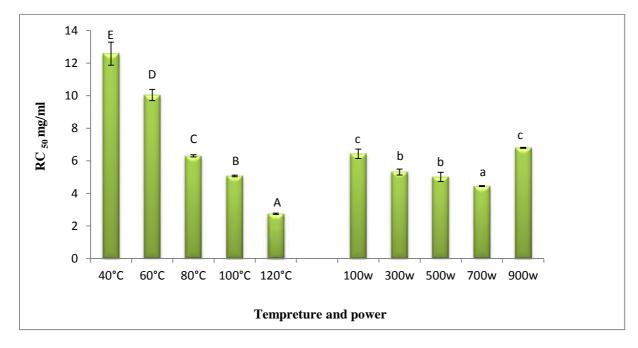


Figure 20: Reducing power (RC<sub>50</sub>) of OFI peel dried in oven and microwave.

For the microwave drying, we show that the OFI peel extracts exhibited higher reducing power at 700W with RC<sub>50</sub>:  $4.45 \pm 0.03$  mg/ml followed by 500W with  $5.01 \pm 0.28$  mg/ml then 300W ( $5.31 \pm 0.17$  mg/ml), 100W ( $6.432 \pm 0.29$  mg/ml) and the lowest activity was obtained from the sample dried at 900W ( $6.8 \pm 0.03$  mg/ml).

This results obtained by reducing power are in good agreement with those obtained by the DPPH° test namely for the temperature of 120°C and power of 500W and 700W. These extracts showed the strongest radical scavenging activity, with the best reducing power.

### III.2. Juice

Results corresponding to moisture, pH and acidity of cactus pear fruits were summarized in table 11.

| Test  | Moisture       | pН          | Brix                    | Titrable acidity              |
|-------|----------------|-------------|-------------------------|-------------------------------|
| Value | 85,73 ± 0,23 % | 6.11 ± 0.11 | $13.5 \pm 0.00^{\circ}$ | $0.055 \pm 0.004 \; g/100 ml$ |

 Table 11: Result of physicochemical analysis of cactus juicy pulp.

### - Humidity

Result indicated higher moisture in the juicy pulp ( $\approx 86 \pm 0.23$  %). This value is in agreement with those previously reported. Indeed, similar data was found by (**Chougui**, **Tamendjari et** *al.* **2013**) 85,22 %, the moisture content in cactus pear pulp was 85.32 ± 0.03 % found by (**Saénz, Tapia et** *al.* **2009**), and (**Roghelia and Panchal**) imported a % of 84 to 87 in moisture content.

### - pH

The pH value the of orange pulp of OFI was  $6.11 \pm 0.11$ , thus it is classified as a low acid fruit (pH > 4.5) (Yahia and Mondragon-Jacobo 2011). This result is in accordance with those recorded by (Medina, Rodríguez et *al.* 2007); and others (El Gharras, Hasib et *al.* 2008; Saénz, Tapia et *al.* 2009) shown a pH value of 6.32, 5.63 respectively.

Pigments (betalains) of cactus pear are stable in this pH. Indeed betalains are soluble in water, and their stability is less affected by pH than anthocyanins, another class of natural red-purple pigments used in foods. They are relatively stable at pHs ranging from 3.0 and 7.0, which allows them to be used in low acid and pH neutral foods, such as dairy products (**Yahia and Mondragon-Jacobo 2011**).

### - Brix degrees

Sugar content is an important criterion of fruit quality for consumers who prefer sweet fruit. The °Bx for OFI juice was  $13.5 \pm 0.00$  °Brix which was higher than the other cultivars 12.53  $\pm 0.01$ ). these results were in agreement with the results published by (**Tlili, El-Guizani et** *al.* **2011**), and the °brix of orange variety of prickly pear fruit obtained by (**Albano, Negro et** *al.* **2015**) was  $12.10 \pm 0.14$  °Brix. The degres brix observed in an orange prickly pear within the specie O. ficus indica was  $14.05 \pm 1.87$  °Brix (**Medina, Rodríguez et** *al.* **2007**).

### - Titrable acidity

The acidity middle of juice analyzed expressed in citric acid is of  $0,055 \pm 0.004$  g /100 ml, this was a very close value to the result given by (Nadia, Hayette et *al.* 2013) (0.04 to 0.07g/100 ml). The acidity of the studied fruit juice, is also in good agreement with data of Mexican and Chilean cultivars (Medina, Rodríguez et *al.* 2007).

