

Ferchichi C, Bessi H, Grissa K, Bikoba V, Mitcham EJ, Bellagha S (2012) Efficiency of ethyl formate and Vapormate® on dates fumigation: effects on fruit quality. In: Navarro S, Banks HJ, Jayas DS, Bell CH, Noyes RT, Ferizli AG, Emekci M, Isikber AA, Alagusundaram K, [Eds.] Proc 9th. Int. Conf. on Controlled Atmosphere and Fumigation in Stored Products, Antalya, Turkey. 15 – 19 October 2012, ARBER Professional Congress Services, Turkey pp: 321-324

## EFFICIENCY OF ETHYL FORMATE AND VAPORMATE® ON DATES FUMIGATION: EFFECTS ON FRUIT QUALITY

Cyrine Ferchichi\*<sup>1</sup>, Haithem Bessi<sup>1</sup>, Kaouthar Grissa<sup>2</sup>, Veronique Bikoba<sup>3</sup>, Elizabeth J. Mitcham<sup>3</sup>, Sihem Bellagha<sup>1</sup>

<sup>1</sup> Department of Food Technology, National Institute of Agronomy of Tunisia, University of Carthage, INAT, 43, Avenue Charles Nicolle, 1082, Tunis Mahrajène, Tunisia  
Laboratory of Economy, Sciences and Food technology, LR03AGR01

<sup>2</sup> Department of Entomology, National Institute of Agronomy of Tunisia

<sup>3</sup> Department of Plant Sciences, University of California, Davis, CA 95616, USA

\*Corresponding author's e-mail: [cyrineferchichi@gmail.com](mailto:cyrineferchichi@gmail.com)

### ABSTRACT

Dates are subject to infestation by several insect species during and after harvest. In Tunisia, the world first producer of Deglet Nour variety, infestation rate may reach 20%, which leads to serious damages to the fruit rendering it unfit for human consumption and unacceptable for marketing in international trade. The main pest of date is carob moth (*Ectomyelois ceratoniae*) which develops inside the fruit and continues its growth upon arrival at the packaging plant and during storage.

In this study, ethyl formate (EF) was chosen, as an alternative to methyl bromide, to fumigate dates at the laboratory scale. For this purpose, three EF concentrations (114.4 g m<sup>-3</sup>; 128.7 g m<sup>-3</sup>; 143 g m<sup>-3</sup>) and two exposure durations (2 and 3 hours) were tested. Results showed that the most efficient combination is 143 g m<sup>-3</sup> EF during 2 hours which ends up with 98.12% mortality. On a commercial scale, EF is available as a mixture of CO<sub>2</sub> and EF: 16.7 wt% EF in liquid carbon dioxide (Vapormate®, LINDE GROUP). Hence, experiments at semi-industrial scale were run in order to validate previous results. Thanks to the CO<sub>2</sub> synergic effect, the mortality rate was confirmed.

In addition, effects of EF on fruit quality were investigated through color, sugar content and microbiological analysis.

**Key words:** Fumigation, Carob moth, Ethyl formate, Vapormate, Methyl bromide, Mortality rate, Fruit quality.

### INTRODUCTION

Carob moth, *Ectomyelois ceratoniae* (Zeller) (Lepidoptera: Pyralidae), is the pest which causes the most damages to harvested date fruits. The rate of infestation may increase from 18% at harvest to 70% during storage. Studies focusing on life cycle development of Carob moth demonstrate that the most resistant phase to EF is the larvae in L5 stage (Bessi et al., 2011).

