Doctoral (Ph. D.) Theses

Application of efficiency enhancer processes in utilisation and treatment of wastes and by-products of the agri-food industry

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1. INTRODUCTION

The continuous growth of the Earth's population goes together with the increased agricultural and food production, as well as with extending amount of energy that is sufficient for the proper functioning of infrastructure and transport.

The advised water usage and the protection of wetlands are very important, that is why there are increasingly strict regulations on wastewaters. Food industry produces large amounts of wastewaters with high level of organic matters. The organic matter of these wastewaters and sludges can be utilised for example biogas production or soil fertilising after composting. Most of the organic matters in sludges are in non-soluble from. Different kinds of pre-treatment types are capable to take a part of these non-soluble compounds into soluble form

Thermal treatments and thermal treatments combined with chemical (alkaline) treatments can increase the biodegradability of wastewaters and waste activated sludges. The microwave (MW) treatment types are proven in several studies to be effective alternatives over classical heating methods. The continuously flow MW treatments are able to model a real industrial treatment process better. In my work I used continuously flow MW treatments combined with alkaline treatments, and I investigated the effects of NaOH-dosage and the amount of applied specific microwave energy in respect of the change of solubility and the biodegradability. My other goal was to find a simple and effective in-line or on-line measurement method what can be applied to estimate the efficiency of the treatments and allows immediate change of the treatment parameters.

The other scope of my work was the bioethanol production from lignocellulosic biomass which is produced in large amounts in the agri-food industry. Production of bioethanol needs a preliminary hydrolysis step. The most environmentally friendly hydrolysis is carried out with enzymes.

Enzymes consumed for hydrolysis have a noticeable cost share of the whole process, so the reuse of the enzymes makes the whole process more cost-efficient., and in addition it is suitable to reduce the chemical and energy requirements of enzyme production, which can also improve the environmental impact of lignocellulose-based bioethanol production.

Removal of enzymes from fermentation broths (i.e., an appropriate downstream process integrated into upstream processes) can also increase the efficiency of further processing operations after fermentation. Membrane separation is a potential process that can be used in

industrial scale with low chemical and energy consumption, but the formation of fouling can be a significant limiting factor, which can radically reduce the permeate flux.
In my work, I used lifetime and performance-enhancing processes (cross-flow and sonication) to make membrane separation more efficient in the ultrafiltration of fermentation broths from lignocellulose hydrolysis and investigated the effect of changing the operating parameters of the methods on the filtration characteristics.

2. OBJECTIVES

The aim of my doctoral dissertation is to investigate the efficiency-enhancing technological processes that can be used in the utilization of biomass-based wastes and by-products.
In my experimental work, I focused on two significant areas

- for the treatment and utilization of food industry waste activated sludge, during which I mainly used the microwave technique and
- for the recovery of enzymes used in the hydrolysis of lignocellulose, where I analysed the effect of the application of ultrasonic force field and crossflow.

I approached my goals in the following steps:

- Investigation of the efficiency of combined treatments (microwave and NaOH dosage) in terms of biological degradability of the organic matter content of waste activated sludge.
- Analysis of the effect of microwave-alkaline treatments on the solubility of waste activated sludge.
- Development and validation of a quick method to monitor the effectiveness of the combined treatments.
- Effect of the applied crossflow speed on the efficiency of separation by ultrafiltration of fermentation broths from lignocellulose hydrolysis.
- Investigation of the usability of ultrasound for increasing of the permeate flux duringultrafiltration.
- Modelling of the fouling mechanisms in filtering real fermentation broths by different models.
- Analysis of the effect of crossflow and ultrasound on the characteristics of fouling.

3. EXPERIMENTAL

In my work I used two types of raw materials: food industry-originated waste activated sludge and fermentation broths from the hydrolysis of lignocellulosic biomass.

Waste activated sludge comes from the wastewater treatment system of a meat processing plant. Sampling was performed after the flocculation step carried out by ferric chloride used in the local wastewater treatment technology. The sludge had very high biochemical oxygen demand (724 ± 51 mgdm-3) and chemical oxygen demand (28300 ± 690 mgdm-3).

