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EXAMINATION OF HEAT TREATMENTS AT PRESERVATION OF GRAPE MUST

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ABSTRACT

Heat treatment is a well-known process in food preservation. It is made to avoid and to slow down food deterioration. The process was developed by Louise Pasteur French scientist to avoid late among others wine further fermentation. The different heat treatments influence the shelf life in food production. In our article we present the process of grape must fermentation, as grape must is the base material of wine production. The treatment of harvested fresh grape juice has a big influence on end product quality. It is our experiments we examined the same grape must with four different methods in closed and in open spaces to determine CO_2 concentration change. There are four different methods for treatment of grape juice: boiling, microwave treatment, treatment by water bath thermostat and a control without treatment. As a result of the comparison it can be stated that the heat treatment delays the start of fermentation, thereby increasing shelf life of grape must. However, no significant differences were found between two fermentation of heat-treated grape must by the microwave and water-bath thermostat. The different heat treatment of grape must base materials was done at the laboratory in Faculty of Mechanical Engineering of Szent István University. The origin of the table grapes used for the examination was Gödöllő-hillside.

Keywords: grape must; preservation; CO_2 concentration; microwave; heat treatment

INTRODUCTION

Grape must is the juice squeezed from grape berries. This juice is the base material of good wine. Grape must is the favourite drink of harvest time, but the fermentation of its sugar content changes the flavour and it's other features. During fermentation the biggest part of sugar content (glucose) converted into ethyl alcohol and carbon dioxide.

$$C_6H_{12}O_6 \rightarrow 2\ C_2H_5OH + 2\ CO_2 + \text{heat}$$

Figure 1 Fermentation of grape must
Source: verlieftwines.com by Dirk Roos **(URL 2)**

Fermentation is very important step in the process of wine production. The yeast in the must starts fermentation. But more and more producers try to preserve the grape must to sell it as a non-alcoholic drink **(Shea, 2008; Lafon-Lafourcade et al., 1984)**.

Heat treatment is a known process in food preservation. It is made to avoid and to slow down food deterioration. The process was worked out by Louise Pasteur French scientist to avoid late wine fermentation (among others). It is interesting, that at the same time as Pasteur, Preysz Móric Hungarian chemist worked out a similar process to avoid quality loss of Tokaj wines **(URL 1)**.

The mild heat treatment treatment (60 - 80 °C) delays the fermentation of grape must, so it is a frequent method at homemade preservation. Industrial heat treatments are made with tubular or plate heat exchangers by convective heat transfer. The heating of liquid food can be done by microwave energy transfer. At our previous researches we examined the effect of heat treatments on milk, egg fluid, beer and orange juice **(Géczi et. al. 2013, Korzenszky et al., 2013, Garnacho et al. 2012)**.

According to the previous mentioned, heat treatment for food preservation gives a good possibility also to examine microwave heat treatment **(Kapcsándi, 2012; Marsellés-Fontanet et al., 2009)**.

MATERIAL AND METHODOLOGY

The different heat-treating of grape must base materials was done at the laboratory in Faculty of Mechanical Engineering of Szent István University. The origin of table grape, which was used for the examination, was Gödöllő-hillside but the grape breed is unknown.

The comparisons were started with the preparation of the samples. The freshly squeezed table grape juice was poured in different tanks according to the different heat treatments. For traceability the samples were marked with unique identifiers. The control sample (untreated) was marked with „U", the boiled with „B", the treated by microwave with „M", and treated by water bath thermostat with „T" according to the Figure 2.

The microwave food treatment researcher group made continuous operating experiment equipment, which is applicable for microwave heating, and heat-treating. In the last period with the development of this equipment we made a measurement circle which is applicable for making comparison experiments of fluid food heating with microwaves and with convective heat transfer (water bath thermostat) **(Géczi and Sembery, 2010)**.

The 'soul' of the comparison measurement circle is a glass spiral in which the liquid food (in our case grape must) is pumped with an adjustable volume flow STENNER 85M5 type (Stenner Pump Compony, Jacksonville, FL, U.S.A.) pump. The glass spiral is put in a Whirlpool AT 314WH (Whirlpool Corporation, U.S.A.) domestic microwave oven or a *T*-PHYWE (Lauda DR. R. Wobser GmbH, Lauda-Königshofen, Germany) water bath thermostat for heating.

We used 900 Watt output power of microwave oven and 70 - 95 °C temperature water bath in the thermostat. With this heat treatment (depending on the length of the glass spiral and the volume flow of the pump) liquid foods can be heated to the needed temperature in the glass spiral.

