

Comparison of Cavitation Effect in Case of Fixed and Free Fibers in an Ultrasound Beaker

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ABSTRACT

This study investigate the impact of high-intensity ultrasound treatment on the mechanical properties of pulp fibers. The pulp fiber samples are sonicated in an acoustically optimized beaker where high-intensity ultrasound is generated using a tuned sonotrode device. The idea is to create a controlled resonance to efficiently enhance the sound pressure in the beaker. Input power was 90 W. The objective is to define the difference between freely suspended fibers in a beaker compared to keeping fibers in a fixed position. The hypothesis is that fiber treatment at a specific input power will be more efficient in the case when fibers are kept in a high pressure zone. Since the fiber wall is a layered structure, it is likely to delaminate internally which will affect the mechanical properties of the fiber. The effect on fiber properties is verified by measuring the ultrasound attenuation spectra for the treated fibers. The attenuation measurements are based on measurements of a low-intensity ultrasound pulse-echo technique. On a macroscopic scale, changes in the attenuation spectra relates to a change in mechanical properties of the fiber wall, since the suspended fibers more or less retain their diameter and length distributions.

Keywords: Ultrasonic, Cavitation, Paper pulp, Cellulose fibers

1. INTRODUCTION

Paper is used worldwide as an everyday commodity that is around us in a spectra of applications, from packaging to construction materials and from bills to receipts. Paper is nowadays generally made from different species of wood but has historically been made from other fibrous materials ever since the paper was first produced around 200 years BC. Today, paper making is a complex, highly automated large scale process industry to continuously evolve to meet the increasing demand for different pulp and paper products.

Pulp and paper production requires a significant part of the total electrical power usage in Sweden. In the production process the most energy-intensive step is refining, which is used to develop desirable fiber properties of the pulp before it is used for production in a paper machine. Therefore, methods achieving a reduction of energy use in the process of refining and development of appropriate fiber properties are highly interesting. The primary idea behind this study is to investigate if new, more energy efficient, techniques for fiber development could be realized by the use of hydrodynamic and acoustic cavitation. To study this, a flow through cavitation reactor has been developed in a previous project (1), as a potential alternative to conventional refining.

In the refining process, the proposed ultrasound technology shows potential improvement since cavitation is relatively gentle to fibers and lead to increased fibrillation without fiber cutting action. Ultrasound refining could be an alternative to conventional refining but is not yet used by the industry, see e.g. (2,3). However, despite some promising results, earlier research show a non-consistency and further investigations are therefore motivated.

The main objective of this paper is to evaluate two different principles of ultrasonic treatment for

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fibrillation of cellulose fibers. The first principle refers to an ultrasound batch reactor where fibers are freely floating in the beaker. The second principle requires fibers to be fixed in the pressure antinode of the beaker volume. The fiber sample is concentrated to a small container fixed in the most intensive cavitation zone of the beaker. One important aspect is to optimize the reactor setup, which combines several resonance phenomena. The idea is to minimize loss of energy transfer from the piezo-driven horn through water volume to cavitation on fiber surfaces. One challenge, in particular, was to optimize the cavitation reactor concerning the effect fiber material has on sound propagation. The aim is to develop an optimization methodology for the design of new reactor types for improvement of energy efficiency by controlled cavitation (1).

2. EXPERIMENTAL SETUP

2.1 Sonoreactor

Figure 1 shows the experimental set-up of the reactor which is composed of a cylindrical glass beaker of 71 mm in inner diameter. The beaker is filled with water representing $5/4^{\text{th}}$ of the acoustic wavelength of the fluid. The water volume was 330ml. The middle plane of the sample container is placed 40mm from the bottom. The sample container is used to keep the fibers under test fixed in the acoustic anti-node of the fluid volume. The beaker volume is acoustically excited by a sonotrode at a frequency of 20.8 kHz. The distance between the tip of the sonotrode and bottom corresponds approximately to one wavelength in the fluid (74 mm). The dimensions of the beaker are chosen to enhance the vertical mode and suppress the radial mode (26.5 kHz). The breathing mode of the beaker is at 18.5 kHz. The exact value of the sonotrode is tuned for each experiment since the wavelength varies depending on the experimental conditions.

