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# Investigation of Osmotic Distillation Technique for Beer Dealcoholization

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In the last years, there has been an increasing interest in the development of techniques for the production of low alcohol or alcohol free beers, due to a significant increase in consumption of this popular beverage. This is mainly due to health and safety reasons, strict social and religious regulations. Moreover, the interest has been focused towards technological and economic tools able to produce low alcohol and alcohol free beers with satisfactory organoleptic characteristics that can be compared with conventional beers. This study investigated osmotic distillation technique for the dealcoholization of a lager beer up to an alcohol content lower than 1.2 %vol which is the residual alcohol content for low alcohol beverages in European countries (Brányik et al., 2012). The dealcoholization process was performed in subsequent cycles using water as stripping agent and different values of volume ratio between beer and water. Beers with different alcohol content (0.47 and 0.89 %vol) were produced and their chemical and physical properties (i.e. colour, pH, volatile compounds) and healthy traits (i.e. polyphenols, antioxidant activity) were evaluated. Results highlighted no significant differences among regular and dealcoholized beers in terms of colour, pH, polyphenols content and antioxidant activity, although dealcoholized beers had a depletion of volatile compounds with respect to the original one.

## 1. Introduction

The non-alcoholic beverages industry is faced with unquenchable expectations from well informed consumers, who are increasingly motivated by cost, innovation and benefit in their product choices. In the last years, soft drinks consume has increased due to several reasons such as the preference of a healthier lifestyle, more restrictive driving/drinking regulations, religion reasons or healthy status (i.e. pregnancy, abstinence, obesity due to energy content of alcoholic intake). The main efforts on the production of low or alcohol free beverages are related to the final quality of products in terms of taste, aroma and flavour, which should be preserved or added to the product in order to obtain a beverage as similar as possible to the alcoholic one. In fact, different techniques can be applied to produce low alcohol beverages, which can be divided in two main groups: biological and physical methods. In the case of low and alcohol free beers production, one of the most common biological method consists on the interruption of the fermentation for keeping the ethanol content very low. This method is simple and uses the same resources of standard fermentation. However, its main drawback is related to beer quality, since the reduction of wort compounds and the interruption in the formation of important beer aroma compounds (Brányik et al. 2012). As a result, alcohol free beer presents a typical worty flavour, very different from the alcoholic beers. On the contrary, physical methods are based on gentle removal of alcohol from regular beer by thermal and membrane processes. The industrially applied methods of beer dealcoholization comprise vacuum

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rectification and evaporation, dialysis, and reverse osmosis, but there have been other methods studied under laboratory conditions such as membrane process as osmotic distillation (Russo et al., 2013), membrane extraction (Matson, 1987; Etuk and Murray, 1990), supercritical $CO_2$ extraction (Mori, 2004), pervaporation (Magalhaes Mendes et al., 2008), adsorption on hydrophobic zeolites (Anglerot, 1994), and freeze concentration (Von Hodenberg, 1991).

The objective of this study was to evaluate the quality of dealcoholized beers with an alcohol content lower than 1.2 %vol, which is the residual alcohol content for low alcohol beverages in European countries (Brányik et al., 2012), produced by means of osmotic distillation. Comparison was made among products obtained by different operating conditions (i.e. volume ratio of beer and water). Dealcoholized beers were evaluated in terms of main quality characteristics (pH, colour and volatile compounds) and healthy treats (i.e. polyphenols, antioxidant activity).

## 2. Materials and methods

### 2.1 Materials

The osmotic distillation processes was carried out using a 1x5.5 Liqui-Cel membrane module with the following characteristics: polypropylene membrane, 1800 $cm^2$ surface area, 42 μm thickness and 14 cm length, 40% porosity, 0.03 μm membrane pore diameter. It consisted of 2300 fibers with dimensions: 11.5 cm length, 220 μm inner diameter and 300 μm outer diameter. A lager beer (5 %vol) was purchased at local market. All reagents used for chemical analyses were analytical grade by Sigma Aldrich.