Because of his weak acidity and his elevated pH, the barbarism fig is classified in the category of the food products weakly acidic (pH> 4,5), what confers him the faculty to undergo thermal treatments to high temperature (passing 115°C) for the industrial valorization (**Saénz**, **Tapia et** *al.* **2009**).

### - Antioxidants contents

Table 12 depicts total bioactive substances (phénolics, flavonoids and betalains), in jucy pulp of priklely pears with antioxidant capacities.

Table 12: Contents of total phenolics, flavonoïd and betalains, in OFI juice.

| Results            |
|--------------------|
| $89,088 \pm 0,686$ |
| $2,\!816\pm0,\!2$  |
| $1.241\pm0.09$     |
| $7.216\pm0.417$    |
| $158.17\pm3.67$    |
| $173.70\pm5.006$   |
|                    |

### - Total phenolic and flavonoïd contents

The total phenolic contents found in fruit extracts was  $89,088 \pm 0,686$  (mg /100g FM), it's approximately similar to that reported by (Albano, Negro et *al.* 2015):  $89.2 \pm 3.6$  mg/100 g and lower than data detected by (Medina, Rodríguez et *al.* 2007; Mabrouki, Zougari et *al.* 2015):  $45.2 \pm 7.4$  mg GAE/100 g;  $54.33 \pm 2.51$  mg GAE/100 g respectively. However this amount is very higher than the polyphenols content of red juice:  $15.34 \pm 0.73$  mg Catechin/ kg of (Khatabi, Hanine et *al.* 2016).

Concerning flavonoids, (Ndhlala, Kasiyamhuru et *al.* 2007; Chougui, Tamendjari et *al.* 2013) found higher values: 25 mg/100 g and  $3.83 \pm 0.36 \text{ mg}$  GAE/100g respectively.

### - Betalains

The quantities measured in the studied juice, are expressed in fresh matter mg/100g. The middle content in betacyanin was  $(1.241 \pm 0.09 \text{ mg}/100\text{ g FM})$ , higher than result obtained by (Nadia, Hayette et al. 2013) (0.90 mg/100g), and lower to result followed by (Butera, Tesoriere et al. 2002) (2.61 mg/100g). However the quantity recorded to betaxanthin (7.216  $\pm$  0.417 mg/100g) was superior to the one returned by (Nadia, Hayette et al. 2013) (6.79 mg/100g) and lower than result of (Butera, Tesoriere et al. 2002) (5.12 mg/100g). (Khatabi, Hanine et al. 2016) found that the average content of the juice of betaxanthin Prickly red analyzed is about 4.587  $\pm$  0.0123 mg/100g of juice, which was higher than our results. These

differences can be explained presumably by period of conservation of our juice, the moment of the harvest or by the local character of the environment of the plant.

### -Antioxidant and antiradical activities

### 1. Scavenging effect on DPPH and reducing power

The ability of the samples to donate hydrogen was checked by using the free radical DPPH•. It is one of the known mechanisms by which antioxidants inhibit lipid peroxidation. The amount of sample needed to decrease the initial DPPH• concentration by 50% (IC<sub>50</sub>) is a parameter widely used to measure the antioxidant activity (**Nadia, Hayette et al. 2013**). The antioxidant activity of OFI fruit(juicy pulp) are show in Table 13.

Table 13: Inhibition of DPPH and reducing power of juicy pulp.

|            | IC <sub>50</sub> g/ml | RC50 g/ml      |
|------------|-----------------------|----------------|
| Juicy pulp | $0.15\pm0.004$        | $0.17\pm0.005$ |

Our result show DPPH - scavenging activity (IC<sub>50</sub>:  $0.15 \pm 0.004$  g/ml FM) lower than result obtained by (**Chougui et al. 2013**) ( $0.26\pm0.01$  g/ml FM). However, a reducing power activity was (RC<sub>50</sub>:  $0.17 \pm 0$  mg/ml FM) which is higher than result followed by (**Chougui et al. 2013**) ( $0.05 \pm 0.00$  g/ml FM).