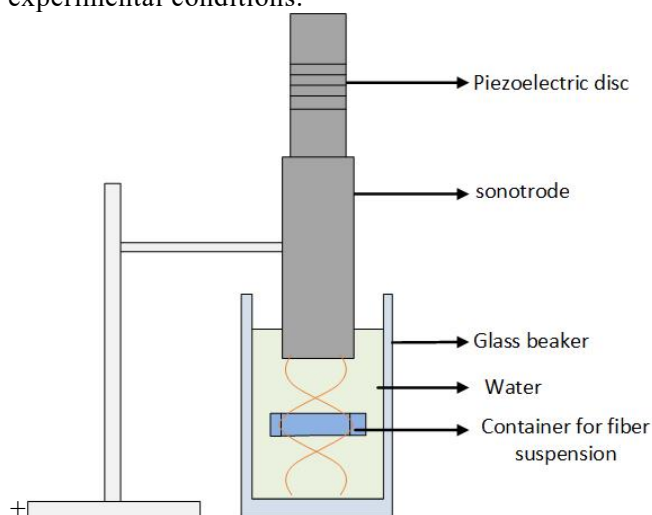


Figure 1 Sonoreactor used for pulp treatment with pressure mode highlighted.

2.2 Apparatus used

The assessment of the reactor cavitation performance requires measurements of the frequency response of sound and vibration signals; input electrical power, electrical impedance; and verification of sound pressure mode shapes. Frequency response measurement in the time and frequency domains were made by the software and hardware (CLIO, $f_s=192$ kHz). The pressure response was measured at 1-80VA electric input power. The pressure sensor (Dytran model 2200V1) and miniature shock accelerometer (PCB 353M15) were conditioned by a B&K NEXUS 2692 amplifier. The pressure transducer has a frequency range of 300 kHz. The pressure response in the time domain has also been monitored using a 1 MHz bandwidth oscilloscope.

3. Experimental Methods

3.1 Aluminum foil test

The aluminum foil test is a common method for characterizing the sound field in an ultrasound reactor. Hydrophones or chemical dosimeters (4) are other methods for measuring cavitation activities.

In this study aluminum foil and hydrophone tests were used.

For the aluminum foil test the beaker was filled with 0.330 liters of water prior to treatment. The test material consisted of 128 circular aluminum film discs, 5 mm in diameter and a thickness of 16 μm . The foil discs were dropped into the beaker to freely float around during the first test. In the second test the foil discs were placed in the sample container (see Figure 1). The aluminum foil discs were taken out and photographed for observation of the damage pattern after exposure. The aluminum foil discs were subjected to the same optimized excitation frequency and electrical energy input.

3.2 Calorimetric test

The input acoustic power in the liquid can be determined by a number of methods. Calorimetry is a common but precise method (5 - 7) to measure the power dissipation in liquids. The dissipated power due to cavitation depends on the acoustic and thermal properties of the fluid. The temperature increase with time (dT/dt) is proportional to the input power. By compensating for heat transfer losses to the surrounding, the power dissipation and efficiency of the ultrasound reactor can be determined, see equations 1-3.

$$P_{diss} = C_p M \frac{dT}{dt} \quad (1)$$

Where C_p is the specific heat capacity ($\text{Jkg}^{-1}\text{K}^{-1}$) of the liquid, M is the liquid mass (kg), and dT represents the temperature change over the time interval dt .

The power density of ultrasound is defined by Equation (2)

$$\Pi = \frac{P_A}{V} \quad (2)$$

The volume of the ultrasonic vessel or reactor is defined either by the liter (L) or (cm^3). P_{diss} is proportional to the applied acoustic power (P_A). The power density unit can, therefore, be either W/L or W/cm^3 . Subsequently, the reactors power conversion efficiency is determined by

$$\eta = \frac{P_A}{\text{Electrical Power}(W)} \quad (3)$$

The properties of the material used in the calorimetric test are shown in the following table. The water absorbed most of the heat dissipated through the cavitation. Moreover, the beaker was heat insulated to minimize the heat transfer to the materials in contact with water. The heat dissipation to the surrounding materials was measured with the ultrasound off. This is an approximation of the true heat dissipation to the surrounding materials. During cavitation, the fluid is subjected to motion which increases the heat transfer efficiency. Therefore, true power conversion efficiency was slightly higher than the estimated values.

Table 1. Physical properties of the material in use for a calorimetric test.

