SEDIMENTATION COMBINED b

Šereš Zita[1], Pajin Biljana[1], Šaranović Žana[2], Ljubica Dokić[1], Šoronja Simović Dragana[1], László Zsuzsanna[3], Jokić Aleksandar[1]

[1] Faculty of Technology, University of Novi Sad, Bul. cara Lazara 1, 21000 Novi Sad, Serbia,
e-mail: zitas@tf.uns.ac.rs
[2] AD Company Fidelinka, Fidelinka – Starch, Subotica, Serbia
[3] Faculty of Engineering, University of Szeged, Szeged, Hungary

ABSTRACT

This paper introduces the research about the possibility of applying microfiltration through a ceramic tubular membrane with 200 nm pore sizes to the wastewater obtained in the production process of wheat starch and vital wheat gluten. The consumption of process water would thus be reduced, the starch would be exploited better to a greater extent and the wastewater problem would consequently be solved. The second issue examined is the efficiency of the combination with sedimentation prior to the microfiltration as to reduce the occurrence of polarization layer on the membrane and to keep the wastewater permeate flux through the membrane constant as possible. The independent variables, the parameters that vary during the course of microfiltration, are the transmembrane pressure, flow rate and the wastewater sedimentation time prior to microfiltration while the dependent parameter that is constantly monitored during the process is the permeate flux. The maximum value of the permeate flux ($24 \ \mathrm{lm^{-2}h^{-1}}$) was achieved at three bars and at a flow rate of $150 \ \mathrm{lh^{-1}}$ after having initially allowed the wastewater to settle for four hours. Microfiltration reduces the wastewater dry matter from 11,000 mg/l to 4,000 mg/l and it also significantly decreases the values of the suspended matter, namely from 9,000mg/l to 300mg/l.

1. INTRODUCTION

In the industry of wheat processing for starch production water is used in the process of flour hydration, starch and gluten separation, starch refinement, for cooling the product of the starch hydrolysis, as well as for cleaning the used equipment and the work space. The greatest part of the wastewater that comes into existence during the process of starch production applying Martin process is obtained during the starch separation process. The obtained water settles, after which it is decanted and after a specific period of time it undergoes a purification procedure [1, 2, 3].

Water elimination and the separation of the product are the two basic processes affecting the quality and economy of the starch industry [1, 2, 3].

For a long time scientists have been investigating the application of membrane processes in wastewater treatment and in the filtering of the intermediate products in the wheat starch production [2, 3, 4, 5].

Governments of the developed countries have tried to increase the pressure on the largest waste producers in order to reduce the undesired environmental pollution. For example, the Commission of the European Communities introduced the Integral Pollution and Prevention Control Directive (Council Directive, 1996). The purpose of the directive is to achieve integrated prevention and the control of pollution arising from the particular activities listed in its Annex I. Among others, the directive defines the Best Available Techniques (BAT) as the most effective and advanced stage in the development of activities and their operation methods which indicate the practical suitability of particular techniques for providing in principle the basis for emission limit values designed to prevent and, where that is not practicable, generally to reduce emissions and the impact on the environment. The European IPPC Bureau published a relevant document (BREF)

where the BAT for the Food, Drink and Milk Industry are presented. To a larger extent, the general techniques commonly used in this industry are described. However, no BAT is described relating specially to sugar beet production. Detailed information can be found in chapter 4, under the title Techniques to Consider in the Determination of BAT. The chapter contains a list of various pollution prevention, waste minimization and energy efficiency techniques applied in industry that are described everywhere, e.g. in books, journals, leaflets, the internet, etc [6, 7].

Membrane separation is a filtration technique in which a solution is forced through a porous membrane. Some of the dissolved solids are held back because their molecular size is too large to allow them to pass through. The size range depends upon the type of membranes used. Fractionation of the feed stream occurs, with some molecules being concentrated on the upstream side of the membrane, which is known as the concentrate or retentate. The smaller molecules pass through the membrane into the permeate stream. The variety of membrane separation techniques can be characterized by their membrane pore size [8, 9, 10].

Membrane filtration is used in order to achieve an increase in the quality of the finished sweetening and syrup products [11]. It has also found its application in the process of water elimination, i.e. dehydration in the course of the production. It is used to isolate proteins from diluted process flows.