The microwave treatments were performed in a continuous flow microwave treatment equipment developed at the Department of Process Engineering of University of Szeged. The continuous flow was provided by a peristaltic pump, with the help of which the flow rate could be adjusted between 6 and 35 dm3h-1. In the microwave equipment, the materials flow in a PTFE tubular spiral. The microwave field is generated by a specially designed 2450 MHz magnetron. The power of the magnetron can be steplessly adjusted between 100 and 850 W, and power being set is delivered in continuous radiation mode.

The alkaline part of the treatment was provided by adding Sodium Hydroxide (NaOH) to the sludge before the microwave treatments. To characterize the NaOH concentration, I used the amount of the weight of NaOH and the dry matter content of the treated material [gNaOH / grm].

To determine and analyze the treatment parameters, I used a central composite factor design, and then, based on the results, I performed analysis of variance and response surface modelling using Statistica software package.

The total chemical oxygen demand (TCOD) was determined by colorimetric method after the dilution of the total sludge matrix. The soluble chemical oxygen demand (SCOD), i.e. the soluble phase of the sludge, was measured from the supernatant after the centrifugation of the sludge for 20 minutes at 10000 rpm with Hanna test cuvettes according to ISO 6060: 1989 by photometric method.

The biochemical oxygen demand (BOD) of the samples was measured using a respirometric measurement system (BOD Oxidirect, Lovibond, Germany). Samples were stored in a 20-degree air thermostat during the 5-day (BOD5) measurement.

The dielectric constant (ε') was determined using a flow-through dielectrometer specially designed for continuous flow microwave treatment. The measuring instrument has its own magnetron, the operating frequency of which is the same as that of the control device,

2450MHz. NRV-Z type power sensors are connected to an NRVD power meter (Rohde & Schwarz, Germany) in the measuring line of the device.

During membrane separation experiments, I used the fermentation broths produced during the enzymatic hydrolysis of Cobex Feeds 12/30 grinded corn cob produced by Cobex Hungária Kft. In one part of the experiments I used fermentation broths prepared with a mixture of cellulase (Trichoderma reesei) and cellobiase (Aspergillus niger) enzymes, while in the other part I performed my experiments with fermentation broths from hydrolysis with β-1,4-xylanase (Aspergillus oryzae).

The fermentation broths were centrifuged at 5400 rpm for 30 min, and the supernatant was used in my further work. The purified fermentation broths had a dry matter content of 1.9 ± 0.07 w/w%.

During the membrane separation experiments, I used a batch laboratory filter cell (Millipore) with a membrane disc with a diameter of 76 mm. Stirring can also be performed in the filter cell with the help of a magnetic stirrer. An ultrasonic generator can be fitted to the filter cell using a special closure. During the ultrasound-assisted membrane separation experiments, I used a Hielscher UP100H ultrasound generator with a frequency of 30 kHz. During filtration, the transmembrane pressure (TMP) was 0.3 MPa. The amount of permeate was recorded by measuring the mass of permeate leaving the filter cell using a balance connected to a computer, and it was also possible to record the temperature of the system.

For the membrane separation I used a Sterlich UF PW polyethersulfone membrane with a cut-off value of 10 kDa.

4. SUMMARY OF NEW SCIENTIFIC RESULTS

T1) It was proven that the disintegration of the sludge can be improved by the combined continuous flow microwave-alkali treatment of the meat industry waste activated sludge, but this improvement cannot be increased above 45%.

As a result of combined microwave and alkaline pre-treatments the disintegration of meat industry waste activated sludge can be increased by 45.1% by increasing the specific microwave energy and alkaline dosage. At medium and high (0.38 and 0.6 g/grm) NaOH dosage, an average disintegration degree of 42.35% can be achieved using a medium specific microwave energy level (5941 Jg-1), that could be increased at high specific microwave energy level (9661 Jg-1) to an average value of 45.05%, but this increase is no longer significant at a 95% significance level compared to the results determined at medium energy level.

T2) It was proven that the biodegradation index characterizing aerobic degradability shows a maximum when examined as a function of the operating parameters of continuous microwave combined alkaline pre-treatments. However, the change in the available aerobic biodegradation index lags behind the increase in the solubility of organic matter of sludge.

The biodegradation index varied between 13 and 27%, while the degree of disintegration ranged from 19.8 to 45.1%.