### III.3. Analysis of prepared stirred yoghurt and milky juice

### **III.3.1.** Physico-chemical analysis of yoghurt

Physico-chemical properties of the manufactured yoghurts (plain flavored yoghurt, plain stirred yoghurt, flavored yoghurt and stirred yoghurt with PJ) and milky juice (plain milky juice and Milky juice with FE) were shown in table 14 and table 15. Results of prepared yogurt (**Tab.14**) revealed that pH, soluble solids, content acidity and protein content determination were conforms to norms. However an increase in total dry extracts, Fat content and viscosity were observed after addition of juicy pulp to yoghurt. On the one hand, this may be related to chemical composition of juicy pulp (protein: 0.21-1.26 %; fat 0.7 %) (Özcan, Haciseferoğulları et *al.* 2005), on the other hand to its impact on the aggregation of casein network in yoghurts via electrostatic interaction and on the resistance for the yoghurt matrix to flow. Indeed the addition of plant extracts generally decreased the consistency of the products owning to reduced waterbinding capacity of proteins (**El-Salid, Nagib et** *al.* **2011).** 

|                           | рН       | Acidity<br>(°D) | Viscosity<br>(CP) | Total dry<br>extract<br>(%) | Fat<br>content<br>(%) | Protein<br>content<br>(%) |
|---------------------------|----------|-----------------|-------------------|-----------------------------|-----------------------|---------------------------|
| plain flavored<br>yoghurt | 4.53     | 80              | 30200             | 20.41                       | 3.2                   | 2.90                      |
| Flavored yoghurt with JP  | 4.48     | 83.3            | 29800             | 21.97                       | 3.4                   | 3.15                      |
| plain stirred<br>yoghurt  | 4.45     | 75              | 24600             | 23.6                        | 2.75                  | 2.62                      |
| Stirred yoghurt with JP   | 4.30     | 79              | 23500             | 24.05                       | 3.02                  | 2.80                      |
| Norms                     | 4.4 -5.7 | 75 -100         |                   | 23.9 -<br>25.15             | 2.75-<br>3.15         | 2.85-<br>3.15             |

Table 14: Physicochemical analysis of yoghurts.

JP: juicy pulp.

Results of milky juice (**Tab.15**), shown that the acidity, brix and viscosity were higher in milky juice with PEP than the standard milky juice; this difference can be related to the composition of PEP and the preparation of recipe.

**Table 15:** Physicochemical analysis of milky juices.

|                       | Acidity (%) | Brix° | Viscosity (CP) |
|-----------------------|-------------|-------|----------------|
| plain milky juice     | 0,3         | 11.58 | 13.5           |
| Milky juice whith PEP | 0,42        | 13.2  | 15.6           |

PEP: Phenolc extract peel.

### **III.3.2.** Microbiological analysis

Microbial quality of the manufactured of dairy products was given in table 16.

Results shown that moulds, yeast and coliforms, are the primary contaminants in yoghurt (**Amakoromo, Innocent-Adiele et al. 2012**), were not detected in yoghurt samples (yogurts with JPBF and plains) and milky juice, were the same results for *Staphilococus aureus* and *Salmonella*. This illustrates the adequate heating treatment of milk under strict aseptic conditions during processing and manufacturing of the different yoghurts and juice.

Yoghurs (favored yoghurt and stirred yoghurt) enriched with PJBF presented a slight increase in viability of lactic acid bacteria Flora (*Streptococcus thermophilus* and *Lactobacillus bulgaricus*) when compared with plains yoghurt, which could be related with the composition of proliferation media (sugar and lipid) of samples after addition of pulp juice. The total viable numbers of lactic flora is an important parameter which contributes in the shelf life of yoghurt. This can be related to the chemical composition of the OFI pulp juice 0.21-1.06 protein, 0.02-3.15 % fibre, 0.7 % fat, and 12-17 % various of carbohydrates and vitamins (K1 and C) (**Feugang, Konarski et al. 2006**).