A key factor determining the performance of micro- and ultrafiltration membranes is concentration polarization, which causes membrane fouling due to deposition of retained colloidal and macromolecular material on the membrane surface. A number of reviews have described the process in detail [9, 10, 12, 13]. The pure water flux of ultrafiltration membranes is often very high.When membranes are used to separate macromolecular or colloidal solutions, the flux falls within seconds.This immediate drop in flux is caused by the formation of a gel layer of retained solutes on the membrane surface due to concentration polarization. This gel layer forms a secondary barrier to flow through the membrane. This first decline in flux is determined by the composition of the feed solution and its fluid hydrodynamics. Sometimes the resulting flux is constant for a prolonged period, and when the membrane is retested with pure water, its flux returns to the original value. However, a further slow decline in flux occurs over a period of hours to weeks, depending on the feed solution. Most of this second decrease in flux is caused by slow consolidation of the secondary layer formed by concentration polarization on the membrane surface. Formation of this consolidated gel layer, called membrane fouling, is difficult to control. Control techniques include regular membrane cleaning, back flushing, or using membranes with surface characteristics that minimize adhesion. Operation of the membrane at the lowest practical operating pressure also delays consolidation of the gel layer [10].

The aim of this research is to examine the possibility of applying microfiltration through a ceramic membrane to the wastewater obtained during the course of the production of wheat starch and vital wheat gluten. The product of filtration, permeate, would return into the production process in the form of purified water playing the role of the process water, while the retentate with a high starch content could return into the drying process in a centrifugal dry-kiln representing technical quality starch. In such a way the consumption of the process water would be reduced, starch would be exploited to a better and greater extent and the wastewater problem would be eventually solved.

2. MATERIALS AND METHODS

The above mentioned researches concerning wastewater purification involve the procedure of primary sedimentation and microfiltration on a single-channel ceramic membrane with 200 nm pore sizes. The dry matter content of the wastewater varies between 0.85 and 1.2%, the suspended matter is between 4000 and 8000 mg. Solid effluents contain mostly starch, proteins, ash and cellulose.

Experiments of microfiltration are conducted on the samples of decanted wastewater following two, three and four hours' sedimentation, respectively. The dry matter content of the wastewater decanted after 2 hours is 0,95%, after 3 hours is 0,85% and after 4 hours of sedimentation the dry matter content is 0,75%.

The independent variables, the parameters that vary during the course of microfiltration, are the transmembrane pressure, flow rate and the wastewater sedimentation time prior to microfiltration while the dependent parameter that is constantly monitored during the process is the permeate flux. After the filtration has been completed, physicochemical parameters of the permeate and the feed are determined: the dry matter, chemical oxygen demand and suspended matter.

The laboratory apparatus for microfiltration is shown in Figure 1. The feed is dosed out into the tank, the membrane is fixed into the module and, after the working parameters have been set, the filtration of wastewater is initiated. Over a determined period of time the permeate volume is recorded, based on which the permeate flux is calculated.
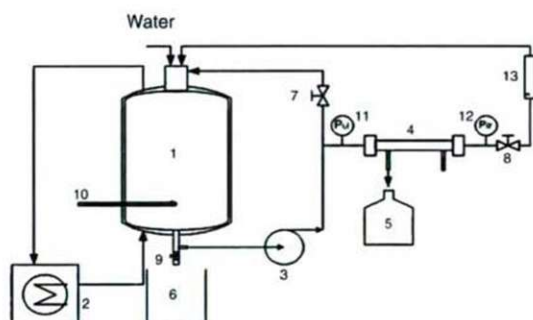

Figure 1. Apparatus for microfiltration (1 – tank, 2 – thermostat, 3 – pump, 4 – module with membrane, 5 –vessel for permeate, 6 – vessel for retentate, 7, 8 – pressure and flow regulation valves, 9 – valve for retentate release, 10 – thermometer, 11, 12 – manometers, 13 – Rotameter)

The central part of the apparatus is the module with membrane inside (Figure 1). In this research the ceramic membrane of manufacturer GEA (Germany) is used. The pore sizes of the membrane are 200 nm. The membrane is single-channel, 250 mm lenght, with inner diameter of 6,8 mm and outer diameter of 10 mm. The membrane is made of α-Al$_2$O$_3$ with TiO$_2$ layer. The active membrane surface equals 0.005m^2.

The microfiltration experiments were planned based on a full 2^3 factorial designed experiment [14]. In this experiment, the factors, i.e. the independent parameters are the following: p -transmembrane pressure, t -wastewater sedimentation time and Q- flow rate. Table 1 shows the values for the independent parameters which varied during the course of filtration.

Table 1. Varied values of independent variables

Independent variables	q [L/h]	t [h]	P [bar]
Varied values	50 / 150	2 / 4	1 / 3

q – flow rate [L/h]
t – decantation time of the wastewater [h]
P – transmembrane pressure [bar]

The dependent parameter monitored during the process of microfiltration is the permeate flux, which is calculated from the active membrane surface and permeate volume got in determined period of time:

$$J = \frac{V_p}{A_m \cdot t} \tag{1}$$

where J is permeate flux [L/m²h], V_p is permeate volume [L], A_m is membrane active surface [m²] and t is time [h].
The dry matter content is determined at the beginning and at the end of microfiltration.
The experimental data are processed with computer programmes Statistica for Windows 8.0 and Origin 6.1.

