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Short Communication

Using cavitation for delignification of wood

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ABSTRACT

The Kraft process is the most widely used chemical process for the removal of lignin and other polymers from wood to obtain cellulosic pulp fibres. In the present study, the effect of cavitation (growth and violent collapse of vapour bubbles in a liquid) on delignification of wood was investigated. Steam was introduced in the reactor in order to study the effect of steam driven hybrid cavitation on delignification. The results obtained were subjected to kinetic analysis. The rates of delignification obtained using hydrodynamic cavitation were about 4–5 orders of magnitude greater than those obtained using acoustic cavitation (rate constants for delignification were 9.78×10^{-6} and 6.8×10^{-1} min^{-1} for acoustic and hydrodynamic cavitation, respectively). The energy imparted by the pump in the hydrodynamic cavitation reactor was much higher than that imparted by the acoustic devices and this was considered to be the cause of the higher delignification rates.

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1. Introduction

The Kraft process is the most widely used chemical process used for the production of cellulosic pulp fibres from various ligno-cellulosic materials (Biswas et al., 2011). In the delignification step of the Kraft process (Austin, 1984), the wood chips are subjected to alkaline hydrolysis in a continuous digestor at a pressure of about 6.6–9.25 atm and a temperature of about 170–176 °C for about 2–5 h. The aim of this study was to explore the possibility of delignifying wood at room temperature and at a pressure of less than 5 bar using cavitation.

Cavitation is the process of the generation, growth and violent collapse of vapour bubbles at millions of locations in the bulk of the liquid. The collapse process is adiabatic and leads to a great rise of the temperature (to about 10^4 K) and pressure (to about 10^3 bar) within the cavity and subsequent return to ambient conditions; within a few microseconds (Pandit and Pinjari, 2010). Thus, without changing the bulk fluid conditions significantly, localized high temperature and pressure can be achieved in the system.

2. Methods

2.1. Experimental setup

Two main types of cavitation, viz. acoustic and hydrodynamic were used in this study. In acoustic cavitation, high frequency sound waves are used for the growth and collapse of the vapour bubbles; whereas variations in the flow geometry are used to achieve the same in hydrodynamic cavitation.

2.1.1. Acoustic cavitation

The horn type device [Dakshin Ltd., Mumbai, India] has an immersion type transducer (Fig. 1). The diameter of the transducer tip in this study was 2.1 cm and the tip was immersed to a depth of 1 cm in the reaction mixture. The device worked at a frequency of 22 kHz and had a rated power output of 240 W. For the horn type device, reactor volumes of 20 ml and 40 ml were used and a magnetic stirrer was used to keep the reaction mixture in uniform suspension.

The bath type device [Supersonics Ltd., Mumbai, India] has three transducers fitted at the bottom of the device in a triangular pitch and the acoustic radiation is transmitted through the bottom surface (Fig. 2). The device used in this study had a cubical cavity of dimensions 15 cm × 15 cm × 15 cm. In order to facilitate the dissipation of acoustic energy to the entire reactor contents, the reactor was placed in the cubical cavity and the annular space in the cavity was then filled with water to the level of liquid in the reactor. The device worked at a frequency of 20 kHz and had a rated power output of 120 W. For the bath type device, reactor volumes of 40 ml and 500 ml were used and an overhead stirrer was used to keep the reaction mixture in suspension.

In order to prepare a baseline for comparison for the acoustic cavitation experiments, 20 ml of the reaction mixture was prepared in a 25 ml beaker and stirred with a magnetic stirrer. The stirring speed was kept constant at about 300 rpm. The magnetic stir bar used for stirring had a diameter of 1.5 cm. This arrangement was called the 'stirred tank (20 ml)' configuration.