Till now, methyl bromide has been the most used fumigant in date postharvest stack disinfestations. However, with the pending phasing-out of methyl bromide, alternatives such as EF need investigation. Research seeking alternative fumigants to methyl bromide has

involved studies with EF as a space fumigant. The practicalities and effects of space fumigation with EF are a highly attractive option for bulk disinfestations of unprocessed dates. The low molecular weight of EF is considered as an advantage over conventionally used chemicals which can persist as residues in food products (Simpson et al., 2004)

EF has been registered for application to dried fruit where it has been used as a post processing disinfestations treatment since 1927 (Simmons and Gertler, 1945) and has proven to be effective on the major insects present in the Australian processing industries. Hence, dried sultana raisins were fumigated in shipping containers using EF and an EF/CO<sub>2</sub> mixture (Tarr et al., 2004).

This study evaluated the use of EF alone or in combination with carbon dioxide (16.7 wt% EF in liquid carbon dioxide called Vapormate® (BOC Gases LINDE Group)) as a postharvest fumigant in order to control Carob moth (*Ectomyelois ceratoniae*), the most common pest on dates in Tunisia. In Vapormate composition, CO<sub>2</sub> is a carrier gas and fire retardant. However, CO<sub>2</sub> presence may enhance EF disinfestations.

The study reported here, summarizes the laboratory work undertaken to determine the concentration of EF and the exposure time required to obtain maximum mortality of Carob moth. The second part of the study relates field trials undertaken to apply the laboratory findings to a semi-industrial scale using a mixture of 16.7% EF in CO<sub>2</sub> (Vapormate®, LINDE Group). The effects of EF and Vapormate treatments on fumigated fruit quality were also investigated through color, sugar content, aromatic composition and microbiological analysis.

## MATERIALS AND METHODS

### 1. Raw materials and target pest

Dates “Deglet Nour” variety in branch were obtained from a local distributor and stored at 4°C. An in vitro infestation of dates by the most EF resistant larvae stage (L5) of Carob moth (*Ectomyelois ceratoniae*) was applied (Bessi et al., 2011).

### 2. EF and Vapormate trials

Dates previously infested by the larvae L5 were exposed to EF in 1 liter glass jars (4 replications; 410g of dates fruits per treatment) sealed with rubber stoppers (Bessi et al., 2011). Three EF concentrations were tested: 114.4 g m<sup>-3</sup>; 128.7 g m<sup>-3</sup>; 143 g m<sup>-3</sup> with two exposure times: 2 and 3 hours. The EF was injected by a microsyringe.

A fumigation pilot plant has been designed by the LINDE Group technical team. It consisted of a stainless steel enclosure (capacity 100 liters) with a sealed cover connected to the bottle of Vapormate by a copper pipe (diameter 1 cm) fitted with a barometer. A precision balance (+/- 0.005kg) was used to determinate the quantity of Vapormate injected in the enclosure. Three trays containing the artificially infested dates were placed respectively on the middle and the two opposite corner of the enclosure for each of three trials.

### 3. Fruit quality analysis

Post-treatment fruit quality evaluations included internal and external dates color using Minolta Chromameter (model CR-300) and sugar content by the colorimetric method. Aerobic mesophilic bacteria (NT 16-14 (2006)) and mold and yeast (NT 16-14 (2006)) were analyzed after EF fumigation and over three weeks storage at 4°C.

## RESULTS AND DISCUSSIONS

### 1. EF effect on carob moth mortality

All EF treatments resulted in significant Carob moth mortality (Table 1). However, the highest mortality, i.e. 98.12%, was achieved with 143 g m<sup>-3</sup> EF during 2 hours. Statistical analysis ( $P > 0.05$ ) has shown that EF exposure time has no significant effect on Carob moth mortality. Table 1 shows no significant difference between the 128.7g.m<sup>-3</sup> and 143 g m<sup>-3</sup> for 2 hours treatments. However, the highest mean is obtained when 143g.m<sup>-3</sup> EF concentration is used. Hence, in order to validate Vapormate assays, 143 g m<sup>-3</sup> EF concentration was adopted.

Table 1. Effect of fumigants on Carob moth mortality in dates.

