Hungarian University of Agriculture and Life Sciences

Studying the Application of Membrane Separation Processes in the Brewing Industry

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LIST OF LEGEND AND ABBREVIATIONS

μ_p	dynamic viscosity of the permeate	(Pas)
μ_w	dynamic viscosity of water at given temperature	(Pas)
a	slope of calibration curve	(mL mg^{-1})
A_{275}	absorbance at 275 nm	
A_{430}	absorbance at 430 nm	
A_{760}	absorbance at 760 nm	
AFB	alcohol-free beer	
A_G	absorbance of the Glycine standard solution at 570 nm	
A_m	membrane active surface area	(m^2)
A_s	absorbance of the sample at 570 nm	
B	bitterness	IBU
BDA	beer dealcoholization	
BMF	beer membrane filtration	
C	colour	EBC
CAPEX	capital expenditures	
C_b	bulk concentration	(g L^{-1})
C_{bi}	bulk concentration of the component *i*.	(g L^{-1})
c_{EtOH}	ethanol content in permeate	(% (w/w))
CFMF	crossflow microfiltration	
CFRO	crossflow reverse osmosis	
C_g	gel-layer concentration	(g L^{-1})
C_{Gly}	concentration of Glycine standard solution	(mg L^{-1})
C_p	permeate concentration	(g L^{-1})
C_{pi}	permeate concentration of the component *i*	(g L^{-1})
d	size of particles in diameter	(μm)
DE	Diatomaceous Earth	
DI	dialysis	
EBC	European Brewery Convention	
ED	electrodialysis	
f	dilution factor	
FAN	free amino nitrogen	(mg L^{-1})
FO	forward osmosis	
FR	flux recovery	(%)

GS	gas separation	
J	flux	(L m^{-2} h^{-1})
J_0	initial flux	(g m^{-2} h^{-1}, L m^{-2} h^{-1})
$J_{b\,0}$	initial beer flux	(L m^{-2} h^{-1})
$J_{b\,ss}$	steady-state beer flux	(L m^{-2} h^{-1})
J_{EtOH}	ethanol flux	(g m^{-2} h^{-1})
$J_{EtOH\,0}$	initial ethanol flux	(g m^{-2} h^{-1})
J_m	mass flux	(g m^{-2} h^{-1})
J_p	permeate flux	(L m^{-2} h^{-1})
J_{ss}	steady-state flux	(L m^{-2} h^{-1})
J_t	flux at any time	(L m^{-2} h^{-1})
$J_{w\,0}$	water flux before separation	(L m^{-2} h^{-1})
$J_{w\,w}$	water flux after membrane cleaning	(L m^{-2} h^{-1})
$J_{wrt\,0}$	initial wort flux	(L m^{-2} h^{-1})
$J_{wrt\,ss}$	steady-state wort flux	(L m^{-2} h^{-1})
K	flux decline coefficient	(h^{-1})
LAB	low-alcohol beer	
LM	liquid membrane processes	
m	permeate mass	(g)
MBR	membrane bioreactor	
MC	membrane contactors	
MD	membrane distillation	
ME	membrane emulsification	
m_{EtOH}	mass of ethanol in permeate	(g)
MEX	membrane extraction	
MF	microfiltration	
MGA	membrane gas absorption	
MR	membrane reactor	
MSP	membrane separation process	
MWCO	molecular weight cut-off	
NF	nanofiltration	
NIR	near-infrared	
NTU	nephelometric turbidity units	
OPEX	operating expenses	
p_0	pressure of the permeate	(bar)
p_1	inlet pressure	(bar)
p_2	outlet pressure	(bar)
PV	pervaporation	
Q	retentate flow rate	(L h^{-1})
Re	Reynolds number	
R_f	fouling layer resistance	(m^{-1})
$R_{f\,0}$	initial fouling layer resistance	(m^{-1})
$R_{f\,ss}$	steady-state fouling layer resistance	(m^{-1})
R_i	retention of the component i	%
R_m	intrinsic resistance of clean membrane	(m^{-1})
R_n	intrinsic resistance of the membrane after membrane cleaning	(m^{-1})

RO	reverse osmosis	
RPM	revolution per minute	(1 min^{-1})
R_t	total resistance	(m^{-1})
S	amount of the sample	(µL)
SG	silica gel	
SGC	silica gel concentration	(g hL^{-1})
STP	static turbulence promoter	
t	time	(h)
t_i	time interval	(h)
TMP	transmembrane pressure	(bar)
TPC	total polyphenol content of the sample	(mg GAE mL^{-1})
UF	ultrafiltration	
V	permeate volume	(L)
VCF	volume concentration factor	(m^3 m^{-3})
V_f	volume of the feed	(m^3)
VFD	variable-frequency drive	
V_r	final volume of the retentate	(m^3)
VSEP	Vibratory Shear Enhanced Process	
V_t	total sample volume	(µL)
WMF	wort microfiltration	
x	distance	
Y	response	
δ	thickness of the boundary layer	m
ΔP	applied pressure	(Pa)
ρ_{EtOH}	ethanol density at given temperature	(g L^{-1})

1. INTRODUCTION

1.1. The importance of the subject

In our time the brewing industry (production of beer), as the part of the food industry, faces several global challenges, and these below-mentioned challenges must be solved as soon as possible.

Firstly, although the art of brewing has a long tradition, there is a growing demand in the brewing industry for developing and applying energy-saving, environmentally friendly and sustainable alternative processes and technologies with the aid of innovation using less energy and no chemicals.

Secondly, brewers reasonably want to be cost effective. Thus, they are making efforts to minimize their capital expenditures (CAPEX) and operating expenses (OPEX).

Thirdly, brewers and brewing scientists are constantly striving to improve product quality because of consumer demands.

Finally, as consumer behaviour and consumer demand are changing, the conscious consumption of products such as low alcohol beer (LAB) and alcohol-free beer (AFB) come to the fore.

Fortunately, membrane separation processes (MSPs), a cleaner technology with efficient separation capability and generally mild operating conditions compared to conventional technologies, can be the solution to the above-mentioned challenges. Thus, it is obvious that MSPs have become an emerging technology in the brewing industry.

Since MSPs are still an emerging technology in the brewing industry, studying the application of MSPs in the brewing industry, the scope of this thesis, is essential and relevant.

1.2. Objectives to achieve

The objectives of the study are discussed in the following subchapters.

1.2.1. Objectives of wort membrane filtration

The objectives of wort membrane filtration (WMF) were the following:

- Complete removal of hot trub and cold trub from pale hopped wort by membrane filtration.
- Studying the effect of membrane filtration on the changes in analytical

parameters.

- Determination of permeate flux values.

1.2.2. Objectives of beer membrane filtration with static turbulence promoter

The objectives of beer membrane filtration (BMF) with static turbulence promoter (STP) were the following:

- Developing a model to describe the BMF with STP process.
- Determination of the effect sizes of the significant parameters of the model.
- Finding the optimum and the optimal values of the significant parameters of the model.

1.2.3. Objectives of beer membrane filtration with silica gel

The objectives of beer membrane filtration (BMF) with silica gel (SG) were the following:

- Developing a model to describe the BMF with SG process.
- Determination of the effect sizes of the significant parameters of the model.
- Finding the optimum and the optimal values of the significant parameters of the model.

1.2.4. Objectives of membrane cleaning for beer membrane filtration

The objectives of membrane cleaning for BMF were the following:

- Recovering the initial intrinsic resistance of the microfiltration membrane.
- Developing a novel and efficient membrane cleaning method for beer membrane filtration.

1.2.5. Objectives of beer dealcoholization by reverse osmosis

The objectives of beer dealcoholization (BDA) by reverse osmosis (RO) were the following:

- Developing a model to describe the BDA by RO process.
- Determination of the effect sizes of the significant parameters of the model.
- Finding the optimum and the optimal values of the significant parameters of the model.

1.2.6. Objectives of membrane cleaning for beer dealcoholization by reverse osmosis

The objectives of membrane cleaning for beer dealcoholization by reverse osmosis

were the following:

- Recovering the initial intrinsic resistance of the reverse osmosis membrane.
- Developing a novel and efficient membrane cleaning method for beer dealcoholization by reverse osmosis.

1.3. Problems to be solved

1.3.1. Problems to be solved of wort membrane filtration

The problems to be solved of the WMF investigation are (i) to determine particle size distributions to study the removal of the hot trub and the cold trub; (ii) to determine analytical properties of original wort (feed) and permeate; (iii) to determine the retentions of different essential components; (iv) to determine initial flux and the steady-state flux values of the WMF with given operating parameters.

1.3.2. Problems to be solved of beer membrane filtration with static turbulence promoter

The problems to be solved of the BMF with SG investigation are (i) to determine the analytical parameters of rough beer and permeate samples (dynamic viscosity values for the physical modelling); (ii) to determine the hydrodynamic parameters of the membrane filtrations for the response (physical modelling) of the experimental design; (iii) to analyse the experimental design (mathematical modelling) of the membrane filtrations (parameter and effect size estimation); (iv) to optimise the objective function (the mathematical model) extracted from the analysis of the experimental design; and (v) to develop an effective membrane cleaning method for MF processes.

1.3.3. Problems to be solved of beer membrane filtration with silica gel

The problems to be solved of the BMF with SG investigation are (i) to determine the analytical parameters of rough beer and permeate samples (dynamic viscosity values for the physical modelling); (ii) to determine the hydrodynamic parameters of the membrane filtrations for the response (physical modelling) of the experimental design; (iii) to analyse the experimental design (mathematical modelling) of the membrane filtrations (parameter and effect size estimation); and (iv) to optimise the objective function (the mathematical model) extracted from the analysis of the experimental design.

1.3.4. Problems to be solved of beer dealcoholization by reverse osmosis

The problems to be solved of the BDA by RO investigation are (i) to determine the analytical parameters of beer and permeate samples (ethanol content values for the

physical modelling); (ii) to determine the hydrodynamic parameters of the membrane separations for the physical modelling; (iii) to calculate the ethanol flux values of the membrane separations for the response (physical modelling) of the experimental design; (iv) to analyse the experimental design (mathematical modelling) of the membrane separations (parameter and effect size estimation); (v) to optimise the objective function (the mathematical model) extracted from the analysis of the experimental design and (vi) to develop an effective membrane cleaning method.

2. OVERVIEW OF LITERATURE

2.1. About beer and brewing

Beer is one of the most popular beverages all over the world (Wunderlich & Back, 2008). The legal definition of beer varies from country to country (Campbell, 2013). According to Codex Alimentarius Hungaricus (Ministry of Agriculture of Hungary, 2013) (regulations about beer in Hungary, the country of this study), beer has to be mashed with water from malt and adjuncts, flavoured with hops, fermented with brewer's yeast, richly carbonated, usually alcoholic beverage. Brewing means making beer (Cambridge Dictionary, 2021).

In order to get a comprehensive picture about brewing, the brewing ingredients and brewing process are discussed in the following subchapters (*Chapter 2.1.1* and *Chapter 2.1.2*).

2.1.1. Brewing ingredients

Water

Beer contains more than 90 % water (Okafor et al., 2016) and brewing is a water-consuming process (Fillaudeau et al., 2006).

Water can be used as an ingredient in several stages of the beer production: product water, sparge water, pushing water and product dilution water (Palmer & Kamiski, 2013).

The production of different beer styles requires different water types, but nowadays the raw water can be treated in several ways (Eumann & Schildbach, 2012). The quality of the water influences the flavour of the beer, the complex enzyme activity of the mash and the other steps of the brewing process (e.g. fermentation) (Comrie, 1967).

Malt

Malt is grain that has been steeped, germinated, and kilned (Mallett, 2014a; Power, 1993). Malt is stable and rich in enzymes and extract. The enzymes derived from malt hydrolase the starch to dissolved sugars and other compounds during mashing (part of brewing process) (Power, 1993).

Usually, malt is made from seeds of the barley plant (Pires & Bránayik, 2015a), but other grains can be used such as wheat, rye, sorghum, oats, triticale, corn, rice and millet. Furthermore, pulses and legumes (like beans and peas) and pseudocereals

(quinoa, buckwheat and amaranth) can also be malted (Cela et al., 2020; Mallett, 2014b).

Base malts supply the essential elements (extract, free amino nitrogen (FAN), and basic malty flavour) needed for beer production, while speciality malts (high-dried, caramelized, roasted) add diversity and complexity to beer (Mallett, 2014c). It is important to note that roasted barley is technically not a malt, and it adds a dry roasted and distinct coffee-like flavour, and significant amount of colour to the final beer (Mallett, 2014c; Mosher & Trantham, 2017a).

Adjuncts

The use of adjuncts (alternative sugar and starch sources) in addition to malt in brewing is not essential, but it can provide benefits in extract cost and beer quality (Lloyd, 1986; Pires & Bránayik, 2015b).

The typically used adjuncts are the following: unmalted barley, wheat, rice and corn. Other sugar sources such as starch, sucrose, glucose, and their syrup also can be used (Pires & Bránayik, 2015b).

Hops

The cones of the female plant of hops (*Humulus lupulus* L.) are grown for the brewing industry (Almaguer et al., 2014).

There are four types hop products that are used in the brewing industry: bale hops, pellets, kettle extracts, postfermentation extracts (Roberts, 2016).

Hops have many attributes that play important role in brewing:

- Bitterness
- Aroma
- Flavour
- Mouthfeel
- Foam and lacing
- Flavour stability
- Anti-microbial effect

  (Hieronymus, 2012)

Yeast

Yeast converts sugar to ethanol, carbon dioxide, and other compounds that influence the taste of the beer (White & Zainasheff, 2010a). There are two main species of brewer's yeast: *Saccharomyces cerevisiae* (ale) and *Saccharomyces pastorianus* (lager) (White & Zainasheff, 2010b). The most important differences between the two main brewer's yeasts are discussed below.

The fermentation temperature of the top fermenter ale yeast is 18 – 22 °C and its maximum growth temperature is 37 °C or higher. The fermentation temperature of the bottom fermenter lager yeast is 8 – 15 °C and its maximum growth temperature is 34 °C. It is important to note that this yeast ferments melibiose. There are further differences in uptake and metabolism of amino acids, yeast flocculation, yeast management between fermentations and yeast strain genetic stability (Stewart et al., 2013).

Process aids

In the brewing industry several process aids are used due to quality and economic aspects (Ryder & Power, 2006). These process aids can be classified as follows:

- Brewing water treatment agents
- Brewing enzymes
- Yeast nutrients
- Defoaming agents
- Clarifiers and fining agents
- Stabilizing agents and filtration aids
- Gases (carbon dioxide and nitrogen)

  (Lewis & Young, 2001; Ryder & Power, 2006)

2.1.2. The brewing process

Figure 1 shows the flow diagram of the brewing process.

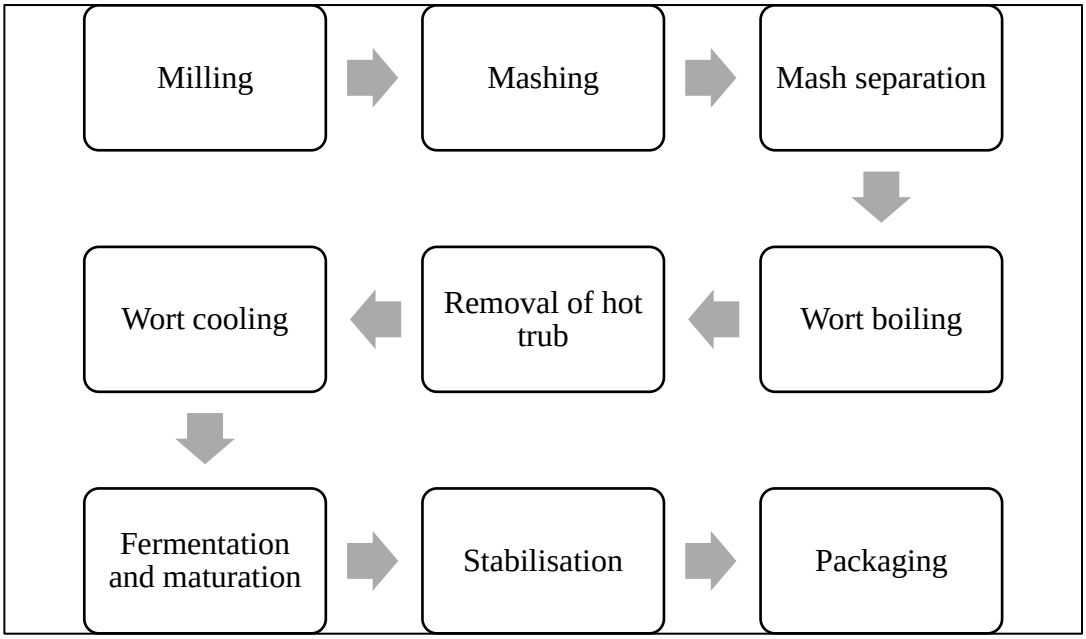

Figure 1: Flow diagram of the brewing process (based on Ambrosi et al., 2014a; Kunze, 2004a; Schneider & Weisser, 2004)

Milling

Before the mashing process the kernels are ground to the suitable size (Kunze, 2004b). It means that milling has to be performed in such a way that the husk of the kernel be intact, which forms a filter bed during the mash separation and lautering, and the starch endosperm be a fine grist to maximize enzymatic hydrolysis and extraction (Bamforth, 2017).

Mashing

During mashing the grist is mixed with water to give as much soluble extract as possible. A gradual increase in temperature is applied to the mash to activate enzymes for the malt (Ambrosi et al., 2014; Kunze, 2004b).

There are three main types of enzymes that play a role in the mashing process: β-glucanase, proteases and amylases (β-amylase, α-amylase) (Gomaa, 2018).

β-glucanase generally hydrolyzes the 1-3 β-glycosidic bonds between glucose molecules in glucans. This reaction is important in mashing because it decreases the viscosity of the wort (Gomaa, 2018). The pH and temperature optimum of β-glucanase are 6.0 and 45 – 55 °C, respectively (Fix, 1999a; Gomaa, 2018).

Proteases catalyses the hydrolysis of peptide bonds in proteins. Protease increases the degree of solubility of the proteins and enhances yeast cell growth by satisfying the availability of FAN. During mashing the hydrolysis of the kernel cell wall proteins by proteases enhances the exposure of the starch to the mashing enzymes, results better mashing and wort fermentability. Furthermore, proteases affect the quality of the beer foam (Gomaa, 2018). Proteases have a tendency for acidic pH and their temperature optimal temperature range is 47 – 52 °C (Fix, 1999a; Gomaa, 2018).

During mashing amylases are utilized to convert the starch into fermentable sugars. β-amylase catalyses the hydrolysis of amylose and amylopectin into maltose by breaking the external α (1-4) glycosidic bonds. The pH and temperature optimum of β-amylase are 5.5 and 60 – 65 °C, respectively (Fix, 1999a; Gomaa, 2018). α-amylase catalyses the hydrolyses of starch's two large macromolecules, amylose and amylopectin into dextrins by breaking the internal α (1-4) glycosidic bonds between the α-glucose molecules (Gomaa, 2018). The pH and temperature optimum of α-amylase are 5.2 and 65 – 70 °C, respectively (Fix, 1999a; Gomaa, 2018).

Mash separation

Mash separation comprises two unit operations (lautering and sparging). Firstly, the soluble extract in the wort (the liquid) is separated from the insoluble material, the spent grains (Bamforth, 2017; Kunze, 2004b; Mosher & Trantham, 2017b; Schneider & Weisser, 2004). Secondly, the grains are washed with water typically at 78 °C to completely deplete the sugars (Ambrosi et al., 2014).

Wort boiling

During the wort boiling the wort is boiled with hops. Furthermore, this process concentrates the wort with evaporation of the water, inactivates the enzymes, sterilises the wort and coagulates proteins (Ambrosi et al., 2014; Kunze, 2004b).

Removal of hot trub

After the wort boiling, the wort is transferred (casting the wort) for removing the hot trub. The hot trub has to be removed because it can cause technological and product quality problems (Kunze, 2004c).

Wort cooling

After the removal of hot trub, the hot wort must be cooled to the temperature of the

yeast pitching. In addition, the wort must be oxygenated to an appropriate level, because in the initial stage of the fermentation the freshly pitched yeast needs dissolved oxygen (Briggs et al., 2004a).

Fermentation and maturation

During the fermentation process, the sugars in the wort are fermented to ethanol and carbon dioxide by yeast and other by-products are also formed (Kunze, 2004d). The (primary) fermentation results the green beer which is hazy and has an unacceptable flavour. During the maturation (after the fermentation) aroma and flavour changes, carbonization and natural sedimentation take place at low temperature (≤ -1 °C) (Briggs et al., 2004b).

Stabilisation

The stability of the beer for the duration of the sell by date is very important. There are three types of stability: microbiological (contaminants), colloidal (haziness) and flavour (changes with time). There are several methods for improving the stability of the rough beer: pasteurisation, filtration and addition of stabilising agents (Kunze, 2004e).

Packaging

Before the sale, the beer has to be packaged. The "package" can be small-pack beer or draught beer. Small-pack beer involves bottle (returnable and non-returnable) and can, while draught beer involves keg and cask (Briggs et al., 2004c).

2.2. Membrane separation processes

2.2.1. Membrane separation processes in the food industry

Membrane separation processes are widely used in the following sub-sectors of food industry: dairy, meat, fruit and vegetables, bread and milling, sugar, fruit juice, beverages (e.g. wine, beer, tea) (Nath, 2017a).

Applications of membrane separation processes in the food industry can be the following:

- Purification: biochemical/chemical stabilization, microbial stabilization, composition correction (demineralization, pH adjustment, dealcoholization)
- Concentration
- Extraction

- Separation
- Effluent treatment: effluent treatment before discharging, effluent treatment to allow water (or solutions) recycling into processes, effluent treatment for by-product valorisation
  (Guiga & Lameloise, 2020)

2.2.2. Fundamentals of membrane separation processes

Membrane is a selective thin layer of a semipermeable material, which separates the undesired materials (components) from the feed solution based on their sizes or affinity by applying potential gradient (pressure, temperature, electrical or concentration difference) as driving force (Asad et al., 2020).

Figure 2 shows the schematic representation of a membrane separation process (MSP) (based on Bélafi-Bakó et al., 2000a).

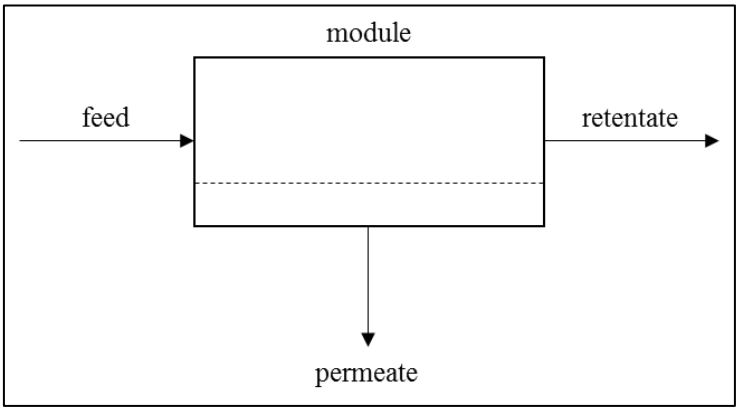

Figure 2: Schematic representation of a membrane separation process (MSP) (based on Bélafi-Bakó et al., 2000a)

During a MSP the feed stream is divided into retentate or concentrate stream and permeate stream.

2.2.3. Basic formulas

Retention of a component during MSP can be determined with *Equation 1* (Basu & Balakrishnan, 2017):

$$R_i = \left(1 - \frac{C_{p_i}}{C_{b_i}}\right) \times 100 \qquad \text{Equation 1}$$

where R_i is the retention (%) of the component i, C_{pi} (g L^{-1}) is the permeate concentration of the component i and C_{bi} (g L^{-1}) is the bulk concentration of the component i.

Permeate flux (volume based) during MSP can be determined with *Equation 2* (Gáspár et al., 2011):

$$J = \frac{V}{A_m \times t_i} \qquad \text{Equation 2}$$

where J (L m^{-2} h^{-1}) is the flux, V (L) is the permeate volume, A_m (m^2) is the membrane active surface area and t_i (h) is the time interval.

Permeate flux (mass based) during MSP can be determined with *Equation 3* (Catarino et al., 2007):

$$J_m = \frac{m}{A_m \times t_i} \qquad \text{Equation 3}$$

where J_m (g m^{-2} h^{-1}) is the mass flux and m (g) is the permeate mass.

