Separation of Solid Residues after SSF and SHF in Fuel Ethanol Production from Spruce

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Abstract

Production of fuel grade ethanol from various lignocellulosic materials using enzymatic hydrolysis has been under development for some time now. Some of the individual steps such as pretreatment, hydrolysis and fermentation have been improved to a great extent. Much effort has also been put into making the process as such more efficient. This includes studies on the effect of recirculation of process streams to minimise fresh water consumption and effluent streams.

However, one major technical obstacle is still to be met, especially in the SSF (simultaneous saccharification and fermentation) process: the separation of the solid residue from the liquid. This residue, which consists mainly of lignin and non-hydrolysed cellulose, tends to be extremely difficult to separate into a solid and a liquid fraction, since it is very sticky. In SSF yeast cells are also present, which complicates the separation.

Common separation methods of these residues often result in a slow filtration rate. When a filter press is utilised an extremely compact filter cake is rapidly formed. Employment of a decanter centrifuge, on the other hand, is rapid but yields a solid phase having a low dry matter content. In this work, a rather new technology has also been studied. A vibrating membrane separation unit (VSEP) was used to separate material from SSF or SHF. Due to high shear forces the membrane surface is not easily fouled, but the liquid flux through the membrane is maintained at a high level. Results from this study are presented.

1. Introduction

The search for alternatives to gasoline is an issue that is being paid more and more attention to. The depletion of oil and the increasing fuel prices, as well as environmental concerns, have increased the interest in fuels derived from various biomass resources. Ethanol, which can be produced from many types of cellulose materials, has been proposed as one of the alternatives (1,2,3,4). It can be manufactured from a whole array of natural materials containing cellulose or starch. The choice of substrate can thus be based on local conditions.

Softwood, which is an abundant material in Sweden, can be used for ethanol production. A couple of alternative process concepts can be designed, of which one is the utilisation of enzymes to hydrolyse the cellulose to monomeric sugars. Since lignocellulosic materials consist of a complex matrix of cellulose, hemicellulose and lignin, a variety of soluble substances and a solid residue will be the result from enzymatic hydrolysis. The solution contains several sugar types that are easily fermented to ethanol using, for example, ordinary baker's yeast.

In Figure 1, a simplified process description is shown. Prior to enzymatic hydrolysis the lignocellulosic material needs to be pretreated at elevated temperatures. This produces a material, which is more accessible to enzymatic hydrolysis, due to removal of most of the hemicellulose fraction from the fibre structure. The softened wood may then be hydrolysed and fermented, either simultaneously (SSF) or in two separate process steps. Downstream from the hydrolysis/fermentation step, separation of the solid residue from the ethanol-containing liquid takes place. The separation is desirable for several reasons: in a large-scale recirculation of process streams must occur to minimise water intake and waste streams, but also the energy-rich lignin-containing residue must be taken care of.

This separation is, however, not an easy task since the filtering properties of the residue are not very favourable. The hydrolysed material is extremely sticky and hard to filter using, for example, a filter press. If the SSF method is employed the situation is also complicated by the presence of yeast cells. Filter rates tend to be very low, which will enforce utilisation of large filter areas to get reasonable filtering rates.

Another possibility is the employment of decanter centrifuges to ensure a rapid dewatering process. This type of equipment is generally very robust and easy to operate and make large throughputs possible. A disadvantage is the relatively low solids content that results and the loss of solid material to the liquid phase, due to unfavourable separation characteristics.

In this study a type of a vibrating membrane unit, VSEP, has been used to separate the lignin-containing residue from the liquid resulting from SSF. Some results are presented and compared with results from a study using a decanter centrifuge.

