NEW METHOD FOR TESTING MILK ACIDITY BASED ON VOLATILE ORGANIC COMPOUNDS EMISSION

Oana OLARIU, Dana COPOLOVICI, Lucian COPOLOVICI*

Faculty of Food Engineering, Tourism and Environmental Protection, "Aurel Vlaicu" University, Romania, 2 Elena Dragoi St., Arad 310330, Romania
**Corresponding author email: lucian.copolovici@uav.ro*

Abstract: *Milk storage is one of the most important condition for people access to healthy food. The volatile organic compounds pattern could be a great tool for determination of fermentation stage. The present paper has been shown that 2-butanone concentration decreased over time of storage while the acetic acid concentration depends not only by time but also by storage temperature. The emission of acetic acid could be used as a marker of fermentation degree and its concentration has been correlated with classical approach of Thorner degrees measurements.*

Keywords: milk fermentation, volatile organic compounds, milk acidity

INTRODUCTION

Milk is one of the basic foods and the way it is stored is very important for its usage by the consumers. The milk taste depends by many factors including cows feeding systems as grazing or conserved forages and concentrates (Aizaki et al., 2013; Ueda et al., 2016). Milk exposed to light and temperature can cause the development of flavour characterised as being *burnt protein, oxidised, cabbage and/or mushroom-like* (Zardin et al., 2016). Even more, other flavour as *cardboard-like, metallic or rancid* taste could be developed (Hedegaard et al., 2006). Many studies have been examined the volatile organic compounds emission from dairy products mainly regarding to terpenes used as tracers of highland milk (see for examples (Tornambe et al., 2006; Ueda et al., 2016; Viallon et al., 2000)). Furthermore, the feeding system or geographical source of milk could be identify using the pattern of volatile organic compounds (Coppa et al., 2011). The pattern of the volatiles has been used to study as well other milk products as Grana Trentino, Grana Padano, Parmigiano Reggiano cheeses (Boscaini et al., 2003), mozzarella cheeses (Gasperi et al., 2001) and even butter from different origin (van Ruth et al., 2008). The volatiles compounds emitted by the milk stored in light have been shown the presence of aldehydes such as formaldehyde acetaldehyde, pentanal, hexanal, heptanal, that increased over time, while the concentration of methanethiol increased rapidly (Zardin et al., 2016).

The aims of this study are to find a volatile organic compound marker for milk freshness and to identify the pattern of the volatile milk emission during storage.

MATERIALS AND METHODS

Milk was taken from a cow farm in Arad County. The milk collection process was carried out with a milking machine under perfect hygienic conditions. Collection of the research material was done in flasks with lid. The sample transportation was carried out in half an hour, which did not affect the physical and chemical properties of milk.

The milk has been divided in 3 parts: one has been kept in refrigerator at +4 $^{\circ}C$ (and used as a control sample), the second one at room temperature +25 $^{\circ}C$ and the third one at +40 $^{\circ}C$ in the full sun. Samples have been collected every 30 minutes for 7 hours. All experiments have been run in triplicate.

For sample collection of volatile organic compounds, 10 mL of milk was placed in a 50 mL plastic tube and a special pump with constant debit (SKC Inc., Houston, TX, USA) has been used to suck the air in the multibead tubes filled with carbotrap (Kannaste et al., 2014). The determinations have been made at +25 ^{o}C. All tubes have been desorbed and analysed using Shimadzu TD20 automated cartridge desorber and Shimadzu 2010 plus GC-

MS instrument (Shimadzu Corporation, Kyoto, Japan). The chromatographic method has been described in (Copolovici et al., 2009).

The acidity of the milk (in Thorner degrees) was determined using classical method by titration with a solution of NaOH in the presence of phenolphthalein.

RESULTS AND DISCUSSIONS

Volatile organic compounds emission

There are some volatile organic compounds which have been detected from fresh milk, so even before fermentation (Figure 1).

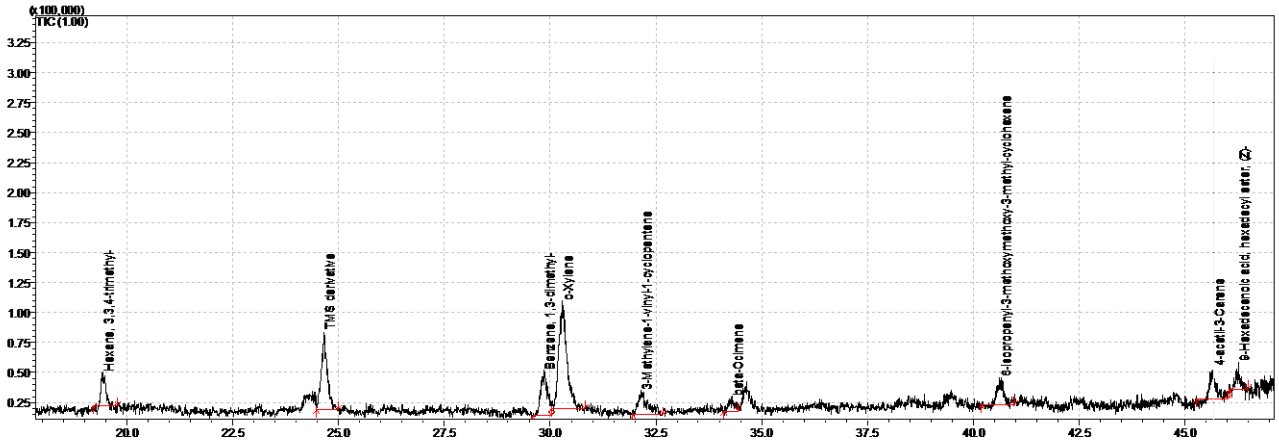

Figure 1. Volatile organic compounds profile of the milk

In the case of the milk which has been kept at +25 °C the concentration of 2-butanone decreased after 5 hours, while for the milk kept at +40 °C after 1 hour (Figure 2). The same pattern has been observed for 2-pentanone (data no show).