Trials conditions			Mortality (%)
Fumigant	Time (h)	Concentration* (g m <sup>-3</sup> )	
EF	2	114.4	91.6±1.32 <sub>a</sub>
	2	128.7	95±1.63 <sub>b</sub>
	2	143	98.12±2.25 <sub>bc</sub>
	3	128.7	95.5±1 <sub>d</sub>
	3	143	96.25±2.5 <sub>c</sub>
	Vapormate	2	856

a,b,c,d Means in a column followed by the same letter are not significantly different at the 5% level.

\*mean of four repetitions.

### 2. effect of Vapormate on carob moth mortality

Vapormate semi-industrial trials were run with 856 g m<sup>-3</sup>, corresponding to 143 g m<sup>-3</sup> of pure EF. Vapormate trials show an increase on insect mortality from 98.12% with pure EF to 100% but statistically there is no significant difference between them. Even though, reports in the literature indicate that elevated CO<sub>2</sub> atmospheres have a positive impact on insect mortality when combined with various fumigants (Bond and Buckland, 1978; Simpson et al., 2004).

### 3. Quality analysis of EF treated dates

Statistical analysis shows that there is no significant ( $p > 0.05$ ) difference between untreated and EF fumigated dates for water content, water activity, glucose content, and color parameters (Table 2).

### 4. Microbiological analysis

Total mesophilic bacteria and mold and yeast count were evaluated over 3 weeks. EF fumigation of dates gave 95% mold and yeast reduction, while over 35% destruction of mesophilic bacteria was noticed. However, after 3 weeks, the rate of microbiological reduction decreases to 77% for the mold and yeast count while it still maintained to 35% for the mesophilic bacteria count.

Table 2. Effects of Ethyl Formate on dates quality.

Samples	EF concentration (g m <sup>-3</sup> )	Water content (%)	Water activity (%)	Glucose Content (g/100g dates)	Color		
					L	a	b
Untreated dates	0	33,70 <sub>a</sub>	71,63 <sub>a</sub>	13,47 <sub>a</sub>	53,95 <sub>a</sub>	7,29 <sub>a</sub>	13,68 <sub>a</sub>
Fumigated dates	143	33,13 <sub>a</sub>	70,05 <sub>a</sub>	13,60 <sub>a</sub>	51,30 <sub>a</sub>	7,69 <sub>a</sub>	13,69 <sub>a</sub>

a Means in a column followed by the same letter are not significantly different at the 5% level.

These findings could be related to the fact that EF residues disappear after 7 to 10 days (Bessi et al., 2011). Nursten, (1970) has shown that EF has insecticidal and fungicidal properties when it is applied on strawberries.

#### ACKNOWLEDGEMENTS

The authors would like to thank Linde Group for their assistance, supply of fruit and premises for the fumigations. This project was partially supported by the National Institute of Agronomy of Tunisia.

#### REFERENCES

- Bessi H, Bellagha S, Grissa K, Bikoba V, Mitcham E (2011) Effects of ethyl formate on dates (deplet nour) quality and carob moth pest. Processing 1<sup>st</sup> Euro-Mediterranean Symposium, Avignon. France 18-21 April 2011.
- Bond EJ, Buckland CT (1978) Control of insects with fumigants at low temperatures: toxicity of fumigants in atmospheres of carbon dioxide. *J Econ Entomol* 71:307–309.
- Nursten HE (1970) *Volatile Compounds: the Aroma of Fruits*. Academic Press, New York.
- Simmons P, Gertler SI (1945) Ethyl formate and isopropyl formate as fumigants for packages of dried fruits. *J Econ Entomol* 38: 715–716.
- Simpson T, Bikoba V, Mitcham EJ (2004) Effects of acetaldehyde on fruit quality and target pest mortality for harvested strawberries. *Postharvest Biol Tech* 28:405–416.
- Tarr C, Reuss R, Clingeffer P, Annis P (2004) The use of ethyl formate for space fumigation of dried vine fruit. FTIC Ltd. Publishing, Israel, pp.193-205.