Transmembrane Pressure of MSP can be determined with *Equation 4* (Ben Hassan et al., 2013):

$$TMP = \frac{p_1 + p_2}{2} - p_0 \qquad \text{Equation 4}$$

where TMP (bar) is the Transmembrane Pressure, p_1 (bar) is the inlet pressure, p_2 (bar) is the outlet pressure and p_0 (bar) is the pressure of the permeate.

Volume concentration factor of MSP can be determined with *Equation 5* (McCarthy et al., 2017):

$$VCF = \frac{V_f}{V_r} \qquad \text{Equation 5}$$

where VCF (m^3 m^{-3}) is the volume concentration factor, V_f (m^3) is the volume of the feed and V_r (m^3) is the final volume of the retentate.

2.2.4. Membrane classification

The membrane is the most important part of the separation process (Scott & Hughes, 1996a). *Table 1* shows the classification of different membranes (based on Hsieh, 1996; Kislik, 2010; Nath, 2017b; Scott & Hughes, 1996b).

Table 1: Classification of different membranes (based on Hsieh, 1996; Kislik, 2010; Nath, 2017b; Scott & Hughes, 1996b)

Classification aspect	**Types**	**Examples**
Method of production	natural	membranes in all life forms
	synthetic	ceramic membranes
State	solid	ceramic membranes
	liquid	emulsion liquid membranes
Material	organic	polyester, polysulfone
	inorganic	ceramic, metal, glass
Structure	microporous	microporous ceramic membranes
	nonporous	RO membranes
	symmetric	symmetric microporous phase cellulosic esters
	asymmetric (skinned)	asymmetric cellulosic esters
	thin film composite	composite polyamide membranes
Charge	neutral	not ion exchange membranes
	electrically charged	electrodialysis membranes

An effective membrane of a specific MSP should meet the following criteria: chemical resistance (to both feed and cleaning fluids), mechanical stability, thermal stability, high permeability, high selectivity, and stable operation (Scott & Hughes, 1996c).

2.2.5. Membrane modules

In practice membranes are installed in a suitable device and this device is termed as membrane module (Nath, 2017c).

There are basically four different designs of membrane modules. These are the following:

1. **Plate and frame (flat sheet):** a flat sheet membrane is pressed into a plate and frame device. The flat sheets can be put close together and they can be removed for cleaning purposes. The membrane packing density of this module is low.
2. **Tubular:** this module has shell-and-tube design. The feed flows through the tube(s) and the permeate passes through the wall of the tubes into the shell side of the module. The membrane contamination can be minimized with the application of high feed flow rate. The main disadvantage of application of this

module is the small membrane area/module space ratio.

3. **Spiral wound:** this module is formed from a flat sheet packed around a centre collection pipe. These modules are compact, because the membrane packing density is high. Thus, application of spiral wound modules is space-saving. Furthermore, the concentration polarization and the pressure drop at the permeate channel are minimum. The tendency of membrane fouling of this module is medium.
4. **Hollow fibre:** this membrane module consists of bundles of individual fibres. This module has the highest membrane packing density. Unfortunately, the flow channel of the fibres can be blocked easily by feed particles.
   (Field & Lipnizki, 2016b; Fonyó & Fábry, 2004a; Nath, 2017d)

2.2.6. Operation modes

Figure 3 shows the operation modes of MSPs (based on Hsieh, 1996b).

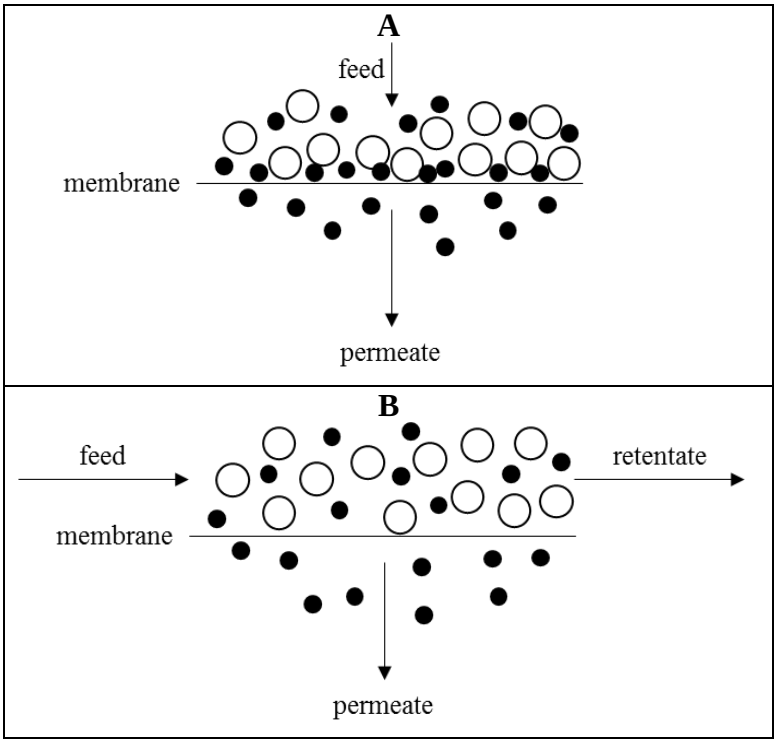

Figure 3: Operation modes of MSPs. A: dead-end; B: crossflow (based on Hsieh, 1996b)

In case of dead-end membrane separation, the feed flow is perpendicular to the membrane surface and the retained particles accumulate on the surface of the membrane forming a filter cake. In crossflow MSP the feed stream flows along almost parallel to the membrane surface. The crossflow mode of operation reduces the effect of a build-up of solid particle cake on the membrane surface (Scott & Hughes, 1996d).

2.2.7. Most common membrane processes

Table 2 shows the summary of the most common membrane processes (Bélafi-Bakó et al., 2000b; Chung, Zhang, Wang, Su, & Ling, 2012; Field & Lipnizki, 2016c; Fonyó & Fábry, 2004b; Ho & Sirkar, 2012; Mimi Sakinah et al., 2014; Piacentini et al., 2014).

Table 2: Summary of the most common membrane processes (Bélafi-Bakó et al., 2000b; Chung et al., 2012; Field & Lipnizki, 2016c; Fonyó & Fábry, 2004b; Ho & Sirkar, 2012; Mimi Sakinah et al., 2014; Piacentini et al., 2014)

Process	**Driving force**	**Mechanism**
microfiltration (MF)	pressure difference	sieving
ultrafiltration (UF)	pressure difference	sieving
nanofiltration (NF)	pressure difference	sieving
reverse osmosis (RO)	pressure difference	diffusion
forward osmosis (FO)	osmotic pressure difference	diffusion
dialysis (DI)	concentration gradient	sieving and hindered diffusion
electrodialysis (ED)	electrical potential difference	counter-ion transport
gas separation (GS)	pressure difference, concentration difference	diffusion
liquid membrane (LM) processes	concentration gradient	diffusion
membrane distillation (MD)	temperature difference	vapour pressure difference
membrane emulsification (ME)	pressure difference	drop-by-drop
membrane extraction (MEX)	concentration gradient	diffusion
membrane gas absorption (MGA)	concentration gradient, solubility difference	selective absorption
membrane reactor (MR)	chemical potential	sorption and diffusion
pervaporation (PV)	concentration gradient, temperature gradient	diffusion

It can be seen that there are many potential processes for the brewing industry.

2.2.8. Most important pressure-driven membrane separation processes

Table 3 shows the characterization of the most importation pressure-driven MSPs (Field & Lipnizki, 2016d; Fonyó & Fábry, 2004b; Mulder & Mulder, 1996; Pal, 2015).

Table 3: Characterization of the most importation pressure-driven MSPs (Field & Lipnizki, 2016d; Fonyó & Fábry, 2004b; Mulder & Mulder, 1996; Pal, 2015)

Process	Pore size	Pressure range (bar)	Application
MF	0.1 – 10 μm	0.1 – 2.0	removal of particles, sterile filtration
UF	5 – 500 nm	1.0 – 5.0	removal of macromolecules
NF	1 – 10 nm	5.0 – 20	removal of sugars, other organic molecules and multivalent salts; concentration
RO	extremely small, < 0.001 μm	10 - 100	removal of monovalent salts and micromolecules

In the investigations of this study, MF and RO MSPs were used.

2.2.9. Membrane fouling

Application of MSPs in the brewing industry has been limited by membrane fouling that results in decrease permeate flux (Kazemi et al., 2013). Thus, serious membrane fouling always lead to high operation costs (Sun et al., 2018).

The reasons of the fouling (flux decline) during BMF illustrated by examples are the following:

1. **Concentration polarization**
2. **Compact cake layer formation** by yeast cells, debris, and coagulated materials on membrane surface
3. **Partial or complete plugging of pore entrances** by suspended particles
4. **Adsorption of macromolecules onto the pore walls** which causes the membrane pore narrowing.
   (Kazemi et al., 2013)

2.2.10. Concentration polarization and gel-layer formation

In gel polarization model, the solute concentration on the membrane surface may be extremely high value and a maximum concentration, the gel-layer concentration (C_g) may be attained for a number of macromolecular solutes. The gel-layer concentration depends on several things: size, shape, chemical structure, and degree of solvation. However, it is independent of the bulk concentration (C_b) (Mulder, 1995a). This model is mainly true for MF and UF (Cheryan, 1998; Fonyó & Fábry, 2004c).

Figure 4 shows the Representation of concentration polarization and gel-layer formation (based on (Fonyó & Fábry, 2004d; Mulder, 1995b)).

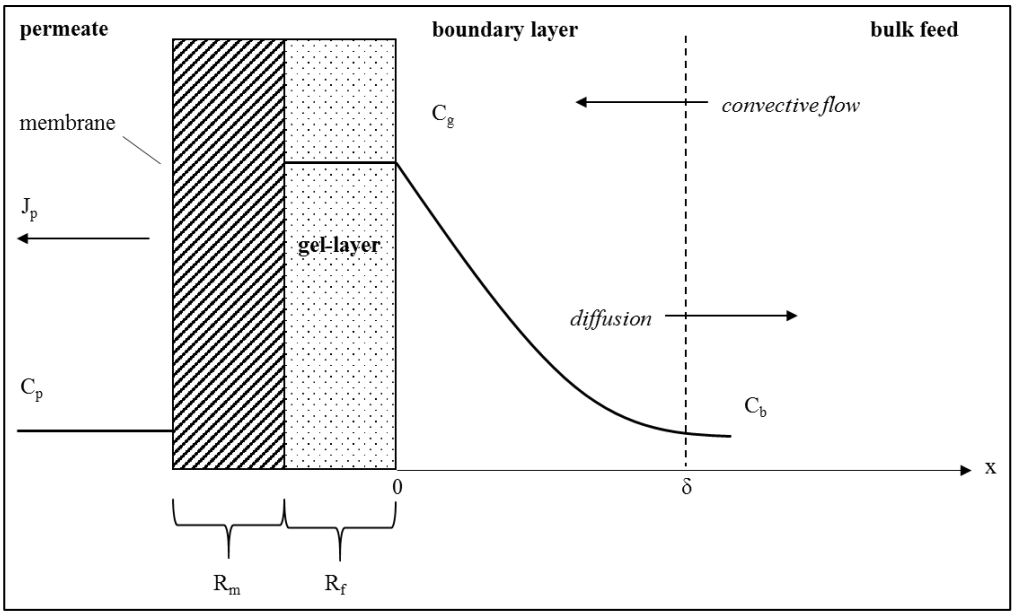

Figure 4: Representation of concentration polarization and gel-layer formation. C_b: bulk concentration; C_g: gel-layer concentration; C_p: permeate concentration; J_p: permeate flux; R_f: fouling layer resistance; R_m: resistance of the membrane; x: distance; δ: thickness of the boundary layer. (based on Fonyó & Fábry, 2004d; Mulder, 1995b).

The process is described by *Equation 6* (Mulder, 1995c):

$$J_p = \frac{\Delta P}{\mu_p \times (R_m + R_f)} \qquad \text{Equation 6}$$

where J_p (L m^{-2} h^{-1}) is the permeate flux, ΔP (Pa) is the applied pressure, μ_p (Pas) is the dynamic viscosity of the permeate, R_m (m^{-1}) is the resistance of the membrane and R_f (m^{-1}) is the fouling layer resistance.

2.2.11. Osmotic pressure model

In case of high flux values, high retentions and low mass-transfer coefficient values, the concentration of macromolecular solutes on the membrane surface can become extremely high. Thus, the osmotic pressures have to be considered (Mulder, 1995d). This model is mainly true for NF and RO (Cheryan, 1998; Cséfalvay, Pauer, & Mizsey, 2009).

The process is described by *Equation 7* (Field, 2016):

$$J_p = \frac{\Delta P - \Delta \pi}{\mu_p \times (R_m + R_f)} \qquad \text{Equation 7}$$

where J_p (L m^{-2} h^{-1}) is the permeate flux, ΔP (Pa) is the applied pressure, $\Delta \pi$ is the

osmotic pressure difference (Pa), μ_p (Pas) is the dynamic viscosity of the permeate, R_m (m^{-1}) is the resistance of the membrane and R_f (m^{-1}) is the fouling layer resistance. If there is no fouling, then $R_f = 0$.

2.2.12. Membrane fouling-prevention and membrane cleaning

As it mentioned in *Chapter 2.2.9,* membrane fouling limits MSPs. Thus, membrane fouling-prevention and membrane cleaning have high impact on membrane process.

Membrane fouling-prevention

There are several ways of preventing membrane fouling and concentration polarization:

1. Feed pre-treatment
2. Boundary layer (velocity) control
3. Application of turbulence inducers and generators
4. Membrane modifications and materials
5. Combined external fields.
   (Lin et al., 2010; Tao et al., 2017)

Membrane cleaning

The aims of the membrane cleaning are to prevent and reduce fouling. Because of the complexity of the fouling, cleaning should involve several variables such as process sequence, hydrodynamic conditions, solution temperature and pH, chemical dosage, and cleaning duration (Shi et al., 2014).

Table 4 shows the summary of the most common membrane cleaning methods (Gao et al., 2019; Lin et al., 2010).

Table 4: Summary of the most common membrane cleaning methods (Gao et al., 2019; Lin et al., 2010)

Cleaning method	**Classification**	**Examples**
physical	hydraulic	forward flushing, backwashing, backflushing backpulsing (backshocking)
	pneumatic	air sparging, air lifting, air scouring, and air bubbling
	mechanical	sponge ball wiping, vibration
	sonication	ultrasound
	combined	air sparging + backflush, ultrasound + forward flushing
chemical	alkaline	NaOH, KOH, NH_4OH
	acids	HCl, HNO_3, H_2SO_4, H_3PO_4, Citric acid, Oxalic acid
	metal chelating agents	EDTA
	surfactants	Alkyl sulfate, SDS, CTAB
	enzymes	Peroxidase
	disinfectants	O_3
	oxidants	H_2O_2, $KMnO_4$
	blended	Ultrasil®

It should be mentioned that chemical cleaning methods can be performed in various ways:

1. Directly immersing the fouled membranes in the chemicals
2. Soaking the fouled membranes in a separate tank with higher concentration cleaning agents
3. Adding chemicals in the feed stream.
   (Lin et al., 2010)

Furthermore, the different membrane cleaning methods can be combined (Lim & Bai, 2003).

Checking of degree of membrane cleanliness

After membrane cleaning, water flux has to be measured at given temperature and Transmembrane Pressure. The purpose of the water flux measurement was checking of degree of membrane cleanliness (Blanpain-Avet et al., 2004). Water flux is affected by temperature and Transmembrane Pressure (Huisman et al., 1997). Thus, the water flux measurement has to be performed with given temperature and Transmembrane Pressure values (same values as the values of the water flux measurement before the separation) to get comparable results.

2.3. Membrane separation processes for the brewing industry

2.3.1. Effect of ingredients and brewing on membrane separation processes

It is important to note that brewing ingredients and brewing process can affect the applications of membrane separation processes for the brewing industry.

As it mentioned in *Chapter 2.2.9*, membrane fouling limits membrane separation processes in the brewery applications. The membrane foulants of brewery products can be the following: carbohydrates (arabinoxylans, β-glucans, starch molecules/particulates), cell debris, minerals, polyphenols, proteins, proteinaceous components, yeast cells (Gan et al., 1997, Stopka et al., 2000, Stkwart et al., 1998).

Thus, brewing ingredients (water, malt, adjuncts, hops and yeast) with low content of foulants should be used during the brewing process and brewery products (e.g. wort, rough beer) with low content of foulants should be used as feed during membrane separation process. Furthermore, the brewery products can be pre-treated with process aids (e.g. enzymes, clarifiers and fining agents) and different unit operations (e.g. centrifugation) (Cimini et al., 2013, Gan et al., 2001).

Proper malting (e.g. starch degradation, degradation of β-glucans), milling (accessibility of extract), mashing (extract conversion, degradation of β-glucans), lautering (appropriate temperature for late saccharification), wort boiling (protein coagulation), whirlpool (hot trub removal), fermentation (with pH decrease proteins can be separated as cold trub), maturation (proteins during maturation adhere onto the yeast and can be discarded with the yeast) during brewing process enhance the application of membrane separation processes (Jin et al., 2004a, Steiner et al., 2010).

2.3.2. Current applications

Microfiltration is the most widely used membrane separation process in the brewing industry because most of the operations related directly to the beer involve solid liquid separation (Ambrosi et al., 2014).

Summary of works and current applications related to MSPs for the brewing industry are shown in *Table 5*.

Table 5: Summary of works and current applications related to MSPs for the brewing industry ('3M - 3M™ Liqui-Cel™ Membrane Contactors Used in the Soft Drink and Brewing Industries to Control Dissolved Gases', 2021; Alcantara et al., 2016; Ambrosi et al., 2014; Catarino et al., 2009; Catarino & Mendes, 2011; Cimini et al., 2014; Cimini & Moresi, 2014, 2015, 2016a, 2016b, 2018, 2020; De Francesco et al., 2014; De Francesco et al., 2020; Eumann & Schildbach, 2012; Halama et al., 2019; Liguori et al., 2015; Liguori et al., 2016; Russo et al., 2013; Stumpf & Schildbach, 2018; Wedel Falkenberg, 2014)

Operation	**Process**	**Module**	**Material**	**Pore size**	**TMP**	**Temperature**	**Feed velocity**
brewing water treatment	RO	ND	PA	[a]	7 – 15 bar	ND	ND
mash separation	MF	tubular	ceramic, PTFE, stainless steel	0.45 – 100 µm	0.35 – 2.1 atm	70 – 80 °C	2 – 8 m s^{-1}
fermentation	MBR	hollow fiber	PES	0.24, 0.4 µm	ND	15 °C	ND
recovery of beer and yeast	MF	ND	ceramic, polymeric	1.0 – 2.0 µm	up to 3 bar	ND	ND
BMF	MF	flat sheet, tubular	ceramic, PC, PSF	0.1 – 4 µm	0.1 – 4.73 bar	0 - 40 °C	0.15 - 6 m s^{-1}
cold sterilisation of beer	MF	flat sheet	CA, ceramic, PA, PC	0.2 – 0.65 µm	0.1 - 2 atm	0 - 25 °C	ND
LAB and AFB production	DI	hollow fibre	cellulose, PSF	500 – 5000 Da	0 – 0.7 atm	5 °C	ND
	MD	spiral wound	PA	[a]	2 – 3 bar feed pressure and 0.49 – 0.66 bar vacuum pressure	ND °C	ND
	NF	flat sheet	PET	[a]	19 bar	14 - 15 °C	ND
	OD	hollow fiber	PP	0.03 µm	1.1 bar feed pressure	10 - 20 °C	ND
	PV	flat sheet	PDMS, PEI, POMS PVA	[a]	(1 – 50) × 10^{-3} atm	5 – 70 °C	0.1 – 3.3 m s^{-1}
	RO	spiral wound	CA, PA, cellulosic, PSF	[a]	3.4 - 50 atm	0 - 30 °C	ND
recovery of aroma compounds	PV	flat sheet	POMS/PEI	[a]	(1 – 20) × 10^{-3} bar	7 - 25 °C	0.1 – 0.5 m s^{-1}

beer gasification, degasification	MC	hollow fiber	ND	ND	ND	ND	ND

ND = no data.

[a] The membrane distillation, nanofiltration, reverse osmosis and pervaporation membranes are not characterised according to pore size, as the selective layers are dense.

The essentials of operations that can be seen in *Table 5* are reviewed below.

For continuous mash separation with reduced cake formation, crossflow MF can be used (Ambrosi et al., 2014).

Combining fermentation with membrane bioreactor allows the adjustment of the cell count on a high level, increasing yield per volume and time (Stumpf & Schildbach, 2018).

The yeast and beer can be recovered from the tank bottom after fermentation with MF (Ambrosi et al., 2014) reducing the losses.

The alternative process to conventional rough beer clarification with Diatomaceous Earth (DE) is BMF because of higher product quality, less environmental issues, less health and safety concerns, simplicity, flexibility, and lower cost (Ambrosi et al., 2014).

Cold sterilisation of beer with MF is an alternative method to pasteurisation. This method can lead to lower deployment cost and fresher-tasting product, eliminating the organoleptic problems induced by heating (Ambrosi et al., 2014).

Different MSPs (DI, MD, NF, OD, PV, RO) provide promising alternatives for low alcohol beer (LAB) and alcohol-free beer (AFB) production with ethanol separation after the fermentation process and include such advantages as lower energy consumption, no chemical additives, and operation at mostly mild temperatures, therefore reducing the impact of heat on the product (Ambrosi et al., 2014).

Aroma compounds can be separated effectively with PV from the beer before BDA process. Then these compounds can be added back to the dealcoholized beer, for the purpose of eliminating the flavour and aroma losses caused by BDA (Catarino et al., 2009; Catarino & Mendes, 2011).

The beer can be gassed and degassed with the application of MC. The main goal of beer gasification with CO_2 or N_2 is the formation of the head when the beer is served

(Ambrosi et al., 2014). Beer is sensitive towards oxidation, especially deteriorating the flavour (Kreim et al., 2018), but O_2 can be removed from the beer with degasification ('3M - 3M™ Liqui-Cel™ Membrane Contactors Used in the Soft Drink and Brewing Industries to Control Dissolved Gases', 2021).

2.3.3. Applications examined in detail in this study

After the detailed literature review, four main research gaps are examined in this study.

Wort membrane filtration

Hopped wort is an intermediate product of brewing. It is the liquid extracted from the mashing process and boiled with hops (Csanádi, 2010). Wort boiling leads to protein coagulation and this coagulation constitutes hot trub (composed of proteins, polyphenols, resins, ash and significant quantities of lipids) (Briggs et al., 2004d; Fix, 1999b). The size of particles in hot trub is 30 – 80 μm in diameter and they settle well. Removing at least some hot trub can decrease the production losses and improve yeast viability, beer filtration performance and the quality of finished beer (Kunze, 2004c). Centrifugation, filtration and sedimentation are some methods used to promote the removal of hot trub (Diakun & Jakubowski, 2013; Leiper & Miedl, 2006).

Wort cooling before fermentation leads to the formation of cold trub (composed of proteins, protein-polyphenol complexes and carbohydrates). The size of particles in cold trub is about 0.5 μm and they settle only with great difficulty (Barchet, 1994; Kunze, 2004f). Removing at least some cold trub can improve yeast viability and the quality of finished beer (Narziß & Back, 2009). Centrifugation, DE filtration, flotation and sedimentation are some methods used to promote the removal of cold trub (Barchet, 1994).

Furthermore, if primary fermentation is performed in Membrane Bioreactor (MBR), wort particles (e.g. cold trub) can cause membrane fouling (Stumpf & Schildbach, 2018).

WMF would be an alternative and novel technology for removal of hot trub and cold trub (Ambrosi et al., 2014) and the two processes can be performed simultaneously with the same crossflow microfiltration (CFMF) equipment. It should be noted that microfiltration is a pure size separation (Van Reis & Zydney, 2001), but different types of fouling (concentration polarization, compact cake layer formation, partial or complete plugging of pore entrances, adsorption of macromolecules onto the pore

walls) (Kazemi et al., 2013) can affect the particle size of the sieving mechanism and it is important to note, that hopped wort is rich in foulants. Thus, the feasibility of the wort membrane filtration process was not obvious.

The main advantages of the WMF are less solid residues, lower energy consumption, lower water requirements, better quality in terms of clarity and homogeneity, microbiologically stable product (Bhayani & Ramarao, 2011; Ghosh et al., 2018; Güell, 1999). The main disadvantage of the WMF is membrane fouling (Field & Lipnizki, 2016a).