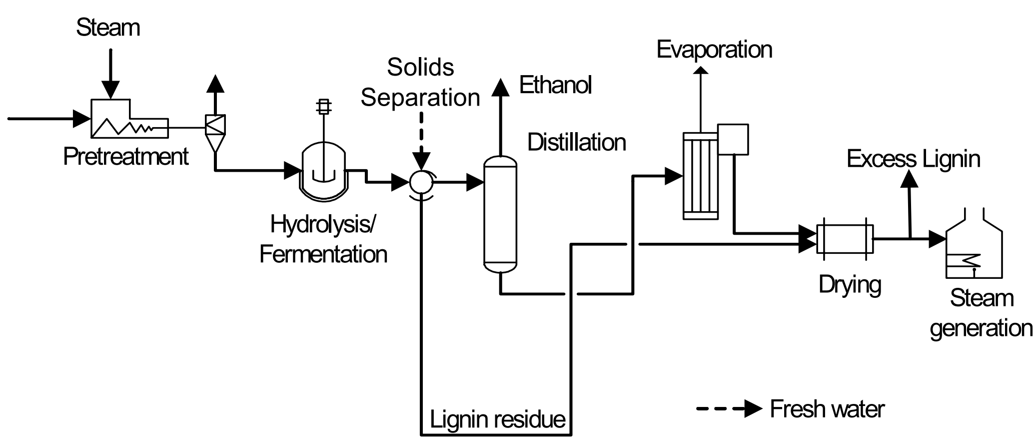

Figure 1 A simplified description of the wood-to-ethanol process

2. Materials and Methods

Fresh, chipped spruce, *Picea abies*, free from bark, was kindly provided by a sawmill (Harry Nilsson, Hästveda, Sweden). The wood chips were rechipped and fractionated further to a chip size between 2 and 10 mm. The material was impregnated with SO_2 (3% w/w water content in the chips). The impregnated material was then pretreated using steam at 215°C for 5 minutes.

After pretreatment, the slurry was collected and transferred to a 100-litre fermenter vessel (Bioengineering AG, Switzerland). The slurry was diluted to a fibre concentration of 5%. The pH was adjusted to 5.0 using solid $Ca(OH)_2$ and then maintained using a 30% (w/w) NaOH solution. Compressed baker's yeast (Jästbolaget, Sweden) was added to a concentration of 5 g/L dry matter (DM). Enzymes, cellulases and β-glucosidase (Novozymes, Denmark) were also added at concentrations of 0.23 g/g fibre and 0.05 g/g fibre, respectively, equivalent to 15 FPU/g DM and 23 IU/g DM, respectively. Simultaneous saccharification and fermentation (SSF) was performed on the pretreated material at 37°C for 72h. After finishing SSF, the fermented slurry was stored in a cold room at 4°C until needed or until new material was produced.

Separation trials were performed using mainly two types of separation equipment:

- Decanter centrifugation
- Vibratory Shear Enhanced Processing (VSEP), which is a type of membrane separation unit

2.1 Separation using decanter centrifugation

A decanter centrifuge, Alfa Laval/Sharples P600 was used to dewater the SSF residue. The slurry was continuously pumped at feed rates between 25 and 800 kg/h to the centrifuge. The separated material was analysed regarding dry matter content and separation efficiency (defined as DM in concentrated material/DM in feed). The rotation speed of the centrifuge bowl was 5300 rpm.

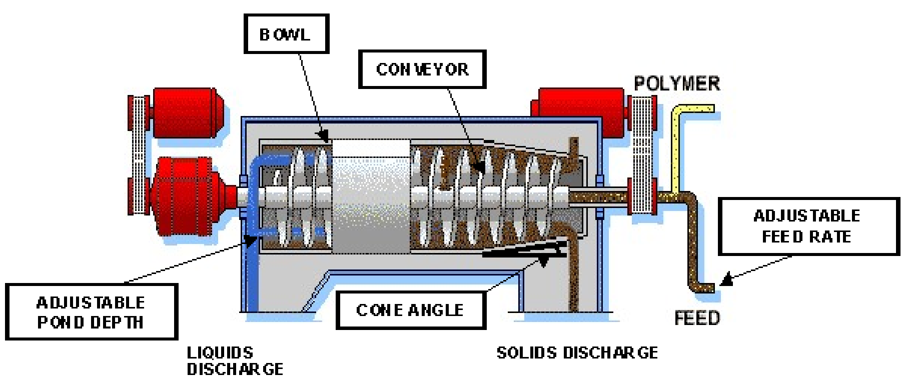

Figure 2 Decanter centrifuge

The construction makes it possible to vary the differential speed between the bowl and the conveyor, which changes the dewatering characteristics. In some experiments, the addition of a cationic polymer (Cytec Superfloc C-494) was added at a concentration of 100 ppm.