|                           | Total coliforms | Yeasts<br>and<br>moulds | Streptococcu<br>s<br>thermophilu<br>s | Lactobacill<br>us<br>bulgaricus | Staphilauc<br>ocus<br>aureus | Salmonelle<br>s |
|---------------------------|-----------------|-------------------------|---------------------------------------|---------------------------------|------------------------------|-----------------|
| plain flavored<br>yoghurt | Absent          | Absent                  | 1.6 ×10 <sup>8</sup>                  | 1.2×10 <sup>5</sup>             | Absent                       | Absent          |
| plain stirred<br>yoghurt  | Absent          | Absent                  | 1.6 ×10 <sup>8</sup>                  | 1.2×10 <sup>5</sup>             | Absent                       | Absent          |
| Flavored yoghurt with JP  | Absent          | Absent                  | 9×10 <sup>8</sup>                     | 1.7×10 <sup>7</sup>             | Absent                       | Absent          |
| Stirred yoghurt with JP   | Absent          | Absent                  | 1.7×10 <sup>8</sup>                   | 1.4×10 <sup>5</sup>             | Absent                       | Absent          |
| plain milky<br>juice      | Absent          | Absent                  | -                                     | -                               | Absent                       | Absent          |
| Milky juice<br>with PEP   | Absent          | Absent                  | -                                     | -                               | Absent                       | Absent          |
| Norms                     | < 1 ufc/g       | Absent                  | $\geq 10^{7}$                         | ≥10 <sup>7</sup>                | Absent                       | Absent          |

 Table 16: Microbiological analysis of dairy products.

JP: Juicy pulp, PEP: Phenolc extract peel.

### III.3.3. Antioxidant activity, total phenolic compounds and betalains:

The antioxidant activity, total phenolic compounds and total betalains of yogurts and milky juice were given in table 17.

It was noticed from table 17 that antioxidant activity of different yoghurts and milky juice revealed that the addition of PJ for the yoghurts and PEP for the milky juice increased significantly the inhibitory activity against DPPH° radical compared with plains yoghurt and plains milky juice.

The JP and PEP contained total phenolic compounds and betalains these compounds were present and significantly highest in supplemented yoghurts and milky juice. In the following order flavored yoghurt with JP > PFY, stirred yoghurt with JP > PSY and milky juice with PEP>PMJ Providing a confirmation of supplementation.

|                          | Total phenolic<br>compound mg GAE<br>/100g | Radical scavenging<br>activity (%) | Total betalains (mg<br>/100g ) |
|--------------------------|--|------------------------------------|--------------------------------|
| Plain flavored yoghurt   | $1.23\pm0.03^{b}$                          | $35.86\pm0.50^b$                   | 0.026±0.0007 <sup>b</sup>      |
| Flavored yoghurt with JP | $1.38\pm0.03^{a}$                          | $46.28\pm0.50^{\text{a}}$          | $0.047\pm0.00^{a}$             |
| Plain stirred yoghurt    | $1.16 \pm 0.02^{b'}$                       | $33.92\pm1^{b'}$                   | $0.028 \pm 0.001^{b^{\prime}}$ |
| Stirred yoghurt with JP  | $1.30 \pm 0.01^{a'}$                       | $42.84 \pm 0.39^{a'}$              | $0.074 \pm 0.006^{a^{\prime}}$ |
| Plain milky juice        | $13.04 \pm 0.15^{b^{''}}$                  | $11.96 \pm 1.09^{b''}$             | $6.86 \pm 0.01^{b''}$          |
| Milky juice with PEP     | $85.16 \pm 3.74^{a''}$                     | $44.11 \pm 0.13^{a''}$             | $11.61 \pm 0.13^{a''}$         |

**Table 17:** Radical scavenging activity, total phenolic compound and total betalains content of prepared yoghurts and milky juice.

Values with different letters (a-b-a'-b'-a''-b'') were significantly different (p < 0.05). JP: Juicy pulp, PEP: Phenolc extract peel.

### Conclusion

### Conclusion

In the present work, the microwave drying was successfully employed to the maximum elimination of humidity from OFI peels comparing to oven drying. The moisture loss of samples using oven at 40 °C was significatively higher (min) than drying time in microwave at 900 W (min). The reduction in the drying time was linked to the improvement in both mass transfer coefficient and the effective moisture diffusivity. Results of the water activities of *OFI* powders were in the range of 0.17–0.47, so relatively stable microbiologically using the both methods of drying (oven and microwave). The color (L\*a\*b\*, values) of prickly pear peels powders was different: drying at 100 W revealed optimum color values, however at 120 °C (oven drying) and at 900 W (microwave drying) developed the least acceptable color values, due to the effect of high temperatures.

