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HEAT TREATMENTS VERSUS FERMENTATION

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Our researches have been determined in the recent years by examining the effects of heat treatment methods on food. In our current article we are presenting the results achieved during storing and fermentation of heat-treated grape must. Heat treatments have been executed by microwave-energy transfer and convective heat transfer carried out in water bath. Identical long treatment time and temperature were applied in both cases. Heat treatment slowed down, respectively prevented the natural fermentation of raw grape juice, which was checked by examining the CO₂ concentration. It was established during the comparison of heat treatment methods, that there is no significant difference between the effects of heat treatments carried out with either microwave energy transfer or water-bath thermostat on grape must.

Introduction

Juice squeezed out of the grape-seeds is called grape must, which is the raw material of wine. It is a popular drink of vintage period, but fermentation of its sugar-content changes both flavour and attributes rapidly. During fermentation process majority of sugar content disintegrates into alcohol and CO₂. It is undoubtedly a – from the aspect of wine production – very favourable process, which is triggered by yeast molecules getting in the grape must, however more and more people are trying to conserve grape must and selling it as a soft drink. (Eperjes, 2010; Mauricio et al., 1997; Lafon-Lafourcade et al., 1984, Rektor 2009)

Heat treatment is a well known procedure in food conservation as well, which is utilised in order to prevent and delay the deterioration processes. This procedure was worked out by Louise Pasteur French scientist – among other purposes – to prevent late fermentation of wine. The mild heat treatment is called pasteurization – named after its inventor. As an interesting fact we would like to mention that simultaneously with Pasteur, Móric Preysz Hungarian chemist, developed a similar method to prevent quality-deterioration of Tokaj wines. ([http1](http://1))

Mild heat treatment (60-80 °C) delays fermentation of grape must, therefore it is a common method used with home-made conservations. Industrial scale heat treatments are carried out in tubular or plate-type heat exchanger, with convective heat transfer. Heating of liquid foods can be executed by microwave energy transfer. During our previous researches we carried out both traditional and microwave-heating of milk, egg juice, beer and orange juice and we examined the effects of heat treatment on the food in all the cases. (Géczi et. al. 2013, Korzenszky et al., 2013, Garnacho et al. 2012). Based on the above, heat treatment for conserving grape must offers a possibility to examine microwave heat treatment as well. (Kapcsándi, 2012)

Material and methods

Comparison-examinations were executed on the experimental instrument set up in the laboratory of Szent István University, Faculty of Mechanical Engineering. Analysing and checking the grape must examples took place at Szent István University, Department of Chemistry and Biochemistry. Examples heat treated with microwave (MH), respectively water bath thermostat (TH) were compared with each other and untreated (WH) example as well. Grape must used for the experiments at the end of July 2013 was prepared from Italian Vittoria white grape and Black Magic red grape. In August was used Bianca white grape and Nero red grape from Hungary. Properties of grape must is summarised in Table 1.

Table 1

Attributes of grape must used in the experiments.

	Vittoria white grape (Italy)	Black Magic red grape (Italy)	Nero red grape (Hungary)	Bianca white grape (Hungary)
Dry matter content [g/100ml]	13.55±0.03	16.63±0.05	23.31±0.12	23.74±0.08
Density [g/ml]	1.0402±0.011	1.0327±0.013	1.083±0.02	1.079
pH	2.08±0.1	2.56±0.08	3.52±0.11	3.26
Conductivity [µS/cm]	1477±11	1497±17	2350±15	1620
Degrees Brix [°Bx]*	13	16	22	23

*determined by OG-101/A type manual refractometer

At the Faculty of Mechanical Engineering of Szent István University a group of researchers dealing with food treatment created an experimental instrument capable of continuous service microwave heating and heat treatment. They managed to create a measuring circuit capable of comparison-examinations, which allowed heating up liquid foods by usage of microwave energy transfer and convective heat transfer, by upgrading this instrument in the recent years.

The „spirit” of the comparison-circuit is a glass spiral, in which the food to be treated is circulated – grape must in our case – by a STENNER 85M5 type pump (Stenner Pump Company, Jacksonville, FL, U.S.A.) which has continuously adjustable flow rate. The glass spiral can be placed into either the Whirlpool AT 314WH type (Whirlpool Corporation, U.S.A.) household microwave device or T-PHYWE (Lauda DR. R. Wobser GmbH, Lauda-Königshofen, Germany) water-bath thermostat for executing the heating procedure. In case of the microwave gadget we applied continuous 900 Watt output power, whereas with the water-bath thermostat a water-bath of appropriate temperature (70-95°C) for the desired target temperature was used. During the heat treatment of continuous flowing, the liquid foods circulating in the glass spiral – depending on the length of the glass spiral and the volume flow of the pump – can be heated to the desired temperature.