2.2 Separation using a VSEP unit.

A membrane unit of VSEP type, called a P-unit, with 0.2 µm Teflon membranes (Nordcap, Gothenburg, Sweden) consisting of 38 membranes and a total filtering area of 1.57 m^2 was also used for dewatering experiments. The main feature of a VSEP equipment, which differ from other membrane-type units, is the strong vibration applied to the membrane stack. The vibrational energy creates high shear forces, which decrease fouling and alleviates the separation and the transport of liquid through the membranes.

In Figure 3, the working principle of the VSEP unit is described. When operating, the on/off valve is set to be closed for a certain time, while permeate is free to pass through the membrane stack. Since feed is continuously pumped to the unit, solid material is accumulated inside the stack during the preset time, after which the valve opens and a purge flow is applied to let the concentrated material out.

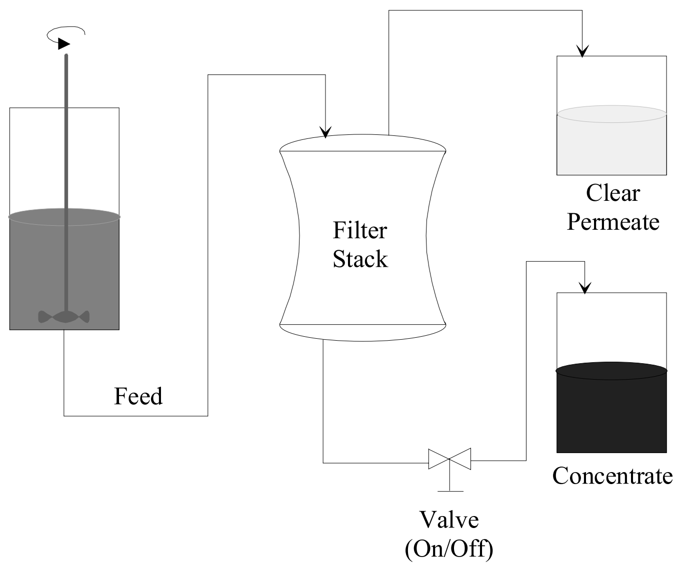

Figure 3. VSEP set-up

Runs were carried out at room temperature, or slightly above (≈25°C). Samples were collected when a stable permeate flow was maintained. The time between purging cycles of the unit was varied between 1 and 9 minutes. The total dry matter contents (DM), as well as the fibre contents, in the concentrate were measured for different settings of the on/off time. The DM was determined by drying at 105°C for at least 2h. The fibre content was determined by washing a small amount of the slurry, to remove soluble substances, and then dried at 105°C for at least 2h. This procedure was carried out for all streams.

A Labox 25 Mini Filter Press (Larox Flowsys OY, Finland) was used to estimate the filter rate employing a filter press equipment. A volume of 150 ml of slurry containing 3% fibrous material was filtered and the filter rate was measured at an applied pressure of 4 bar.

3 Results and Discussion

Some initial trials using a Labox 25 were performed to estimate the filtering properties of the slurry. In Figure 4 the time course for a filtration run at 25°C is presented. The average flux during the linear part of the process was approximately 0.6 l/(min m^2).