Material used in a calorimetric setup	Specific heat capacitance (JKg/K)	Density of material (kg/m^3)	Mass (kg)
Aluminum	336.3	2700	0.02
Water	4190.2	998.27	0.33
Quartz glass	700	2200	0.05

3.3 Fiber qualities

The fibers used in the experiments were hardwood fibers in never-dried form. Hardwood pulp (birch), totally chlorine-free (TCF) having a mean length of 0.932 mm (8). These fibers were not pre-treated before exposure to ultrasound. After ultrasonic cavitation processing, the samples were analyzed with respect to various fiber properties and the energy used for processing.

The fiber experiments were carried out in three ways: (i) fibers freely floating in the beaker, (ii) fibers fixed in the small container and (iii) no treatment. Only one pulp quality was used. The pulp was industrially manufactured hardwood pulp, sampled at the end of the bleaching process. The details of the experiments are tabled in Table 2.

Table 2 the experimental design for beaker reactor (335 ml) where fibers are free and fixed.

Test	Type of treatment	Conc	f _{us}	signal	Temp	EXP Time	P _{input} *
Nr		%	kHz		°C	sec	VA _{rms}
REF	free	0.43	20.9	-	-	-	-
BEXP1	free	0.32	20.9	sine	50-52	167	45
BEXP2	free	0.49	20.5-21.4	chirp	50-51	163	43
BEXP3	free	0.33	20.5-21.5	chirp	51-52	322	43
BEXP4	free	0.40	20.9	sine	50-52	295	45
BEXP5	free	0.33	20.5-21.5	chirp	50-51	92	76
BEXP6	fixed	0.55	20.6	sine	49-50	188	40
BEXP7	fixed	0.55	20.5-21.4	chirp	50-51	150	47
BEXP8	fixed	0.64	20.5-21.5	chirp	51-52	360	38
BEXP9	fixed	0.59	20.6	sine	50-52	333	40
BEXP10	fixed	0.50	20.5-21.5	chirp	51-52	91	77

*The chirp signal peak power is more than two times higher than the rms power.

4. Results and discussions

4.1 Aluminium foil test

The aluminum foil test was performed for both of the test conditions (free/fixed) at the same level of electrical power input to the sonotrode. The exposure time was 33 seconds and the electrical input power was 75 W_{RMS}. Irradiation of the water filled beaker was based on an excitation signal with a pure sinusoidal frequency of 20.8 kHz at 22°C.

The beaker experiments, both for freely floating foil discs and in a fixed position, gave very minute erosion, which can almost not be seen with the naked eye. However, the foil pieces in a fixed position were slightly more affected. When an aluminum foil is exposed longer time, a greater erosion effect will be observed. In case of a flow-through type of reactor evaluated in parallel, a more pronounced effect was found (1).



Figure 2 Aluminum foil discs sonicated by 33 sec in the beaker reactor. (a) Freely dropped (b) In container.

4.2 Calorimetric test

The glass beaker was filled with 330 ml of water at 55°C and the fluid was sonicated with pure sine signal at 20.8 kHz and an input power of 51.5 W_{rms} to the sonotrode. The heat insulated beaker remained static until a thermal balance at 45°C was achieved. The ultrasound was then activated for 20 minutes and at each fifth minute, the beaker's temperature rise was recorded with a precision of 0.1°C by using two thermocouple thermometers. Figure 3 showed a nearly linear 20 minutes rise in

temperature and exponential 20-minutes decay in temperature when the ultrasound was turned off. The acoustic power was then determined by Eq. 1. The experiment was repeated to verify the reaction stability. With a power conversion efficiency of 25%, the acoustic input power was found to be 13.2 W (Eq 3). The corresponding power density was 0.16 W/cm³ (Eq 2). However, the acoustic pressure varies extensively within the water volume due to the standing wave, and acoustic properties of the beaker.

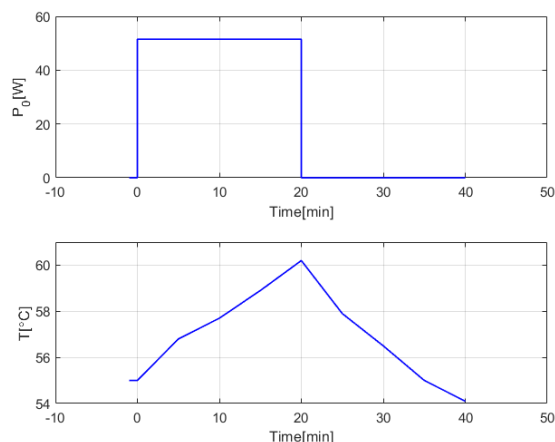


Figure 3. Electric input power to the beaker reactor and corresponding internal temperature variations.