The highest biodegradation value was obtained at medium specific microwave energy level (5941 Jg-1) combined with high (0.6 g/grm) NaOH dosage, however it did not differ from results obtained at medium (0.38 g/grm) alkaline dosage level, at the significance level of 95%. In both cases, at high (9661 Jg-1) specific energy level, a decrease in the degree of biodegradation was already observed.

T3) It was proven that both the solubility and the biodegradability of the microwave-alkali combined pre-treatments of meat industry waste activated sludge in the continuous flow treatment system are more affected by the parameters of the applied specific microwave energy transfer than the parameters of the alkaline

In the case of disintegration degree of sludge solubility and aerobic biodegradability index determined as a function of combined treatments with a specific microwave energy of 2221 to 9661 Jg-1 and NaOH dosage of 0.15 to 0.6 g/grm using a central composite factor design I performed the analysis of variance in the case of values, which showed the highest F values at the linear term of the microwave pre-treatments, thus proving the highest efficacy

T4) It was proven that there is a correlation between the degree of disintegration and the change of the dielectric constant in the case of the combined alkaline-microwave treatment of meat industry waste activated sludge, the value of the dielectric constant is suitable for characterizing the solubility of the sludge, i.e. for predicting the treatment efficiency.

The disintegration degree of sludge obtained during the combined microwave-alkaline pre-treatments and the dielectric constant values measured at 2450 MHz were compared by linear regression, and I found that there is a close relationship between them, which is also confirmed by the high coefficient of determination ($R2 = 0.9517$).

T5) It was proven that during ultrasonic filtration of fermentation broths containing a mixture of cellulase and cellobiase enzymes and xylanase enzyme, the sonication can increase the permeate flux. However, the flux-increasing effect cannot be detected if the crossflow in the system exists simultaneously.

At ultrafiltration of the centrifugally purified fermentation broths from the hydrolysis of both maize cob flakes studied by me, higher flux values were achieved in the ultrasound-assisted separations at a frequency of 30 kHz compared to the control. However, when stirring was used, the application of sonication did not increase the permeate flux.

T6) It was proven that the most important enzymes in the breakdown of lignocellulose structures, cellulase, cellobiase and xylanase, can be recovered by ultrafiltration, they do not lose their activity during the processes, even under the sonication used together with the separation.

Enzyme activity tests following the separations confirmed that the recovered enzymes are able to hydrolyse new substrates, their activity is the same as that of the unexposed enzymes in the separation and sonication processes performed at 30 kHz with average specific power of 4.08·10-5 and 8.16·10-5 W/cm3, so the separated enzymes can be used in another hydrolysis process.

T7) It was proven that at separation of real fermentation broths from the hydrolysis of grinded corn cob containing cellulase and cellobiase enzymes, the cake filtration model is suitable to describe the fouling mechanism in case of ultrafiltration, andduring cross-flow or sonication connected to it, but when both of the mentioned methods are applied, the permeate flux varies according to the dynamics described by the intermediate blocking model.

The Hermia fouling models used to describe the ultrafiltration process showed that the best-fitting fouling model is the cake layer filtration model. This model showed the best fit both without stirring and sonication and when using ultrasound only or stirring only. However, when sonication and mixing were used together, the intermediate blocking model showed the best fit.

5. PUBLICATIONS RELATED TO THE PRESENT THESIS

1. Microwave–alkaline treatment for enhanced disintegration and biodegradability of meat processing sludge
   Balázs, Lemmer; Petra, Veszelovszki-Kovács; Cecilia, Hodúr; Sándor, Beszédes
   Desalination And Water Treatment (2017) 98: 130-136. (IF: 1,38, Q2)

2. The effect of sonication and stirring on ultrafiltration of fermentation broth
   Lemmer, Balázs; Jákói, Zoltán; Gulyás, Nikolett; Kertész, Szabolcs; Beszédes, Sándor; László, Zsuzsanna; Hodúr, Cecilia
   Environment Protection Engineering (2020) 46(1). 49-62. (IF:0,812, Q3)

3. Sonicated Membrane Separation
   Balázs, Lemmer; Szabolcs, Kertész; Gábor, Keszthelyi-Szabó; Kerime, Özel; Cecilia, Hodúr
   Progress In Agricultural Engineering Sciences (2018)14: 89-99 (Q3)

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