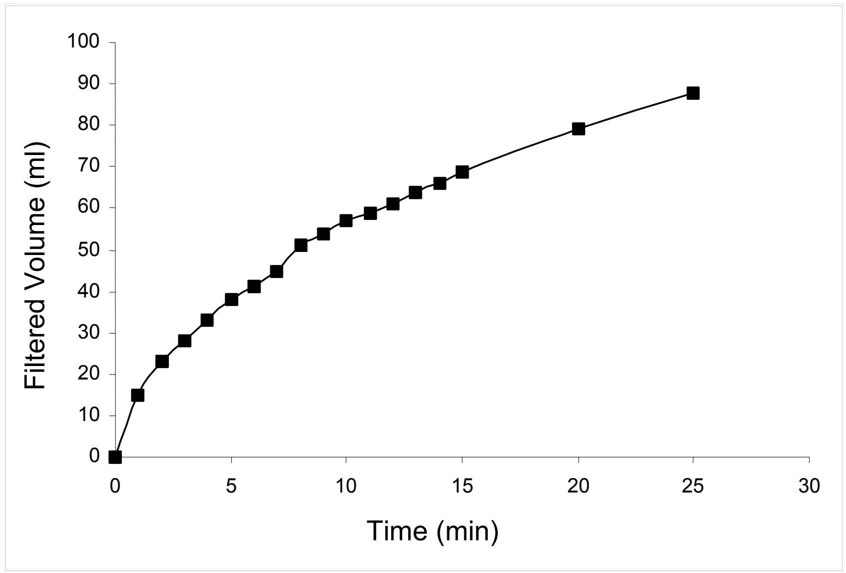

Figure 4 Filtration using a Labox 25 Mini Press Filter

Separation in a decanter centrifuge is rapid and allows for high throughputs, while at the same time the construction of the equipment is very robust. It is easy to clean and maintain and causes no problems if different slurries are to be separated in a short period of time.

The recovery of fibrous material in the concentrated slurry is not very much influenced by the feed rate. The recovery of fibrous material, based on the content in the feed stream, was about 70% for the whole feed-rate range from 25 to 800 kg/h. This means that the clarified liquid contains a considerable amount of particles, which will be brought on to other process steps, such as the distillation unit, and may cause problems, e.g. fouling of heating areas.

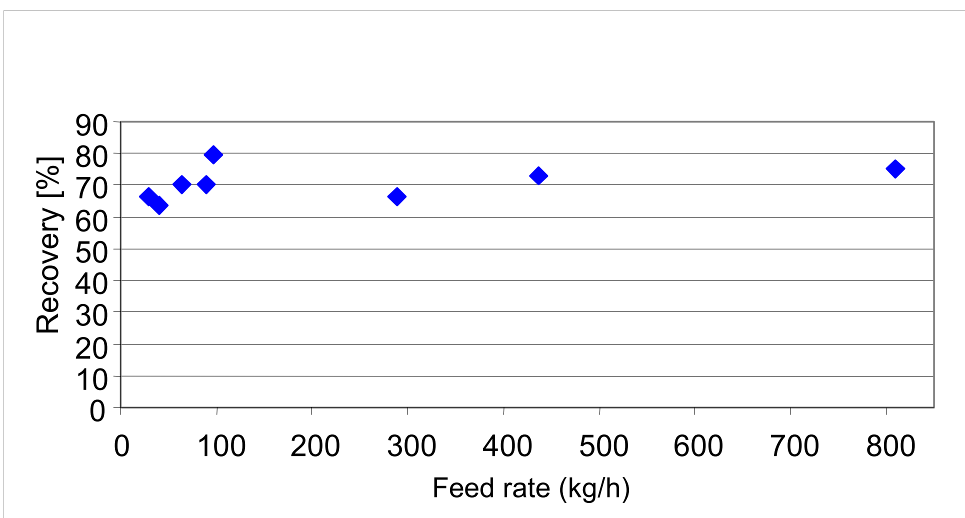

Figure 5 Influence of feed rate on recovery

In Figure 6, the dry matter contents of the concentrated slurry at various feed rates are shown. At low feed rates the DM of the concentrate is approximately 18%, while already at moderate flow rates the DM decreases rapidly. At the highest feed rates almost no concentration takes place. The settling velocity of the residual wood fibres is obviously too low to make a proper separation possible.