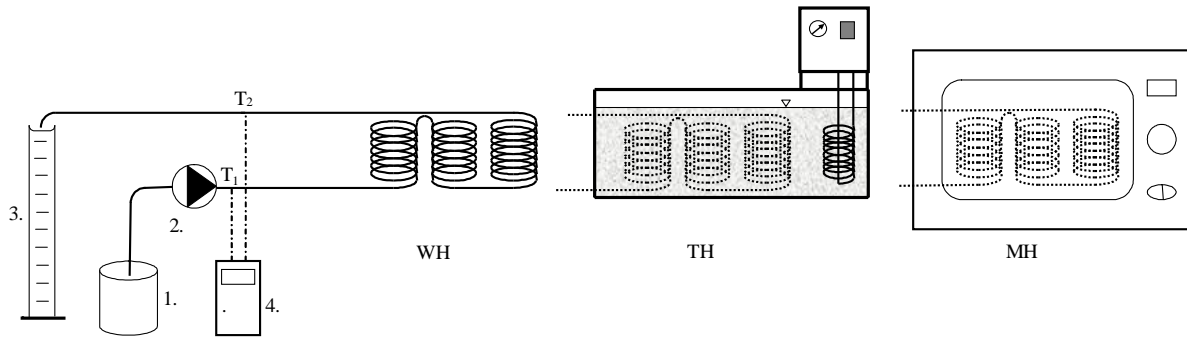


Figure 1

Structuring of heat-treated sets of samples

1- Grape must tank (5 litres); 2-STENNER 85M5 pump; 3-Heat treated sample; 4-ALMENO 2590-9 monitoring and data collection system, WH – untreated control; TH – heat treated with water-bath; MH – microwave heat treated

One of the advantages of this method is that heating process in the electromagnetic field of the microwave instrument is continuous and outlet temperature is constant by using glass spiral. Therefore the fluctuations of temperature inside the product, which is highly characteristic for microwave heating, could be avoided. Temperature – before and after microwave field and water bath as well – is easily to supervise, the procedure can be controlled easily. For measuring the temperature ALMEMO 2590-9 type (Ahlborn, Holzkirchen, Germany) data collection system was applied with NiCr-Ni temperature sensors.

We aimed at a target temperature of $T_2=70^\circ\text{C}$ for the heat treatment of grape must, but we did not hold this temperature. The desired temperature could be realised at $Q=2,69\text{cm}^3/\text{s}$ flow rate and at $T_{\text{bath}}=78^\circ\text{C}$ water temperature in case of convective heating. We poured 1-1 dl of the control and heat treated samples in the commercially available 3,3dl PET bottles. The samples cooled down to the storage temperature of 22°C in a natural way in all cases. Sample bottles were closed one day after the treatment in order to avoid pressure decrease due to cooling. Samples were stored 7 days long at the indicated storage temperature. The CO_2 determination, which is fairly characteristic to the fermentation process, was executed at Szent István University, Faculty of Chemistry and Biochemistry. Gas analysis and control of samples, were carried out as a blind test, the examiner received the samples encrypted and mixed, the person carrying out the heat treatment of the grape must did not participate in the examinations.

During the fermentation we took samples from the atmosphere above the liquid level of the PET bottles. Determination of the samples' CO_2 content was carried out by means of Hewlett Packard 5890 series II. (Germany, SN.: 3203616265) type gas chromatograph, by using thermal conductivity measuring (TCD) universal scanner and helium carrier gas. For data display we used HP GC Chem Station Rev. A. 08.03 software, which is capable of visualising the area under the peak, characteristic of CO_2 content and retention time. (Kristóf 2009; Erdey & Mázor 1974, Buffington & Wilson 1991, Burger 1992)

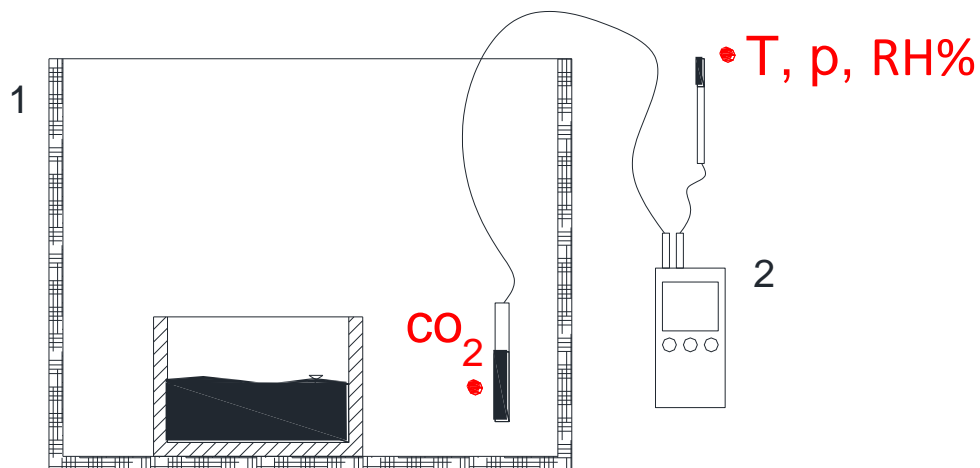


Figure 2

Double vessel for examining grape must fermentation

1-Double vessel with grape must in the inside one, 2- ALMEMO 2590-9 monitoring and data collection system with sensors