The preliminary study gave a satisfactory description in order to select the suitable extraction method which may maximize the yield of TPC, flavonoids, betalains and antioxidant activity of prickly pear peels, showing that UAE method appeared to be better than MAE and CS, CM (p<0.05), allowing for higher recovery yield and specific antioxidant activity (76.6 ± 1.13 % (100 W), 51.91 ± 2.84 % (40 °C) at p <0.05).

This study indicates that prickly pear peels can be considered as a good source of antioxidants. The results of these bioactive components determination for OFI peels, dried with oven and microwave, were statistically different (p < 0.05). In oven drying, the temperature of 120 °C provides a highest recovery of TPC (2734.28±178.15 mg GAE/100 g DM), total betalains 93.10±7.49 mg/100 g DM. Even in the antioxidant assays 120°C extracts revealed better activities: radical scavenging test DPPH• (IC<sub>50</sub>= 0.58 ± 0.01 mg/ml), reducing power (RC<sub>50</sub> = 2.75 ± 0.04 mg/ml). Whereas, TFC shown the highest value at 80°C (815.30 QE mg/100 g DM). However with the microwave drying technique, powder extract obtained at 100W was the best power in terms of extraction of TPC, but lower than conventional oven drying (2556.38 mg GAE/100g DM), the extraction of TFC with maximal amount was attributed to 500W (984.46 ± 34.32 mg/100 g). The power of 900W provides a highest recovery of total betalains (68.95 ± 2.89 mg/100g). Data of assessment of antioxidant activities of 500 W showed the same tendencies of the results obtained in 120 °C.

On the other hand, the juicy pulp was characterized by a high humidity ( $85.73 \pm 0.23$  %) high pH ( $6.11 \pm 0.11$ ), low acidity ( $0.055 \pm 0.004$  g/100ml), and high amount of °Bx ( $13.5 \pm 0.00$ ). The quantification of the antioxidants in the pulp juice presented high yields in TPC ( $89,088 \pm 0,686$  GAE mg /100 g DM) and pigments (betacyanins  $1.241\pm0.09$  mg/100g FM, betaxanthins  $7.216\pm0.417$  mg/100g FM), with an antioxidant capacity (IC<sub>50</sub>:  $158.17\pm3.67$  mg/ml and RC<sub>50</sub>:  $173.70\pm5.006$  mg/ml).

The addition of juicy pulp and phenolic extract peels of OFI in the dairy products (yoghurts and milky juice), could offer practical and economic sources of betalains and antioxidants in dairy industry.

Commercialization of cactus pears based on their antioxidant properties can generate competitive advantages, and these can turn into business opportunities and the development of new products and a high-value ingredient for the food industry. Proper utilization of OFI peels (by-products) could reduce waste disposal problems and serve as a potential of new natural source of bioactive compounds.

Our study suggested that prickly pear peels and pulps are rich in polyphenols and present high antioxidant activity effects, it would be necessary to support it by:

- ✓ Introduction of the culture of barbarism fig in the development projects of the agriculture ministry.
- ✓ Its utilization as novel natural antioxidants for use in functional foods like a fresh juice, fruit yoghurt, or medicine.
- ✓ Ethno-botanical study for a good determination and classification of species and existing varieties in Algeria.
- $\checkmark$  Extraction of pectin fiber from the peels.
- ✓ Assessment of sensory analyses of dairy product.

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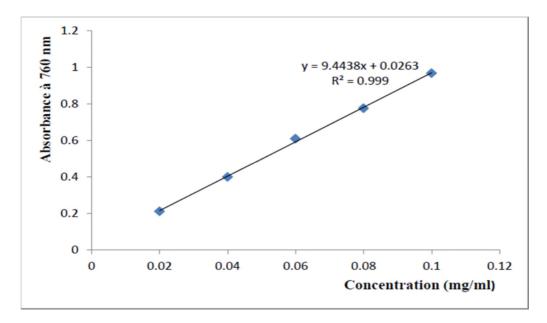
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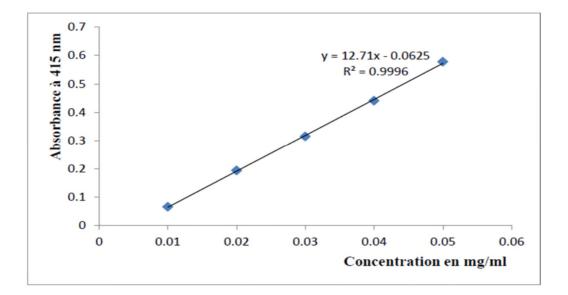
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Appendix



Appendix 01: Calibration courbe of gallic acid.