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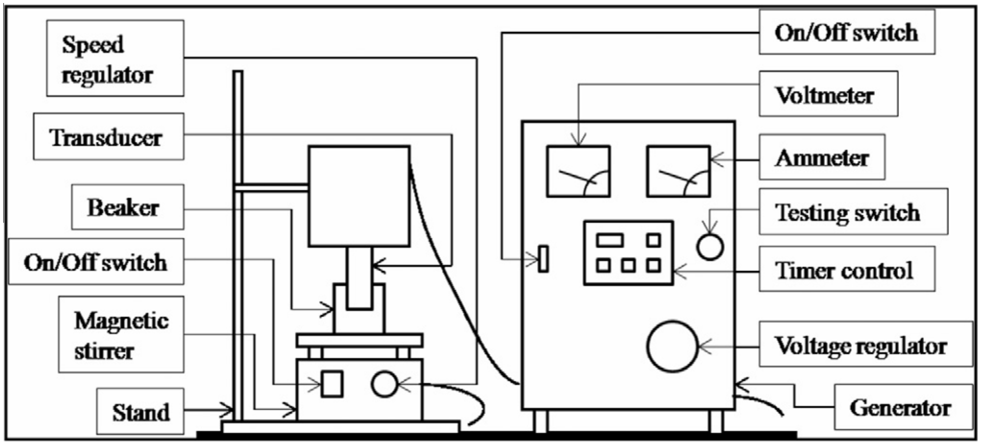

Fig. 1. Horn type acoustic cavitation device (schematic).

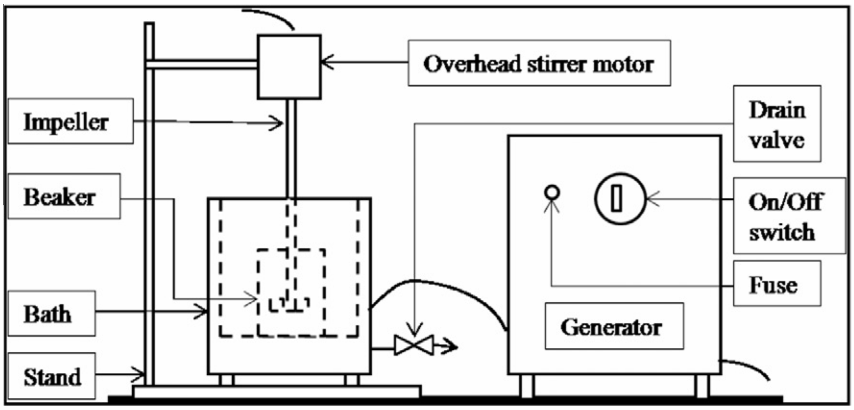

Fig. 2. Bath type acoustic cavitation device (schematic).

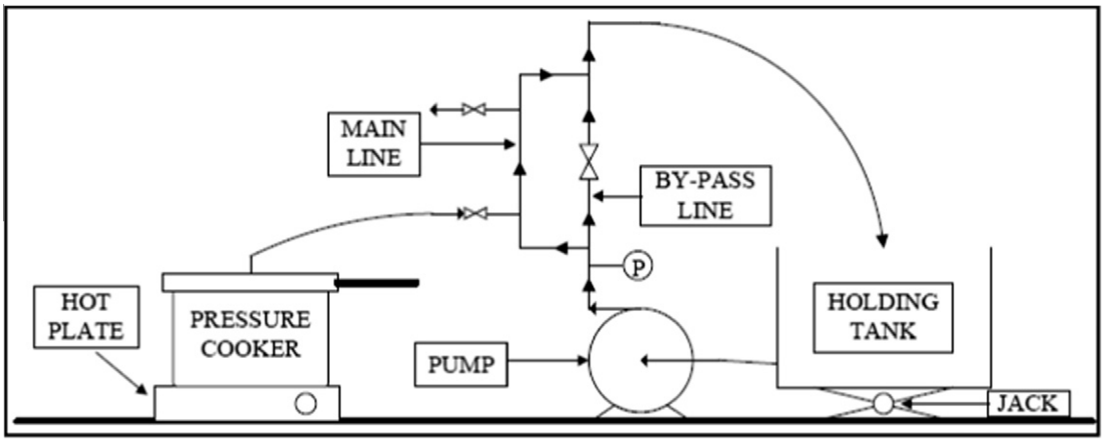

Fig. 3. Hydrodynamic cavitation setup (schematic).