It is advantageous that with the utilization of glass spirals the heating is gradual and outcome temperature is constant. We managed to avoid temperature variation, which is common at microwave heating. The temperature - before and after the microwave equipment and water bath thermostat - can be easily controlled and regulated. We used an ALMEMO 2590-4S type (Ahlborn, Holzkirchen, Germany) thermometer and data logger system with NiCr-Ni heat elements.

We selected T_{target} = 70 °C target temperature for grape must heat treatment. We did not keep this temperature for any length of time, but we cooled it down immediately.
The target temperature was achieved at Q =2.5cm^3.s^{-1} volume flow of peristaltic pump both of microwave and water bath thermostat heat treatment. In the latter case, we applied T_{bath} = 78 °C water temperature.

In case the making of boiled control sample the grape must was heated up gradual and after boiling was cooled.

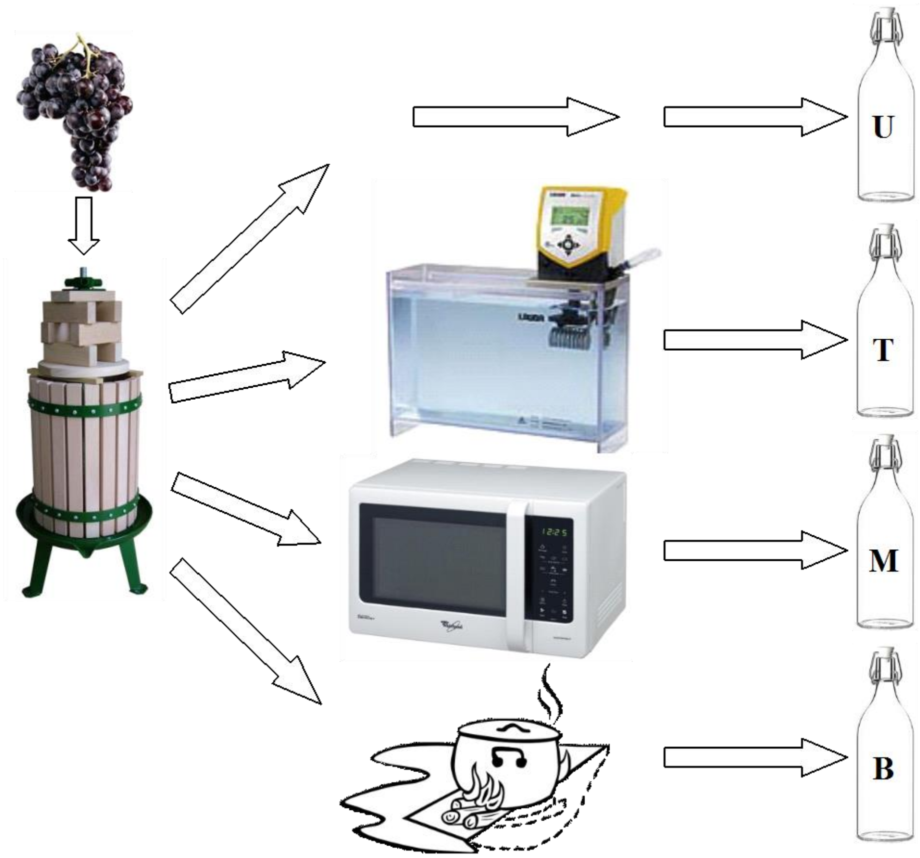

Figure 2 Forming of treated sample groups

„U" – untreated 'control'; „T" – heat treated with water bath;
„M" – microwave treated; „B" – heat treated with boiling 'control'

Figure 3 Examination methods for fermentation

We poured 1-5 dL of heat treated and untreated samples in bottles according to analysis. In all cases the samples were naturally cooled to 24 °C storage temperature.

The carefully prepared heat-treated samples were used for the definition of physical parameters in the laboratory with standardized methods. The analysis and control of grape must samples was done at the Department of Chemistry and Biochemistry, on Szent István University of Gödöllő. During the analysis of grape we must defined dry matter content, density, pH-level and conductivity. For the physical measures we used 0.5 - 0.5 L samples.

The fermentation process can be characterized to determine CO_2 concentration. It was done from closed bottle by sampling (Figure 3-I.) and by examination of airspace of opened barrels (Figure 3-II.). The analysis and control of samples were blind probes; the examiner person got coded and mixed samples. The person who heat-treated the samples did not take part in the measures.

For the examination I. we measured 170 - 170 mL of the samples with a measuring cylinder. These samples were put in gastight septum closed glass bottles. These were used for the CO_2 analysis.

During fermentation we took samples from the 1.2 L septum closed glass bottles, from the space above the fluid (with a 1ml Hamilton syringe). The CO_2 concentration of these samples was defined with a Hewlett Packard 5890 series II. gas chromatograph (Germany, SN.:3203616265), with an universal detector, TCD (used for measuring heat conduction) and with helium gas. For data visualization we used the HP GC Chem Station Rev. A. 08.03 software, which is able to present the values under the maximum of CO_2 concentration and retention time **(David Del Pozo-Insfran et al., 2006)**.

