



The Preservation of *Thymus satureioides* by convective solar dryer

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Abstract

The medicinal and aromatic plants (MAP) are widely used not only for culinary application but also in traditional medicine. It allows an important economic value-added. However, the dreadful quality management in manufacturing phases and the multiplication of actors and intermediaries raise risks to the hygiene, safety and health by microbiological, physical, and chemical contamination. Faced up to these failures and increasing customer demands, manufacturers face the challenge of ensuring the safety of products, reducing manufacturing costs and respecting the environment. Moreover, it's now widely accepted that solar energy provides valuable energy. The industrial integrated it in conservation in order to preserve and store the seasonal plants and make them available to consumers all year round. Our approach is curative. We proposed a nondestructive innovative preservation process integrating sustainable energy. It is a thermobiochemical process for Eco-conservation of commercialized thyme "Thymus satureioides" contaminated by *Escherichia coli* (E. Coli). It is based on a pretreatment with a natural organic acid followed by thermal drying by convective dryer using solar energy. The modeling of the process parameters and microbiological analysis has proven the effectiveness of this process against E. coli. Similarly, the color and the total phenol content of treated thyme remain intact compared to the witness thyme. Therefore, the thermobiochemical treatment studied may be used in industry not only for drying plants, but also to preserve and promote the hygienic quality level.

Keywords: Solar energy, Convective dryer, Conservation, *Thymus satureioides*, Quality.

1. Introduction

Nowadays, the food safety developments joined the spirit of conservation processes improvement [1-2]. However, problems related to the contamination by microorganisms such as *Escherichia coli*, *Salmonella* and *Clostridium perfringens*, in addition to the vulnerability of aromatic plants and their compounds degradation alongside existing processes makes this task more difficult [3-5]. Moreover, the use of the developed process in the industry as the gamma radiation is very encouraging but expensive [2]. Hence, small and medium company in Morocco opted to bulk marketing whose relevance was questionable.

Conventional drying processes are not suitable for plant conservation. They may yield a less quality product and affect its characteristics. Establishing a conservation approach taking into account the various factors (health safety, sensory quality, chemical and cost) is critical. Moreover, the high quantity of energy needed for conservations process thrust industry to develop methods based on solar energy. Several, researches had been developed by means of solar energy as source for drying and conserving food products [6-7].

The relevance of our eco-process lies precisely in our overall approach to conservation, packaging and quality through the use of solar energy. The aim of our study is the experimental determination of the drying kinetics of preserved *Thymus satureioides* by thermo-biochemical processing and the identification of the process parameters (temperature and air flow, concentration and volume citric acid) for the better conservation. We used a thin layer

solar dryer partially operating in forced convection. The influence of temperature, air flow and conservative on the drying rate of these products is investigated. The experimental results are used to evaluate the effect of these parameters on measurable quality indicators such as the debacterization of thyme contaminated with *Escherichia coli* as sign of food sanitary quality [8], change in color and phenolic compounds.

2. Materials and methods

2.1. Sample preparation and conservation process

Thymus saturoioides Coss. was collected and identified from Marrakech region Morocco. The herbs were dried under ambient conditions and packed under vacuum packaging in high density polyethylene (HDPE) bags.

The drying kinetics analysis is conducted at the beginning to determine the drying time for each parameter duration-air rate- concentration. The samples were distributed uniformly as a thin layer onto the stainless steel trays inside the drying chamber (Figure 1) [9]. The change in the product moist mass throughout time $M_m(t)$ is determined by the readings on a precision balance ± 0.001 g each 5 minutes.

At the end of the experiment, the dry mass M_d was determined by drying the sample at 105 °C for 24 hours. The water content $X(t)$ of the product at an instant t is calculated by equation (1):

$$X(t) = \frac{M_m(t) - M_d}{M_d} \quad (1)$$



Figure 1: Convective solar dryer (LISPAM, ENS Marrakech)

Then, a batch of samples was intentionally contaminated with *Escherichia coli* (SCTC471) with a dose of about 10^6 UFC determined by colony counting equipment [10]. This lot was split into four lots, a control group and three batches were sprayed with distilled water and citric acid at 0.25% and 0.5%. At the end of drying, each sample is packed under vacuum.