### 2.2 Experimental units design

The dealcoholization tests were carried out in a lab scale plant equipped with the membrane module in which feed (i.e. beer) and stripping (i.e. water) streams flowed in recycle mode, respectively, in the lumen and shell side. Lager beer (5%vol) was used as regular beer.

The temperature of streams was set at 10°C by a thermostatic water bath and the temperatures of retentate (i.e. dealcoholized beer) and permeate (i.e. water enriched in ethanol) were monitored by K-type thermocouples. Feed pressure was measured by a manometer. The feed and stripping flow rates were respectively 70 and 140 mL/min. Water was used as stripping agent with different volume ratio ($V_s/V_f$) with respect to feed: 1 and 2.

Preliminary dealcoholization tests were performed on hydroalcoholic solutions (0.5 L) as feed stream ($V_f$).

The ethanol concentration profile during time in both streams was evaluated to determine the process time for beer dealcoholization tests. As will be discussed later, the beer dealcoholization process was carried out in four cycles at the end of which the stripping solution was renewed with water. At the end of each cycle, low alcoholic beer samples were obtained and codified as $B_{ij}$ where i indicates the cycle number and j the $V_s/V_f$ ratio (1 or 2) used in the tests.

### 2.3 Analyses

Chemical and physical analyses were carried out on regular and dealcoholized beer samples. The pH was measured by pHmeter, whereas the alcohol content was measured by pycnometer. Beer colour was analyzed according to the standard method (EBC, 2008) using an UV/Vis spectrophotometer (Perkin Elmer). The phenolic content was determined by Folin-Ciocalteau method (Singleton & Rossi, 1965) as described in Russo et al. (2013). After 2 h of reaction at room temperature, in the dark, the absorbance at 765 nm was determined. DPPH radical scavenging activity of different samples of beer was determined according to the method of Brand-Williams et al. (1995) with minor changes: diluted beer sample (0.1 ml) was added to 2.9 ml of 6 $*10^{-5}$ mol/l DPPH solution (dissolved in methanol). The absorbance at 515 nm was measured after the solution was stored in the dark for 40 min. A blank experiment was also carried out applying the same procedure to a solution without the test material. The antioxidant activity was expressed as percentage inhibition of DPPH and then calculated according to the following equation:

$$(\%)\ \text{inhibition of DPPH} = 100 * \left( \frac{A_{blank} - A_{sample}}{A_{blank}} \right) \quad (1)$$

The final results were expressed as percentage of antioxidant activity/µl of beer.
Volatile compounds were determined according to Vesely et al. (2003) and Malfliet et al.(2008) by a Gas Chromatograph 6850 (Agilent Technologies) equipped with a Mass Spectrometer 5975C coupled with Maestro Autosamples Gerstel Multi-Purpose Sampler. The GC-MS was equipped with a glass direct inlet liner (1.5 mm inner diameter and 140 µL volume) and a DB-5MS capillary column, 60 m x 0.32 mm x 1 µm (J&W Scientific Folsom, CA) consisting of cross linked 5% phenyl methyl siloxane. Helium was the carrier gas at a flow rate of 1.1 mL/min. The front inlet temperature was 230°C. The injection was in the splitless mode with the purge valve set at 20 mL/min. The detector temperature was at 280°C. The ionization energy was 70eV. Detection and data acquisition was performed in Scan mode, from 30 to 660 m/z. The 20 mL vial containing the sample was heated for 30 min at 40 °C and stirred at 250 rpm for 30 s every minute. The PDMS/DVB SPME fibre was then placed in the sample headspace for 2 min at 40 °C. The fibre desorption time was 120 s. For aldehydes determination, 1 mL of PFBOA solution (4 g/L), 500µL of 2-chlorobenzaldehyde (10mg/L) and 5 mL of sample were placed in a 20mL glass vial; for higher alcohols and esters, 500 µL of the two internal standard solutions (1-butanol, 60 mg/L and ethyl tridecanoate, 60 µg/L) were added to 5 mL of sample and placed in a 20 mL glass vial. GC Conditions: for aldehydes analysis, the oven temperature program used was 40 °C for 2 min, followed by an increase of 10 °C/min up to 140 °C and 7 °C/min up to 250 °C. The final temperature was maintained constant for 10 min. For higher alcohols and esters analysis, the oven temperature was held at 40°C for 3 min, then increased to 200°C at 6°C/min, followed by an increase up to 250°C at 15°C/min and finally held at 250°C for 6 min. Data analysis was performed using the MSD Chemstation Data Analysis Software (Agilent).