2.1.2. Hydrodynamic cavitation

The hydrodynamic cavitation reactor (Fig. 3) mainly consists of a holding tank, a pump and a piping system. The holding tank was a stainless steel jacketed vessel of about 8 l volume, having an internal diameter of 30 cm and jacket thickness of 1 cm. To avoid spilling over of the reaction mixture, the volume of the reaction mixture was kept constant at 5 l. The pump used in this study was a 1 hp, open impeller pump driven at an impeller speed of 1425 rpm by an electric motor having a rated power of 750 W. The piping system used in this study consisted of a main line and a by-pass line, both having an inside diameter of 2.54 cm. The output from the pump was split into the main line and the by-pass

Table 1
Operating parameters in hydrodynamic cavitation.

Reactor configuration	P (Pa)	$\dot{V}_m$ (m^3/s)	$\dot{V}_b$ (m^3/s)	υ_m (m/s)	υ_b (m/s)
Stirred tank	1×10^5	0	2×10^{-3}	0	3.95
Cavitating venturi	5×10^5	4×10^{-4}	0	0.79	0
Cavitating venturi with indirect cooling	5×10^5	4×10^{-4}	0	0.79	0
Cavitating venturi with steam	3×10^5	5×10^{-4}	1.5×10^{-3}	0.99	2.96

Where p = discharge pressure (absolute) of pump, Pa.
$\dot{V}_b$ = volumetric flow rate in by-pass line, m^3/s.
$\dot{V}_m$ = volumetric flow rate in main line fitted with cavitating venturi, m^3/s.
υ_b = average velocity in by-pass line, m/s.
υ_m = average velocity in main line fitted with cavitating venturi, m/s.

line. The main line was fitted with a cavitating venturi. The by-pass line was fitted with a ball valve for controlling the flow through the set-up.

In order to isolate the effect of the cavitation achieved by the venturi from the effect of increase in temperature, cooling water was circulated at a flow rate of 1.5 ml/s through the jacket of the holding tank.

Steam was fed to the reactor at the throat of the venturi in order to study the effect of steam driven hydrodynamic cavitation. A pressure cooker was used to generate steam at a rate of 1.11×10^{-4} kg/s. The heat necessary to generate steam with a pressure cooker was supplied by a hot plate.

In order to prepare a baseline for comparison for the hydrodynamic cavitation experiments, all the flow was directed through the by-pass line by keeping the control valve fully open. This arrangement was called the 'stirred tank' configuration. The various reactor configurations used for hydrodynamic cavitation are summarized in Table 1.

Here, it must be noted that 5×10^5 Pa (a) was the maximum operating pressure, which was achieved when the valve on the by-pass line was kept completely shut. Also, steam was introduced at a lower operating pressure of 3×10^5 Pa (a) because it was found that maximum suction was created at the throat of the venturi at this pump discharge pressure and so, adequate quantity of steam could be introduced.

2.2. Experimental procedure

Aqueous alkaline solution of 5% w/w concentration was prepared by dissolving sodium hydroxide flakes in tap water. The experiments involving hydrodynamic cavitation required pumping of the reaction mixture and so, sawdust was used instead of the wood chips which are conventionally used for the pulping process. Sawdust, screened using a 30 mesh sieve (diameter of opening = 6×10^{-4} m), was added to the alkali solution such that the concentration of solids in the slurry was 5% w/w. The reaction mixture thus prepared was subjected to continuous stirring, to avoid settling of the slurry, and was allowed to react with/without cavitation for 30 min at room temperature and atmospheric pressure.

2.3. Methods of analysis

The reaction samples were analysed for delignification by using the acid-insoluble lignin method (T 222 om-02, 2002). The value of acid-insoluble lignin content obtained using this method has an accuracy of ±0.17 wt.%.

In order to compare the various parametric variations tried in this study on a uniform basis, cavitational yield is defined using the following expression:

$$\text{Cavitational yield} = \frac{\text{reduction in lignin content}}{\text{energy delivered}}$$

The least squares method was used for the kinetic analysis of the experimentally obtained data, in order to determine the mathematical form of the rate equation that gives the best correlation for the data.

3. Results and discussion

3.1. Experimental results

The acid-insoluble lignin content of untreated sawdust was found to be 32%. The acid-insoluble lignin content of commercial wood pulps exists between 2.6% and 19.1% (T 222 om-02, 2002) and it can be seen from Table 2 that the desired level of delignification (<19.1%) cannot be achieved by acoustic cavitation within 30 min of treatment time; whereas, it can be obtained by using hydrodynamic cavitation and especially steam driven hydrodynamic cavitation.