Beer membrane filtration with static turbulence promoter

The purpose of BMF is to eliminate yeast and colloidal particles responsible for haze. Furthermore, BMF should ensure the microbiological stability of beer (Daufin et al., 2001). The alternative process to conventional clarification with DE is BMF because of higher product quality, less environmental issues, less health and safety concerns, simplicity, flexibility, and lower cost (Ambrosi et al., 2014). However, one of the main problems of ordinary application of BMF is fouling mechanisms (flux decline during BMF). Thus, it is essential to reduce membrane fouling during BMF.

One of the hydrodynamic techniques for fouling reduction is increase of the turbulence intensity with use of STPs in the flow channel of the membrane (Popović et al., 2011; Popović & Tekić, 2011). In case of membrane filtration of other liquids than rough beer, effect of STP on permeate flux enhancement have been reported (Ikonić et al., 2012; Liu et al., 2012; Rai et al., 2010).

Optimisation of operating parameters can be a solution for reducing membrane fouling. Fortunately, full factorial experimental design can be used successfully to optimise the operating parameters of membrane filtration and study the process (Azizi Namaghi & Mousavi, 2016; Banvolgyi et al., 2016) with minimal number of experiments (Hamdi et al., 2016). In addition, the effect of STP on fouling during membrane filtration can be studied with the application of factorial experimental design (Ikonić et al., 2012).

Beer membrane filtration with silica gel

The purpose of BMF is to eliminate yeast and colloidal particles responsible for haze. Furthermore, BMF should ensure the microbiological stability of beer (Daufin et al., 2001). The alternative process to conventional clarification with DE is BMF because

of higher product quality, less environmental issues, less health and safety concerns, simplicity, flexibility, and lower cost (Ambrosi et al., 2014). However, one of the main problems of ordinary application of BMF is fouling mechanisms (flux decline during BMF). Thus, it is essential to reduce membrane fouling during BMF. Optimisation of operating parameters can be a solution for reducing membrane fouling. Fortunately, full factorial experimental design can be used successfully to optimise the operating parameters of membrane filtration and study the process (Azizi Namaghi & Mousavi, 2016; Banvolgyi et al., 2016) with minimal number of experiments (Hamdi et al., 2016).

Application of filtration aids, e.g. Silica Gel (SG), can also be a solution for reducing membrane fouling. However, the effect of SG is questionable. In one case, silica had an interactive or little effect on normalized fouling rate during dead-end microfiltration of synthetic mixtures (Kim et al., 2015). In another case, SG had mainly positive effect on filtration rate during conventional beer filtration (Leiper et al., 2002). The properties and mechanism of SG are discussed below. SG has a very large surface area containing a network of pores and this surface of SG is covered in silanol (SiOH) groups which form interactions with proline residues in haze-active proteins (Benítez et al., 2016). The mechanism of action of SG is via hydrogen bonding of protein carbonyl groups to hydroxyl groups on SG (Ryder & Power, 2006).

Generally polymeric membranes are used for industrial BMF (e.g. Pentair‘s Beer Membrane Filtration System - BMF (‘Pentair - Beer Membrane Filtration System - Beverage Filtration Solutions | Pentair Food & Beverage Process Solutions’, n.d.) and SG products are developed for these types of membranes. However, ceramic membranes are suitable to be used in extreme conditions which could not be achieved by traditional polymer membranes (Elaine Fung & Wang, 2013). The advantages of ceramic membranes include high chemical, microbial, physical and thermal stability, insensitivity to swelling and ease of cleaning (Das & Maiti, 1998; Elaine Fung & Wang, 2013).

Cleaning of microfiltration membranes after beer membrane filtration

Based on the literature, beer membrane filtration membranes can be cleaned with internal and external cleaning procedures, and caustic and acid components can be combined in the cleaning agents for recovering water permeabilities of ceramic and polymeric membranes (Wenten, et al., 1994). Furthermore, a cleaning procedure with

0.3 % (w/w) Sodium hydroxide and Hydrogen peroxide 0.5 % (w/w) (T = 80 °C, TMP =0.2 bar, Re = 1552) can be applied effectively (Gan et al., 1999). It is important to note that ceramic membranes are much more resistant to chemicals and high temperatures than polymeric membranes (Stopka et al., 2000). Thus, the application of ceramic membranes during beer membrane filtration is more advantageous in term of membrane cleaning.

Beer dealcoholization by reverse osmosis

Moderate beer consumption has health benefits, but these benefits are restricted by the negative consequences of ethanol (alcohol) content of beer. However, there is potential to reduce ethanol content of beer through innovation (Salanță et al., 2020).

The production of beers with reduced ethanol content is a fast-growing segment in the global beer market (Salanță et al., 2020).

The legal definitions of low-alcohol beer (LAB) and alcohol-free beer (AFB) vary from country to country (Sohrabvandi et al., 2010). For example, in Hungary (the country of this study) the ethanol content of LAB must be between 0.51 and 1.50% (V/V) and AFB must contain maximum ethanol level of 0.50% (V/V) (Ministry of Agriculture of Hungary, 2013).

There can be several reasons for LAB or AFB production. The reasons are the following: increase in the overall production by introduce new products in countries with highly competative markets; provide beer consumers with products prior or during their activites (driving motor vehicles, operating machinery, doing sports) or under conditions (pregnancy, medication) irreconcilable with alcohol consumption; penetrate beverage markets in countries, where alcohol consumption is forbidden for religious reasons (Brányik et al., 2012).

The aim of LAB or AFB production is to reduce the ethanol content of beer while maintaining other characteristics (Salanță et al., 2020).

There are different methods for LAB or AFB production. *Figure 5* shows the scheme of LAB and AFB production methods (based on Brányik et al., 2012; Conidi et al., 2020; De Francesco et al., 2020; Purwasasmita et al., 2015; Sohrabvandi et al., 2010).

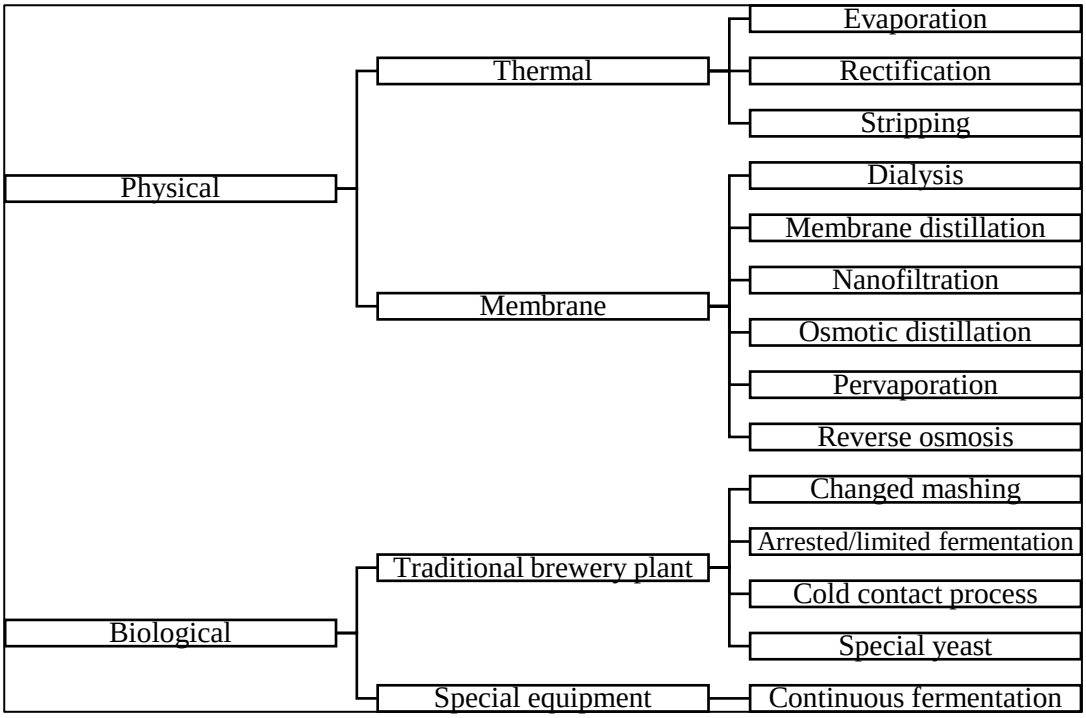

Figure 5: The scheme of low-alcohol beer (LAB) and alcohol-free beer (AFB) production methods (based on Brányik et al., 2012; Conidi et al., 2020; De Francesco et al., 2020; Purwasasmita et al., 2015; Sohrabvandi et al., 2010)

As it can be seen in *Figure 5*, one of the groups of the methods are the MSPs. MSPs provide promising alternatives for separating the ethanol after the fermentation process and include such advantages as lower energy consumption, no chemical additives, and operation at mild temperatures, therefore reducing the impact of heat on the product (Ambrosi et al., 2014). In this study, a MSP for BDA is investigated, namely RO. The most important parameters of the BDA by RO are the permeate flux and the ethanol concentration in the permeate. These parameters can be combined into one parameter: ethanol flux (Catarino et al., 2007; Halama et al., 2019; L. Liguori et al., 2015; Russo et al., 2013).

The operating parameters affect the permeate flux and the ethanol concentration in the permeate (Catarino et al., 2007); therefore the operating parameters also affect the ethanol flux. Thus, the optimisation of the operating parameters is essential to achive ethanol flux enhancment. Fortunately, full factorial experimental design can be used successfully to optimise the operating parameters of membrane separation and to study the process (Azizi Namaghi & Mousavi, 2016; Banvolgyi et al., 2016; Habibi, Aroujalian et al., 2011; Nor et al., 2017) with minimal number of experiments (Hamdi et al., 2016).

Cleaning of reverse osmosis membranes after beer dealcoholization

Based on the literature, the cleaning process of polyester-sulfone reverse osmosis membrane after beer dealcoholization was the following: 0.1 M Sodium hydroxide solution for 60 min at room temperature with 2 kg cm^{-2} pressure followed by distilled water with 2 kg cm^{-2} pressure. The filtrate was discarded and the procedure ended when the filtrate reached a pH of between 7 and 7.1 (Alcantara et al., 2016).

The manufacturer of RO99 polyester reverse osmosis membrane (Alfa Laval, Sweden) suggests the following operating parameters for membrane cleaning: pH range = 1.5 – 11, pressure = 1 – 5 bar, temperature = 30 – 50 °C (Alfa Laval, n.d.).

3. MATERIALS AND METHODS

3.1. Materials

3.1.1. Brewing water

Water (Fővárosi Vízművek, Hungary) with 13°dH total hardness (classified as average hardness (Kunze, 2004g)) was used during mashing and sparging of hopped wort and brewing of rough beers. This water met the drinking water quality standards ('Fővárosi Vízművek - Vízminőség, Vízkeménység', n.d.), based on the water analysis (*Chapter 1.M2.1*).

3.1.2. Malts

Pilsner Malt

Pilsner Malt (Boortmalt, Hungary) was used during mashing of hopped wort. This malt met the typical specification for a lager malt (O'Rourke, 2002), based on the batch analysis (*Chapter 1.M2.2*).

Extra Pale Premium Pilsner Malt

Extra Pale Premium Pilsner Malt (Weyermann, Germany) was used during mashing of rough beers. This malt met the typical specification for a lager malt (O'Rourke, 2002), based on the batch analysis (*Chapter 1.M2.3*).

3.1.3. Hops

Hallertauer Tradition hops

Hallertauer Tradition T90 hop pellets (HVG, Germany) with 10.0% (w/w) alpha acid content was used during wort boiling of production of hopped wort. Characteristics of this hop variety can be found in *Chapter 1.M2.4*.

Hallertauer Magnum hops

Hallertauer Magnum T90 hop pellets (HVG, Germany) with 14.6% (w/w) alpha acid content was used during wort boiling of production of rough beers. Characteristics of this hop variety can be found in *Chapter 1.M2.5*.

3.1.4. Yeast

Third generation liquid lager yeast (*Saccharomyces pastorianus*) (Cara Technology, United Kingdom) was used for fermentation of pilot beers. Properties of the used yeast can be found in *Chapter 1.M2.6*.

3.1.5. Beers

0.5 L canned Soproni Klasszikus pale lager bright beers (HEINEKEN Hungária, Hungary) with 4.5% (V/V) ethanol content were used during beer dealcoholization by reverse osmosis. The ingredients of this beer are water, malted barley, maize grits, hops and hop extract.

3.1.6. Membranes

The characteristics of the applied membranes for the membrane separation experiments (*Chapter 3.3.2*) are shown in *Table 6*.

Table 6: The characteristics of the applied membranes for the membrane separation experiments

Application	Process	Manufacturer	Type	Module	Material	Pore size	Active surface
WMF	MF	Pall, United States of America	Membralox T1-70	tubular	ceramic	0.2 µm	0.005 m^2
BMF with STP	MF	Pall, United States of America	Membralox T1-70	tubular	ceramic	0.5 µm	0.005 m^2
BMF with SG	MF	Pall, United States of America	Membralox T1-70	tubular	ceramic	0.5 µm	0.005 m^2
BDA by RO	RO	Alfa Laval, Sweden	RO99	flat sheet	polyester	[a]	0.05 m^2

[a] The reverse osmosis membrane is not characterised according to pore size, as the selective layer is dense. $R_{NaCl} \geq 98$ %, measured on 2000 ppm NaCl, 16 bar, 25 °C.

3.1.7. Static turbulence promoter

The SPIRAL LD2 STP (Inox, Serbia) was used during beer membrane filtration with static turbulence promoter. It was chosen from several STPs with different geometries because compared with other STPs the highest initial flux had been achieved during membrane filtration of oil-in-water emulsions (Gáspár, 2016). This stainless-steel spiral STP (*Figure 6*) has a pitch diameter ratio of approximately 2, 13.2 mm pitch length, 6.5 mm diameter, 241 mm total length and 1.2 mm thickness. This STP can be inserted in Membralox T1-70 tubular membrane (*Table 6*).

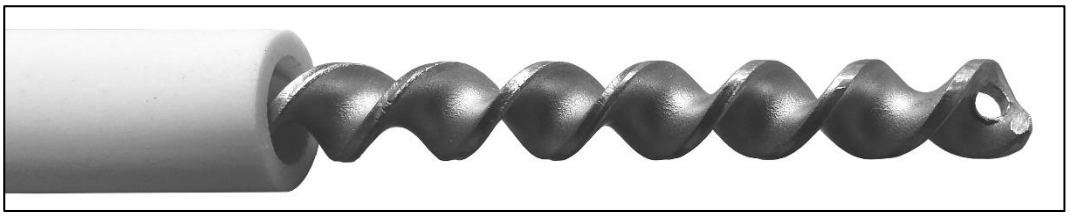

Figure 6: The SPIRAL LD2 STP (Inox, Serbia) is inserted in Membralox T1-70 tubular membrane

3.1.8. Silica gel

Stabifix W MF (Stabifix Brauerei-Technik, Germany) was used during beer membrane filtration with silica gel. Stabifix W MF is a hydrogel and white powder with SiO_2 content approximately 99% (w/w) in residue on ignition and moisture content up to 65% (w/w), and designed for filtration with polysulfone based membranes.

3.2. Types of equipment

3.2.1. Pilot-scale brewery

Wort and beer productions were performed in the 50 L pilot-scale brewery (HBH, Hungary) of Department of Bioengineering and Alcoholic Drink Technology, Hungarian University of Agriculture and Life Sciences (Budapest, Hungary).

3.2.2. Crossflow microfiltration equipment

WMF and BMF experiments were carried out with bench scale in-house developed crossflow microfiltration (CFMF) equipment (*Figure 7*).

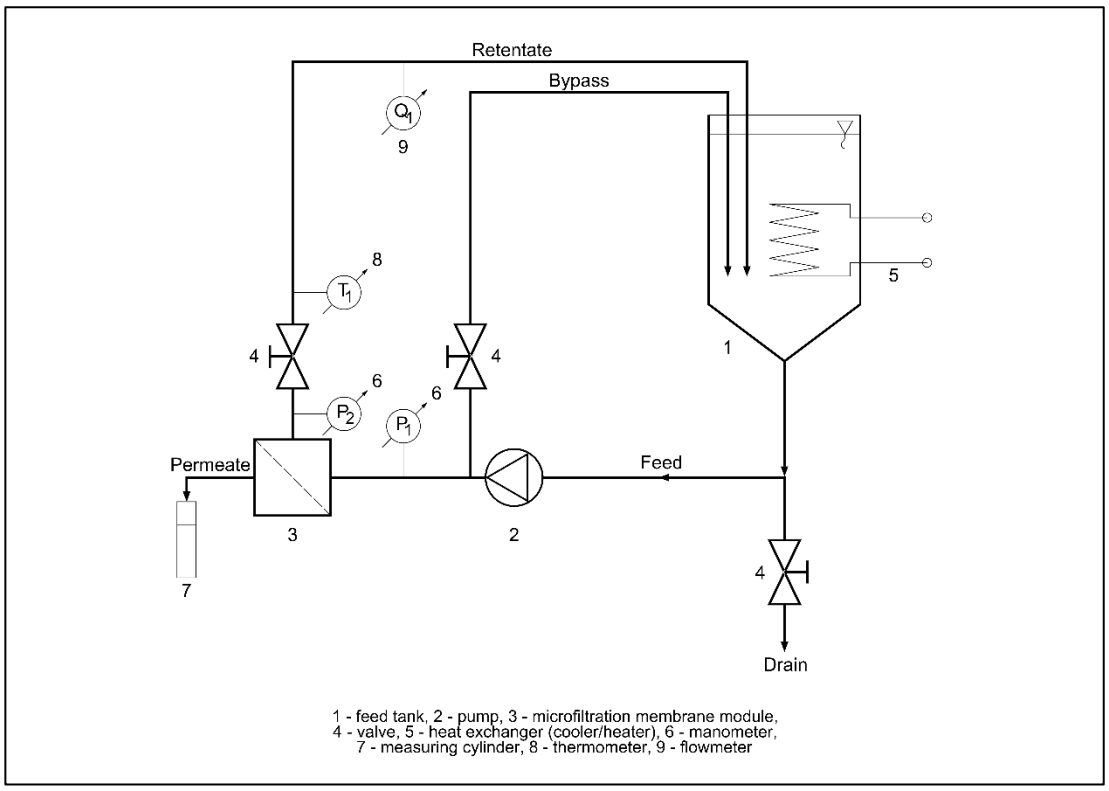

Figure 7: Schematic flow diagram of CFMF equipment

The pressure can be adjusted with the valve following the microfiltration membrane module. The flow rate can be adjusted with pump with variable-frequency drive (VFD). The bypass part of the CFMF equipment can be used with the opening of the valve at beginning of the bypass pipeline.

3.2.3. Crossflow reverse osmosis equipment

BDA experiments were carried out with bench scale "HF-528/08." crossflow reverse osmosis (CFRO) equipment (Hidrofilt, Hungary) (*Figure 8*).

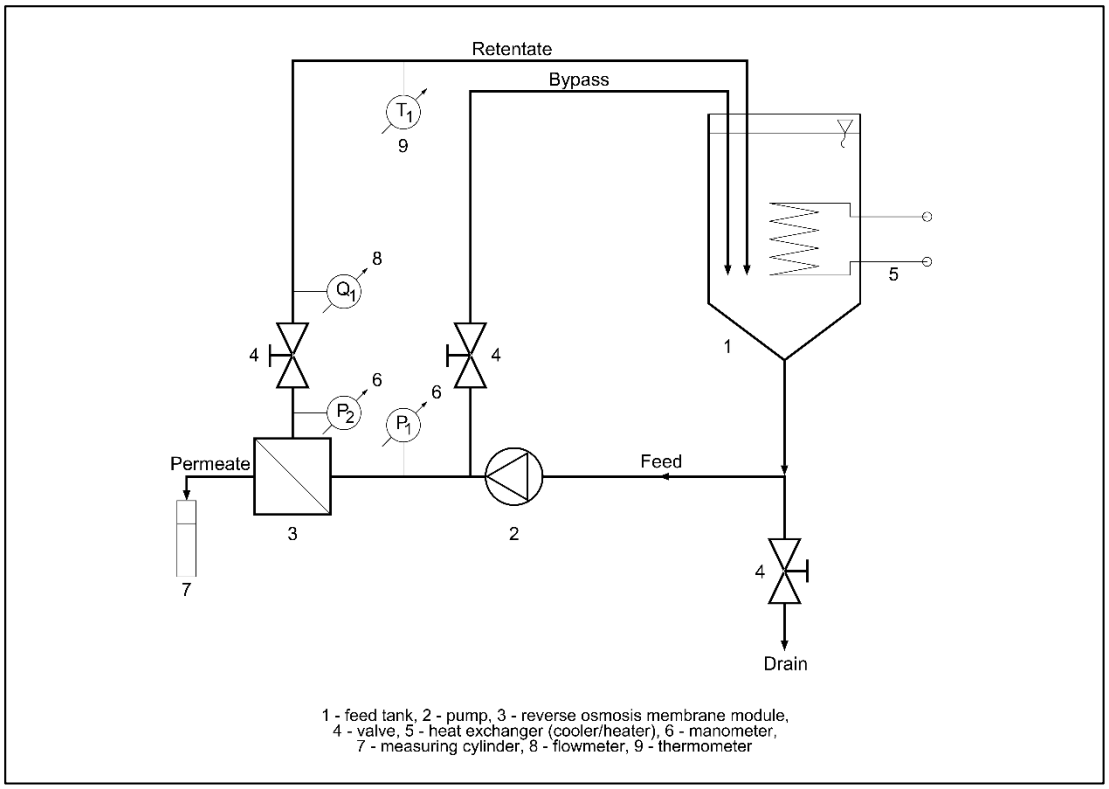

Figure 8: Schematic flow diagram of CFRO equipment

The pressure can be adjusted with the valve following the reverse osmosis membrane module. The flow rate can be adjusted with pump with variable-frequency drive (VFD). The bypass part of the CFRO equipment can be used with the opening of the valve at beginning of the bypass pipeline.

3.3. Methods

3.3.1. Brewing

Hopped wort and rough beers were produced for the membrane filtration investigations in the pilot-scale brewery of Department of Bioengineering and Alcoholic Drink Technology, Hungarian University of Agriculture and Life Sciences (Budapest, Hungary).

The rough beer recipe was designed based on "2A. International Pale Lager" from Beer Judge Certification Program (BJCP) ('Beer Judge Certification Program 2015 Style Guidelines', 2015).

All multistep mashing programs were performed with 1 °C/min temperature increases and ± 0.5 °C temperature accuracy. Lautering was carried out in a lauter tun.

Production of hopped wort for wort membrane filtration

7 kg Pilsner Malt and 28 L brewing water were used during mashing-in. The following

multistep mashing program was used: 20 min at 51 °C, 45 min at 63 °C, 15 min at 72 °C, and 1 min at 78 °C. Sparging water with temperature of 78°C was added in such a way to reach a final wort volume before boiling of 40 L. 40 g of Hallertauer Tradition hops were added at the start of 60 min boiling, aiming for 35 IBU in the theoretical final beer. After boiling, most of the hot trub was separated from bitter wort by whirlpool in 10 min.

Production of rough beer for beer membrane filtration with static turbulence promoter

11 kg Extra Pale Premium Pilsner Malt and 40 L brewing water were used during mashing-in. The following multistep mashing program was used: 20 min at 50 °C, 40 min at 63 °C, 20 min at 72 °C, and 1 min at 78°C. Sparging water with temperature of 78 °C was added in such a way to reach a final wort volume before boiling of 55 L. 28 g of Hallertauer Magnum pellet hops were added at the start of 90 min boiling, aiming for 20 IBU. After boiling, the hot trub was separated from bitter wort by whirlpool in 20 min and with the addition of water the final volume (56 L) of hopped wort and original real extract (11.5% (w/w)) were adjusted. Then the wort was cooled to 11 °C and oxygenated. The yeast was pitched at the rate of 15 million cells/mL. The fermentation was carried out at 11 ± 1 °C for seven days, followed by maturation at 4 ± 1 °C under 0.5 bar overpressure for 14 days.

Production of rough beer for beer membrane filtration with silica gel

11 kg Extra Pale Premium Pilsner Malt and 40 L brewing water were used during mashing-in. The following multistep mashing program was used: 20 min at 50 °C, 40 min at 63 °C, 20 min at 72 °C, and 1 min at 78°C. Sparging water with temperature of 78°C was added in such a way to reach a final wort volume before boiling of 65 L. 28 g of Hallertauer Magnum pellet hops were added at the start of 90 min boiling, aiming for 22 IBU. After boiling, the hot trub was separated from bitter wort by whirlpool in 20 min. Then the wort was cooled to 12 °C and oxygenated. The yeast was pitched at the rate of 15 million cells/mL. The fermentation was carried out at 11 ± 1 °C for eight days, followed by maturation at 4 ± 1 °C under 0.5 bar overpressure for 14 days.