We analyzed the CO_2 concentration parallel at open tanks during fermentation (Figure 3-II.). CO_2 is heavier than air, so during the fermentation a constant increase of CO_2 could be measured inside the open barrels. For the measurement we used an ALMEMO 2590-4S type (Ahlborn, Holzkirchen, Germany) data logger with FYAD 00 CO2 B10 and FHA646E1C. We measured during 240 hours, and recorded the concentration. During the measures we examined storage temperature, humidity and air pressure (data not shown). The fermentation of different sample groups were examined parallel.

Table 1 Features of grape must treated with different heat treatments

Sample identifiers	**Untreated „U”**	**Mikrowave „M”**	**Thermostat „T”**	**Boiling „B”**
Dry matter content [%]	24.74	26.11	27.22	30.21
Density (20°C) [g/cm^3]	1.079	1.073	1.073	1.081
pH	2.76	2.81	2.98	3.05

*Average of three measurements

RESULTS AND CONCLUSION

We used standardized measurement methods for examining the grape must's physical features. The results of the experiments can be seen in Table 1. The physical parameters (density, dry matter content, pH) of grape must were changed slightly with heating and boiling. The change is explained with evaporation and CO_2 stripping. It can be seen that the dry matter content of grape must in case boiled sample "B" nearly 5% higher than in the untreated sample "U". The difference of samples density are minimum, the pH value in case the untreated sample "U" is 2.76, whereas for boiled sample "B" increased to 3.05. The values in the Table 1 are the average of three measurements.

The CO_2 concentrations of the samples were controlled during fermentation at laboratory conditions. (Fig. 3-I.) The CO_2 concentration change of the microcosmos closed samples can be seen on Figure.4. On the logarithmic scale it can be seen that CO_2-concentration of untreated grape must sample shows significantly bigger values as at the other samples.

In the untreated case CO_2 concentration starts from 1200 µg/L value and after 50 hours it reaches 550000 µg/L.

The CO_2 concentration of boiled must does not change

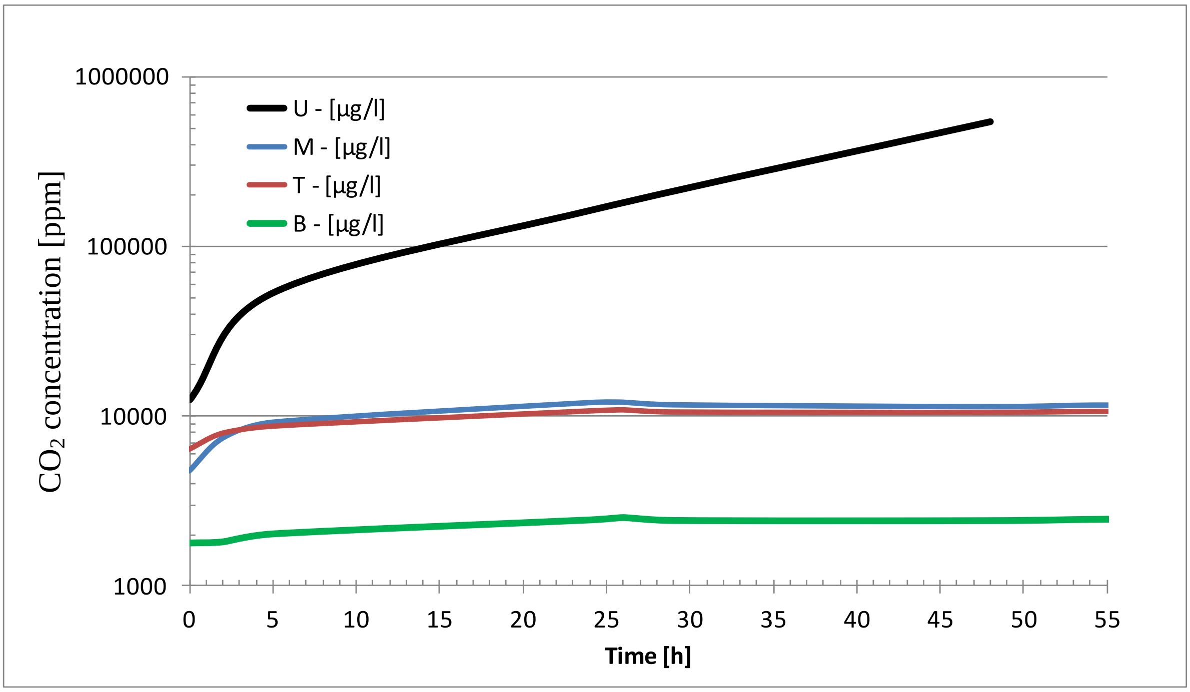

Figure 4 CO_2 concentration of grape must samples in sealed bottle as a function of the time