2.1. Quality analysis

a) Microbiological analysis:

The enumeration of *E. coli* was performed on MacConkey agar medium and incubated at 44 ° C. After 24 hours of incubation, we counted the number of red colonies [11-12].

b) Color intensity

The powdered plant (2 g) was extracted with 20 ml of acetone at room temperature for 24h under magnetic stirring in the dark. The extract is filtered and adjusted to 50ml with acetone / water 80% (v/v) and then diluted 1:20. The Spectrum of chlorophyll extract was evaluated by UV-visible spectrophotometer UV-2550. 3 extractions are carried out per sample.

c) Phenolic contents extraction and measure

100 mg of ground thyme sample is mixed with cold methanol 4°C (80% v/v). The resulting product is centrifuged (ROTANTA 460R) at a speed of 4600 RPM for 30 min at 4 ° C. The supernatant is recovered and the extraction process is repeated three times on the respective residues to ensure complete extraction of phenol compounds. The supernatants obtained were piled up and stored at -20 ° C until use.

Total phenolic content (TPC) was measured using the Folin– Ciocalteu FC colorimetric method [13]. The extracted phenolic material 50 µl was mixed with 1.7ml of distilled water and 0.25ml of FC reagent. After

rigorous agitation, 0.5ml of sodium carbonate solution was added and the mixture was immediately incubated at 40°C during 30 min. Absorbance at 765nm was then recorded for the mixture, this was performed in triplicate with each sample. The total phenolic content was expressed as catechin equivalent (in mg) per 100 g of dry plant.

d) CPG-MS Analysis:

The plant was placed in contact with a cooled hexane at 4 °C under magnetic stirring for a few minutes. The extract was filtered through the filter paper followed by the evaporation of the solvent.

The Gas chromatography/mass spectrometry (GC/MS) analyses were carried out using a gas chromatograph coupled with Mass Spectrophotometer (MS)(GC–MS Trace GC ultra – ITQ900, ThermoScientific, USA) operating in electron- impact mode (70eV, m/z 40–450). The capillary column used was 1MS (30m_0.25 mm_0.25 mm film thickness). Injector and transfer line temperatures were 250 and 300 °C, respectively. The oven temperature was programmed from 50 to 200°C at 10°C/min and from 200 to 290°C at 35 °C/min, carrier gas helium, at 1mL/min, injection of 1 ml (10%hexanesolution), and the split ratio was1:20. The identification of the compounds was performed by comparing their mass spectra with data bank (NISTMS searchV.2.0) and homemade library mass spectra built up from pure substances and components of known essential oils and MS literature data and by co-injection with an authentic sample [14].

3. Results and discussion

3.1. Drying kinetic

Thyme drying kinetic was studied using a solar dryer operated in forced convection. The purpose of this study is to understand the mechanisms affected the treatment duration. Drying experiments were performed for four drying air temperatures (50, 60, 70 and 80 ±0.1 ° C) and two air flow rates (150 and 300 ± 0.002 m³/ h). Thus, the experimental curves describing the evolution of the drying time as a function of drying and treatment parameters are shown respectively in Figures 2 and 3.

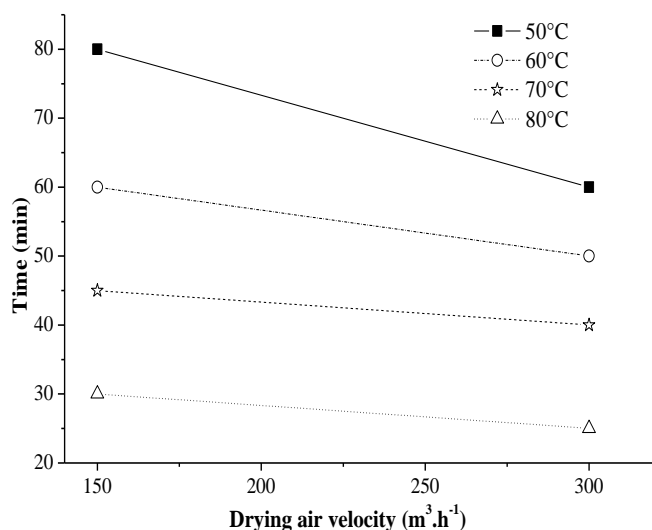


Figure 2: Drying time function of temperatures and drying air velocity for treated thyme (0.25% citric acid)