3.3.2. Membrane separation processes

The feed volumes of WMF, BMF with STP, BMF with SG and BDA by RO were 3 L, 3 L, 3 L and 5 L respectively.

Before each separation experiment, water flux was measured at given temperature and transmembrane pressure.

Microfiltration experiments were performed at 10 ± 1 °C, while reverse osmosis experiments were performed at 15 ± 1 °C.

During separations pressures at both ends of the membrane module were measured.

Wort membrane filtration

Membralox T1-70 membrane with 0.2 μm pore size (*Table 6*) was used for the filtration.

During the membrane filtration process transmembrane pressure (TMP) and retentate flow rate (Q) were maintained 0.4 bar (relatively low driving force) and 50 L h^{-1} (resulted in relatively low velocity), respectively.

Following the water flux measurement, in order to avoid the dilution of original wort with water, the water from CFMF equipment was drained with the valve at the bottom (*Figure 7*). Furthermore, the residual water was carefully run off with wort.

At the beginning of the filtration, the first collected permeate sample (10 mL) was ignored to eliminate the dilution of bright wort with water. During the rest of the time, permeate samples were continuously collected with constant volume (10 mL). Whenever the steady-state flux was achieved and the required volume of permeate sample was collected the filtration was finished. The VCF of the process was 1.04.

Beer membrane filtration with static turbulence promoter

Membralox T1-70 membrane with 0.5 μm pore size (*Table 6*) was used for filtrations with silica gel.

Filtration experiments were performed according to the experimental design (*Table 10*) discussed in *Chapter 3.3.9*. The three factors were static turbulence promoter (STP), transmembrane pressure (TMP) and retentate flow rate (Q).

Following the water flux measurement, in order to avoid the dilution of rough beer with water, the water from CFMF equipment was drained with the valve at the bottom (*Figure 7*). Furthermore, the residual water was carefully run off with rough beer.

At the beginning of the filtrations, the first collected permeate samples (10 mL) were ignored to eliminate the dilution of bright beer with water. During the rest of the time,

permeate samples were continuously collected with constant volume (10 mL). Whenever the steady-state fluxes were achieved and the required volumes of permeate samples were collected, the filtrations were finished at the same VCF (VCF = 1.1).

Beer membrane filtration with silica gel

Membralox T1-70 membrane with 0.5 μm pore size (*Table 6*) was used for filtrations with silica gel.

Filtration experiments were performed according to the experimental design (*Table 11)* discussed in *Chapter 3.3.9.*. The three factors were silica gel concentration (SGC), transmembrane pressure (TMP) and retentate flow rate (Q).

Following the water flux measurement, in order to avoid the dilution of rough beer with water, the water from CFMF equipment was drained with the valve at the bottom (*Figure 7*). Furthermore, the residual water was carefully run off with rough beer.

According to the experimental design (*Table 11)*, the required amount of SG (Stabifix W MF (*Chapter 3.1.8*)) was added to the rough beer in the feed tank. After the addition, the rough beer was circulated for 2 min through the bypass (*Figure 7*) for the mixing and effect of the SG.

At the beginning of the filtrations, the first collected permeate samples (10 mL) were ignored to eliminate the dilution of bright beer with water. During the rest of the time, permeate samples were continuously collected with constant volume (10 mL). Whenever the steady-state fluxes were achieved and the required volumes of permeate samples were collected, the filtrations were finished at the same VCF (VCF = 1.1).

Beer dealcoholization by reverse osmosis

RO99 membrane (*Table 6*) was used for dealcoholization processes.

Dealcoholization experiments were performed according to the experimental design (*Table 12*) discussed in *Chapter 3.3.9.* The two factors were transmembrane pressure (TMP) and retentate flow rate (Q). Generally, in case of RO process, the retentate flow rate (Q) is lower than feed flow rate by permeate flow rate (flow drop) (Salamon et al., 2018). In this study, the permeate flow rates were less than 0.4 % of the feed flow rates, thus the flow drops were negligible.

Before each dealcoholization experiment, in order to avoid foaming during dealcoholization process, beer was decarbonated by stirring for 30 min with LR40

stirrer (MLW, German Democratic Republic) with marine propeller impeller with 2 blades at the lowest RPM to prevent vortex formation. After the water flux measurement, in order to avoid the dilution of beer with water, the water from CFRO equipment was drained with the valve at the bottom (*Figure 8*). Furthermore, the residual water was carefully run off with beer.

At the beginning of the filtrations, the first collected permeate samples (10 mL) were ignored to eliminate the dilution of permeate with water. During the rest of the time, permeate samples were continuously collected with constant volume (10 mL). Whenever the fluxes declined steadily and the required volumes of permeate samples were collected, the dealcoholization processes were finished at the same VCF (VCF = 1.06). It should be noted that the properties of the beer samples did not change significantly, because the volume concentration factors of the membrane separations were only 1.06.

3.3.3. Membrane cleaning

The process of development of a membrane cleaning method is detailed below. Based on the literature and suggestions of the membrane manufacturers I have created an initial cleaning procedure. After a membrane separation process, I tested and modified this cleaning procedure. After the cleaning, I measured pure water flux, thus I was able to calculate the membrane cleaning efficiency. If it was necessary, I modified the types of the chemicals, the concentration of the cleaning solutions, the temperature of the cleaning solutions and the cleaning times.

Cleaning of microfiltration membranes

After each microfiltration experiment, the used membrane was cleaned thoroughly by deionized water for 5 min at a temperature of 25 °C and then by 1 % (w/w) Sodium hydroxide for 60 min at a temperature of 60 °C. After cleaning by alkali, the membrane was rinsed again by deionized water for 10 min at a temperature of 25 °C followed by cleaning with 1 % (w/w) Hydrogen peroxide for 60 min at a temperature of 25 °C. Finally, the membrane was cleaned thoroughly with deionized water for 10 min at a temperature of 25 °C. In all cases transmembrane pressure (TMP) and retentate flow rate (Q) were maintained at 0.2 bar and 50 L $h^{-}1$, respectively. Sodium hydroxide was purchased from Reanal, Hungary and Hydrogen peroxide from Hungaro Chemicals, Hungary. After each membrane cleaning, water flux was measured at given temperature and transmembrane pressure (TMP). The purpose of the water flux

measurement was checking of degree of membrane cleanliness (Blanpain-Avet et al., 2004). Water flux is affected by temperature and transmembrane pressure (TMP) (Huisman et al., 1997). Thus, the water flux measurement has to be performed with given temperature and transmembrane pressure (TMP) values (same values as the values of the water flux measurement before the filtration) to get comparable results.

The above-mentioned membrane cleaning procedure was developed based on the literature of cleaning after BMF (Gan et al., 1999).

Cleaning of reverse osmosis membrane

After each dealcoholization experiment, the membrane was cleaned thoroughly by deionized water for 10 min at a temperature of 25 °C and then by 0.2 % (w/w) Sodium hydroxide for 60 min at a temperature of 25 °C. After cleaning by alkali, the membrane was rinsed again by deionized water for 10 min at a temperature of 25 °C. In all cases transmembrane pressure (TMP) and retentate flow rate (Q) were maintained at 6 bar and 240 L h^{-1}, respectively. Sodium hydroxide was purchased from Reanal, Hungary. After each membrane cleaning, water flux was measured at given temperature and transmembrane pressure (TMP).

The above-mentioned membrane cleaning procedure was developed based on the cleaning recommended by the membrane manufacturer (Alfa Laval, n.d.).

3.3.4. Analytical parameters

Ethanol and extract content

Alcohol, real extract and apparent extract contents of wort, beer and permeate samples were measured with Alcolyzer Plus (Anton-Paar, Austria).

Measurement principles

The measuring system consists of the Alcolyzer Plus basic instrument, a DMA 4500 density meter. While the Alcolyzer Plus determines the alcohol content with near-infrared (NIR) method, an Anton Paar oscillating U-tube density meter determines the density of samples.

Other parameters are calculated as a function of alcohol and density by Tabarie's formula.

Apparatus

- Alcolyzer Plus (Anton-Paar, Austria)

Measurement procedure

First, 20 mL of bubble-free sample was injected into the density meter. Secondly, the measurement started by pressing the "Start" button. Before measurement started, the instrument automatically adjusted the temperature of the sample to 20 °C. The instrumented was controlled and data were acquired using *VisioLab 1.0*, n.d.

Bitterness

The bitterness (concentrations of iso-alpha acids in ppm) of samples were measured according to 'Analytica EBC | Wort | 8.8 - Bitterness of Wort', 2003 and 'Analytica EBC | Beer | 9.8 - Bitterness of Beer (IM)', 2020.

Measurement principles

The bitter substances are extracted from acidified sample with Isooctane. After centrifugation, the absorbance of the isooctane layer is measured at 275 nm against a reference of pure Isooctane.

Reagents

- Diatomaceous earth (Reanal, Hungary)
- Hydrochloric acid (Reanal, Hungary), c(HCl) ≈ 6 M
- Isooctane (Reanal, Hungary)

Apparatus

- 50 mL centrifuge tubes
- DR 6000 spectrophotometer (Hach, USA)
- Glass balls
- Heraeus Megafuge 16R Centrifuge (Thermo Fisher Scientific, USA)
- Pipettes
- Silica cuvettes, 10 mm optical path length

Measurement procedure

Firstly, the sample was degassed and filtered with pleated filter paper and diatomaceous earth (DE). Secondly, 10 mL of degassed sample was pipetted exactly

into a 50 mL centrifuge tube. After that, 0.5 mL of 6 M Hydrochloric acid was added, followed by 20 mL of Isooctane. 2 to 3 glass balls were placed in the centrifuge tube. Then a cap was screwed onto the centrifuge tube and the centrifuge tube was shaken by hand for 15 min. After the shaking, the sample was centrifuged for 3 min at 3000 RPM. Finally, absorbance of the Isooctane layer in a 10 mm cuvette at 275 nm was measured, using pure Isooctane in the reference cuvette.

Calculation

Bitterness values were determined with *Equation 8*:

$$B = 50 \times A_{275} \qquad \text{Equation 8}$$

where B is the bitterness of the sample (IBU) and A_{275} is the absorbance at 275 nm measured against a reference of pure isooctane. The results have to reported as IBU values to the nearest whole number.

Colour

The colour of samples were measured according to 'Analytica EBC | Wort | 8.5 - Colour of Wort', 2000 and 'Analytica EBC | Beer | 9.6 - Colour of Beer', 2000.

Measurement principles

The absorbance of the sample is measured at a wavelength of exactly 430 nm. The colour in EBC units is obtained by multiplying the absorbance by a factor.

Reagents

- Diatomaceous earth (Reanal, Hungary)
- Distilled water

Apparatus

- Cuvettes, 10 mm optical path length
- DR 6000 spectrophotometer (Hach, USA)

Measurement procedure

First, the sample was diluted if the absorbance at 430 nm was higher than 0.8. Secondly, the sample was filtered with pleated filter paper and diatomaceous earth (DE). Finally, absorbance of the sample in a 10 mm cuvette at 430 nm was measured, using distilled water in the reference cuvette.

Calculation

Colour values were determined with *Equation 9*

$$C = f \times 25 \times A_{430} \qquad \text{Equation 9}$$

where C is the colour of the sample (EBC), f is the dilution factor and A_{430} is the absorbance at 430 nm measured against a reference of distilled water. The results have to be expressed in EBC units to 2 significant figures.

Dynamic viscosity

Dynamic viscosity values of wort, beer and permeate samples were measured with Physica MCR 51 Rheometer (Anton-Paar Hungary, Hungary) with DG27 double gap concentric cylinder measurement system.

Measurement principles

The rotational viscometer measures the viscosity of the sample by turning a spindle in a cup. The viscosity is determined through the measurement of the torque on a vertical shaft that rotates a spindle.

Apparatus

- Physica MCR 51 Rheometer (Anton-Paar Hungary, Hungary) with DG27 double gap concentric cylinder measurement system

Measurement procedure

First, ~30 mL of sample was poured into the cup. Secondly, the probe was set to measurement position. After that, the temperature was set. When the sample temperature had reached the desired value (temperatures of 0, 5, 10, 15 and 20 °C, the samples from BDA were also measured at 25 °C), the measurement was started, and flow curve of the sample was measured by increasing the shear rate from 500 to 1000 s^{-1}. After the measurement sets (measurements of a sample at different temperatures), the probe was set to lift position and the sample was removed from the cup. The instrumented was controlled, and data were acquired and analysed using *Rheoplus/32*, 2008.

Calculation

Dynamic viscosity values of samples were calculated based on Herschel-Bulkley model (Mezger, 2006) fitted to the measured data of flow curve (shear stress in

function of shear rate).

Free amino nitrogen content

Free amino nitrogen (FAN) content of samples were measured according to ‘Analytica EBC | Wort | 8.10.1 - Free Amino Nitrogen in Wort by Spectrophotometry - Manual Method (IM)’, 2015.

Measurement principles

The sample and a standard solution are heated in the presence of ninhydrin at pH 6,7 and the absorbances at 570 nm are measured against a reagent blank. For dark coloured worts (higher than 100 EBC units) a correction for the colour of the sample is applied.

Reagents

- Ninhydrin colour reagent: 100 g di-Sodium hydrogen phosphate dodecahydrate, 60 g Potassium dihydrogen, 5 g Ninhydrin and 3 g D-(-)-Fructose are dissolved in distilled water and diluted to 1 L. The pH of the solution must be between 6.6 and 6.8 and it can be kept for 2 weeks if stored cold in an amber bottle.
- Diluting solution: 2 g Potassium iodate is dissolved in 600 mL water and 400 mL of 96 %(V/V) Ethanol is added. Cold storing is necessary.
- Glycine standard solution: 107.2 mg Glycine is weighted accurately and dissolved in distilled water and diluted to 100 mL. This solution is has to be stored at 0 to 4 °C. For use 1 mL to 100 mL is diluted with water so that the diluted solution contains 2 mg amino nitrogen L^{-1}.

All reagents for FAN measurement were purchased from Reanal, Hungary.

Apparatus

- 1100 H pH meter (VWR, USA)
- AX200 analytical balance (Shimadzu, Japan)
- Boiling water bath
- Cuvettes, 10 mm optical path length
- DR 6000 spectrophotometer (Hach, USA)
- Glass balls, 20 to 25 mm diameter.
- Pipettes with rubber suction bulbs
- Test tubes, 16 x 150 mm

- Volumetric flasks fitted with glass stoppers, 50 mL, 100 mL and 1000 mL
- Water bath at 20 ± 1 °C

Measurement procedure

Firstly, 1 mL wort sample was diluted with distilled water to 100 mL. Secondly, 2 mL of the diluted sample, standard solution and distilled water were taken, each in sperate test tubes and mL of colour reagent was added. After that, the test tubes were covered with glass balls and placed in a boiling water bath for exactly 16 min and then cooled in a water bath at 20 °C for 20 min. After the cooling, 5 mL of diluting solution was added to each test tube. Finally, absorbance of the sample in a 10 mm cuvette at 570 nm was measured against a reagent blank prepared from 2 mL of distilled water, 2 mL of colour reagent and 5 mL of diluting solution.

Calculation

FAN contents were determined with *Equation 10*:

$$F = f \times C_{Gly} \times \frac{A_s}{A_G} \qquad \text{Equation 10}$$

where F (mg L^{-1}) is the FAN content of the sample, f is the dilution factor, C_{Gly} (mg L^{-1}) is the concentration of Glycine standard solution, A_s is the absorbance of the sample at 570 nm measured against a reagent blank. A_G is the absorbance of the Glycine standard solution at 570 nm measured against a reagent blank. If necessary, the absorbance value of the blank have to be subtracted. The results have to be reported in mg L^{-1} to the nearest whole number.

Particle size distribution

Particle size distributions of original wort (feed) and permeate were measured with Fritsch Analysette 22 (Fritsch, Germany) and Malvern Zetasizer Nano-ZS (Malvern Instruments, United Kingdom).

Measurement principles

Both instruments operating on the principle of laser diffraction.

Apparatus

- Fritsch Analysette 22 (Fritsch, Germany)
- Malvern Zetasizer Nano-ZS (Malvern Instruments, United Kingdom)

Measurement procedure

Sample preparation was not necessary. The samples were placed in the sample holders of the instruments. The volume of sample holder of Fritsch Analysette 22 is 100 mL, while the minimum sample volume of Malvern Zetasizer Nano-ZS is 12 μL. Data were acquired and analysed using *Analysette 22*, 2006 (in case of Fritsch Analysette 22) and *Zetasizer 6.32*, 2011 (in case of Malvern Zetasizer Nano-ZS) software packages.

pH

The pH value of wort, beer and permeate samples were determined at 20 °C with 1100 H pH meter (VWR, USA). Before each measurement, the device was calibrated with three standard buffers (pH 4.00, 7.00 and 10.00) (VWR, USA).

Total polyphenol content

Total polyphenol content (TPC) content of samples were measured according to (Singleton & Rossi, 1965).

Measurement principles

The principle of the method is the reduction of the Folin–Ciocalteu reagent in the presence of phenolics resulting in the production of molybdenum–tungsten blue that is measured spectrophotometrically at 760 nm and the intensity increases linearly with the concentration of phenolics in the reaction medium.

Reagents

- MeOH:DW = 4:1 (methanol and distilled water)
- Folin-Ciocalteu solvent: 1:10 in DW
- 0.7 M Na_2CO_3 solution (7,42 g/100 mL DW)
- 0.3 M Gallic acid (5.1 mg/10 mL MeOH-DW solution)
  All reagents for TPC measurement were purchased from Reanal, Hungary.

Apparatus

- AX200 analytical balance (Shimadzu, Japan)
- Boiling water bath
- Cuvettes, 10 mm optical path length
- DR 6000 spectrophotometer (Hach, USA)
- Pipettes

- Test tubes

Measurement procedure

Firstly, a calibration curve had to be made with diluted Gallic acid. Secondly, the sample had to be prepared. The total sample volume is 2500 µL, consisting of 1250 µL of Folin-Ciocalteu reagent, 200 µL of Methanol:distilled water, 50 µL of sample and 1000 µL of Na_2CO_3 solution. After that, the solutions were then placed in a 50 ° C water bath for 5 min. Finally, the absorbance of the samplewas measured at 760 nm.

Calculation

The TPC concentration was expressed as Gallic acid equivalent and determined with *Equation 11*:

$$TPC = \frac{A_{760} \times V_T \times f}{S \times a \times 1000} \qquad \text{Equation 11}$$

where *TPC* (mg GAE mL^{-1}) is the total polyphenol content of the sample, A_{760} is the absorbance of the sample at 760 nm, V_t (µL) is the total sample volume, f is the dilution factor, S (µL) is the amount of the sample and a (mL mg^{-1}) is the slope of calibration curve.

Turbidity

The turbidity of wort, beer and permeate samples were measured at a temperature of 20 °C (permanent haze) with 2100P Turbidimeter (Hach, USA) in NTU and converted to EBC (Cimini & Moresi, 2014). The device was calibrated with Gelex Secondary Standards Kit (Hach, USA).

β-glucan content

The β-glucan content of wort samples was measured with Enzytec™ Color GlucaTest® (R-Biopharm, Germany).

Measurement principles

Enzytec™ Color GlucaTest® (R-Biopharm, Germany) is a colorimetric assay for the quantitative determination of high molecular weight β-glucan from barley in malt-mash and wort.

Reagents

- Enzytec™ Color GlucaTest® (R-Biopharm, Germany):

- Solution A: 1 bottle with approximately 125 mL
- Calibrator set: 6 vials with approximately 1.0 mL each (0, 100, 200, 300, 400 and 500 mg L^{-1} of β-glucan)

Apparatus

- DR 6000 spectrophotometer (Hach, USA)
- Pipettes
- Cuvettes, 10 mm optical path length
- Plastic spatula

Measurement procedure

Firstly, 200 µL of wort sample was put into the cuvette and 200 µL of calibrators (1-6) were put into other cuvettes (samples have to be tested directly, they cannot be diluted.). Secondly, 3 mL of Solution A was added to each cuvette. After that, the samples were mixed with plastic spatula and incubated for 30 min at room temperature. Finally, the absorbance values of the samples were measured at a wavelength of exactly 550 nm against a reference of distilled water.

Calculation

The calibration curve was plotted using the absorbance of the calibrators 1 to 6. The β-glucan content values of the samples were calculated from the resulting equation (linear regression).

3.3.5. Separation characteristics parameters

Retentions of different components were calculated with *Equation 12* (Basu & Balakrishnan, 2017):

$$R_i = \left(1 - \frac{C_{p_i}}{C_{b_i}}\right) \times 100 \qquad \text{Equation 12}$$

where R_i is the retention (%) of the component i, C_{pi} (g L^{-1}) is the permeate concentration of the component i and C_{bi} (g L^{-1}) is the bulk concentration of the component i.

3.3.6. Hydrodynamic parameters

Water, wort, beer and permeate fluxes were determined with *Equation 13* (Gáspár et al., 2011):

$$J = \frac{V}{A_m \times t_i} \quad \text{Equation 13}$$

where J (L m^{-2} h^{-1}) is the flux, V (L) is the permeate volume, A_m (m^2) is the membrane active surface area and t_i (h) is the time interval.

Permeate fluxes (mass based) were determined with *Equation 3*.

To describe the permeate flux during WMF and BMF processes, a mathematical model *Equation 14* was used (Varga & Márki, 2019):

$$J_t = J_0 + (J_{ss} - J_0) \times (1 - e^{-K \times t}) \quad \text{Equation 14}$$

where J_t (L m^{-2} h^{-1}) is the flux at any time (wort or beer), J_0 (L m^{-2} h^{-1}) is the initial flux (wort or beer), J_{ss} (L m^{-2} h^{-1}) is the steady-state flux (wort or beer), K (h^{-1}) is the flux decline coefficient (wort or beer) and t (h) is the time.

Ethanol fluxes were determined with *Equation 15* (based on Gnus et al., 2018):

$$J_{EtOH} = \frac{m_{EtOH}}{A_m \times t_i} = \frac{J \times c_{EtOH} \times \rho_{EtOH}}{100} \quad \text{Equation 15}$$

where J_{EtOH} (g m^{-2} h^{-1}) is the ethanol flux, m_{EtOH} (g) is the mass of ethanol in permeate, c_{EtOH} (% (w/w)) is the ethanol content in permeate and ρ_{EtOH} (g L^{-1}) is the ethanol density at given temperature.

To describe the flux during the early stage of BDA process, a mathematical model *Equation 16* was developed:

$$J_t = K \times t + J_0 \quad \text{Equation 16}$$

where J_t (L m^{-2} h^{-1}) is the flux at any time (BDA permeate), J_0 (L m^{-2} h^{-1}) is the initial flux (BDA permeate), K (h^{-1}) is the flux decline coefficient (BDA permeate) and t (h) is the time.

Transmembrane pressures were determined with *Equation 4.*

Then intrinsic resistances of the clean membranes before separations were determined with *Equation 17* (Ben Hassan et al., 2013):

$$J_{w\,0} = \frac{TMP}{\mu_w \times R_m} \quad \text{Equation 17}$$

where $J_{w\,0}$ (L m^{-2} h^{-1}) is the water flux before separation, μ_w (Pas) is the dynamic viscosity of water at given temperature and R_m (m^{-1}) is the intrinsic resistance of clean

membrane. Then total resistances were determined with *Equation 18* (Ben Hassan et al., 2013):

$$J_p = \frac{TMP}{\mu_p \times R_t} \quad \text{Equation 18}$$

where J_p (L m^{-2} h^{-1}) is the permeate flux (beer), μ_p (Pas) is the dynamic viscosity of the permeate (beer) at given temperature and R_t (m^{-1}) is the total resistance. Then fouling layer resistances were determined with *Equation 19* (Ben Hassan et al., 2013):

$$R_t = R_m + R_f \quad \text{Equation 19}$$

where R_f (m^{-1}) is the fouling layer resistance. For each BMF $R_{f\,0}$ (m^{-1}) initial fouling layer resistance and $R_{f\,ss}$ (m^{-1}) steady-state fouling layer resistance values were calculated with J_0 and J_{ss} values from *Equation 14*.

3.3.7. Evaluation of cleaning efficiency

Then intrinsic resistances of the membranes after cleanings were determined with *Equation 20* (Blanpain-Avet et al., 2004):

$$J_{w\,w} = \frac{TMP}{\mu_w \times R_n} \quad \text{Equation 20}$$

where $J_{w\,w}$ (L m^{-2} h^{-1}) is the water flux after membrane cleaning and R_n (m^{-1}) is the intrinsic resistance of the membrane after membrane cleaning.