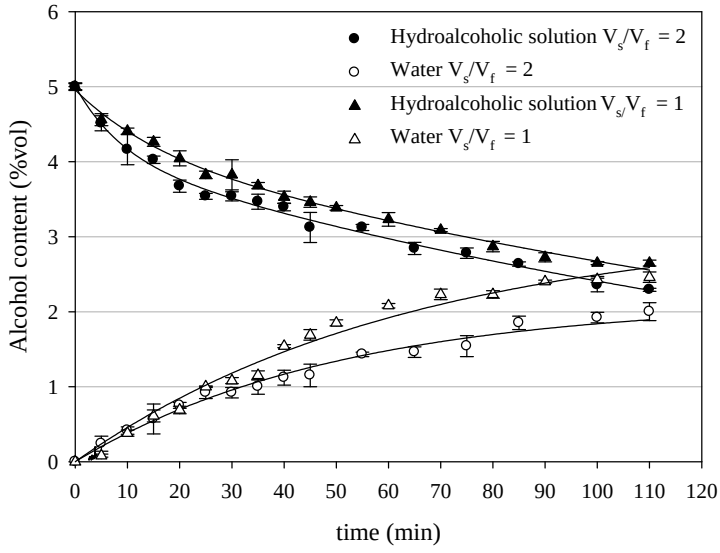
### 2.4 Statistical Analysis

Dealcoholization trials and analytical measurements were carried out in triplicate and mean values and standard deviation values were reported. Monofactorial variance analysis was used to determine significant differences ($p<0.05$) among original (5 %vol) and dealcoholized beer samples by Analysis Lab software.