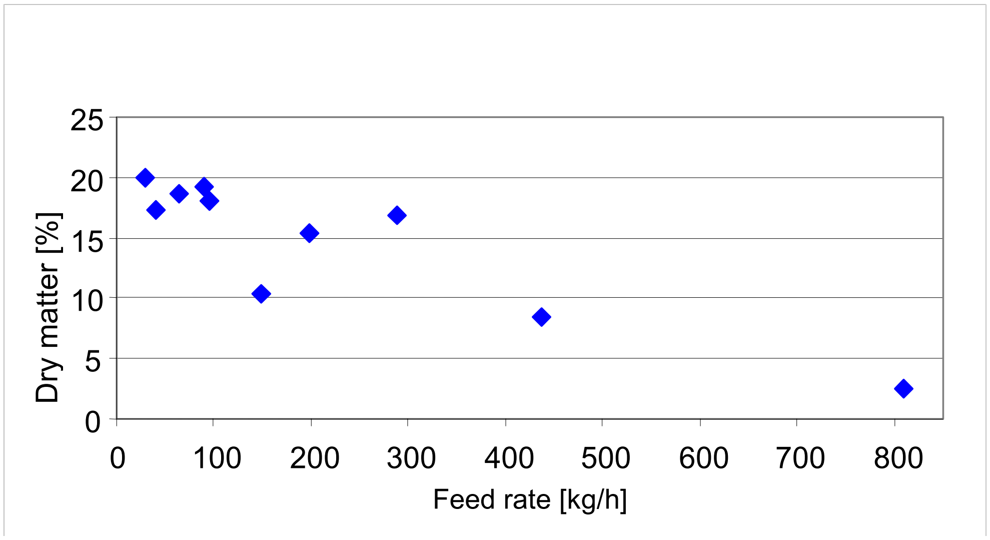

Figure 6 Influence of feed rate on dry matter content

Addition of flocculating agents, such as anionic or cationic detergents, has in many instances a huge impact on the agglomeration of particles. The formation of floccules makes separation a much easier and rapid procedure. The addition of a cationic flocculant improves the recovery of fibrous material in the concentrated stream as is shown in Figure 7.

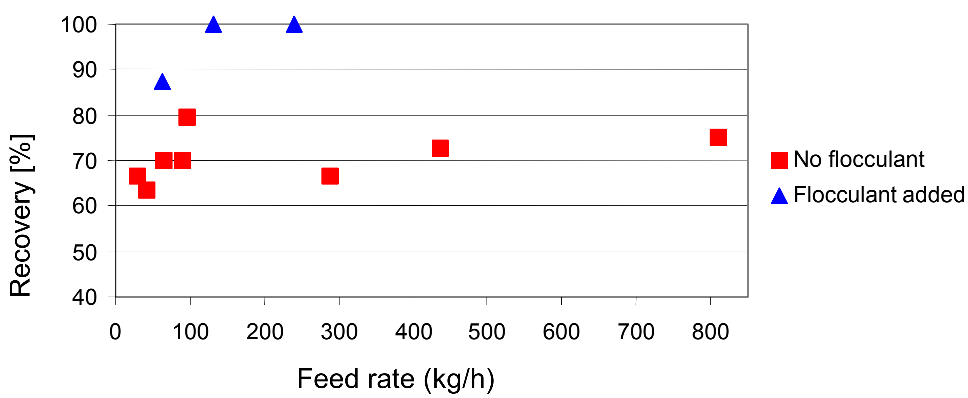

Figure 7 Influence of addition of flocculant on recovery

However, the dry matter content of the concentrated slurry changed in a way that was undesired. The dry matter content decreased when the flocculant was added. The reason for the decrease is the flocculant's (a polymer) ability to bind water and form a gelatinous slurry with high water-binding capacity. This is shown in Figure 8.