Flux recoveries were calculated with *Equation 21* (Blanpain-Avet et al., 2004):

$$FR = \frac{R_m}{R_n} \times 100 \quad \text{Equation 21}$$

where *FR* is the flux recovery (%).

3.3.8. Regressions

Nonlinear regression

Based on *Equation 14* and time - flux data, $J_{wrt\,0}$, $J_{wrt\,ss}$ (for WMF); $J_{b\,0}$, $J_{b\,ss}$ (for BMF) and *K* values of the individual filtrations (WMF and BMF processes) were determined with iterations by using *SPSS Statistics 25.0*, 2017 software. Significances of parameter estimates, F values and determination coefficients (R^2) of the models were evaluated. Normality of the residuals was accepted by the absolute values of their skewness and kurtosis as they all were below 1 (Tabachnick & Fidell, 2013).

Linear regression

Based on *Equation 16* and time - flux data (with the exclusion of the first five unstable data points), J_0, (for BDA by RO); and *K* values of the seven individual filtrations (BDA process) were determined by regression, using *SPSS Statistics 25.0*, 2017 software. Significances of parameter estimates, F values and determination coefficients (R^2) of the models were evaluated. Normality of the residuals was accepted by the absolute values of their skewness and kurtosis as they all were below 1 (Tabachnick & Fidell, 2013).

3.3.9. Modelling

Experimental designs

BMF with STP, BMF with SG and BDA by RO experiments were performed according to 2^p full factorial experimental design (Kemény, 1985), because application of experimental design minimizes the required number of experiments (Akcal Comoglu et al., 2016). The aims of the application of the experimental design were the following: (i) to formulate an objective function that describes the relationship between the factors and the response, and (ii) to determine the significant parameters and the effect sizes.

The general mathematical model for a 2^3 full factorial experimental design (three factors, each at two levels) (*Equation 22*) is the following (Kemény, 1985):

$$Y = b_0 + \sum_{i=1}^{3} b_i \times x_i + \sum_{i=1}^{3} \sum_{j=1, i \neq j}^{3} b_{ij} \times x_i \times x_j + b_{123} \times x_1 \times x_2 \times x_3 \qquad \text{Equation 22}$$

where *Y* is the response; b_0 is the constant; b_i *(i = 1, 2, 3)* are the regression coefficients of the main factor effects; b_{ij} *(i = 1, 2, 3; j = 1, 2, 3; i ≠ j)* and b_{123} are the regression coefficients of the interactions and x_i *(i = 1, 2, 3)* are the coded factors.

The factors and levels of the 2^p full factorial experimental designs are shown in *Table 7*, *Table 8* and *Table 9*.

Table 7: The factors and levels of the 2^p full factorial experimental design of BMF with STP experiments

Factor	**Abbreviation**	**Code**	**Unit**	**Factor levels**		
				Low (-1)	Central (0)	High (+1)
Static turbulence promoter	STP	x_{STP}	-	no	-	yes
Transmembrane pressure	TMP	x_{TMP}	bar	0.4	0.8	1.2
Retentate flow rate	Q	x_Q	L h^{-1}	50	125	200

Table 8: The factors and levels of the 2^p full factorial experimental design of BMF with SG experiments

Factor	**Abbreviation**	**Code**	**Unit**	**Factor levels**		
				Low (-1)	Central (0)	High (+1)
Silica gel concentration	SGC	x_{SGC}	g hL^{-1}	0	40	80
Transmembrane pressure	TMP	x_{TMP}	bar	0.4	0.8	1.2
Retentate flow rate	Q	x_Q	L h^{-1}	50	125	200

Steady-state beer flux is the most important hydrodynamic parameter of BMF, because generally, most of the time of the filtration run is operated with this flux value or when it is achieved permeate backflow techniques are applied. But the steady-state fouling layer resistances ($R_{f\,ss}$) describe more accurately the fouling characteristics than the steady-state beer flux values (*Chapter 4.2.2* and *Chapter 4.3.2*). Thus, $R_{f\,ss}$ was considered as the response of the 2^p full factorial experimental designs of BMF with STP and BMF with SG.

Table 9: The factors and levels of the 2^p full factorial experimental design of BDA by RO experiments

Factor	**Abbreviation**	**Code**	**Unit**	**Factor levels**		
				Low (-1)	Central (0)	High (+1)
Transmembrane pressure	TMP	x_{TMP}	bar	10	20	30
Retentate flow rate	Q	x_Q	L h^{-1}	120	180	240

Initial ethanol flux ($J_{EtOH\,0}$) is the most important parameter of BDA by RO. Thus, $J_{EtOH\,0}$ was considered as the response of the 2^p full factorial experimental design of BDA by RO.

The design matrix of the 2^p full factorial experimental designs were generated in *Statistica 12.0*, 2012 software and they are shown in *Table 10*, *Table 11* and *Table 12*. The experiments were run in random order to reduce the potentials for biases.

Table 10: The design matrix of the 2^p full factorial experimental design of BMF with STP experiments

Standard order number	**Actual value**			**Coded value**		
	STP	TMP (bar)	Q (L h^{-1})	x_{STP}	x_{TMP}	x_Q
1	no	0.4	50	-1	-1	-1
2	yes	0.4	50	+1	-1	-1
3	no	1.2	50	-1	+1	-1
4	yes	1.2	50	+1	+1	-1
5	no	0.4	200	-1	-1	+1
6	yes	0.4	200	+1	-1	+1
7	no	1.2	200	-1	+1	+1
8	yes	1.2	200	+1	+1	+1
9 (C)	no	0.8	125	-1	0	0
10 (C)	yes	0.8	125	+1	0	0

C = center point.

Table 11: The design matrix of the 2^p full factorial experimental design of BMF with SG experiments

Standard order number	Actual value			Coded value		
	SGC (g hL^{-1})	TMP (bar)	Q (L h^{-1})	x_{SGC}	x_{TMP}	x_Q
1	0	0.4	50	-1	-1	-1
2	80	0.4	50	+1	-1	-1
3	0	1.2	50	-1	+1	-1
4	80	1.2	50	+1	+1	-1
5	0	0.4	200	-1	-1	+1
6	80	0.4	200	+1	-1	+1
7	0	1.2	200	-1	+1	+1
8	80	1.2	200	+1	+1	+1
9 (C)	40	0.8	125	0	0	0

C = center point.

Table 12: The design matrix of the 2^p full factorial experimental design of BDA by RO experiments

Standard order number	Actual value			Coded value
	TMP (bar)	Q (L h^{-1})	x_{TMP}	x_Q
1	10	120	-1	-1
2	10	240	-1	+1
3	30	240	+1	+1
4	30	120	+1	-1
5 (C)	20	180	0	0
6 (C)	20	180	0	0
7 (C)	20	180	0	0

C = center point.

Analysis of the experimental designs

The results of the experimental designs were analysed in various steps.

First, the parameters of the objective functions were estimated (the non-significant parameters were eliminated), and model accuracies and determination coefficients were evaluated in *R-3.5.1*, 2018 software using *RcmdrPlugin.DoE 0.12-3*, 2014 package.

Secondly, after the standardization of the response values, the effect sizes of the significant parameters were calculated (linear regressions without constants), and

model accuracies and determination coefficients were evaluated in *R-3.5.1*, 2018 software using *RcmdrPlugin.DoE 0.12-3*, 2014 package.

Finally, normalities of the residuals were checked by Shapiro-Wilk normality test in *RStudio 1.2.1335*, 2015 software.

In case of BMF with STP, according to Shapiro-Wilk normality tests, the normalities of residuals of the objective functions and functions for effect size determinations were accepted ($p = 0.67$).

In case of BMF with SG, according to Shapiro-Wilk normality tests, the normalities of residuals of the objective functions and functions for effect size determinations were accepted ($p = 0.23$).

In case of BDA by RO, according to Shapiro-Wilk normality tests, the normalities of residuals of the objective functions and functions for effect size determinations were accepted ($p = 0.72$).

Optimisation

In case of BMF with STP and BMF with SG, it was essential to find the global minima of the objective functions, because the lower steady-state fouling layer resistance ($R_{f\,ss}$) is better from technological point of view.

In case of BDA by RO, it was essential to find the global maximum of the objective function, because the higher initial ethanol flux ($J_{EtOH\,0}$) is better from technological point of view.

Global optimisation method 'Grid Search' (G.-Tóth & Tűz, 2009) was used for these purposes. Aspects and comments about Grid Search optimisation method applied for response surface objective function are shown in *Table 13*.

Table 13: Aspects and comments about Grid Search optimisation method applied for response surface objective function

Method	Comments	Conclusion
Response surface method	- The objective function is continuous.	Using Grid Search optimisation of response surface objective function can provide an optimal parameter set which can be directly applied in MSP.
	- Analytical optimisation of the objective function results in a parameter set that does not necessarily fit to the parameter settings available for MSP.	
Grid Search optimisation method	- It is a numerical method with brute force (exhaustive) search (global optimisation method on a grid).	
	- It does not get stuck at a local optimum.	
	- The set of optimisation grid points can be adjusted to the resolution of the parameter ranges available for membrane process.	

Based on the literature (Parkhomenko, 2017), the Grid Search algorithm was implemented in *Scilab 6.1.0*, 2020 software. Furthermore, the response surfaces of the effects of significant parameters for responses were plotted in *Scilab 6.1.0*, 2020 software.

3.4. Assumptions

No unpredictable factors affected the courses of the experiments. The equipment was functioning well, and no technical/equipment problems occurred. The samples were homogeneous and there were no sampling problems.

3.5. Limitations

In case of WMF experiments, full factorial experimental design was not used for membrane filtration to get comprehensive results, because only the feasibility of the process was important at the first stage of the study.

In case of BMF with STP and BMF with SG experiments, filtrations were conducted as single trials, because in a pilot-scale brewery small amount of rough beer can be produced compared to the demand of multiple trials, and the same product quality between different batches of rough beer cannot be guaranteed. However, based on literature (Cimini et al., 2014; Cimini & Moresi, 2014, 2015, 2016a, 2016b), some measurements were replicated for studying the reproducibility potential of the process

(error variance for all the experimental campaign). The average coefficient of variation (10 %) in the estimated beer flux values within data population was appropriate and this value is very similar to the value in the literature (Cimini et al., 2014; Cimini & Moresi, 2014, 2015, 2016a, 2016b).

In case of BDA by RO experiments, the alcohol content limit (0.5% (V/V)) of beer was not reached, because the process times of the dealcoholization trials would have been too long (measurable in days) due to the extremely low and continuously decreasing ethanol fluxes. Thus, the dealcoholization processes were carried out until the beginnings of the preconcentrations at the same volume concentration factor. However, valuable information could have been gained about the process. Furthermore, according to the literature (Li, 2011), the specific energy consumption of RO processes can be determined by a formula including retentate flow rate (one of the factors of modelling of this study), difference in the system pressure and permeate flux (one of the investigated parameters and optimised response of this study). In this study, difference in the system pressure could not be determined exactly. Firstly, the difference in the system pressure was extremely low because of the small size of the membrane module, thus it could not be measured. Secondly, the difference in the system pressure could not be calculated, because of the flat sheet design of the membrane module. Fortunately, specific energy consumption can be deduced from retentate flow rate value, estimated difference in the system pressure and permeate flux.

The main objectives of all studies were not the statistical evaluation or validation of the measured analytical parameters. Only small amounts of samples could be collected, thus just a few parallel analytical experiments were conducted; statistical analyses with validation aims were not performed in these cases.

4. RESULTS AND THEIR DISCUSSION

4.1. Wort membrane filtration

According to the current literature, only I studied wort membrane filtration for the purpose of hot and cold trub separation. Thus, other results can not be compared to my results.

4.1.1. Analytical parameters

Particle size distributions of the original wort (feed) and the permeate are shown in *Figure 9.*

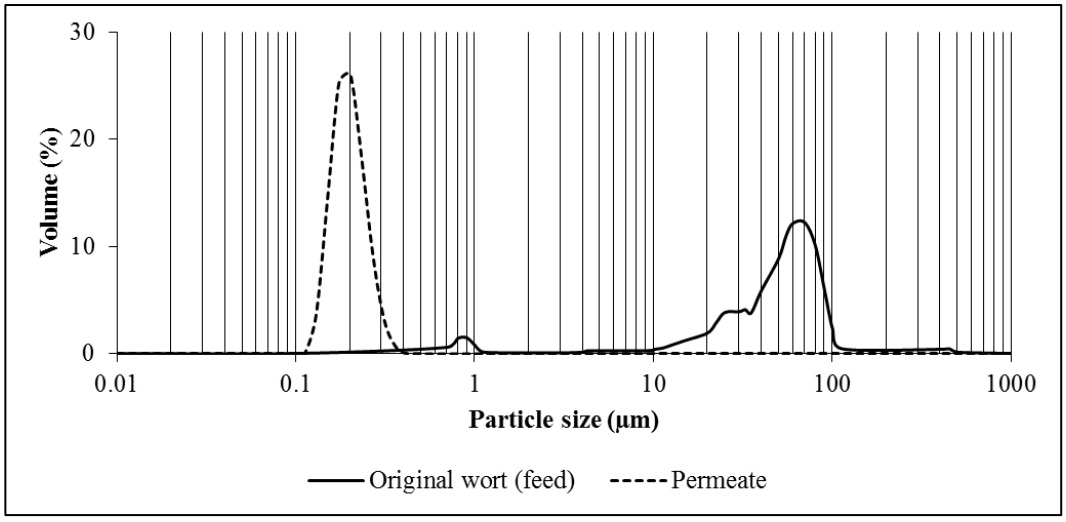

Figure 9: Particle size distributions of the original wort (feed) and the permeate

As it can be seen the original wort contained high volume of hot trub. Because of the very high volume of the hot trub, the volume of the cold trub is low. Maybe the main reason of the fouling mechanism and the low flux values (*Chapter 4.1.3*) is the high volume of the hot trub. Hot trub (particles 30 - 80 µm in diameter) and cold trub (particles about 0.5 µm in diameter) were completely removed by MF. The average particle size of the permeate is around 0.2 µm that corresponds to the nominal pore size of the membrane.

Measured analytical parameters of original wort (feed) and permeate of WMF investigation are shown in *Table 14*.

Table 14: Measured analytical parameters of original wort (feed) and permeate of WMF investigation

Parameter	**Original wort (feed)**	**Permeate**
β-glucan content (mg L^{-1})	117 ± 14	70 ± 9
Bitterness (IBU)	49 ± 4	44 ± 4
Colour (EBC)	12.98 ± 0.09	8.98 ± 0.06
Dynamic viscosity at 20 °C (mPas)	5.43 ± 0.06	4.95 ± 0.01
Extract content (w/w %)	11.16 ± 0.01	10.34 ± 0.01
FAN content (mg L^{-1})	159 ± 8	159 ± 7
pH	6.02 ± 0.03	6.42 ± 0.01
TPC (mg GAE L^{-1})	573.33 ± 9.40	541.22 ± 5.91
Turbidity at 20 °C (EBC)	106.75 ± 5.50	7.88 ± 0.38

β-glucan content decreased dramatically that leads to less fouling during fermentation in MBR. Furthermore, the lower β-glucan content can improve clarification of rough beer (higher filtration throughput and less haze problems in the final product). The bitterness decreased by approximately 5 unit, but this difference cannot be evaluated with sensory analysis in case of these bitterness values. Colour became paler, supposedly due to notable retention of carbohydrates and Maillard reaction products. The dynamic viscosity decreased, this is mainly because of the lower β-glucan content. The dynamic viscosity values are slightly high, but the reasons for this phenomenon are discussed below. The rotary viscometer was chosen because it provides a rapid measurement of the flow curve of the sample tested with high reproducibility. The shear rate used in the test was rather high (when compared to shear rate occurring in a falling ball or capillary viscometer) and therefore shear stress values were also higher. However, all of the samples proved to show Newtonian behaviour (linear flow curve). Therefore, the measured viscosity values (~5 mPas) are appropriate values and are in the proper range (10^{-3} Pas) (Jin et al., 2004b). The extract content decreased by reason of retention of different compounds (e.g. carbohydrates). FAN content did not change that is essential, because adequate level of FAN (150 – 200 mg L^{-1} (Hornsey, 2013)) in wort ensures efficient yeast cell growth and desirable fermentation performance. The reason of the pH change is questionable. According to the literature (Mathias et al., 2015), the pH of the hot trub is acidic (4.62). Hot trub had been completely removed during wort membrane filtration, this may be the reason of the pH increase. However, pH change during microfiltration with this order of magnitude is not entirely unusual (Pagliero et al., 2011). The pH increase negatively affects the microbiological

stability of the permeate. TPC decreased by approximately 5.6 %, because hot trub and cold trub (partly composed of polyphenols) were completely removed. This decrease is beneficial in terms of colloidal stability, because polyphenols play a decisive role in haze formation. However, this decrease is not beneficial in terms of flavour stability, because polyphenols hinder and prevent the oxidation of other molecules present in beer (Aron & Shellhammer, 2010). The turbidity decreased by nearly two orders of magnitude, because of removal of hot break and cold break. This results in less haze problems in the final product.

4.1.2. Separation characteristics parameters

Retentions of different components during WMF investigation are shown in *Table 15*.

Table 15: Retentions of different components during WMF investigation

Component	Retention (%)
β-glucan	40.17
Iso-alpha acids (bitterness)	10.20
Extract	7.65
FAN	0

From a technological point of view, the retention values of β-glucan and FAN are suitable. The retention values of iso-alpha acids and extract are acceptable.

β-glucans are important haze-forming compounds (Mastanjević et al., 2018), thus 40.17 % β-glucan loss positively affects the colloidal stability of the final beer. However, β-glucans are shown to enhance palate fullness (Krebs et al., 2019) and have beneficial health effects (Rondanelli et al., 2009).

According to the literature the iso-α-acid loss is 9-12 %, if the hot wort is treated in the traditional way (using whirlpool) (Jaskula et al., 2009). During my experiments, this value was 10.20 %.

4.1.3. Hydrodynamic parameters

The initial wort flux and the steady-state wort flux of WMF were 16.75 L m^{-2} h^{-1} and 4.89 L m^{-2} h^{-1}, respectively. These values are quite low, because of fouling mechanism. High fouling resistance always leads to high operation costs, which restrict the application of the microfiltration technology (Sun et al., 2018). However, higher flux values and stable fluxes can be achieved with optimisation of the process and pre-treatment of the wort.

4.1.4. Nonlinear regression

According to Student's *t*-test, the parameter estimates were all statistically highly significant ($p < 0.001$). Similarly, F value and R^2 value ($F(3;8) = 516,9$; $p < 0.001$; $R^2 > 0.9$; $p < 0.001$) of the model were also statistically highly significant.

4.2. Beer membrane filtration with static turbulence promoter

Unfortunately, there is no literature about the application of static turbulence promoter during beer membrane filtration. Thus, other results can not be compared to my results.

4.2.1. Analytical parameters

As it can be seen in *Table 16*, the analytical parameters of the rough beer that was produced for the BMF with STP investigations correspond to BJCP vital statistics of 2A. International Pale Lager.

Table 16: BJCP vital statistics of 2A. International Pale Lager and measured analytical parameters of the rough beer (feed) produced for the BMF with STP investigations

Name of parameter	**BJCP vital statistics**	**Rough beer (feed) for BMF with STP**
Alcohol content (V/V %)	4.6 - 6.0	4.58
Original real extract (w/w %)	10.5 - 12.5	11.44
Final real extract (w/w %)	ND	4.48
Final apparent extract (w/w %)	2 - 3	2.82
Bitterness (IBU)	18 - 25	18
Colour (EBC)	3.9 - 11.8	6.53
pH	ND	4.55
Turbidity at 20 °C (EBC)	ND	2.50
Dynamic viscosity at 20 °C (mPas)	ND	5.12

ND = no data.

Because of the high apparent attenuation (75 %) of the used lager yeast, the final apparent extract was low. Generally lower final extract content could lead to lower fouling resistances.

The bitterness of beer comes from a group of substances that are extracted components of hops during wort boiling (Popescu et al., 2013). The bitterness of the rough beer

was not so high, because the wort had been hopped moderately. About 20 % of phenolic compounds present in beer are derived from hops (Fărcaş et al., 2013) and polyphenols are membrane foulants (Stopka et al., 2000).

The colour of the rough beer was light, because Extra Pale Pilsner Malt had been used for the brewing.

The pH of the rough beer was slightly higher than the normal pH values (4.2 - 4.4) of lager beers at the end of the fermentation (Kaneda et al., 1997), but this small pH difference has no significant effect on beer membrane filtration.

According to the EBC standard (Hanna Instruments, n.d.), the rough beer was slightly hazy (2.0 - 4.0 EBC). It appeared that the reason of high fouling resistances was the slightly high turbidity in the rough beer.

The dynamic viscosity values of rough beer and permeate samples of BMF with STP at the filtration temperature are shown in *Table 17*.

Table 17: Dynamic viscosity values of rough beer and permeate samples of BMF with STP at the filtration temperature

Sample (BMF with STP)		**Dynamic viscosity at 10 °C (mPas)**
Rough beer (feed)		5.72 ± 0.04
Standard order number (permeate)	1	5.44 ± 0.87
	2	5.13 ± 0.22
	3	4.75 ± 0.22
	4	4.62 ± 0.21
	5	4.60 ± 0.07
	6	5.39 ± 0.11
	7	5.64 ± 0.17
	8	5.37 ± 0.13
	9	5.82 ± 0.07
	10	5.45 ± 0.03

The dynamic viscosity values were slightly high, but the reasons for this phenomenon are discussed below. The rotary viscometer was chosen because it provides a rapid measurement of the flow curve of the sample tested with high reproducibility. The shear rate used in the test was rather high (when compared to shear rate occurring in a falling ball or capillary viscometer) and therefore shear stress values were also higher.

However, all of the samples proved to show Newtonian behaviour (linear flow curve). Furthermore, at lower temperature the dynamic viscosity values of beer samples are higher (Severa & Los, 2008). Therefore, the measured viscosity values (~5.3 mPas) are appropriate values and are in the proper range (10^{-3} Pas).

4.2.2. Hydrodynamic parameters

Figure 10 shows the hydrodynamic parameters of BMF with STP.

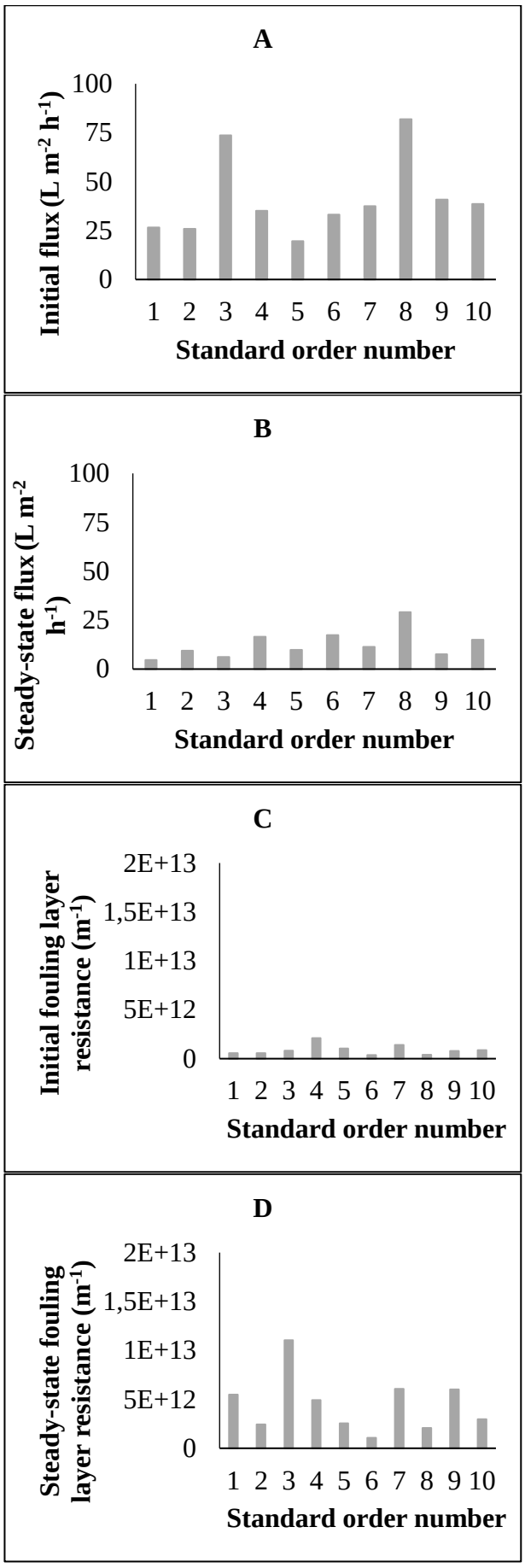

Figure 10: Hydrodynamic parameters of BMF with STP. A: initial beer flux; B: steady-state beer flux; C: initial fouling layer resistance; D: steady-state fouling layer resistance.