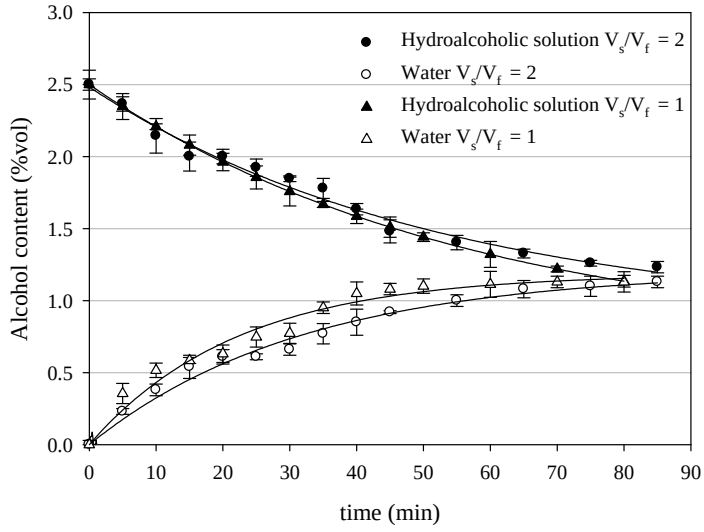
## 3. Results and discussion

### 3.1 Dealcoholization kinetics

The ethanol removal from beverages using osmotic distillation takes place by the alcohol transfer across the membrane due to different concentration of volatile components between the two sides of the membrane module. In order to fix the exchange time of alcohol from feed to stripping stream, dealcoholization kinetic tests were performed. Hydroalcoholic solutions at decreasing concentrations of ethanol (starting from 5.0 vol%) were dealcoholized using water as stripping agent with a volume ratio $V_s/V_f$ equal to 1 and 2. The figure 1a reported the comparison of the temporal profiles of alcohol concentration in the feed and stripping streams using the volume ratio $V_s/V_f$ = 1 and 2. During time, a decreasing ethanol concentration in feed and an increasing alcohol content of water up to an equilibrium condition occurred. As expected a slightly lower decrease in ethanol happened in feed when the ratio $V_s/V_f$ is equal to 1, because of the smallest ethanol difference between feed and stripping streams. The exchange time was stopped at 60 min when alcohol content difference was equal to 1.5 %vol, because further process time determines only a slight decrease of ethanol concentration in feed. For this reason, the 1$^{st}$ cycle time lasted 60 min. Afterwards, the dealcoholization kinetic of 2.5 %vol hydroalcoholic solution was performed using pure water as ethanol stripper (figure 1b). Ethanol trend is similar to the previous cycle but the duration of cycle was fixed at 45 min for the same reason above (figure 1b). The subsequent cycles have the same trend, similar to that of 2$^{nd}$ cycle and the duration was chosen equal to 45 min (data not reported). Totally, 4 cycles were used in both conditions ($V_s/V_f$ = 1 and 2) necessary to reach the alcohol content lower than 1.2 %vol in dealcoholized beer.




a)




b)

*Figure 1. Comparison of dealcoholization kinetics of hydroalcoholic solution at 5%vol (a) and 2.5%vol (b) using $V_s/V_f$ = 1 and 2.*

The process conditions established by previous dealcoholization kinetic tests were applied for the production of dealcoholized beers: 4 cycles of total duration of 195 min. The evolution of alcohol content in beer and water during the process was shown in figure 2. The lager beer ($B_0$) was 5 %vol and the ethanol content decreased progressively up to reach at the end of process a concentration of 0.47 %vol ($B_{42}$) when $V_s/V_f$ was equal to 2. Higher ethanol concentrations were measured in all beer samples ($B_{11}$-$B_{31}$) at the end of each cycle when the volume ratio $V_s/V_f$ = 1 was applied, obtaining a low alcohol beer ($B_{41}$) of 0.89 %vol.
As reported in previous papers about beverage dealcoholization (Liguori et al., 2012, 2013; Russo et al., 2013), the stripping agent (i.e. water) from zero alcohol content at the beginning of each cycle was enriched in ethanol up to a concentration equal to or about a half of the percentage ethanol loss observed in the beer depending on which volume of stripping agent was used (respectively for $V_s/V_f$ equal to 1 and 2).

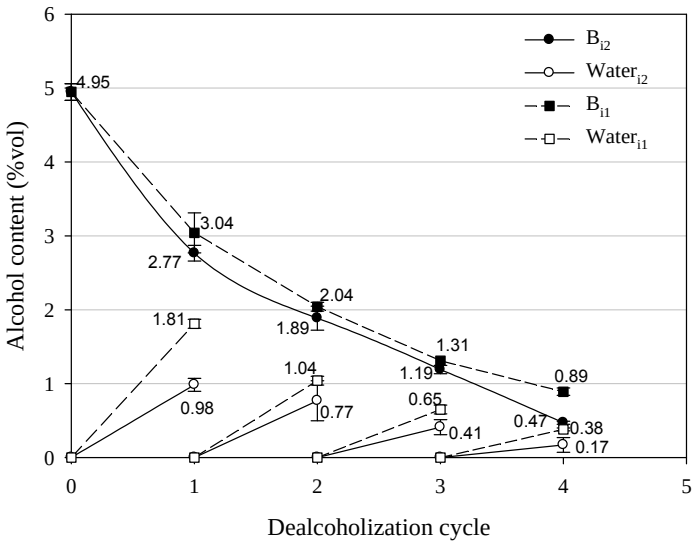



*Figure 2. Comparison of alcohol content in feed and stripping streams during beer dealcoholization using $V_s/V_f$ = 1 and 2.*

### 3.2 Chemical and physical analyses

Regular beer ($B_0$) and low alcohol ($B_{41}$ and $B_{42}$) beer samples, obtained in the two different operating conditions, were analyzed for some chemical properties such as pH, colour and volatile compounds content and healthy treats as antioxidant activity and polyphenols.