4.3 Fiber treatment

The fiber treatment conditions are summarized in Table 3. The average fiber geometrical properties were characterized by a pulp analyzer. The impact on pulp quality was determined by measuring the ultrasound absorption according to a validated method (9). The experimental pulp used for the verification of the cavitation effect on fibers properties was a never dried type of birch fiber. The fiber suspension concentration was 0.3-0.7%, see Table 3.

The results in Figures 4 to 7 shows that the ultrasound controlled cavitation can alter the fiber properties, both in the case of fixed fibers and free fibers. Figure 4 and 5 shows the results of all completed absorption measurements characterizing changes in the fibers mechanical properties.

Table 3 Energy conversion in the conducted experimental series

Test	Fiber position	f_{US}	signal	Temp interval	EXP-time	Electric Power	conc fibers	Energy	Energy/ton dry fiber
Nr		kHz		°C	s	W	%	kWs	kWh/bdt
Ref				21			0.43		
BExp1	Free	20.9	sine	50-52	167	45	0.32	7515	1941
BExp2	Free	20.5-21.4	chirp	50-51	163	43	0.49	6993	1191
BExp3	Free	20.5-21.4	chirp	51-52	322	43	0.33	13813	3452
BExp4	Free	20.9	sine	50-52	295	45	0.40	13275	2711
BExp5	Free	20.5-21.4	chirp	50-51	92	76	0.33	6992	1773
BExp6	Fixed	20.6	sine	49-50	188	40	0.55	7520	1140
BExp7	Fixed	20.5-21.4	chirp	50-51	150	47	0.55	7050	1068
BExp8	Fixed	20.5-21.4	chirp	51-52	360	38	0.64	13788	1784
BExp9	Fixed	20.6	sine	50-52	333	40	0.59	13320	1851
BExp10	Fixed	20.5-21.4	chirp	51-52	91	77	0.50	7007	1166

Figure 4 shows that the spread within a specific pulp sample is small, except for sample "BExp2" where absorption is reduced. Test BExp1 and BExp5 have approximately the same attenuation. BExp3 has the greatest attenuation, which indicates a clear change of fiber properties at an energy equivalent to 3452 kWh / bdt. In case of Test BExp3 the fiber suspension was exposed by a chirp signal within the frequency band of 20.5-21.5 kHz. Increased attenuation relates most likely to internal and external fibrillation, as other fiber parameters do not change significantly by ultrasonic treatment. However, test BExp2 has a significantly lower absorption which may be related to lower sonification energy, since the sonification signal type is the same. However, Figure 5 shows that BExp2 deviates from the other samples by having a greater mean width and a shorter mean length. In some earlier experiments, absorption values lower than the reference was related to damage of fiber walls. If that the case here, it may be related to the chirp signal, which creates a pulse like rise of acoustic power. At 60°C the cellulose structure is still brittle and could be damaged by misguided and too powerful cavitation.

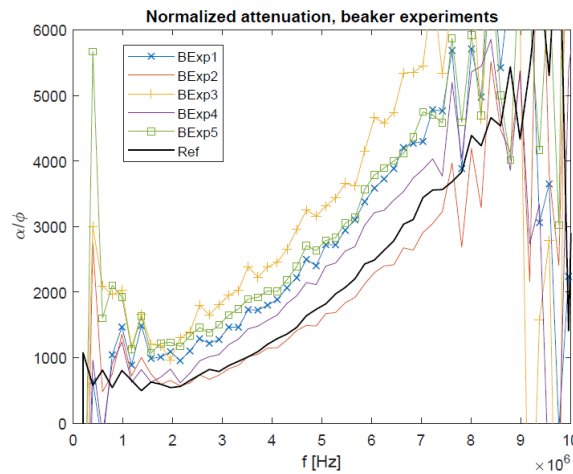


Figure 4. Measured absorption of fiber suspensions treated in a beaker. The change of the normalized attenuation rate indicates a change of the fiber mechanical properties.