3 RESULTS AND DISCUSSION

Experiments are started with comparition of water flux and wastewater flux. On the Figure 2 the water flux and permeate of wastewater decanted after 4h, depending on the tranmembrane pressure at microfiltration on ceramic membrane with pore sizes of 200 nm at flow rate of 50 L/h and at room temperature can be seen. The water flux is the basic parameter for flux comparition with permete wastewater flux. It can be seen that the permeate flux of wastewater decanted after 4 h is 15 times reduced at transmembrane pressure of 1 bar compared to water flux (21 L/m²h). Such an effect can be ascribed to an increased adsorption and adhesion of particles and solutes on the membrane, which leads to an effective decrease in the diameter of the pores and a decline in the permeate flux. Such a change, i.e. flux decline is explained by the concentration polarization and the formation of a layer containing wastewater compounds on the membrane surface.
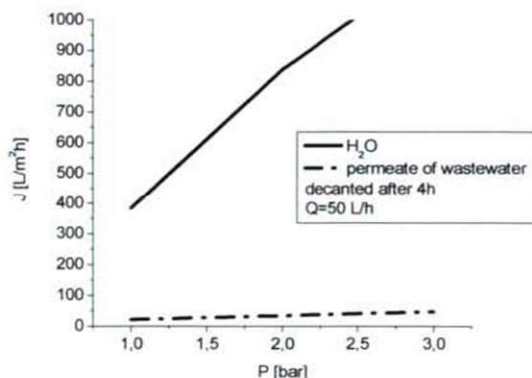

Figure 2. Water flux and wastewater permeate flux after 4 h of deposition, depending on the tranmembrane pressure at microfiltration on ceramic membrane with pore sizes of 200 nm at flow rate of 50 L/h and at room temperature

From the aspect of filtration, allowing sedimentation to take place first positively affects the microlevel since larger particles, mainly the starch ones, settle after which the dry matter content of wastewater is decreased. To examine how the sedimentation time of wastewater affects in microfiltration phase on the permeate flux, the Figure 3 has to be observed. It can be seen that with longer time of sedimentation due to the decrease of the dry matter in the wastewater from 0.95% (wastewater decanted after 2h of sedimentation) to 0.75% (wastewater decanted after 4h of sedimentation), the wastewater permeate flux increse. At transmembrane pressure of 1 bar the permeate flux of decanted wastewater after 2h sedimentation is 10 L/m²h, which is 2 times less then the permeate flux of decanted wastewater after 4h sedimentation. On the 3 bars that difference is more effective. At transmembrane pressure of 3 bars the permeate flux of decanted wastewater after 2h sedimentation is 17.5 L/m²h, which is 2.7 times less then the permeate flux of decanted wastewater after 4h sedimentation (46 L/m²h). Based on the above mentioned facts, the sedimentation phase before microfiltration has significant effect on the wastewater permeate flux.
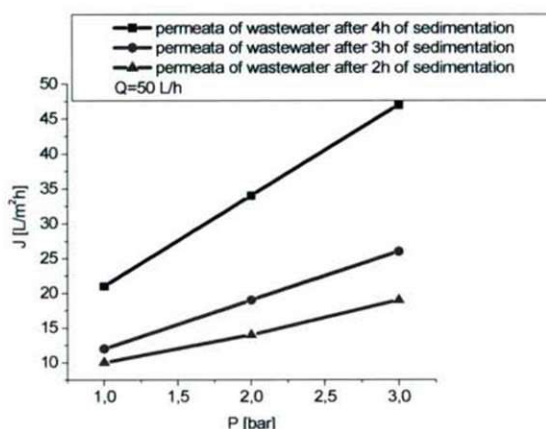

Figure 3. Wastewater permeate flux after different time of sedimentation, depending on the tranmembrane pressure at microfiltration on ceramic membrane with pore sizes of 200 nm at flow rate of 50 L/h and at room temperature