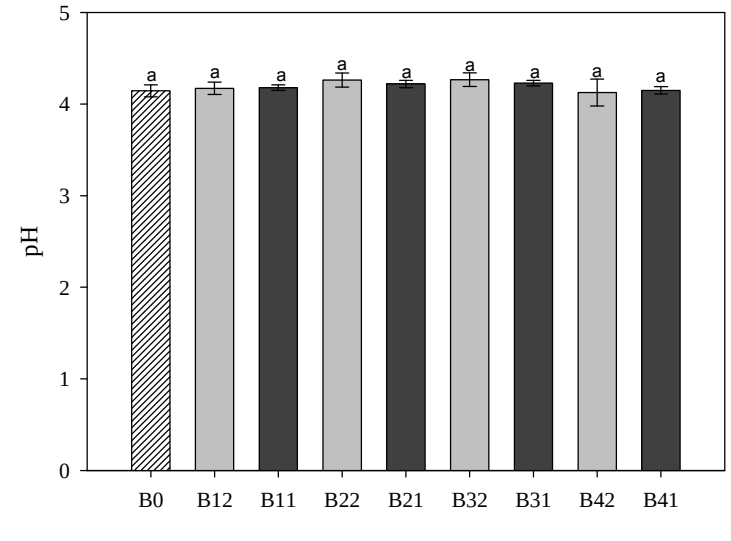
The pH of beer is an important parameter because low value causes a good aroma stability and resistance to microbial contaminations (Smedley, 1992). The pH of different beer samples was evaluated and reported in figure 3a. During the process, no difference of pH was observed among regular beer and low alcohol beer samples obtained using $V_s/V_f$ = 1 and 2. The values were similar to those reported in other studies (Russo et al., 2013; Branyik et al., 2012; Harmanescu et al., 2006).

One of the first indications of wholesomeness of product is the visual appearance that, in case of beer, is due to reproducible foam and colour which are considered key quality targets (Smedley, 1992). The colour was measured on regular and dealcoholized beer samples at different concentrations, reported in figure 3b as EBC units. The obtained values were in the range 4-18 (EBC scale), typical of lager beers (Shellhammer, 2009). The dealcoholization process had not influenced on the beer colour, which differences among dealcoholized beers appeared to be not statistically significant ($p < 0.05$).