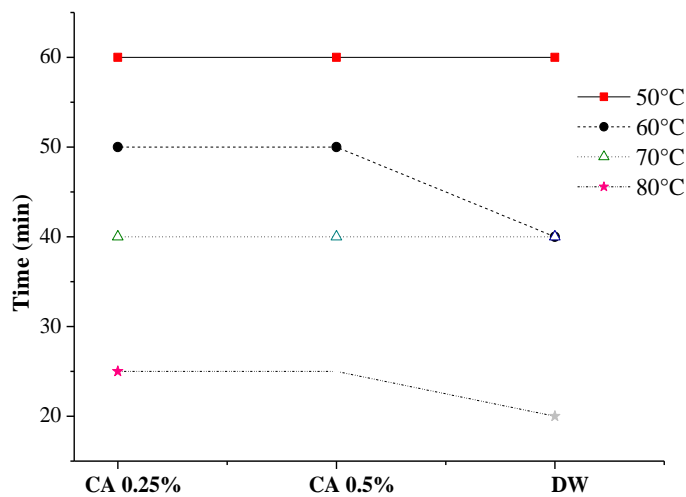


Figure 3: Drying time function of treatment “Distilled water DW and citric acid CA at 0.25% and 0.5%” at different temperatures

The effects of the temperature and flow on the drying process are remarkable. Drying time decreased with the increase of the drying temperature and the air flow. The diffusion process of water through the dry outer layers to the surface is influenced by high temperatures. Also, the increase of air rate boosts contact with the product which allows faster elimination of water. The same results were obtained for various plant products [15-17].

The drying speed is governed by the diffusion of water in the solid. This fact could be influenced by the use of citric acid which create links with water and compounds of the product.

3.2. *Quality analysis*

a) Microbiological analysis :

The drying is a hopeful method for the preservation of food products. In fact, it trimmed down their water content until microbial spoilage and deterioration reactions are minimized [5]. Drying influenced the microbiological quality of thyme in concert with the temperature and the citric acid concentration (Table 1).

Drying at 50°C led a significant bacterial elimination of 98% and 99% respectively with a citric acid concentration of 0.25% and 0.5%, but it is not absolute. Otherwise, spraying the sample contaminated with citric acid 0.25% followed by drying at 80 °C for 30 min showed a significant efficiency of debacterization of approximately 6 logarithmic units. However, the exclusion of citric acid allows the growth of mold after 15 days of storage.

Table 1: Effect of pretreatment combined to drying on the microbiological quality of thyme

Initial bacterial load (Witness)	Debacterization rate				
	T (°C)	Drying	Drying after pulverization of		
			Distilled water	Citric acid at 0.25%	Citric acid at 0.50%
1.4 10 ⁶	50	81%	84%	98%	99%
	80	87%	98%	100%	100%

The combined pretreatment with drying affects the rate of microorganisms destruction. This process, allows first of all a choc to microorganisms due to the use of citric acid. Then, drying raises the effect by decreasing the available amount of water necessary for the bacterial growth. Similar results were discovered. Chkir et al (2015) showed the effect of drying on stability of cactus/brewer’s grains mixture. This research clarified that the increase of temperature from 40 to 60°C at the air velocity 1.0 m/s reduced considerably the number of lactic acid bacteria, mesophilic total flora, and yeasts and molds. This effect was justified by the effect of drying on cells composition. Indeed, drying changes the physical state of lipids membrane and causes important conformational changes of proteins inducing their denaturation and loss of their biological activity. The temperature rise also promotes chemical reactions leading to the formation of free radicals which cause damage to the cell membrane. It can also affect various molecular processes such as replication, transcription and protein synthesis, and inducing a dysfunction of specific enzymes leading to a disruption of the cell equilibrium [18].

b) Color intensity

The effect of drying process can be evaluated by the plant color as a quality indicator due to the relation between the color amount and the structure of the pigment. Higher temperature could heave color deteriorations and burning of the product. The obtained spectrum put on show peaks at around 337nm and 672 nm, which match up the presence of chlorophyll a, b and β-carotene reported in thyme [1]. The obtained spectrum of the pigments extracted from witness and treated thyme demonstrated no dissimilarity between them. Hence, the process used does not improve an impact on the degradation of total pigments.

c) Phenolic contents extraction and measure

Usually, drying temperature influences essential oil quality and quantity in medicinal and aromatic plants not only during drying but also the reduction continues during the storage period. Hence, the total phenolic content was studied by Spectrophotometry during the storage of witness and treated thyme (Figure 4).

We observe that the treated thyme shows higher concentration than the witness thyme during storage. The TPC of witness thyme stills stable because of packing under vacuum. Moreover, the treated thyme with distilled water presents an increase of TPC during storage but after three weeks this concentration decrease until it became stable. However, the treated thyme with citric acid shows a boost of TPC until the concentration became stable after 60 days.

This effect can be explained by the activation of the L-Phenylalanine ammonia-lyase PAL enzymatic activation, responsible of setting-up phenolic compounds.