On the Figure 4 the wastewater permeate flux after 4 h of sedimentation are shown, depending on the tranmembrane pressure at microfiltration on ceramic membrane with pore sizes of 200 nm at different flow rates at room temperature. It can be seen that on the wastewater microfiltration flow rate has also great influence, which are more intensive on higher transmembrane pressures. On the transmembrane pressure of 1 bar if flow rate is held on 50 L/h, the permeate flux is 20 L/m²h, while increasing the flow rate on 150 L/h the pereate flux can be increased for 30%.
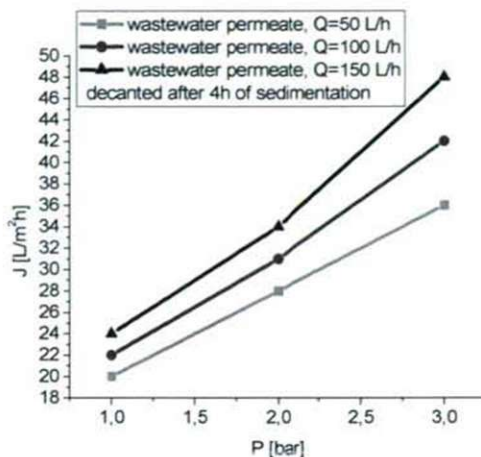
Figure 4.Wastewater permeate flux after 4 h of deposition, depending on the tranmembrane pressure at microfiltration on ceramic membrane with pore sizes of 200 nm at different flow rates at room temperature

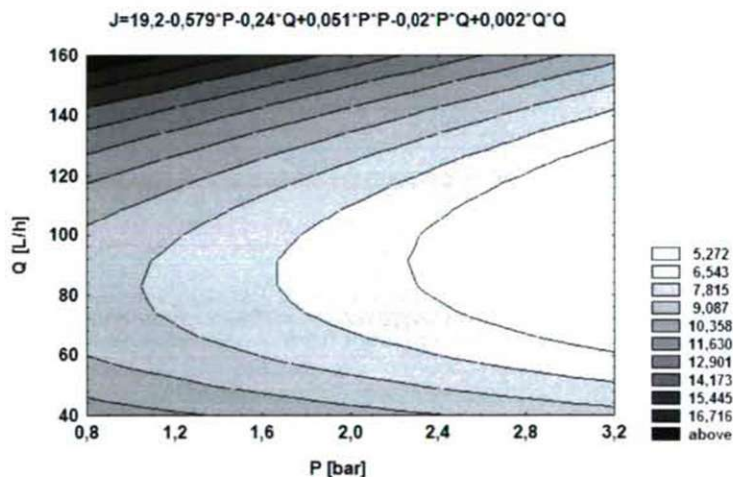$$J=19,2-0,579 \cdot P-0,24 \cdot Q+0,051 \cdot P \cdot P-0,02 \cdot P \cdot Q+0,002 \cdot Q \cdot Q$$

Figure 5. Wastewater permeate flux dependence on the tranmembrane pressure and on the flow rate at microfiltration on ceramic membrane with pore sizes of 200 nm at room temperature

On the basis of the obtained experimental values graphs depicting two dependent variables are drawn. Based on these values and using the programme *Statistica 8.0* a regression equation was obtained, which best describes the function of the flux dependency upon the transmembrane pressure and flow rate (Figure 5). Figure shows that the highest flux values caan be achieved (over 15 L/ m²h) when flow rate is held over 140 L/h and the transmembrane pressure is held under 2 bars.

Using the sedimentation in combuination with microfiltration with ceramic membrane of 200 nm pore sizes the dry matter of the wastewater decreses from 11000 mg/l to 4000 mg/l, which is 64% decrease. The suspended solids are also decresed. They decrease from 9000 mg/l to 300 mg/l.

4. CONCLUSIONS

On grounds of the research into the effects of the conditions of microfiltration of the wastewater obtained during the technological process of wheat starch processing the following conclusions have been reached:

- The permeate flux of wastewater decanted after 4 h is 15 times reduced at transmembrane pressure of 1 bar compared to water flux (21 L/m²h). Such an effect can be ascribed to an increased adsorption and adhesion of particles and solutes on the membrane, which leads to an effective decrease in the diameter of the pores and a decline in the permeate flux. Such a change, i.e. flux decline is explained by the concentration polarization and the formation of a layer containing wastewater compounds on the membrane surface.

- The initial sedimentation of wastewater significantly aproove the microfiltration of wastewater. At transmembrane pressure of 3 bars the permeate flux of decanted wastewater after 2h sedimentation is 17.5 L/m²h, which is 2.7 times less then the permeate flux of decanted wastewater after 4h sedimentation (46 L/m²h).

- From the beginning of microfiltration, the maximum value of the permeate flux (24 lm⁻²h⁻¹) was achieved at the pressure under 2 bars and the flow rate over 140 lh⁻¹ after having initially allowed the wastewater to settle for few hours.

- By using a 200nm membrane the wastewater dry matter is decreased from 11,000 mg/l na 4,000 mg/l, which is a reduction of about 60%.

- The wastewater suspended matter also declines significantly from 9,000mg/l to 300mg/l.

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