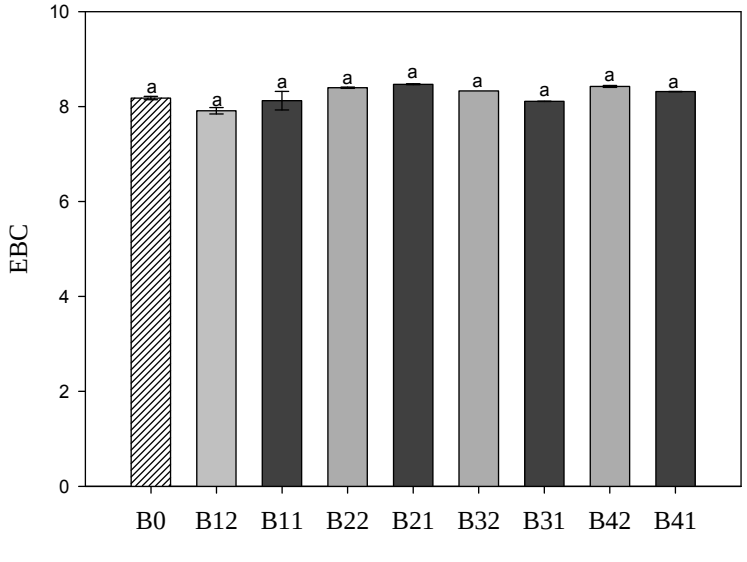
The antioxidant capacity of beverages, and especially of food, has attracted considerable interest, since antioxidants are believed to possess protective properties against numerous pathologies. Beer is increasingly recognized as a rich source of bioavailable dietary antioxidants (Montanari *et al* ., 2009; Nardini and Ghiselli, 2004), which are able to exert several positive effects on health (Halliwell and Gutteridge, 1999). The antioxidant power of beer therefore presumably depends on the antioxidant content of barley and hop and, on a number of parameters involved in brewing process (i.e. malting process, temperature, pH, mashing, boiling). The main antioxidant compounds in beer are phenolic and Maillard compounds. In addition, some antioxidant additives used in beer (i.e. vitamin C) may also contribute to its antioxidant capacity (Saura-Calixto F. et al., 2009). Polyphenols and melanoidins are the major natural antioxidants in beer, which content is largely influenced by genetic and agricultural factors in the raw material and by technological factors in the brewing because Maillard reaction products are largely formed during the malting and brewing process (Saura-Calixto F. et al., 2009). The most widely studied constituents of beer's antioxidant fraction are phenolic compounds, which derived from both the hop and malt components (Gerhauser, 2005). Common beer polyphenols include flavonols, phenolic acids, catechins, procyanidins, tannins and chalcones. The polyphenols amount in regular beer ($B_0$) was 236 mg GAE/L and it remained almost unchanged in all beer samples ($B_{11}$-$B_{41}$; $B_{12}$-$B_{42}$) (figure 4a).

Beer has greater antioxidant capacity than white wine, but less than red wine (Saura-Calixto F. et al., 2009) and various assays are described in literature for the determination of antioxidant capacity (Ribeiro Tafulo et al. 2010).

The antioxidant activity of beer was measured by DPPH assay and results pointed out that the lager beer used in the tests displayed antioxidant properties, comparable to those reported by Harmanescu et al. (2006). The antioxidant capacity remained almost unchanged in the different alcoholic beer samples (figure 4b),.

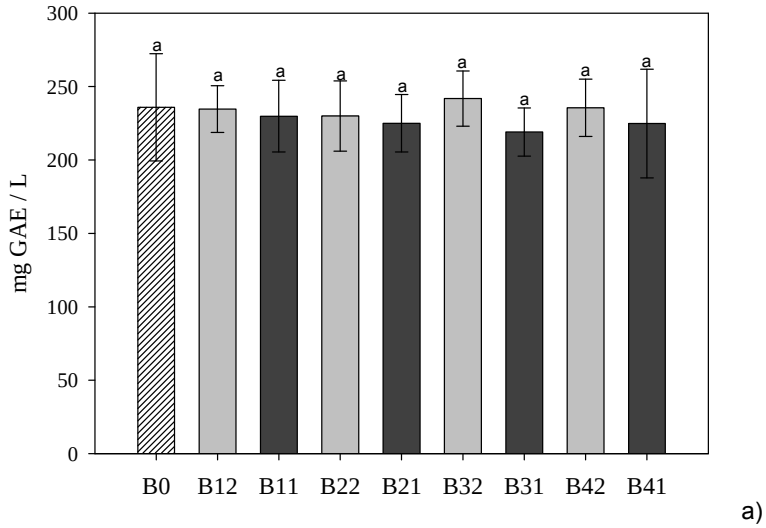



a)