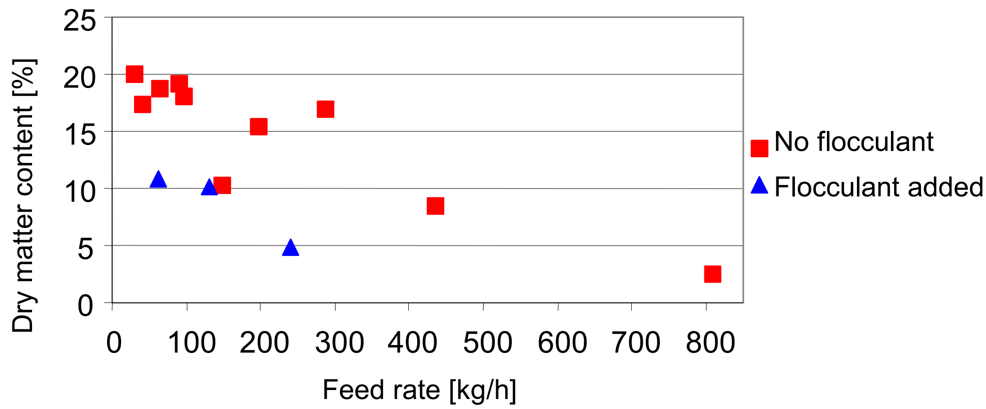

Figure 8 Influence of flocculant on dry matter content

Concentration of the feed stream utilising the VSEP unit is an on-going study. Currently, two different feed concentrations have been studied. Prior to starting the actual measurements slurry was recirculated through the VSEP unit to equilibrate the membranes and to reach a stable permeate flux. In Figure 9, the results from a run where a fibre concentration of 3% was employed are presented. The feed also contained some soluble material, such as sugars, which contributes to the total dry matter content.

The permeate flux changed by about 20%, from 1.4 to 1.1 l/(min m^2) when the cycle time was extended from 1 to 9 minutes. The recovered permeate was a clear liquid free from particles.

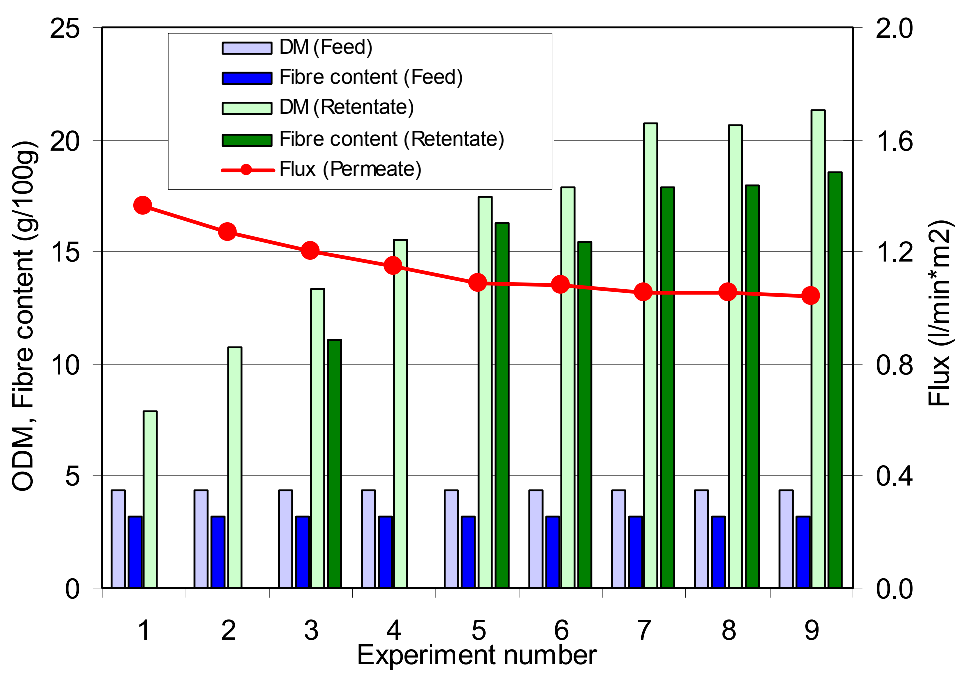

Figure 9 Separation using a VSEP unit

The fibre content in the retentate increased as expected when the outlet valve was closed for a longer time. The fibre content after a run where the valve was closed for 9 minutes resulted in about 18% fibre content in the concentrated slurry. This yields a concentration factor, relative to the feed concentration, of 6 times. In this study the pore size of the membrane was 0.2 µm, which is not necessary to reach a high separation. A 2.0 µm membrane has also been tested under different conditions, which resulted in a higher flux, and a clear permeate free from particles.

The final concentration of fibres is about the same whether a decanter centrifuge or a VSEP unit is utilised. However, there is one major difference: the permeate stream from the VSEP unit does not contain any solid particles. Since the fibre residue is intended for production of process heat in a burner the liquid must be evaporated prior to the incineration. It is, therefore, important to reach high dry matter content prior to evaporation. This is a criterion that is met both by decanter centrifugation and by the VSEP membrane. However, some fibrous material is not separated by the decanter centrifuge, but found in the liquid stream. This represents either a loss of material, or the addition of another step for recovery of the fibrous material.

Acknowledgements

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