As it can be seen in *Figure 10*, the flux declines were significant, because the differences between initial flux and steady-state flux values were high.

Unfortunately, the membrane resistance changes in time (Jeison, 2007) because of membrane ageing (Robinson et al., 2016) and membrane cleaning efficiency (Jeison, 2007). Thus, the initial and steady-state fouling layer resistances described more accurately the fouling characteristics than the initial and steady-state beer flux values, because during the determination of the fouling layer resistances the actual intrinsic resistance of clean membrane were taken into consideration (*Equation 19*).

4.2.3. Nonlinear regression

According to Student's *t*-test, the parameter estimates were all significant ($p < 0.01$). Similarly, F values and R^2 values ($F(3;8) > 659.1$; $p < 0.001$; $R^2 > 0.8$; $p < 0.001$) of the models were also significant.

However, in cases of 'Standard order number 5' and 'Standard order number 9', the very first flux values were extreme which made their estimations slightly biased.

4.2.4. Modelling

Analysis of the experimental design

Parameter estimates of the significant parameters of the objective function of BMF with STP are shown in *Table 18.*

Table 18: Parameter (coefficient) estimates of the significant parameters of the objective function of BMF with STP

Term	Estimate	Standard error	t	Pr(>|t|)
b_0	4.4630×10^{12}	7.9097×10^{10}	56.425	***
b_{STP}	-1.7662×10^{12}	7.9097×10^{10}	-22.330	***
b_{TMP}	1.5702×10^{12}	8.8434×10^{10}	17.755	***
b_Q	1.5166×10^{12}	8.8434×10^{10}	-17.150	***
$b_{STP:TMP}$	-6.9648×10^{11}	8.8434×10^{10}	-7.876	**
$b_{STP:Q}$	4.6600×10^{11}	8.8434×10^{10}	5.269	*
$b_{TMP:Q}$	-4.3718×10^{11}	8.8434×10^{10}	-4.944	*

Response: $R_{f\ ss}$

Significance codes: *** 0.001 ** 0.01 * 0.05

Effect size estimates of the significant parameters of the objective function of BMF with STP are shown in *Table 19*.

Table 19: Effect size estimates of the significant parameters of the objective function of BMF with STP

Parameter	Estimate	Standard error	t	Pr(>|t|)
STP	-0.60520	0.02347	-25.785	***
TMP	0.53802	0.02624	20.502	***
Q	-0.51967	0.02624	-19.803	***
STP:TMP	-0.23865	0.02624	-9.094	***
STP:Q	0.15967	0.02624	6.085	**
TMP:Q	-0.14980	0.02624	-5.708	**

Response: standardized $R_{f\ ss}$

Significance codes: *** 0.001 ** 0.01 * 0.05

There was no significant three-way interaction between the three factors. From the final model, the three-way interaction term was omitted while the significant coefficients of STP, TMP, Q, STP:TMP, STP:Q and TMP:Q are represented in *Table 18*. Model accuracy and determination coefficients of the objective function were also significant ($F(6;3) = 203.7$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$). The objective function (*Equation 23*) which exactly included the parameters determined as significant in *Table 18* was the following:

$$\begin{aligned} R_{f\ ss} = {} & 4.4630 \times 10^{12} - 1.7662 \times 10^{12} \times x_{STP} \\ & + 1.5702 \times 10^{12} \times x_{TMP} - 1.5166 \times 10^{12} \times x_{Q} \\ & - 6.9648 \times 10^{11} \times x_{STP} \times x_{TMP} \\ & + 4.6600 \times 10^{11} \times x_{STP} \times x_{Q} \\ & - 4.3718 \times 10^{11} \times x_{TMP} \times x_{Q} \end{aligned} \qquad \text{Equation 23}$$

The linear model which includes three factors (STP, TMP and Q) is quite accurate.

A positive sign of the effect size indicates an interactive effect of the factors, while a negative sign of the effect size indicates an antagonistic effect of the factors. Thus, TMP and STP:Q had interactive effects and STP, Q, STP:TMP and TMP:Q had antagonistic effects on $R_{f\ ss}$. The possible reasons for these phenomena are discussed below.

Firstly, turbulence promoter (STP) increases the tangential velocity of the flowing rough beer and this flow could sweep the membrane and affect the build-up of the gel layer. Therefore, the usage of turbulence promoter (STP) led to lower steady-state fouling layer resistance ($R_{f\ ss}$).

Secondly, transmembrane pressure (TMP) is the driving force of the membrane filtration. It appears, that TMP pressed the foulants on the membrane surface and into the membrane pores. Maybe higher TMP pressed more the foulants. Therefore, higher TMP led to higher steady-state fouling layer resistance ($R_{f\,ss}$).

Thirdly, retentate flow rate (Q) determines directly the crossflow velocity and turbulence of the feed in the flow channel of the membrane. It appears, that flowing feed could sweep the membrane. Maybe feed with higher crossflow velocity swept more the foulants. Therefore, higher retentate flow rate (Q) led to lower steady-state fouling layer resistance ($R_{f\,ss}$).

Finally, the absolute value of the effect size of STP was higher than the absolute value of the effect size of TMP and the absolute value of the effect size of Q. This implied that STP had higher effect on $R_{f\,ss}$ than TMP and Q had. The absolute values of the effect sizes of two-way interactions were significantly lower than the absolute values of the effect sizes of main factors. This implied that main factors had higher effect on $R_{f\,ss}$ than two-way interactions had.

Model accuracy and determination coefficients of the effect size estimation were significant ($F(6;4) = 271.6$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$).

Optimisation

Figure 11 shows the response surface of the effects of significant parameters (x_{STP}, x_{TMP}, x_Q) and their significant interactions for $R_{f\,ss}$ of BMF with STP.

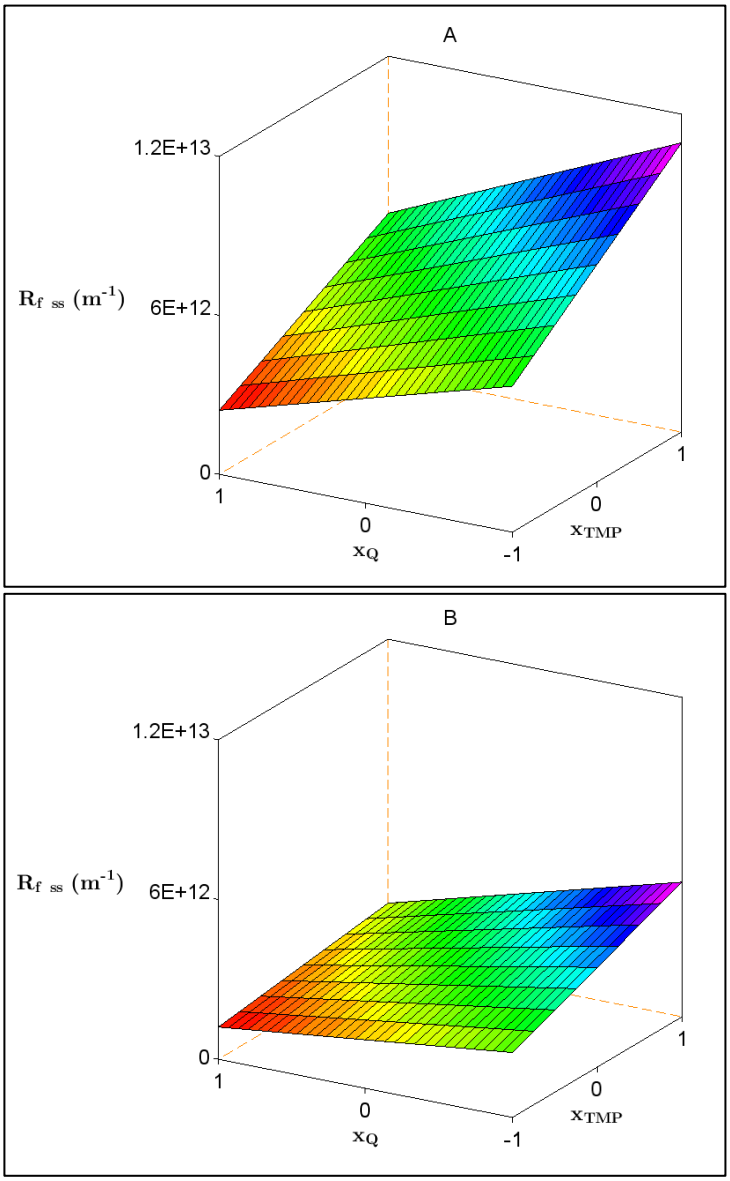

Figure 11: Response surface of the effects of significant parameters (x_{STP}, x_{TMP}, x_Q) and their significant interactions for $R_{f\,ss}$ of BMF with STP. A: without STP; B: with STP.

The optimal values of the factors amounted to respectively STP = yes, TMP = 0.4 bar, Q = 200 L h^{-1}. The predicted $R_{f\,ss}$ under the above condition was 1.2097×10^{12} m^{-1}. Therefore, lowest steady-state fouling layer resistance ($R_{f\,ss}$) could be achieved with the usage of turbulence promoter (STP), the lowest transmembrane pressure (TMP) and the highest retentate flow rate (Q).

4.3. Beer membrane filtration with silica gel

4.3.1. Analytical parameters

As it can be seen in *Table 20*, the analytical parameters of the rough beer that was produced for the BMF with SG investigations correspond to BJCP vital statistics of 2A. International Pale Lager.

Table 20: BJCP vital statistics of 2A. International Pale Lager and measured analytical parameters of the rough beer (feed) produced for the BMF with SG investigations

Name of parameter	**BJCP vital statistics**	**Rough beer (feed) for BMF with SG**
Alcohol content (V/V %)	4.6 - 6.0	4.74
Original real extract (w/w %)	10.5 - 12.5	11.58
Final real extract (w/w %)	ND	4.10
Final apparent extract (w/w %)	2 - 3	2.37
Bitterness (IBU)	18 - 25	24
Colour (EBC)	3.9 - 11.8	5.25
pH	ND	4.63
Turbidity at 20 °C (EBC)	ND	18.0
Dynamic viscosity at 20 °C (mPas)	ND	4.82

ND = no data.

Because of the high apparent attenuation (79 %) of the used lager yeast, the final apparent extract was low. Generally lower final extract content could lead to lower fouling resistances.

The bitterness of beer comes from a group of substances that are extracted components of hops during wort boiling (Popescu et al., 2013). The bitterness of the rough beer was not so high, because the wort had been hopped moderately. About 20 % of phenolic compounds present in beer are derived from hops (Fărcaş et al., 2013) and polyphenols are membrane foulants (Stopka et al., 2000).

The colour of the rough beer was light, because Extra Pale Pilsner Malt had been used for the brewing.

The pH of the rough beer was slightly higher than the normal pH values (4.2 - 4.4) of

lager beers at the end of the fermentation (Kaneda et al., 1997), but this small pH difference has no significant effect on beer membrane filtration.

According to the EBC standard (Hanna Instruments, n.d.), the rough beer was very hazy (> 8.0 EBC). It appeared that the reason of high fouling resistances was the high turbidity in the rough beer.

The dynamic viscosity values of rough beer and permeate samples of BMF with SG at the filtration temperature are shown in *Table 21*.

Table 21: Dynamic viscosity values of rough beer and permeate samples of BMF with SG at the filtration temperature

Sample (BMF with SG)		**Dynamic viscosity at 10 °C (mPas)**
Rough beer (feed)		5.57 ± 0.01
Standard order number (permeate)	1	5.55 ± 0.19
	2	5.23 ± 0.03
	3	6.11 ± 0.11
	4	5.69 ± 0.12
	5	5.66 ± 0.09
	6	5.60 ± 0.06
	7	5.31 ± 0.05
	8	5.30 ± 0.20
	9	5.48 ± 0.43

The dynamic viscosity values were slightly high, but the reasons for this phenomenon are discussed below. The rotary viscometer was chosen because it provides a rapid measurement of the flow curve of the sample tested with high reproducibility. The shear rate used in the test was rather high (when compared to shear rate occurring in a falling ball or capillary viscometer) and therefore shear stress values were also higher. However, all of the samples proved to show Newtonian behaviour (linear flow curve). Furthermore, at lower temperature the dynamic viscosity values of beer samples are higher (Severa & Los, 2008). Therefore, the measured viscosity values (~5.5 mPas) are appropriate values and are in the proper range (10^{-3} Pas).

4.3.2. Hydrodynamic parameters

Figure 12 shows the hydrodynamic parameters of BMF with SG.

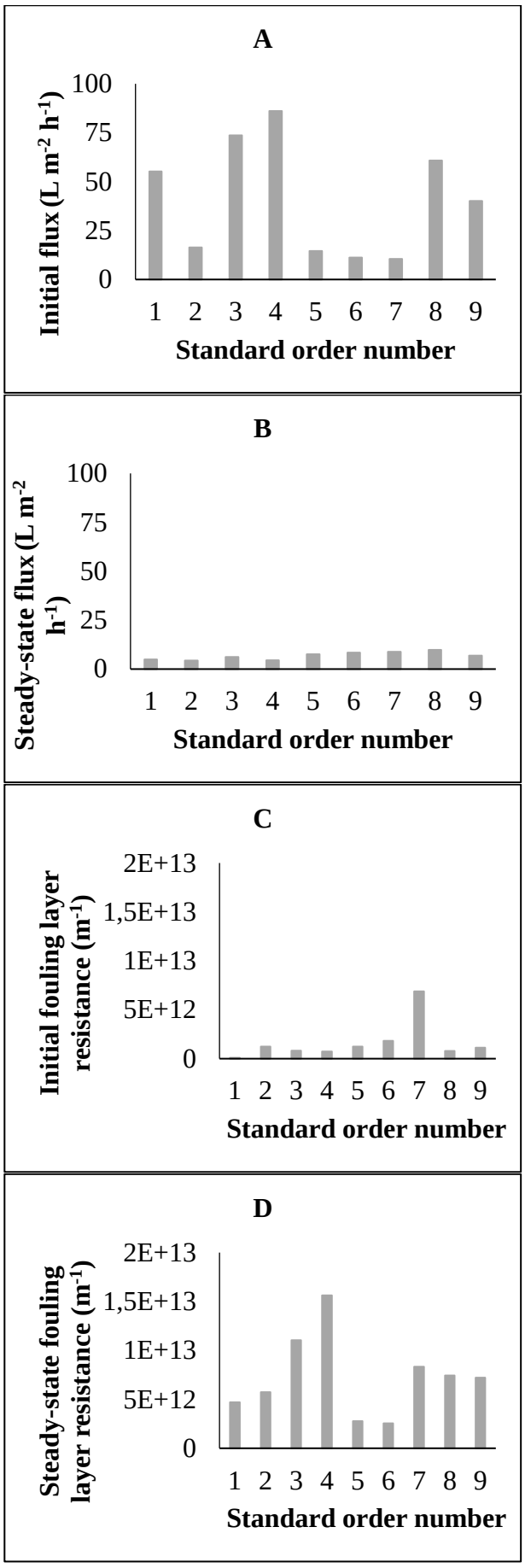

Figure 12: Hydrodynamic parameters of BMF with SG. A: initial beer flux; B: steady-state beer flux; C: initial fouling layer resistance; D: steady-state fouling layer resistance.

As it can be seen in *Figure 12*, the flux declines were significant, because the differences between initial flux and steady-state flux values were high.

Unfortunately, the membrane resistance changes in time (Jeison, 2007) because of membrane ageing (Robinson et al., 2016) and membrane cleaning efficiency (Jeison, 2007). Thus, the initial and steady-state fouling layer resistances described more accurately the fouling characteristics than the initial and steady-state beer flux values, because during the determination of the fouling layer resistances the actual intrinsic resistance of clean membrane were taken into consideration (*Equation 19*).

According to the literature, in case of beer membrane filtration with silica gel, flux values (24 hr average flux) showed a general increase at all operating conditions ($T = 2.0 \pm 0.9$ °C, TMP = 0.8 bar, Re = 1552; operation modes: conventional crossflow microfiltration, with backflush, with backflush and reversed configuration) and chill haze level was substantially reduced (Gan et al., 2001).

4.3.3. Nonlinear regression

According to Student's *t*-test, the parameter estimates were all significant ($p < 0.05$). Similarly, F values and R^2 values ($F(3;8) > 99.4$; $p < 0.001$; $R^2 > 0.9$; $p < 0.05$) of the models were also significant. There were two exceptions when a bootstrapping was necessary with 60 samples. In the case of setting 'Standard order number 3', the estimation of the coefficient of $J_{b\ ss}$ was close to significant ($p = 0.06$), while for 'Standard order number 7', R^2 was as low as 0.51, though still significant ($p < 0.05$). Having such a low number of observations, it can be considered as very good results of fit.

4.3.4. Modelling

Analysis of the experimental design

Parameter estimates of the significant parameters of the objective function of BMF with SG are shown in *Table 22*.

Table 22: Parameter (coefficient) estimates of the significant parameters of the objective function of BMF with SG

Term	Estimate	Standard error	t	Pr(>|t|)
b_0	7.2678×10^{12}	5.3865×10^{11}	13.4925	***
b_{TMP}	3.3383×10^{12}	5.7133×10^{11}	5.8431	**
b_Q	-2.0083×10^{12}	5.7133×10^{11}	-3.5072	*

Response: $R_{f\ ss}$

Significance codes: *** 0.001 ** 0.01 * 0.05

Effect size estimates of the significant parameters of the objective function of BMF with SG are shown in *Table 23*.

Table 23: Effect size estimates of the significant parameters of the objective function of BMF with SG

Parameter	Estimate	Standard error	t	Pr(>|t|)
TMP	0.8069	0.1278	6.311	***
Q	-0.4843	0.1278	-3.788	**

Response: standardized $R_{f\ ss}$

Significance codes: *** 0.001 ** 0.01 * 0.05

SGC had no significant effect on $R_{f\ ss}$. Furthermore, there were no significant interactions between the factors. From the final model, SGC and the interaction terms were omitted while the significant coefficients of TMP and Q are represented in *Table 22*. Model accuracy and determination coefficients of the objective function were also significant ($F(2;6) = 23.22$; $p < 0.01$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$). The objective function (*Equation 24*) which exactly included the parameters determined as significant in *Table 22* was the following:

$$R_{f\ ss} = 7.2678 \times 10^{12} + 3.3383 \times 10^{12} \times x_{TMP} - 2.0038 \times 10^{12} \times x_Q \quad \text{Equation 24}$$

The linear model which includes merely two factors (TMP and Q) is quite simple and accurate at the same time.

A positive sign of the effect size indicates an interactive effect of the factors, while a

negative sign of the effect size indicates an antagonistic effect of the factors. Thus, TMP had interactive effect and Q had antagonistic effect on $R_{f\,ss}$. The possible reasons for these phenomena are discussed below.

Firstly, transmembrane pressure (TMP) is the driving force of the membrane filtration. It appears, that TMP pressed the foulants on the membrane surface and into the membrane pores. Maybe higher TMP pressed more the foulants. Therefore, higher TMP led to higher steady-state fouling layer resistance ($R_{f\,ss}$).

Secondly, retentate flow rate (Q) determines directly the crossflow velocity and turbulence of the feed in the flow channel of the membrane. It appears, that flowing feed could sweep the membrane. Maybe feed with higher crossflow velocity swept more the foulants. Therefore, higher retentate flow rate (Q) led to lower steady-state fouling layer resistance ($R_{f\,ss}$). Furthermore, the absolute value of the effect size of the TMP was higher than the absolute value of the effect size of the Q. This implied that TMP had higher effect on $R_{f\,ss}$ than retentate flow rate (Q) had.

Model accuracy and determination coefficients of the effect size estimation were significant ($F(2;7) = 27.09$; $p < 0.001$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$).

Optimisation

Figure 13 shows the response surface of the effects of significant parameters (x_{TMP}, x_Q) for $R_{f\,ss}$ of BMF with SG.

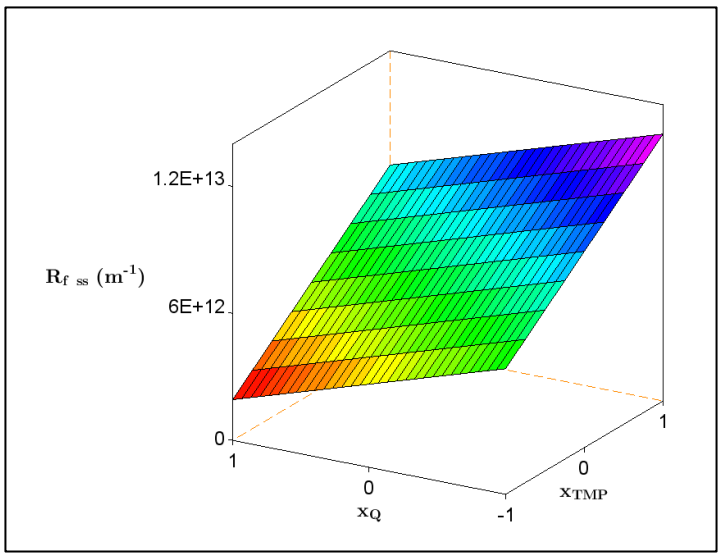

Figure 13: Response surface of the effects of significant parameters (x_{TMP}, x_Q) for $R_{f\,ss}$ of BMF with SG

The optimal values of the factors amounted to respectively TMP = 0.4 bar,

Q = 200 L h^{-1}. The predicted $R_{f\,ss}$ under the above condition was 1.9257×10^{12} m^{-1}. Therefore, lowest steady-state fouling layer resistance ($R_{f\,ss}$) could be achieved with the lowest transmembrane pressure (TMP) and the highest retentate flow rate (Q). Furthermore, silica gel free BMF can be performed.

4.4. Beer dealcoholization by reverse osmosis

4.4.1. Analytical parameters

Measured analytical parameters of the beer (feed) (*Chapter 3.1.5*) used for the BDA by RO investigations are shown in *Table 24*.

Table 24: Measured analytical parameters of the beer (feed) used for the BDA by RO investigations

Name of parameter	Beer (feed) for BDA by RO
Alcohol content (V/V %)	4.34
Original real extract (w/w %)	10.28
Final real extract (w/w %)	3.63
Final apparent extract (w/w %)	2.04
Bitterness (IBU)	12
Colour (EBC)	8.89
pH	4.23
Turbidity at 20 °C (EBC)	0.48
Dynamic viscosity at 20 °C (mPas)	5.48

Because of the high apparent attenuation (80 %) of the used lager yeast, the final apparent extract was low. Generally lower final extract content could lead to lower fouling resistances and lower osmotic pressure of the feed.

The bitterness of beer comes from a group of substances that are extracted components of hops during wort boiling (Popescu et al., 2013). The bitterness of the beer was not so high, because the wort had been probably hopped moderately. About 20 % of phenolic compounds present in beer are derived from hops (Fărcaş et al., 2013) and polyphenols are membrane foulants (Stopka et al., 2000).

The colour of the beer was pale, and the colour of beer is mostly attributed to melanoidins, product of the final phase of the Maillard reaction (Steiner et al., 2011). The melanoidins have foam stabilising properties (Bamforth, 1985) and foaming can cause problems during MSP (Chang & Lee, 1998). The colour values of the permeate

samples were 0.00 EBC. This means that the RO99 membrane (*Table 6*) completely rejected the colour compounds of the beer.

The pH value of the beer was in the pH interval (4.2 - 4.4) of lager beers at the end of the fermentation (Kaneda et al., 1997). The pH levels of the permeate samples (3.80 ± 0.01 – 4.07 ± 0.01) were slightly lower than the pH level of the beer. This may be because the acids of the beer passed through the RO99 membrane (*Table 6*).

According to the EBC standard (Hanna Instruments, n.d.), the beer was brilliant (0.0 – 0.5 EBC). Generally, if a beer is brilliant in terms of haziness, it leads to lower fouling resistances. Turbidity values of the permeate samples were low (0.2 – 0.3 EBC), because the RO99 membrane (*Table 6*) rejected most of the haze-active compounds of the beer.