Appendix 02: Calibration courbe of quercitin.

### Résumé

L'objectif de ce travail est l'étude de l'effet de séchage des sous-produits (pelure) de la figue de barbarie (opuntia ficus indica), en utilisant deux méthodes de séchage: micro-onde (100, 300, 500, 700 et 900 W) et étuve (40, 60, 80, 100 et 120 ° C). Ainsi, la cinétique du séchage a été effectuée conformément à la perte de masse de la pelure de la figue de barbarie. Pour chaque technique, l'analyse physico-chimique (teneur en humidité, test de couleur, la détermination des composés phénoliques et de l'activité anti-oxydante) des poudres a été évaluée. Microonde a réduit significativement les temps de séchage comparativement au séchage conventionnel de l'étuve. Les résultats de la détermination des composants bioactifs de la pelure de la figue de barbarie, séchée à l'étuve et au micro-onde, ont été statistiquement différents (p < 0.05). Dans le séchage à l'étuve, la température de 120 °C a donné le taux le plus élevé en polyphénols, mais avec la technique micro-onde, l'extrait de poudre obtenue à 100 W a enregistré la meilleure puissance en terme d'extraction de composés phénoliques, mais qui reste inférieur au séchage de l'étuve. Les données d'activité antioxydante des échantillons séchés à 900 W et 120 ° C, ont montré les mêmes tendances qu'aux résultats des teneurs en polyphénols. Le jus pulpeux a été caractérisée par une haute humidité, haut pH, basse acidité, la quantification des antioxydants suivi par le jus pulpeux a présenté de plus hauts rendements dans les TPC (89,09  $\pm$  GAE /100g FM), betalains (8,46  $\pm$  0.50 mg /100g FM) et aussi activité antioxydante. Donc, yaourt avec jus pulpeux ajoutée et jus lacté avec extrait phénolique, a augmenté considérablement (p < 0.05) l'activité inhibitrice contre radical DPPH° et le contenu en betalains comparé avec le standard des produits laitiers préparé.

**Mots-clés:** *Opuntia ficus indica*, Micro-onde, Etuve, Séchage, Polyphenols, essais Antioxydants, Yaourt, produits laitiers.

### Abstract

The aim of this study was to investigate the drying effect of by product (peels) of Prickly pear (Opuntia ficus indica), using two drying methods: microwave (100, 300, 500, 700 and 900 W) and ventilated oven (40, 60, 80, 100 and 120 °C) and the valorisation of Prickly pear juice. Thus, kinetic drying was performed according to the mass loss of Prickly pear peel. For each technique, the physico-chemical analysis (moisture content, color test, determination of phenolic compounds, betalians and the antioxidant activity) of the powders were evaluated. Microwave provided significantly shorter drying time than conventional oven drying. The results of bioactive components determination for Prickly pear peels, dried with oven and microwave, were statistically different (p < 0.05). In oven drying, the temperature of 120 °C provides a highest recovery of polyphenols, betalians, however with the microwave technique, powder extract obtained at 100 W was the best power in terms extraction of phenolic compounds but lower than oven drying. The data of antioxidant activities of dried samples at 900 W and 120 °C showed the same tendencies of the results obtained in polyphenols. The juicy pulp was characterized by a high humidity, high pH, low acidity, the quantification of the antioxidants followed by the juicy pulp presented higher yields in TPC  $(89,09 \pm 0.69 \text{ mg GAE}/100 \text{g FM})$ , betalains  $(8,46 \pm 0.50 \text{ mg}/100 \text{g FM})$  and also antioxidant activity. Thus, yoghurt with added juicy pulp and milky juice with phenolic extract, increased significantly (p < 0.05) the inhibitory activity against DPPH<sup>o</sup> radical and betalains contents compared with standard prepared milk products.

**Key words**: *Opuntia ficus indica*, Microwave, Oven, Drying, Prickly pear peels, Polyphenols, Antioxidant assays, Milk products.