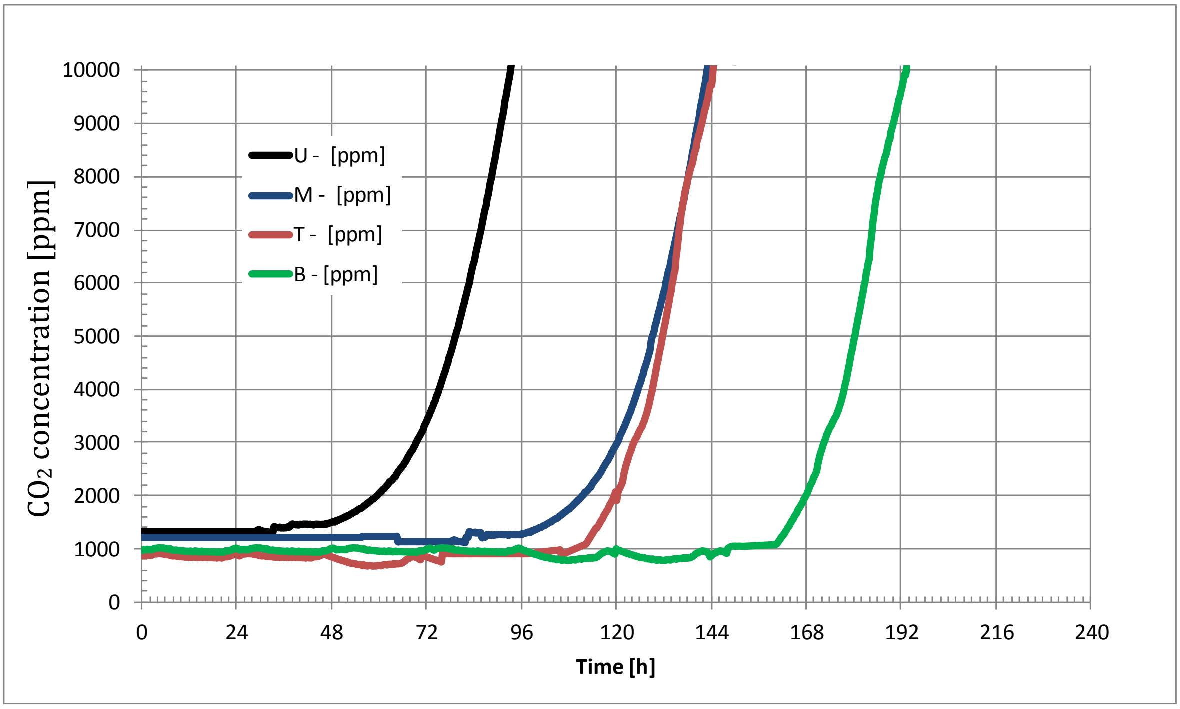

Figure 5 CO_2 concentration of must samples in airspace of opened barrels as a function of the time

significantly compared to the start value. The value changes from1750 µg/L to 2450 µg/L.

After microwave and water bath thermostat treatment the CO_2 concentration is similar. The starter value was 5000 µg/L and it changes to10000 µg/L.

In this treatment temperature (T_{target} = 70 °C) with two different processes no significant difference can be seen in CO_2 concentration growth of closed space.

The result of examination methods Figure 3-II for CO_2 concentration is shown on Figure 5. On the figure it can be seen that we measured different values at the four samples. The CO_2 concentration is showed from 0 to 10000 ppm - in accordance with the measurement range of the device - as a function of the time. The fermentation started earliest at the untreated sample "U", after 48 hours, CO_2 was observed intensive growth. The fermentation of more samples delayed by heat treatments, greatest extent was at boiled sample "B". The fermentation of 70 °C heat treated samples by microwave energy "M" and convective way in water bath thermostat "T" started on 5th day, while the fermentation of the boiled sample begin on 7th day only. The slopes which show the increase of CO_2 concentration are very similar all four cases, therefore we can conclude that the heat treatments do not influence for process of fermentation but delay the formation. Based on Figure 5 it can be stated that there is no difference between effect of the microwave and convective heat treatment methods for must preservation. We got similar results with the experiments of Nero and Bianca grape must (data not shown).

It can be stated that at similar treatment temperature the CO_2 concentration of "M" and "T" samples does not show significant difference during the fermentation process in open tank. We got similar results with the experiments of NERO must (data not shown).

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