The dynamic viscosity values of beer and permeate samples of BDA by RO at the separation temperature are shown in *Table 25*.

Table 25: Dynamic viscosity values of beer and permeate samples of BDA by RO at the separation temperature

Sample (BDA by RO)		**Dynamic viscosity at 15 °C (mPas)**
Beer (feed)		5.85 ± 0.03
Standard order number (permeate)	1	5.50 ± 0.03
	2	5.43 ± 0.01
	3	5.07 ± 0.04
	4	5.04 ± 0.03
	5	5.37 ± 0.03
	6	5.14 ± 0.02
	7	5.13 ± 0.02

The dynamic viscosity values were slightly high, but the reasons for this phenomenon are discussed below. The rotary viscometer was chosen because it provides a rapid measurement of the flow curve of the sample tested with high reproducibility. The shear rate used in the test was rather high (when compared to shear rate occurring in a falling ball or capillary viscometer) and therefore shear stress values were also higher. However, all of the samples proved to show Newtonian behaviour (linear flow curve). Furthermore, at lower temperature the dynamic viscosity values of beer samples and permeate samples (ethanol-water mixture) are higher (Severa & Los, 2008; ‘Anton

Paar – Viscosity of Ethanol – viscosity table and viscosity chart', n.d.; 'Anton Paar – Viscosity of Water – Viscosity Table and Viscosity Chart', n.d.). Therefore, the measured viscosity values (~5.9 mPas for beer samples and ~5.2 mPas for permeate samples) are appropriate values and are in the proper range (10^{-3} Pas).

The ethanol content values of beer and permeate samples of BDA by RO at 20 °C are shown in *Table 26*.

Table 26: Ethanol content values of beer and permeate samples of BDA by RO at 20 °C

Sample (BDA by RO)		**Ethanol content at 20 °C (% (V/V))**
Beer (feed)		4.34 ± 0.02
Standard order number (permeate)	1	2.56 ± 0.02
	2	2.75 ± 0.01
	3	1.45 ± 0.01
	4	1.82 ± 0.01
	5	1.92 ± 0.01
	6	2.10 ± 0.01
	7	2.07 ± 0.05

The alcohol content values of the permeate samples were low. Thus, the optimisation of the operating parameters and proper membrane area are required for the short dealcoholization process time. Short dealcoholization process is important in terms of sustainability and cost efficiency.

4.4.2. Separation characteristics parameters

The retention values of the real extract were ~99 % and retention values of the Iso-alpha acids (bitterness) were 100 % because of the application of the RO99 membrane (*Table 6*). The retention values of Iso-alpha acids were lower in the literature than the values of this study, because membrane with lower molecular weight cut-off (MWCO) was used in this study (Alcantara et al., 2016).

Furthermore, the organoleptic properties of the dealcoholized beer can be predicted well, because besides the measured analytical parameters of permeate samples (colour, pH value, turbidity, dynamic viscosity), the calculated retention of the different components (real extract, Iso-alpha acids) significantly determine the sensory characteristics of this type of product.

4.4.3. Hydrodynamic parameters

Figure 14 shows the hydrodynamic parameters of BDA by RO.

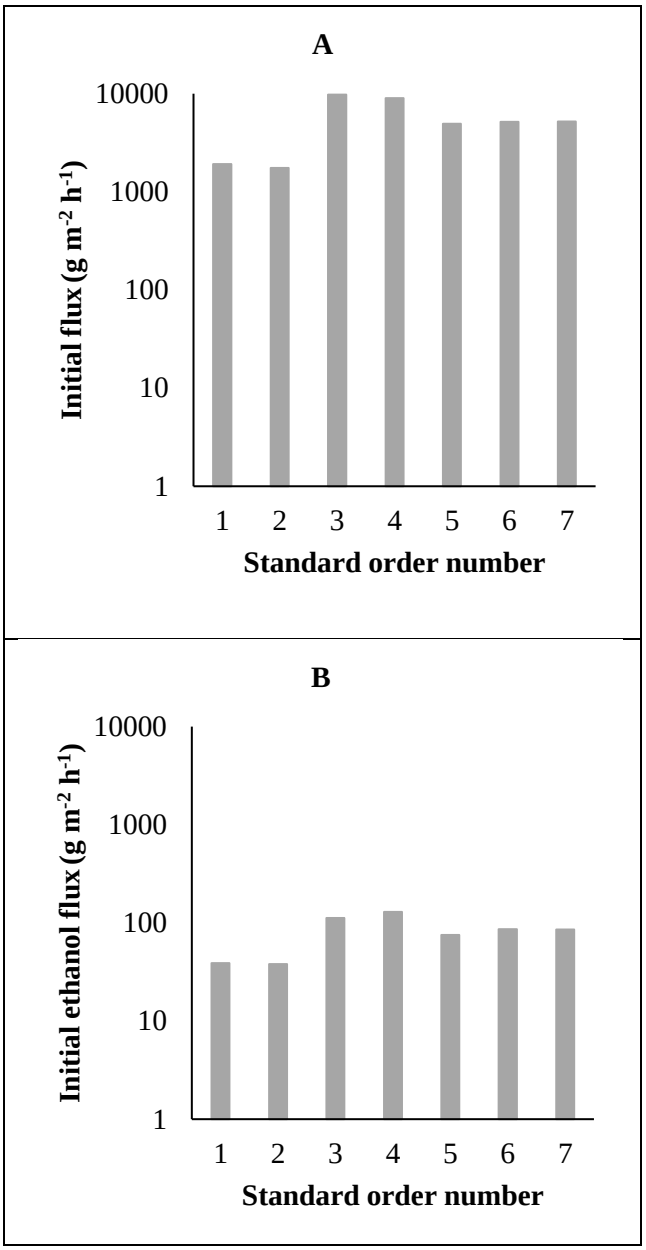

Figure 14: Hydrodynamic parameters of BDA by RO. A: initial flux (log); B: initial ethanol flux (log).

The flux values were very low, especially the initial ethanol flux values. In order to get adequate amount of permeate and separated ethanol, large membrane area is required. However, a larger membrane area results higher investment and operating costs.

4.4.4. Linear regression

According to Student's *t*-test, the parameter estimates were all highly significant in five cases ($p < 0.001$). Similarly, F values and R^2 values ($F > 27.9$; $df_1=1$; $24<df_2<26$; $p < 0.001$; $R^2 > 0.5$; $p < 0.001$) of the models were also highly significant. For the

settings ‘Standard order number 1‘ and ‘Standard order number 5’, the parameter estimates were all significant, though less highly ($p < 0.05$; $p<0.01$, respectively). In these two cases, we obtained some less but still significant F values ($F(1;23) > 4.4$; $p < 0.05$; $F(1;24) > 14.7$; $p < 0.01$, respectively) and R^2 values ($R^2 > 0.2$; $p < 0.05$; $R^2 > 0.4$; $p < 0.01$) as well.

4.4.5. Modelling

Analysis of the experimental design

Parameter estimates of the significant parameters of the objective function of BDA by RO are shown in *Table 27*.

Table 27: Parameter (coefficient) estimates of the significant parameters of the objective function of BDA by RO

Term	Estimate	Standard error	t	Pr(>|t|)
b_0	80.871	2.597	31.14	***
b_{TMP}	41.094	3.435	11.96	***

Response: $J_{EtOH\ 0}$

Significance codes: *** 0.001 ** 0.01 * 0.05

Effect size estimate of the significant parameter of the objective function of BDA by RO are shown in *Table 28*.

Table 28: Effect size estimate of the significant parameter of the objective function of BDA by RO

Parameter	Estimate	Standard error	t	Pr(>|t|)
TMP	1.20389	0.09187	13.1	***

Response: standardized $J_{EtOH\ 0}$

Significance codes: *** 0.001 ** 0.01 * 0.05

TMP had significant effect on $J_{EtOH\ 0}$. As it can be seen in the literature (Catarino et al., 2007), the effect of TMP on ethanol retention and permeate flux values were significant. Thus, it is clear why TMP had significant effect on $J_{EtOH\ 0}$. retentate flow rate (Q) had no significant effect on $J_{EtOH\ 0}$. As it can be seen in the literature (Catarino et al., 2007), the effect of Q on ethanol retention and permeate flux values were close to negligible with wider Q range (Q: 120, 270, 420 L h^{-1}) than the applied Q range (Q: 120, 240 L h^{-1}) in this study. Thus, it is clear why Q had no significant effect on $J_{EtOH\ 0}$. Furthermore, there was no significant interaction between the factors. From the final model, Q and the interaction terms were omitted while the significant coefficient of

TMP is represented in *Table 27*. Model accuracy and determination coefficients of the objective function were also significant ($F(1;5) = 143.1$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$). The objective function (*Equation 25*) which exactly included the parameters determined as significant in *Table 27* was the following:

$$J_{EtOH\ 0} = 80.871 + 41.094 \times x_{TMP} \qquad \text{Equation 25}$$

The linear model which includes merely one factor (TMP) is quite simple and accurate at the same time.

A positive sign of the effect size indicates an interactive effect of the factors, while a negative sign of the effect size indicates an antagonistic effect of the factors. Thus, TMP had interactive effect on $J_{EtOH\ 0}$. The possible reasons for these phenomena are discussed below.

Firstly, the difference of transmembrane pressure (TMP) and osmotic pressure difference is the driving force of RO. Therefore, higher TMP led to higher total initial flux (*Chapter 4.4.3*).

Secondly, higher transmembrane pressure (TMP) presses more the foulants on the membrane surface, forming thicker fouling layer. Maybe the ethanol molecules are captured into the fouling layer. Therefore, higher TMP led to higher ethanol retention (results lower ethanol concentration in permeate) (*Chapter 4.4.1*).

Summarizing it can be said that higher transmembrane pressure (TMP) led to higher total initial flux and higher alcohol retention, but the effect of the TMP on total initial flux is higher than the effect of the TMP on ethanol retention. Thus, higher TMP led to higher initial ethanol flux ($J_{EtOH\ 0}$). The facts about the effect sizes of the TMP on total initial flux and ethanol retention that have been mentioned in this paragraph are not obvious.

Model accuracy and determination coefficients of the effect size estimation were significant ($F(1;6) = 171.7$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$).

Optimisation

Figure 15 shows the 2D response plot of the effect of significant parameter (x_{TMP}) for $J_{EtOH\ 0}$ of BDA by RO.

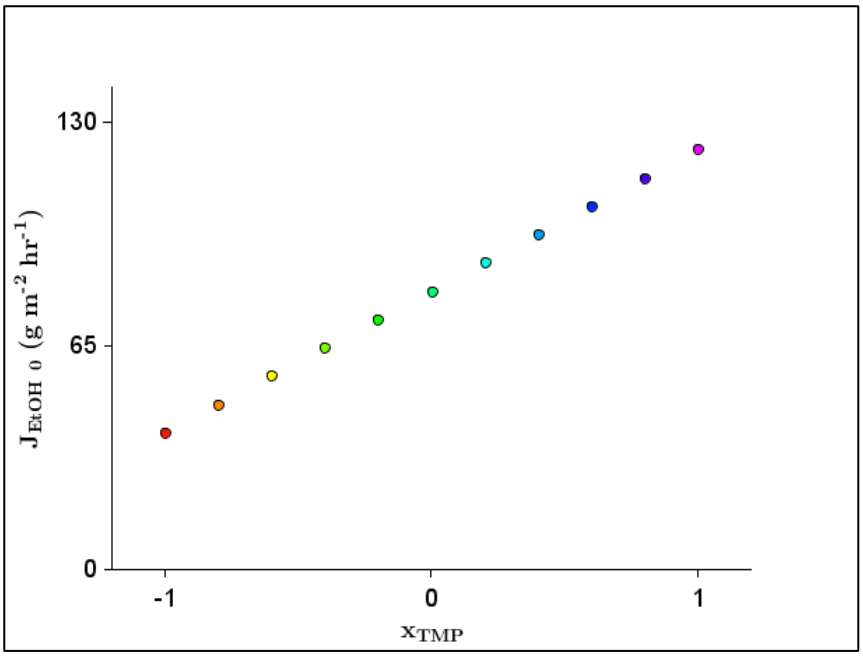

Figure 15: 2D response plot of the effect of significant parameter (x_{TMP}) for $J_{EtOH\ 0}$ of BDA by RO

The optimal value of the factor amounted to TMP = 30 bar. The predicted $J_{EtOH\ 0}$ under the above condition was 121.965 g m^{-2} h^{-1}. Therefore, highest initial ethanol flux ($J_{EtOH\ 0}$) could be achieved with the highest transmembrane pressure (TMP) and the lowest retentate flow rate (Q) can be applied.

4.5. Membrane cleaning

4.5.1. Membrane cleaning of microfiltration experiments

In case of microfiltration experiments, it was a great challenge to find the optimal temperature (60 °C) of the Sodium hydroxide solution and the relatively high concentration (1 % (w/w)) of the cleaning solutions.

The proposed cleaning method can be considered to be efficient, because the average of flux recoveries was higher than 97 %.

4.5.2. Membrane cleaning of reverse osmosis experiments

In case of reverse osmosis experiments, I had to ignore the citric acid cleaning, because it had no effect on membrane cleanliness and I was able to reduce the concentration of the NaOH solution. Furthermore, the low cleaning temperature was effective.

The proposed cleaning method can be considered to be efficient, because the average of flux recoveries was 109 %.

5. CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions and recommendations of wort membrane filtration

All of the problems of the WMF investigation mentioned in *Chapter 1.3.1* have been completely solved: (i) valuable information about the removal of hot trub and cold trub was gained from determined particle size distribution; (ii) valuable information was gained from determined analytical parameters of original wort and permeate; (iii) the determined retentions of different essential components described well the separation; (iv) determined initial flux and the steady-state flux values of the WMF gave important information of the fouling mechanism.

It has been proven that hot trub and cold trub can be completely removed by WMF and the changes in the analytical parameters are appropriate. The changes in the analytical parameters could be improved by the optimisation of operating parameters (e.g. TMP and crossflow velocity) and application of permeate backflow techniques, enzymes, filtration aids, flow pulsation, gas sparging, static turbulence promoter (STP), Vibratory Shear Enhanced Process (VSEP) etc. Flux values of the membrane filtration experiment were quite low, but fluxes could be enhanced by the above-mentioned optimisations and applications.

5.2. Conclusions and recommendations of beer membrane filtration with static turbulence promoter

All of the problems of the BMF with STP investigation mentioned in *Chapter 1.3.2* have been completely solved: (i) valuable information for membrane filtrations was gained from determined analytical parameters of rough beer and viscosity values of permeate samples could be used for the physical modelling; (ii) the determined values of hydrodynamic parameters of the membrane filtrations could be used for the physical modelling and the experimental design; (iii) the experimental design was analysed, parameters of the objective function and effect sizes were estimated; (iv) the global minimum of the objective function was successfully found and the results of the optimisation can directly be applied in practice; (v) an effective membrane cleaning method was developed for MF processes.

The most important findings of this investigation are summarized, and conclusions are drawn below.

According to the analysis of the experimental design, STP, TMP, Q, STP:TMP, STP:Q

and TMP:Q had significant effect on $R_{f\,ss}$ with the given parameters. Furthermore, there was no significant three-way interaction between the factors. This means that the commercial breweries should focus on the optimisation of usage of STP, TMP and Q too. In this research, an turbulence promoter (STP) with a specific geometry was tested. However, in a later exercise, a wider range of operating parameters and several STPs with different geometries could be tested with the aid of lowering fouling layer resistances.

TMP and STP:Q had interactive effects and STP, Q, STP:TMP and TMP:Q had antagonistic effects on $R_{f\,ss}$. Furthermore, the effect size of STP was the highest among the effect sizes of the significant main factors and interactions.

Based on the results of the optimisation, the lowest steady-state fouling layer resistance ($R_{f\,ss}$) could be achieved with the usage of turbulence promoter (STP), lowest transmembrane pressure (TMP) and the highest retentate flow rate (Q). Thus, commercial breweries should use turbulence promoter (STP) and set the operating parameters at these levels.

Furthermore, a novel and efficient (average of flux recoveries was higher than 97 %) membrane cleaning method was developed and applied to recover the initial intrinsic resistance.

The laboratory measurements, modelling and optimisation method that were detailed in this research can be implemented by turbulence promoter (STP) manufacturers, membrane researchers and commercial breweries during product and technology development because of the simplicity and relatively low resource demand.

5.3. Conclusions and recommendations of beer membrane filtration with silica gel

All of the problems of the BMF with SG investigation mentioned in *Chapter 1.3.3* have been completely solved: (i) valuable information for membrane filtrations was gained from determined analytical parameters of rough beer and viscosity values of permeate samples could be used for the physical modelling; (ii) the determined values of hydrodynamic parameters of the membrane filtrations could be used for the physical modelling and the experimental design; (iii) the experimental design was analysed, parameters of the objective function and effect sizes were estimated; (iv) the global minimum of the objective function was successfully found and the results of the

optimisation can directly be applied in practice.

The most important findings of this investigation are summarized, and conclusions are drawn below.

According to the analysis of the experimental design, TMP and Q had significant effect, while SGC had no significant effect on $R_{f\ ss}$ with the given parameters. Furthermore, there were no significant interactions between the factors. This means that the commercial breweries should only focus on the optimisation of TMP and Q, and silica gel (SG) free BMF can be performed. The SG free BMF is important because of environmental issues. However, filtration aids other than silica gel (SG) can be developed and tested to intensify BMF.

TMP had interactive effect and Q had antagonistic effect on $R_{f\ ss}$. Furthermore, the effect size of TMP was higher than the effect size of Q.

Based on the results of the optimisation, the lowest steady-state fouling layer resistance ($R_{f\ ss}$) could be achieved with the lowest transmembrane pressure (TMP) and the highest retentate flow rate (Q). Thus, commercial breweries should set the operating parameters at these levels.

The laboratory measurements, modelling and optimisation method that were detailed in this research can be implemented by membrane researchers and commercial breweries during product and technology development because of the simplicity and relatively low resource demand.

5.4. Conclusions of and recommendations beer dealcoholization by reverse osmosis

All of the problems of the BDA by RO investigation mentioned in *Chapter 1.3.4* have been completely solved: (i) valuable information for membrane separations was gained from determined analytical parameters of beer and ethanol content values of permeate samples could be used for the physical modelling; (ii) the determined values of hydrodynamic parameters of the membrane separations could be used for the physical modelling; (iii) the calculated ethanol flux values of the membrane separations could be used for the physical modelling and the experimental design; (iv) the experimental design was analysed, parameters of the objective function and effect sizes were estimated; (v) the global maximum of the objective function was successfully found and the results of the optimisation can be applied in practice; (vi)

an effective membrane cleaning method was developed

The most important findings of this investigation are summarized, and conclusions are drawn below.

According to the analysis of the experimental design, TMP had significant effect, while Q had no significant effect on $J_{EtOH\ 0}$ with the given parameters. Furthermore, there was no significant interaction between the factors. This means that the commercial breweries should only focus on the optimisation of TMP. BDA by RO can be performed with lowest required retentate flow rate (Q), which results in lower energy consumption. The lower energy consumption is important because of environmental and economic issues.

Furthermore, TMP had interactive effect on $J_{EtOH\ 0}$.

Based on the results of the optimisation, the highest initial ethanol flux ($J_{EtOH\ 0}$) could be achieved with the highest transmembrane pressure (TMP). Thus, commercial breweries should set the TMP at this level.

Furthermore, a novel and efficient (average of flux recoveries was 109 %) membrane cleaning method was developed and applied to recover the initial intrinsic resistance.

In a later exercise, beers with different alcohol and extract content could be dealcoholized by RO.

5.5. General conclusion

From my experiments, beer membrane filtration (rough beer clarification with microfiltration) with static turbulence promoter would be applied mostly at the brewing industry. The reasons of this are as follows: the process is much more sustainable than the conventional diatomaceous earth (DE) filtration (Ambrosi et al., 2014) and according to my results, the process can be intensified with static turbulence promoter.

The benefits of my research outcomes from an industrial point of view are detailed below. Based on my results, lower fouling rates and higher flux values can be achieved in industrial scale that result much more sustainable processes (less energy consumption, much more easier cleaning procedures) and shorter shifts.

6. NEW SCIENTIFIC RESULTS

1.

I have proven that hot trub (d = 30 – 80 μm) and cold trub (d = ~0.5 μm) can be completely removed from pale hopped wort (extract content = 11.16 ± 0.01 w/w %, bitterness = 49 ± 4 IBU, turbidity at 20 °C = 106.75 ± 5.50 EBC) by microfiltration with the application of Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.2 μm pore size and 7 mm channel diameter) and with the following operating parameters: T = 10 ± 1 °C, transmembrane pressure (TMP) = 0.4 bar, retentate flowrate (Q) = 50 L h^{-1}.

According to the results of the analytical measurements, the changes in analytical parameters were appropriate: the bitterness decreased by approximately 5 unit, TPC decreased by approximately 5.6 %, retention of β-glucan was 40.17 % and free amino nitrogen content did not change.

The initial and steady-state fluxes, with the above-mentioned conditions, were 16.75 and 4.89 L m^{-2} h^{-1}, respectively.

2.

I have developed a model that describes rough beer ("2A. International Pale Lager" (BJCP); alcohol content = 4.58 V/V %, final real extract content = 4.48 w/w %, bitterness = 18 IBU, turbidity at 20 °C = 2.50) membrane filtration at a temperature of 10 ± 1 °C with static turbulence promoter (SPIRAL LD2 STP from Inox, Serbia) and Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.5 μm pore size and 7 mm channel diameter).

The model (objective function) (*Equation 26*) was the following:

$$\begin{aligned} R_{f\,ss} = {} & 4.4630 \times 10^{12} - 1.7662 \times 10^{12} \times x_{STP} \\ & + 1.5702 \times 10^{12} \times x_{TMP} - 1.5166 \times 10^{12} \times x_{Q} \\ & - 6.9648 \times 10^{11} \times x_{STP} \times x_{TMP} \\ & + 4.6600 \times 10^{11} \times x_{STP} \times x_{Q} \\ & - 4.3718 \times 10^{11} \times x_{TMP} \times x_{Q} \end{aligned} \qquad \text{Equation 26}$$

where $R_{f\,ss}$ (m^{-1}) is the steady-state fouling layer resistance; x_{STP} is the coded factor for static turbulence promoter (STP) with the factor values: -1, +1; x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1 and x_Q is the coded factor for retentate flow rate (Q) with the factor interval: -1 – +1. The range of validity: STP = no or yes; TMP = 0.4 – 1.2 bar, Q = 50 - 200 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant (F(6;3) = 203.7;

$p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$).

I have determined the effect sizes of the significant parameters and they were the following: STP = -0.61, TMP = 0.54, Q = -0.52, STP:TMP = -0.24, STP:Q = 0.16 and TMP:Q = -0.15. Model accuracy and determination coefficients of the effect size estimation were significant ($F(6;4) = 271.6$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$).

I have found the optimum (global minimum) of the objective function. The optimal values of the factors amounted to respectively STP = yes, TMP = 0.4 bar, Q = 200 L h^{-1}. The predicted $R_{f\,ss}$ under the above condition was 1.2097×10^{12} m^{-1}.

3.

I have developed a model that describes rough beer ("2A. International Pale Lager" (BJCP); alcohol content = 4.74 V/V %, final real extract content = 4.10 w/w %, bitterness = 24 IBU, turbidity at 20 °C = 18.00) membrane filtration at a temperature of 10 ± 1 °C with silica gel (Stabifix W MF from Stabifix Brauerei-Technik, Germany) and Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.5 µm pore size and 7 mm channel diameter).

The model (objective function) (*Equation 27*) was the following:

$$R_{f\,ss} = 7.2678 \times 10^{12} + 3.3383 \times 10^{12} \times x_{TMP} - 2.0038 \times 10^{12} \times x_Q \qquad \text{Equation 27}$$

where $R_{f\,ss}$ (m^{-1}) is the steady-state fouling layer resistance, x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1 and x_Q is the coded factor for retentate flow rate (Q) with the factor interval: -1 – +1. The range of validity: silica gel concentration (SGC) = 0 – 80 g hL^{-1}; TMP = 0.4 – 1.2 bar, Q = 50 - 200 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(2;6) = 23.22$; $p < 0.01$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$).

I have determined the effect sizes of the significant parameters and they were the following: TMP = 0.81, Q = -0.48. Model accuracy and determination coefficients of the effect size estimation were significant ($F(2;7) = 27.09$; $p < 0.001$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$).