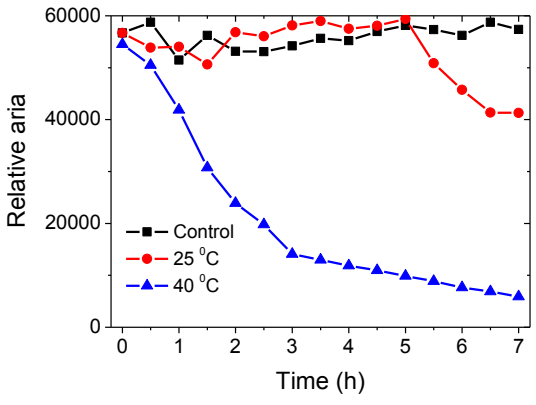

Figure 2. The evolution of 2-butanone emission

In contrast, in the same time, the emission of acetic acid is increasing (Figure 3). The emission of acetic acid is correlated with the *oxidised* taste. Such increase over time could be as well due to transformation of acetaldehyde through oxidation reaction in light (Zardin et al., 2016).

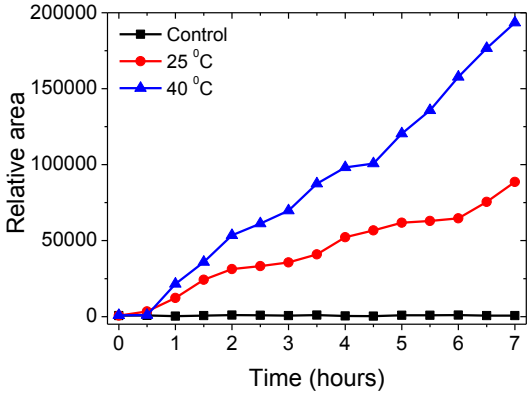

Figure 3. The evolution of acetic acid emission

While hexanal is associated with the oxidation of linoleic acid (Nukuntornprakit et al., 2015) the emission of Strecker aldehydes formed in Maillard reactions is due to thermal treatments (Makhoul et al., 2016). The formation of acetic acid in milk due to lactic acid bacteria (as *Streptococcus thermophilus* or *Lactobacillus delbrueckii ssp. bulgaricus*) has been shown for all milk products (Dan et al., 2017).

In Figure 4 it has been shown the 3 D trend of the emission of acetic acid. The trend

of concentration emission is increasing with the time and storage temperature.

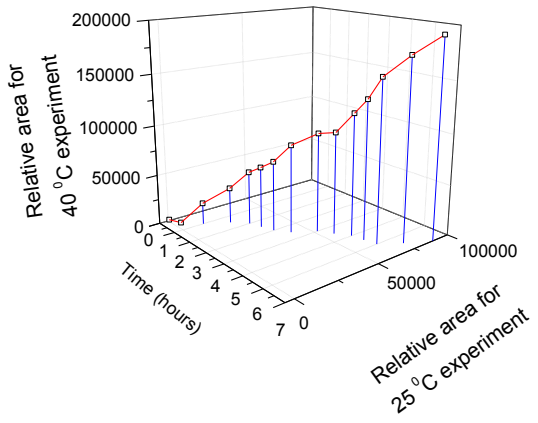

Figure 4. The 3D plot of acetic acid emission function of time

Correlation between acetic acid emission and milk acidity

A very good correlation between acetic acid emissions with the milk acidity (in Thorner degrees) has been found for both storage temperatures (Figure 5).

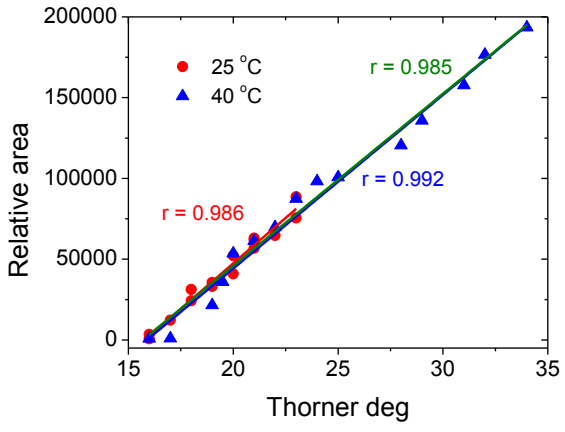

Figure 5. The correlation between acetic acid emission and Thorner degree

The slopes for both storage temperatures are statistically equal with $p < 0.05$ (11247 ± 530 for +25 °C compared with 10741 ± 378 for +40 °C). Even more, concatenated linear fitting (the green line in figure 5) has been shown a very good correlation (r = 0.985).

CONCLUSIONS

Different volatile organic compounds have been identified from milk during storage at two different temperatures. One candidate as possible marker of milk fermentation, acetic acid, has been detected and its concentration has been correlated with the classical method of determination in Thorner degrees. More work is necessary to identify more stress markers and difference in the pattern of volatile organic compounds emitted by milk during storage time.

ACKNOWLEDGEMENTS

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