3.2. Cavitational yield

The values of cavitational yield calculated from the data are shown in Table 3. It can be seen from Table 3 that the cavitational yield is the highest in the stirred tank (20 ml) configuration. In fact, all the other values of yield are smaller by 3–4 orders of magnitude. Hence, it can be concluded that a stirred tank is the most energy efficient reactor configuration to initiate the process of delignification, but it cannot achieve a commercially acceptable extent of delignification in the specified time. In order to increase the delignification and make it commercially acceptable, additional energy is added by means of the acoustic/hydrodynamic cavitation devices.

3.3. Kinetic analysis

For the data obtained from the acoustic cavitation experiments, the least error was observed when the 3rd order rate equation was used. The values of the rate constant for 3rd order kinetics are shown in Table 4. Bland and Logan (1965) have reported that all

Table 2
Results from acoustic and hydrodynamic cavitation.

Acoustic cavitation		Hydrodynamic cavitation	
Reactor configuration	Acid-insoluble lignin content after 30 min (% w/w)	Reactor configuration	Acid-insoluble lignin content after 30 min (% w/w)
Stirred tank (20 ml)	28.00	Stirred tank	17.86
Acoustic horn with stirring (20 ml)	21.57	Cavitating venturi	14.95
Acoustic horn with stirring (40 ml)	25.74	Cavitating venturi with indirect cooling	16.43
Acoustic bath with stirring (40 ml)	27.24	Cavitating venturi with steam	13.08
Acoustic bath with stirring (500 ml)	26.47		

Table 3
Cavitational yield.

Acoustic cavitation		Hydrodynamic cavitation	
Reactor configuration	Cavitational yield (%wt/J)	Reactor configuration	Cavitational yield (%wt/J)
Stirred tank (20 ml)	6.55	Stirred tank	1.05×10^{-5}
Acoustic horn with stirring (20 ml)	2.41×10^{-5}	Venturi	1.27×10^{-5}
Acoustic horn with stirring (40 ml)	1.45×10^{-5}	Venturi with cooling	1.16×10^{-5}
Acoustic bath with stirring (40 ml)	2.20×10^{-5}	Venturi with steam	1.41×10^{-5}
Acoustic bath with stirring (500 ml)	2.54×10^{-5}		

Table 4
Results of kinetic analysis.

Acoustic cavitation (3rd order kinetics)		Hydrodynamic cavitation (0th order kinetics)	
Reactor configuration	k (min^{-1})	Reactor configuration	k (min^{-1})
Stirred tank 20 ml	5.27×10^{-6}	Stirred tank	0.475
Horn with stirring 20 ml	2.35×10^{-5}	Venturi	0.587
Horn with stirring 40 ml	1.17×10^{-5}	Venturi with cooling	0.530
Bath with stirring 40 ml	7.49×10^{-6}	Venturi with steam	0.680
Bath with stirring 500 ml	9.78×10^{-6}		

natural lignins are phenylpropane polymers, which are mainly formed by coupling reactions of three main types of monomer units, viz. *p*-hydroxyphenyl, guaiacyl (4-hydroxy-3-methoxyphenyl) and syringyl (4-hydroxy-3,5-dimethoxyphenyl). Hence it was deduced that all the three building blocks of lignin react differently with the alkaline solution in the presence of acoustic cavitation.

For the data obtained from the hydrodynamic cavitation experiments, the least error was observed when the 0th order rate equation was used. The values of the rate constant for 0th order kinetics are shown in Table 4. As mentioned earlier in Section 2.2.2, a 1 hp (746 W) pump was used to circulate the reaction mixture through the setup. Due to the energy imparted by the pump, the reaction mixture remained completely well mixed. Hence, the delignification became independent of the lignin content and remained a function of only the energy supplied and/or the hydrodynamics of the operating conditions.

4. Conclusions

This study proves that the lignin content of wood can be reduced to its commercially required value, at room temperature and low pressure, using hydrodynamic cavitation.

Further study is required to determine whether the process of delignification using hydrodynamic cavitation is economically and energetically feasible. It remains to be seen whether the advantage of operating the process at room temperature and low pressure with a smaller holding tank can balance the increased energy consumption for size reduction of the wood and pumping of the slurry.

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