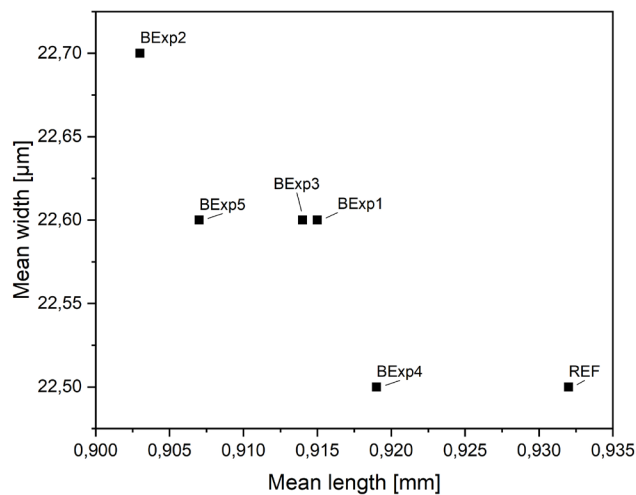


Figure 5. Fiber length and width distribution of fibers moving freely during sonification.

The fiber types tested have been analyzed with Lorentzen&Wettré pulp analyzer, see Figure 5. After the cavitation treatment, fiber suspensions were tested and analyzed to see define the size and shape distributions. Figure 5 shows that all samples in case of freely moving fibers except BExp 4 has an increase fiber width. BExp2 has the most pronounced change in fiber properties likely related to a

destruction of the fibers walls, since the absorption was lower than reference. Test BExp1, BExp3 and BExp5 were almost equal in geometrical distribution, see Figure 6.

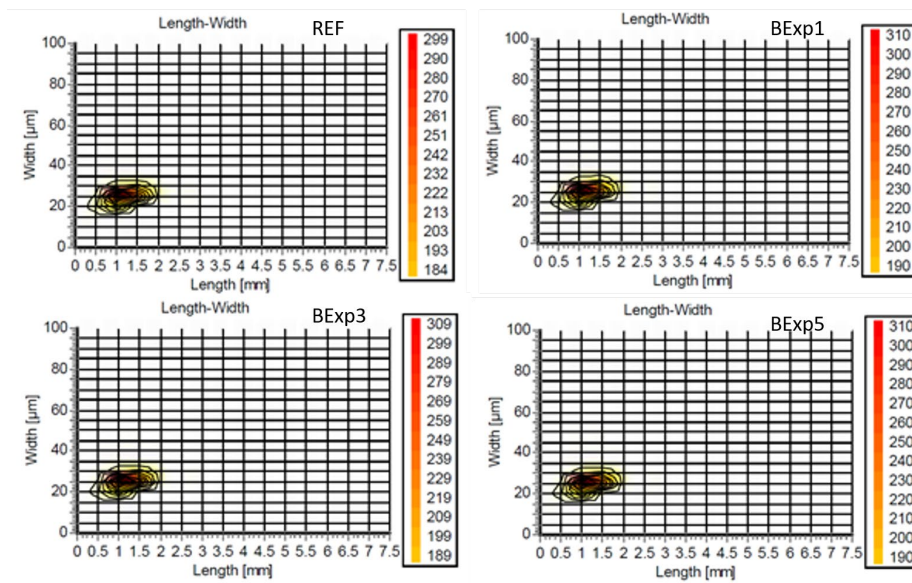


Figure 6. Length and width distribution of test samples: Ref, BExp1, BExp3, and BExp 5.

Figure 7 shows the result of sonification of fixed fibers, denoted test BExp6 to BExp10. Test BExp6 has the greatest attenuation, which indicates a clear change of fiber properties at an energy equivalent to 1140 kWh/bdt. In case of Test BExp6 the fiber suspension was exposed by a sinusoidal signal at 20.6 kHz. Increased attenuation relates most likely to internal and external fibrillation, as other fiber parameters do not change significantly by ultrasonic treatment. Test BExp10 has a lower absorption, that could be related to a delamination of the fibers walls, caused by the chirp signal with highest rms power. The chirp signals characteristics having a sudden increase of power when hitting the systems resonance frequency, may have exceeded the suitable cavitation intensity and caused fiber wall destruction.

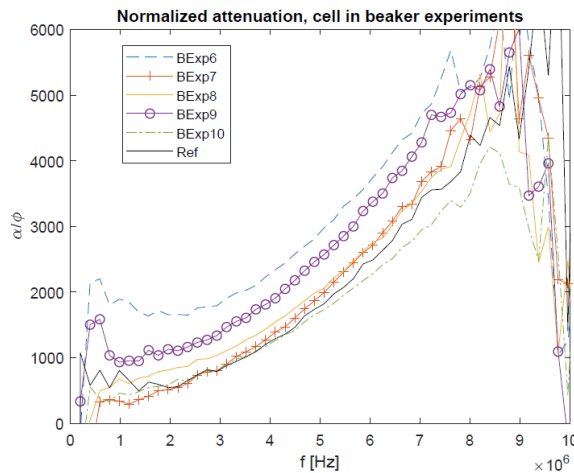


Figure 7. Measured absorption of fiber suspensions sonicated in a container fixed in the pressure anti-node of the beaker. The change of the normalized attenuation rate indicates a change of the fiber mechanical properties. Reduced absorption indicates deteriorated fiber properties.