An increase in drying temperature has an important effect on the total phenolic content. Vega-Galvez et al. (2009) [19] investigated the effect of drying temperature on phenolic content and antioxidant capacity of red pepper. This research showed that products dehydration at high temperatures (80 and 90°C) shows higher

antioxidant activity rather than at low temperatures (i.e. 50, 60 and 70°C). This behavior was related to drying process at low temperatures, which implies long drying times that may promote a decrease of antioxidant capacity. Moreover, the formation of phenolic compounds at high temperatures might be because of the availability of precursors of phenolic molecules by non-enzymatic interconversion between phenolic molecules. Arslan et al (2010) demonstrated the effect of drying temperature on TPC in function of drying processes [20]. They related the increase in total phenolics to the liberation of phenolic compounds from the matrix during the process. Also, drying might have accelerated more bound phenolic compounds releasing from the breakdown of cellular constituents.

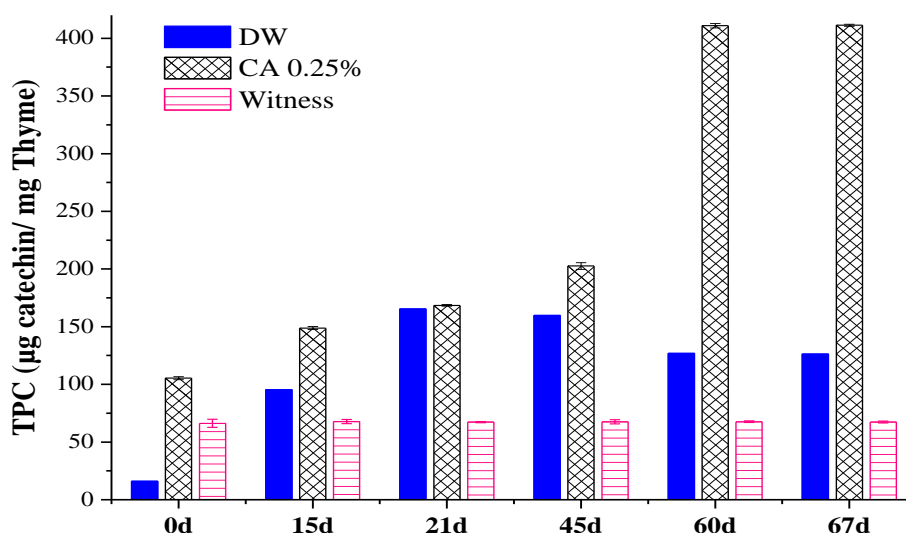


Figure 4: Effect of the treatment on Total phenolic compound during storage

d) CPG-MS Analysis:

The essential oils compositions of thyme were studied by Gas chromatography/mass spectrometry (GC/MS) before and after treatment. The results illustrated the presence of fourteen compounds that were identified by mass spectrometry (Table 2). The analyze shows an increase of borneol and terpeniol and a decrease of carvacrol (Table 2). This modification may be justified by the L-Phenylalanine ammonia-lyase PAL enzymatic activation [21].

Table 2: Thyme composition before and after treatment

TR	Compound	Witness	Treated
5.75	α -Pinene	0.12	0.12
6.00	Camphene	0.28	0.26
7.13	<i>p</i> -cymene	0.21	0.17
7.65	γ -Terpinene	0.12	0.09
7.80	(E)-Sabinene hydrate	0.08	0.04
8.23	Linalol	0.43	0.27
9.03	Camphor	1.74	0.48
9.38	Borneol	9.89	44.21
9.68	α -Terpineol	1.87	6.16
10.08	Bornyl acetate	0.01	1.30
10.50	NI	10.30	0.13
11.04	Thymol	2.14	1.35
11.24	Carvacrol	45.86	23.22
13.12	β -caryophyllene	6.88	4.60

Conclusion

1. Thermobiochemical treatment based on using solar drying is part of the approach to the recovery and conservation of aromatic plants.
2. Different factors affecting the drying kinetics and the quality of an endemic plant of morocco *Thymus satureioides* were studied.
3. The drying kinetics study of witness and treated thyme with a solution of citric acid is influenced by the temperature and the air flow drying. However the effect of the concentration is negligible.
4. This eco-process allows a total decontamination of E. coli and the conservation of the plant quality.
5. The thermobiochemical process conducts to an amelioration of the total phenolic content during storage.
6. The use of solar convective drying allowed us to dry and preserve our samples cheaply.

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