I have found the optimum (global minimum) of the objective function. The optimal values of the factors amounted to respectively TMP = 0.4 bar, Q = 200 L h^{-1}. The

predicted $R_{f\,ss}$ under the above condition was 1.9257×10^{12} m^{-1}.

4.

I have developed a model that describes pale lager bright beer (Soproni Klasszikus from HEINEKEN Hungária, Hungary; alcohol content = 4.34 V/V %, final real extract content = 3.63 w/w %, bitterness = 12 IBU, turbidity at 20 °C = 0.48) dealcoholization by reverse osmosis at a temperature of 15 ± 1 °C with RO99 flat sheet polyester membrane (Alfa Laval, Sweden; $R_{NaCl} \geq 98$ %).

The model (objective function) (*Equation 28*) was the following:

$$J_{EtOH\,0} = 80.871 + 41.094 \times x_{TMP} \qquad \text{Equation 28}$$

where $J_{EtOH\,0}$ (g m^{-2} h^{-1}) is the initial ethanol flux and x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1. The range of validity: TMP = 10 – 30 bar, Q = 120 - 240 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(1;5) = 143.1$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$).

I have determined the effect size of the significant parameter and it was the following: TMP = 1.20. Model accuracy and determination coefficients of the effect size estimation were significant ($F(1;6) = 171.7$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$).

I have found the optimum (global maximum) of the objective function. The optimal value of the factor amounted to TMP = 30 bar. The predicted $J_{EtOH\,0}$ under the above condition was 121.965 g m^{-2} h^{-1}.

5.

I have developed a novel and efficient (average of flux recoveries > 97 %) membrane cleaning method for "2A. International Pale Lager" (BJCP) rough beer (alcohol content = 4.58 V/V %, final real extract content = 4.48 w/w %, bitterness = 18 IBU, turbidity at 20 °C = 2.50) membrane filtration at a temperature of 10 ± 1 °C with Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.5 μm pore size and 7 mm channel diameter).

The developed membrane cleaning method is detailed below. After the membrane filtration experiment, the used membrane was cleaned thoroughly by deionized water for 5 min at a temperature of 25 °C and then by 1 % (w/w) Sodium hydroxide (Reanal,

Hungary) for 60 min at a temperature of 60 °C. After cleaning by alkali, the membrane was rinsed again by deionized water for 10 min at a temperature of 25 °C followed by cleaning with 1 % (w/w) Hydrogen peroxide (Hungaro Chemicals, Hungary) for 60 min at a temperature of 25 °C. Finally, the membrane was cleaned thoroughly with deionized water for 10 min at a temperature of 25 °C. In all cases transmembrane pressure (TMP) and retentate flow rate (Q) were maintained at 0.2 bar and 50 L h^{-1}, respectively.

I have developed a novel and efficient (average of flux recoveries = 109 %) membrane cleaning method for pale lager bright beer (Soproni Klasszikus from HEINEKEN Hungária, Hungary; alcohol content = 4.34 V/V %, final real extract content = 3.63 w/w %, bitterness = 12 IBU, turbidity at 20 °C = 0.48) dealcoholization by reverse osmosis at a temperature of 15 ± 1 °C with RO99 flat sheet polyester membrane (Alfa Laval, Sweden; $R_{NaCl} \geq 98$ %).

The developed membrane cleaning method is detailed below. After the dealcoholization experiment, the used membrane was cleaned thoroughly by deionized water for 10 min at a temperature of 25 °C and then by 0.2 % (w/w) Sodium hydroxide (Reanal, Hungary) for 60 min at a temperature of 25 °C. After cleaning by alkali, the membrane was rinsed again by deionized water for 10 min at a temperature of 25 °C. In all cases transmembrane pressure (TMP) and retentate flow rate (Q) were maintained at 6 bar and 240 L h^{-1}, respectively.

7. SUMMARY

The scope of this thesis is studying the application of membrane separation processes (MSPs) in the brewing industry. Since MSPs are cleaner technology with efficient separation capability and generally mild operating conditions, and still an emerging technology in the brewing industry, the above-mentioned topic of the thesis is essential and relevant.

After the detailed literature review, four main research gaps were examined in this study: wort membrane filtration (WMF), beer membrane filtration (BMF) with static turbulence promoter (STP), beer membrane filtration (BMF) with silica gel (SG) and beer dealcoholization (BDA) by reverse osmosis (RO).

The objectives of the different investigations are the determination of the analytical parameters of feed and permeate samples, determination of the hydrodynamic parameters of the membrane separations for the responses of the experimental designs, to analyse the experimental designs, optimisation of the objective functions, to develop effective membrane cleaning methods for microfiltration (MF) and RO processes.

It has been proven that hot trub and cold trub can be completely removed by WMF and the changes in the analytical parameters are appropriate. Flux values of the WMF experiment were quite low.

According to the analysis of the experimental design of BMF with STP; STP, transmembrane pressure (TMP), retentate flow rate (Q), STP:TMP, STP:Q and TMP:Q had significant effect on steady-state fouling layer resistance ($R_{f\,ss}$) with the given parameters. TMP and STP:Q had interactive effects and STP, Q, STP:TMP and TMP:Q had antagonistic effects on $R_{f\,ss}$. Furthermore, the effect size of STP was the highest among the effect sizes of the significant main factors and interactions.

According to the analysis of the experimental design of BMF with SG, TMP and Q had significant effect, while silica gel concentration (SGC) had no significant effect on $R_{f\,ss}$ with the given parameters. TMP had interactive effect and Q had antagonistic effect on $R_{f\,ss}$. Furthermore, the effect size of TMP was higher than the effect size of Q.

According to the analysis of the experimental design of BDA by RO, TMP had significant effect, while retentate flow rate (Q) had no significant effect on initial ethanol flux ($J_{EtOH\,0}$) with the given parameters. Furthermore, TMP had interactive

effect on $J_{EtOH\ 0}$.

In addition, a novel and efficient membrane cleaning methods were developed and applied to recover the initial intrinsic resistances of MF and RO membranes.

In case of WMF, the changes in the analytical parameters could be improved by the optimisation of operating parameters (e.g. TMP and crossflow velocity) and application of permeate backflow techniques, enzymes, filtration aids, flow pulsation, gas sparging, static turbulence promoter (STP), Vibratory Shear Enhanced Process (VSEP) etc. Fluxes could be enhanced by the above-mentioned optimisations and applications.

In case of BMF with STP, the commercial breweries should focus on the optimisation of usage of STP, TMP and Q too. In this study, a turbulence promoter (STP) with a specific geometry was tested. However, in a later exercise, a wider range of operating parameters and several STPs with different geometries could be tested with the aid of lowering fouling layer resistances.

In case of BMF with SG, the commercial breweries should focus on the optimisation of TMP and Q, and silica gel (SG) free BMF can be performed. The SG free BMF is important because of environmental issues. However, filtration aids other than silica gel (SG) can be developed and tested to intensify BMF.

In case of BDA by RO, the commercial breweries should focus on the optimisation of transmembrane pressure (TMP). BDA by RO can be performed with lowest required retentate flow rate (Q), which results in lower energy consumption. The lower energy consumption is important because of environmental and economic issues. In a later exercise, beers with different alcohol and extract content could be dealcoholized by RO.

8. ÖSSZEFOGALÁS

E dolgozat témája a membránműveletek (MSPs) alkalmazásának a vizsgálata a söriparban. Mivel a membránműveletek egy környezetkímélő technológia, ami hatékony szétválasztási képeséggel és enyhe működési körülményekkel rendelkezik, és feltörekvő technológia a söriparban, az értekezés témája fontos és releváns.

A részletes szakirodalmi feltárás után, négy főbb hiányos témát vizsgáltam meg, ezek a következőek voltak: sörlé membránszűrése (WMF), sör membránszűrése (BMF) statikus keverő (STP) segítségével, sör membránszűrése (BMF) szilikagél (SG) segítségével, sör alkoholmentesítése (BDA) reverz ozmózis (RO) segítségével.

A céljaik a különböző kísérletsorozatoknak a következőek voltak: a betáplált anyagoknak és a permeátum minták analitikai tulajdonságainak a meghatározása, a különböző membránszeparációk hidrodinamikai paramétereinek a meghatározása a kísérlettervek válaszaihoz, kísérlettervek elemzése, célfüggvények optimalizálása, hatékony membránmosási eljárások kifejlesztése mikroszűréses (MF) és RO műveletnél.

Megállapítottam, hogy a forró seprő és a hideg seprő teljesen eltávolítható WMF-el és hogy az analitikai paraméterek változása megfelelő volt. A WMF kísérletnél a fluxusok igen alacsonyak voltak.

Az STP-vel történő BMF kísérlettervének az elemzése alapján elmondható, hogy az STP-nek, a transzmembránnyomáskülönbségnek (TMP), a retentátum térfogatáramának (Q), az STP:TMP kölcsönhatásnak, az STP:Q kölcsönhatásnak és a TMP:Q kölcsönhatásnak szignifikáns hatása volt az állandósult gélréteg ellenállásra ($R_{f\ ss}$) az adott működési paraméterek mellett. A TMP-nek és az STP:Q kölcsönhatásnak interaktív hatása volt az $R_{f\ ss}$-re, míg az STP-nek, a Q-nak, az STP:TMP és TMP:Q kölcsönhatásoknak antagonisztikus hatása volt az $R_{f\ ss}$-re. Továbbá az STP hatásnagysága volt a legnagyobb a szignifikáns fő faktorok és kölcsönhatások hatásnagyságai közül.

Az SG-vel történő BMF kísérlettervének az elemzése alapján elmondható, hogy a TMP-nek és a Q-nak szignifikáns hatása volt az $R_{f\ ss}$-re, míg a szilikagél koncentrációnak (SGC) nem volt szignifikáns hatása az $R_{f\ ss}$-re az adott működési paraméterek mellett. A TMP-nek interaktív hatása volt az $R_{f\ ss}$-re, míg a Q-nak

antagonisztikus hatása volt az $R_{f\ ss}$-re. Továbbá a TMP hatásnagysága nagyobb volt, mint a Q hatásnagysága.

Az RO-val történő BDA kísérlettervének az elemzése alapján elmondható, hogy a TMP-nek szignifikáns hatása volt a kezdeti etanol fluxusra ($J_{EtOH\ 0}$), míg a Q-nak nem volt szignifikáns hatása a $J_{EtOH\ 0}$-ra az adott működési paraméterek mellett. Továbbá a TMP-nek interaktív hatása volt a $J_{EtOH\ 0}$.

Ezeken túlmenően, kifejlesztettem olyan újszerű és hatékony membránmosási eljárásokat MF és RO membránokhoz, amelyekkel vissza lehet nyerni a membránok kezdeti ellenállásait.

A WMF esetében elmondható, hogy az analitikai paraméterek változását lehetne javítani a működési paraméterek optimalizálásával (pl. TMP, áramlási sebesség), vagy permeátum visszaáramoltatás módszerekkel, enzimek és szűrési segédanyagok használatával, az áramlás pulzáltatásával, gáz permetezéssel, statikus keverő (STP) alkalmazásával, vibrációval (VSEP) stb. A fluxusokat is lehetne növelni a fent említett optimalizálással és alkalmazásokkal.

Az STP-vel történő BMF esetében elmondható, hogy a kereskedelmi sörfőzdéknek hangsúlyt kell fektetniük a statikus keverő (STP) használatának és a transzmembránnyomáskülönbségnek (TMP), illetve a retentátum térfogatáramának (Q) optimalizálására. Ebben a tanulmányban egy adott geometriájú statikus keverő (STP) volt tesztelve. Azonban jövőbeli kísérleteknél a működési paraméterek széles tartományát és számos STP-ét lehetne vizsgálni, azzal a céllal, hogy csökkenjenek a gélréteg ellenállások.

Az SG-vel történő BMF esetében elmondható, hogy a kereskedelmi sörfőzdéknek hangsúlyt kell fektetniük a transzmembránnyomáskülönbségnek (TMP) és a retentátum térfogatáramának (Q) optimalizálására. Továbbá fontos megjegyezni, hogy a BMF az szilikagél (SG) nélkül megvalósítható. Az szilikagél (SG) mentes BMF környezetvédelmi okok miatt fontos. Azonban az SG-től különböző szűrési segédanyagokat lehetne kifejleszteni és tesztelni a BMF intenzifikálására.

Az RO-val történő BDA esetében elmondható, hogy kereskedelmi sörfőzdéknek a transzmembránnyomáskülönbség (TMP) optimalizálására kell hangsúlyt fektetniük. Az RO-val történő BDA a lehető legkisebb retentátum térfogatárammal (Q) megvalósítható, ami kisebb energiafelhasználást eredményez. A kisebb

energiafelhasználás környezetvédelmi és gazdasági okok miatt fontos. Jövőbeli kísérletek során különböző alkohol- és extrakttartalmú söröket lehetne alkoholmentesíteni RO-val.

APPENDICES

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M2. Additional appendices

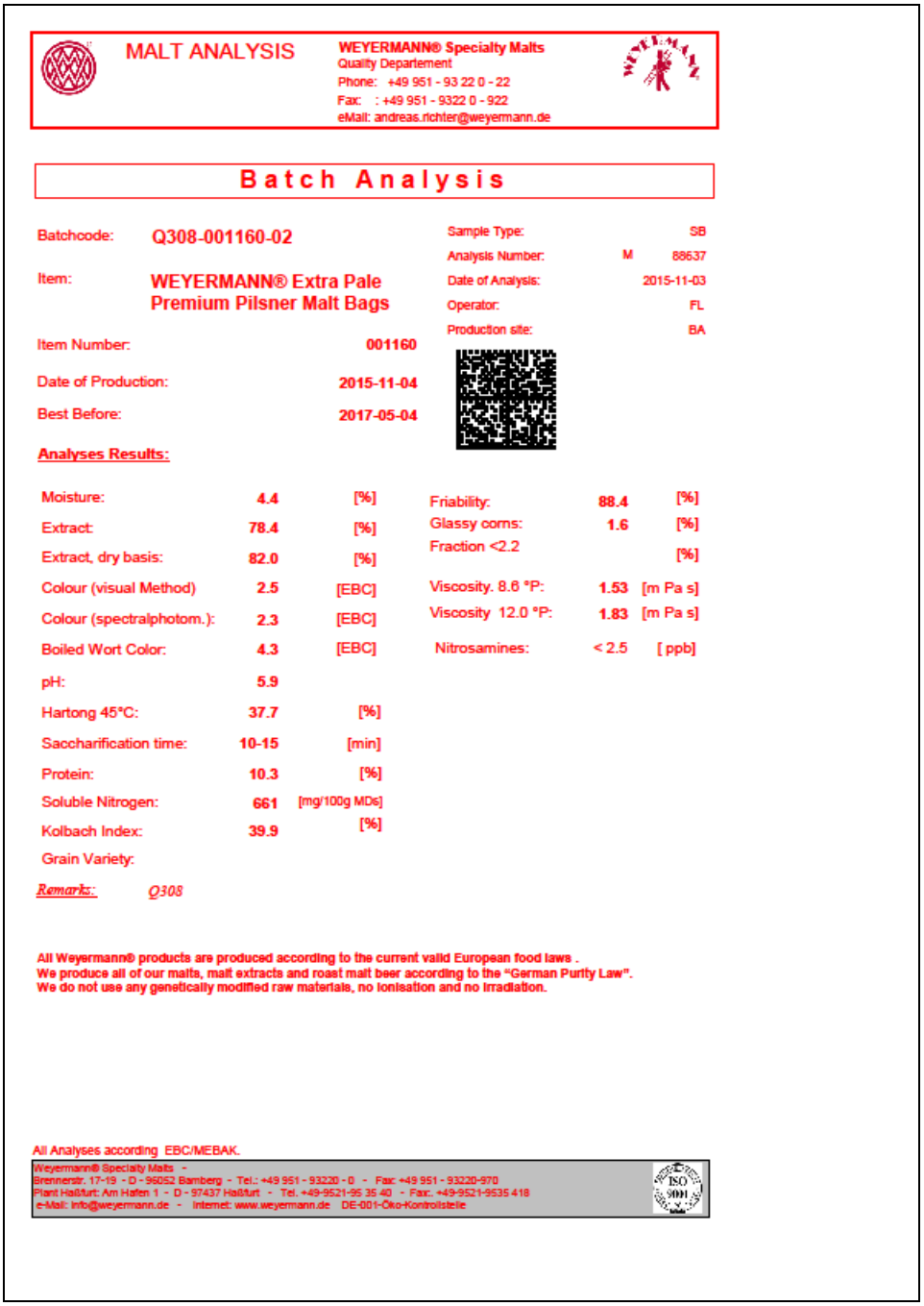
M2.1. Water analysis

Free active Cl (mg mL^{-1})	0.18
Cl^- (mg mL^{-1})	24
Total Fe (µg L^{-1})	11
Mn^{2+} (µg L^{-1})	2
NH_4^+ (mg mL^{-1})	<0.04
NO_2^- (mg mL^{-1})	<0.03
NO_3^- (mg mL^{-1})	9
Total hardness (°dH)	13
Electrical conductivity (µS cm^{-1})	465
pH	8

M2.2. Batch analysis of Pilsner Malt

Manufacturer	Boortmalt, Hungary
Batch number	05/2017
Moisture content (w/w %)	4.4
Extract content (w/w %)	83.1
Protein content (w/w %)	11.0
FAN content (mg L^{-1})	171
Colour (EBC)	3.8
Dynamic viscosity at 8.6% (w/w) at 20 °C (mPas)	1.51
Sorting >2.5 mm (%)	90.2
pH	6.00

M2.3. Batch analysis of Extra Pale Premium Pilsner Malt



MALT ANALYSIS

WEYERMANN® Specialty Malts
Quality Departement
Phone: +49 951 - 93 22 0 - 22
Fax: : +49 951 - 9322 0 - 922
eMail: andreas.richter@weyermann.de

Batch Analysis

Batchcode:	Q308-001160-02
Item:	WEYERMANN® Extra Pale Premium Pilsner Malt Bags
Item Number:	001160
Date of Production:	2015-11-04
Best Before:	2017-05-04

Sample Type:		SB
Analysis Number:	M	88637
Date of Analysis:		2015-11-03
Operator:		FL
Production site:		BA

Analyses Results:

Moisture:	4.4	[%]	Friability:	88.4	[%]
Extract:	78.4	[%]	Glassy corns:	1.6	[%]
Extract, dry basis:	82.0	[%]	Fraction <2.2		[%]
Colour (visual Method)	2.5	[EBC]	Viscosity. 8.6 °P:	1.53	[m Pa s]
Colour (spectralphotom.):	2.3	[EBC]	Viscosity 12.0 °P:	1.83	[m Pa s]
Boiled Wort Color:	4.3	[EBC]	Nitrosamines:	< 2.5	[ppb]
pH:	5.9				
Hartong 45°C:	37.7	[%]			
Saccharification time:	10-15	[min]			
Protein:	10.3	[%]			
Soluble Nitrogen:	661	[mg/100g MDs]			
Kolbach Index:	39.9	[%]			
Grain Variety:					

Remarks: *Q308*

All Weyermann® products are produced according to the current valid European food laws .
We produce all of our malts, malt extracts and roast malt beer according to the "German Purity Law".
We do not use any genetically modified raw materials, no ionisation and no irradiation.

All Analyses according EBC/MEBAK.

Weyermann® Specialty Malts -
Brennerstr. 17-19 - D - 96052 Bamberg - Tel.: +49 951 - 93220 - 0 - Fax: +49 951 - 93220-970
Plant Hallstadt: Am Hafen 1 - D - 97437 Haßfurt - Tel. +49-9521-95 35 40 - Fax: +49-9521-9535 418
e-Mail: info@weyermann.de - Internet: www.weyermann.de DE-001-Öko-Kontrollstelle

M2.4. Characteristics of Hallertauer Tradition hops

Hallertauer Tradition

Characteristics

Bitter substances

alpha-acids	4.0 – 7.0 % *
beta-acids	3.0 – 6.0 % *
cohumulone	24 – 30 % x
colupulone	41 – 49 % x

Polyphenols

total content	4.0 – 5.0 % *
xanthohumol	ca. 0.4 % *

Aroma susbstances

total oil	0.5 – 1.0 % *
myrcene	17 – 32 % x
linalool	0.7 – 1.2 % x
beta-caryophyllene	10 – 15 % x
aromadendrene	< 0.2 % x
humulene	35 – 50 % x
farnesene	< 1 % x
beta-selinene	0.5 – 1.0 % x
alpha-selinene	0.5 – 1.0 % x

(*)weight-% – (x)% relative – (+)ml/100g hops

Very fine aroma variety

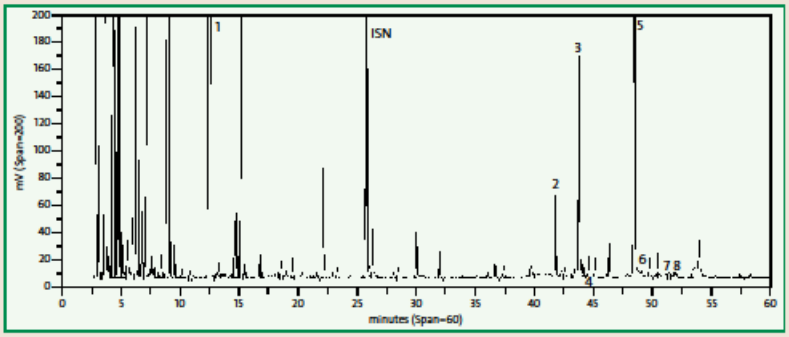
- average to higher bitter value
- very good aroma
- good storage stability

Resistance to disease:

• wilt	good
• downy mildew	good to very good
• powdery mildew	average

Ripening time	medium early
Storage stability	good
Average yield	1,850 kg/ha

Headspace Gaschromatogramme



1 = myrcene · ISN = internal standard · 2 = linalool · 3 = beta-caryophyllene · 4 = aromadendrene · 5 = humulene · 6 = farnesene
7 = beta-selinene · 8 = alpha-selinene

M2.5. Characteristics of Hallertauer Magnum hops

Hallertauer Magnum

Characteristics

Bitter substances

alpha-acids	11.0 – 16.0 % *
beta-acids	5.0 – 7.0 % *
cohumulone	21 – 29 % x
colupulone	38 – 48 % x

Polyphenols

total content	2.0 – 3.0 % *
xanthohumol	0.4 – 0.5 % *

Aroma substances

total oil	1.6 – 2.6 % *
myrcene	30 – 45 % x
linalool	0.2 – 0.7 % x
beta-caryophyllene	8 – 13 % x
aromadendrene	< 0.2 % x
humulene	30 – 45 % x
farnesene	< 1 % x
beta-selinene	0.3 – 0.8 % x
alpha-selinene	0.3 – 0.8 % x

[*]weight-% – [x]% relative – [x]ml/100g hops

High-alpha variety

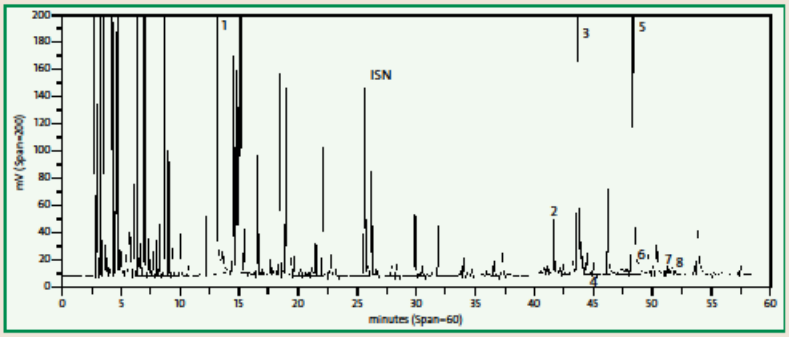
- very high bitter value
- average aroma
- good storage stability

Resistance to disease:

• wilt	good to very good
• downy mildew	good
• powdery mildew	very low

Ripening time	medium late to late
Storage stability	good
Average yield	2,000 kg/ha

Headspace Gaschromatogramme



1 = myrcene · ISN = internal standard · 2 = linalool · 3 = beta-caryophyllene · 4 = aromadendrene · 5 = humulene · 6 = farnesene
7 = beta-selinene · 8 = alpha-selinene

M2.6. Properties of the used yeast

Manufacturer	Cara Technology, United Kingdom
Species	*Saccharomyces pastorianus*
Total esters	low
Total superior alcohols	low
Apparent attenuation (%)	80 - 84
Flocculation	high
Sedimentation	fast
Ethanol tolerance (% (V/V))	9 - 11
Ideal fermentation temperature (°C)	12 - 15

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Finally, thank you for reading my dissertation.

SOLI DEO GLORIA!