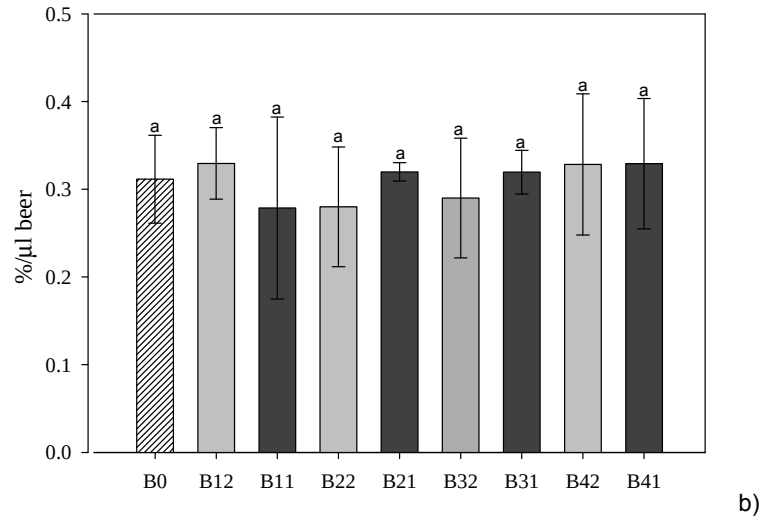



b)

*Figure 3. pH (a) and EBC colour (b) of regular ($B_0$) and low alcohol beer samples ($B_{ij}$) using $V_s/V_f = 2$ and 1.*




a)




b)

*Figure 4. Polyphenols (mg GAE/L) (a) and antioxidant activity (b) of regular ($B_0$) and low alcoholic beer samples ($B_{ij}$) using $V_s/V_f = 2$ and 1.*

Volatile composition of beer is another important parameter that affects the perceived quality by consumers during its consumption. The volatile profile of regular beer was reported in table 1. Totally, 21 compounds were detected belonging to the chemical classes such as alcohols, esters, aldehydes and ketons.

The main volatile compounds are formed during beer fermentation and are characteristic of a finished beer, especially higher alcohols and esters, which concentrations have a great impact on beer organoleptic and sensory quality (Kunze, 1999). They were respectively 53 and 10% over the total volatile compounds, followed by acetaldehyde, the most abundant aldehyde detected in beer; a low concentration of ketons (about 3%) was detected

The dealcoholization of beer by osmotic distillation involved volatile compounds transfer together with ethanol during the process, causing their depletion in the final beer. In fact, the table 1 highlighted high percentage of volatile compounds loss in dealcoholized samples using both $V_s/V_f$ equal to 1 and 2.

Among alcohols, in regular beer ($B_0$) amyl alcohols, propanol and 2-phenylethanol were found in higher concentration with respect to isobutanol and furfuryl alcohol. Usually, their concentration in beer depends on the

yeast used and, in particular, on the fermentation conditions. The decrease of higher alcohols occurred in beer at the end of the process in the same extent (about 60%) for both conditions evaluated.
Esters affect mainly the floreal, fruity and solvent-like flavours (Hughes, 2009). The most abundant of five esters detected in regular beer was ethyl acetate. As observed for higher alcohols, the percentage decrease was similar in $B_{41}$ and $B_{42}$ samples about 89.4% and 92.5%, respectively.
Aldehydes are flavour-active compounds in beer that can contribute to the negative flavour of beer (i.e. green apple, cardboard-like flavours) (Hughes, 2009). Acetaldehydes in regular beer was 52.6 mg/L and together with other aldehydes, responsible of the "aged" flavour of beer (Gijs et al., 2000), were reduced of about 97% in both beer after the dealcoholization process. Only two ketones were detected in beer: diacetyl and 2,3-pentanedione, which are considered off-flavours of beer. They were almost completely lost in dealcoholized beer samples