Carbon-dioxide concentration forming during fermentation was checked with the arrangement displayed on Figure 2. as well. Carbon-dioxide has higher density than air therefore during fermentation of the grape must placed in the inside vessel, a continuous increase of carbon-dioxide concentration in the outside vessel can be measured. For measurement we used ALMEMO 2590-9 type (Ahlborn, Holzkirchen, Germany) data collection system with connectable FYAD 00 CO₂ B10 és FHA646E1C type sensor, plus the concentration was continuously measured and recorded for 4 days. Storage temperature, humidity, and atmospheric pressure were also monitored during the measuring process. Fermentation of the three sample groups was observed simultaneously in a parallel way.

Results

There was a transformation in the heat treated (MH és TH) and untreated (WH) samples' atmosphere above liquid level during the 7 days long storage because of the fermentation procedures. This transformation is nicely demonstrated in Figure 3, by the photo taken before measuring the CO₂ concentration. On the photo can be see in PET bottle Nr. 23 heat treated sample by microwave-energy transfer, in PET bottle Nr. 35 an untreated control- and in PET bottle Nr. 41 a heat treated grape must sample by water-bath thermostat. It is visible, that due to the formed CO₂, pressure increased in the middle bottle (35), PET bottle lost its original shape.



Figure 3

Grape must samples after 7 days storage.

Nr.23 heat-treated by microwave energy (MH), Nr.35 without heating, as a control (WH), Nr.41 heat treated by water-bath thermostat (TH)

We took samples from the atmosphere above the grape must by piercing through the bottles and we determined the CO₂ content of the atmosphere by using gas chromatograph examination. On Figure 4 we depicted the CO₂ content formed in the closed atmosphere above the liquid level of 8-8 pcs of sample grape musts. The diagram confirms the pressure difference experienced on the photo of Figure 3. CO₂ concentration originating from the atmosphere of untreated sample (WH) (938,57±36,21SD) is more than four times of that of the heat treated samples.

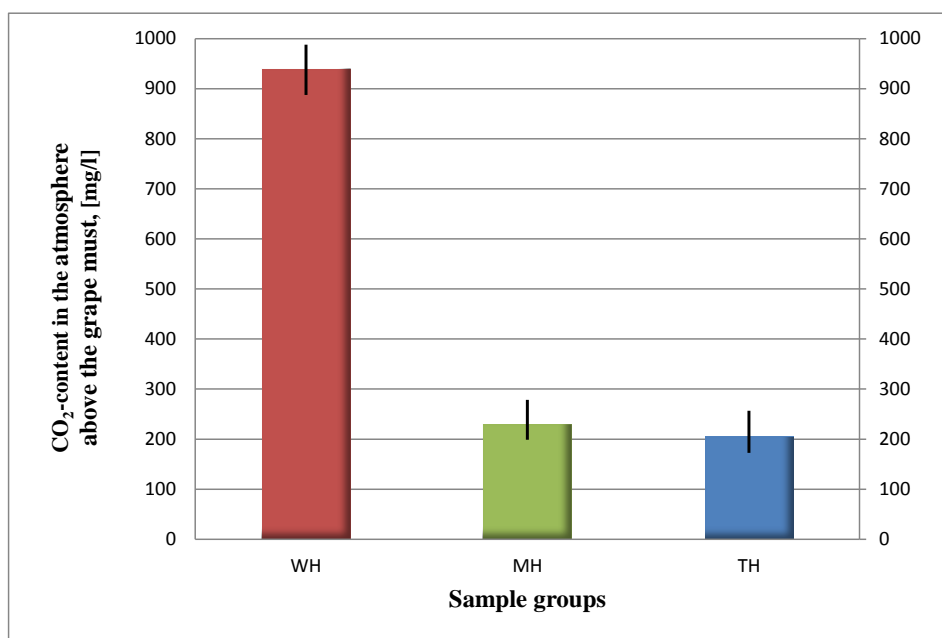


Figure 4

CO₂ content in the PET bottle atmosphere above the grape must after 7 days storage