Figure 8 shows the comparison of the cavitation effect with fixed and free fibers in an ultrasound beaker. The maximum absorption was with test BExp3 and BExp6. The power used for these experiments was 3452 kWh/bdt (BExp3) and 1140 kWh/bdt (BExp6). This proves that with fibers at a fixed position and using pure sinusoidal signal give most energy efficient change of fiber properties.

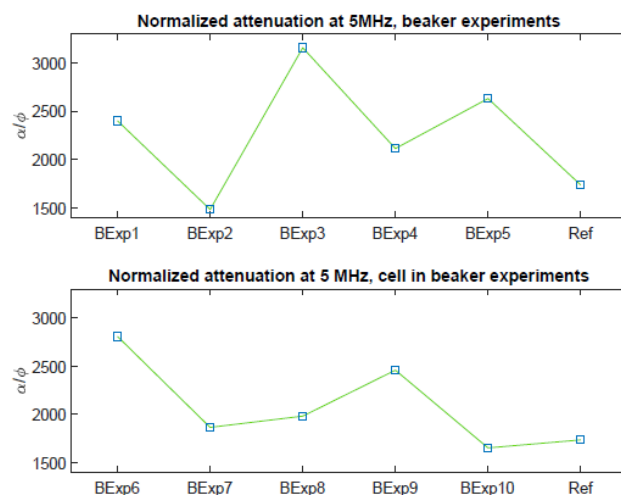


Figure 8. Comparison of measured absorption at 5MHz. Free fibers (BExp1 - 5). Fixed fibers (BExp6 - 10).

5. CONCLUSIONS

The results of a small scale experiment on the effectiveness of resonance amplified cavitation with respect to hardwood fiber fibrillation gave promising results. The hypothesis that fixed fibers in an acoustic antinode would be more effectively processed is validated. Foil test results indicated that there is only a minor difference between free and fixed in the acoustic antinode. The reason for smaller effect using foil discs is because they were not as fixed as the fibers were, and by that, the discs were moved to the circumference by the max pressure in the center of the container. In the fiber tests, the container was packed and only allowed for minor movement of the fibers. The change in absorption of the fiber mass verifies a significant modification of the fibers mechanical properties for all samples. The increase was most pronounced in the case when fibers are fixed in the acoustic pressure anti-node in the beaker. The fibers were also more evenly modified when fixed in the beaker as indicated by the absorption spectrum.

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REFERENCES

1. Ö. Johansson, T. Pamidi, T. Löfqvist. Design of a high-intensity ultrasound reactor. 2017 IEEE International Ultrasonics Symposium (IUS) 6-9 Sept. 2017 Washington, DC, USA
2. Thompson R, Manning A. A review of ultrasound and its applications in papermaking. Prog Pap Recycl. 2005;14(2):26–42.
3. Laine J, MacLeod J, Bolker H, Goring D. Applications of ultrasound in pulp and paper technology. Pap ja puu, 1977.
4. Ley S V., Low CMR. Ultrasound in Synthesis . Berlin, Heidelberg: Springer Berlin Heidelberg; 1989.
5. Contamine RF, Wilhelm AM, Berlan J, Delmas H. Power measurement in sonochemistry. Ultrason Sonochem. 1995 Jan 1;2(1):S43–7.
6. Margulis IM, Margulis MA. Measurement of acoustic power in studying cavitation processes. Acoust Phys. 2005 Nov;51(6):695–704.
7. Kimura T, Sakamoto T, Leveque J-M, Sohmiya H, Fujita M, Ikeda S, et al. Standardization of ultrasonic power for sonochemical reaction. Ultrason Sonochem. 1996 Nov 1 3(3):S157–61.
8. Paper and textile from pulp. Available from: <https://www.sodra.com/en/pulp/products/>
9. Aitomäki, Yvonne, and Torbjörn Löfqvist. "Estimating suspended fibre material properties by modelling ultrasound attenuation." AIP Conference Proceedings. Vol. 834. No. 1. AIP, 2006.