*Table 1. Volatile compounds content in regular ($B_0$), and low alcohol ($B_{42}$, $B_{41}$) beers.*

| **Volatile compounds (mg/L)** | **Regular beer ($B_0$)** | | **Low alcohol beer ($B_{42}$)** | | **Low alcohol beer ($B_{41}$)** | |
|---|---|---|---|---|---|---|
| | x | σ | x | σ | x | σ |
| **Alcohols** | | | | | | |
| n-Propanol | 18.14 | 0.23 | 1.39 | 0.267 | 1.42 | 0.019 |
| Isobutanol | 3.02 | 0.03 | 1.92 | 0.633 | 2.08 | 0.074 |
| 3-Methyl-1-butanol | 23.61 | 0.50 | 7.00 | 2.075 | 7.78 | 0.488 |
| 2-Methyl-1-butanol | 9.39 | 0.13 | 2.39 | 0.811 | 2.71 | 0.194 |
| Furfuryl alcohol | 2.18 | 0.09 | 1.30 | 0.465 | 0.83 | 0.057 |
| 2-phenylethanol | 26.63 | 2.37 | 17.66 | 2.735 | 19.02 | 0.877 |
| **Sum** | **82.97** | **3.35** | **31.66** | **6.99** | **33.84** | **1.71** |
| **Esters** | | | | | | |
| Ethyl acetate | 13.61 | 0.15 | 1.01 | 0.704 | 1.44 | 0.080 |
| Ethyl butyrate | 0.04 | 0.01 | 0.00 | 0.004 | 0.01 | 0.000 |
| Isoamyl Acetate | 1.31 | 0.08 | 0.13 | 0.081 | 0.18 | 0.004 |
| Ethyl caproate | 0.17 | 0.02 | 0.02 | 0.004 | 0.03 | 0.000 |
| Ethyl caprylate | 0.60 | 0.06 | 0.02 | 0.007 | 0.02 | 0.000 |
| **Sum** | **15.74** | **0.31** | **1.18** | **0.80** | **1.67** | **0.08** |
| **Aldehydes** | | | | | | |
| Acetaldehyde | 52.616 | 3.90 | 1.294 | 0.259 | 1.601 | 0.176 |
| 2-Methylbutanal | 0.023 | 0.00 | 0.005 | 0.002 | 0.006 | 0.000 |
| 3-Methylbutanal | 0.131 | 0.01 | 0.015 | 0.005 | 0.018 | 0.001 |
| Hexanal | 0.002 | 0.00 | 0.002 | 0.001 | 0.002 | 0.000 |
| Furfural | 0.309 | 0.01 | 0.029 | 0.010 | 0.034 | 0.002 |
| methional | 0.057 | 0.00 | 0.011 | 0.003 | 0.012 | 0.001 |
| Phenylacetaldehyde | 0.123 | 0.00 | 0.015 | 0.004 | 0.017 | 0.001 |
| Trans-2-nonenal | 0.000 | 0.00 | 0.000 | 0.000 | 0.000 | 0.000 |
| **Sum** | **53.26** | **3.93** | **1.37** | **0.28** | **1.69** | **0.18** |
| **Ketons** | | | | | | |
| diacetyl | 2.33 | 0.01 | 0.023 | 0.008 | 0.027 | 0.003 |
| 2,3 pentanedione | 2.33 | 0.06 | 0.008 | 0.003 | 0.010 | 0.001 |
| **Sum** | **4.65** | **0.07** | **0.03** | **0.01** | **0.04** | **0.00** |

## 4. Conclusions

The osmotic distillation allows to obtain low alcohol beers with chemical and physical properties similar to the original one, but a lack of volatiles compounds occurred during the process. In particular, no significant differences of all investigated parameters (pH, colour, polyphenols content and antioxidant activity) were detected on beer samples dealcoholized using a volume ratio between stripping agent and feed solution (i.e. beer) equal to 1 and 2. Among the volatile compounds, the lowest percentage losses were found for alcohols, while the highest for esters aldehydes. In particular, the significant reduction of aldehydes is advantageous because they are responsible of the “aged” flavour of beer.
Hence, the low alcohol beers maintain the properties (i.e. polyphenols) of beer that exerts healthful effects on the human body and does not provide the adverse effects of high alcohol intake. The retention of volatile compounds remains a very difficult task to achieve in order to produce a beer as close as possible to the conventional types from sensory point of view and future work will be devoted to this aim.

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