Table 2

Two sample t-test, for comparing the effects of heat treatment methods

Measured values CO ₂ [ppm]			Calculated values (data analysis*)		
WH	MH	TH		MH	TH
987,65	236,53	213,86	Expected value	229,16	204,23
945,60	278,65	176,43	Variance	1016,78	903,69
887,65	275,65	179,55	F-value	1,13	
917,69	198,76	256,53	P(F<=f)	0,44	
972,92	214,42	218,79	F-critical	3,79	
897,22	205,11	187,44	t-value	1,61	
933,38	220,67	228,88	P (T<=t) two-tailed	0,13	
966,44	203,45	172,38	t critical two-tailed	2,14	

*data analysis by means of Excel2007 Analysis Toolpack

Based on Table 2, a two-sample t-test did not show a significant difference ($|t_{\text{value}}| < t_{\text{critical}}$) between the mean CO₂ concentration in the two heat-treatment conditions at a significance level of $p=0.05$.

On the diagram of Figure 5 we present the results of the experiment executed under the conditions described in Figure 2. It can be seen that during the experiment which was carried out in the double vessel under open-air conditions the CO₂ concentration measured in the outside vessel is continuously increasing from the ~520 ppm initial concentration. Pressure (986mbar) and temperature (20,2°C) of the storage can be considered as constant. We can establish that the fermentation process starts earlier and gets accomplished quicker in case of the untreated (WH) sample. The procedures of carbon-dioxide generation in the heat treated samples are very similar; we did not detect any remarkable deviations this time either. Examinations were repeated three times each with Nero and Bianca grape types in August 2013; formations of the curves were similar in all cases.

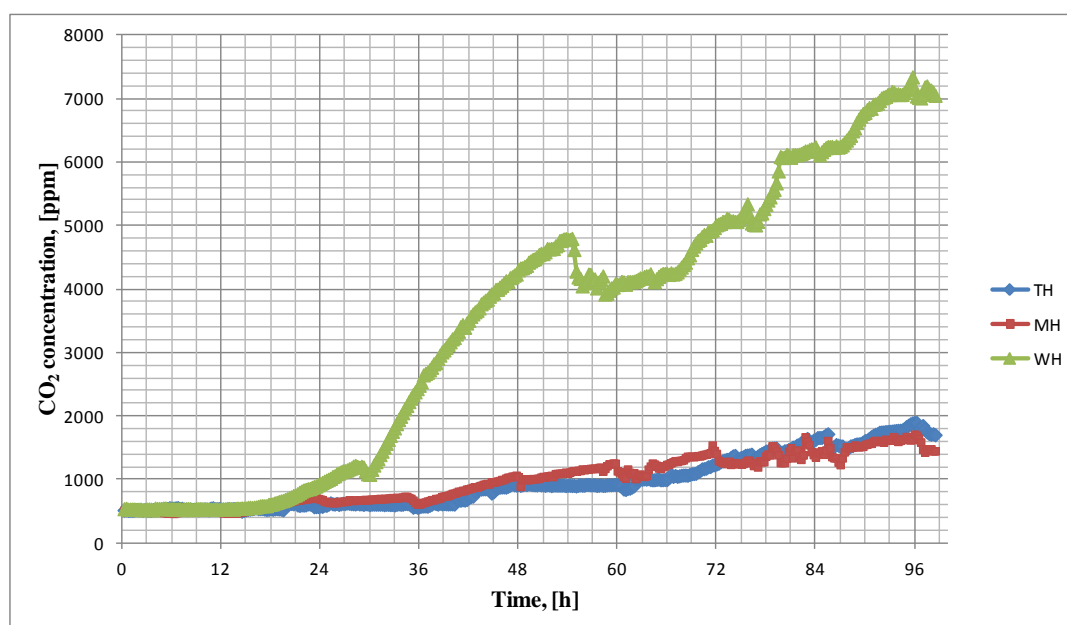


Figure 5

CO₂ change of concentration during fermentation of Nero grape must

Conclusions

Grape must proved to be an appropriate subject for examining microwave heat transfer. During vine-making technology grape must starts to ferment and its sugar content transforms into alcohol and carbon-dioxide. Fermentation of grape must can be tracked by measuring the latter component. Fermentation process might be delayed, slowed down by using heat treatment methods. (A known method for home-made grape must conservation.) Just as during our previous examinations, we were searching for an answer whether microwave heat treating can be matched with any further advantages or disadvantages apart from the known thermal-effect. Fermentation of the grape must heat treated with microwave method was compared to fermentation of samples heat treated with convective method (water bath thermostat). We examined fermentation in PET bottle, closed conditions and in double glass vessel fermentation under natural conditions. It can be established that the effect of heat treatment is obviously detectable in both examinations; however we could not find any significant difference between the effects of heat treatments in either of the cases.

Acknowledgments

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