Chemical and Morphological Changes in Fibre Structure due to Material Heating during Ultrasonic-assisted Embossing of Cardboard

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Embossing is a commonly used design element on printed products and packaging. It enhances the product impression with optical and haptic effects. The material deformation during the embossing of cardboard is normally done using high mechanical pressure between two dies. The use of ultrasound in the embossing process leads to a noticeable reduction of the embossing pressure and a greater embossing precision. However, there is a noticeable heating of the cardboard during the ultrasonic-assisted embossing process. This work aimed to characterise the effects of heating and to understand the reasons for the greater precision with decreased force when ultrasound is used. Therefore, the effects of the thermal ultrasonic energy on the chemical composition and the morphological properties of the fibres were investigated. The findings showed that no noticeable changes occurred in the chemical composition or fibre geometry as a result of the embossing process with ultrasound.

Keywords: Cardboard; ATR-IR spectroscopy; Raman spectroscopy; Single fibre analysis; Ultrasound; Embossing

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INTRODUCTION

Embossing is a common technique used to enhance the optical and haptic qualities of packaging. The three-dimensional effect is often used to highlight lettering and logos on cardboard packaging and printed products such as books to attract a consumer's attention. Embossing is a design element that needs no additional materials such as printing ink or coatings to provide an optical or haptic effect. Hence, the embossing has little to no influence on the recycling process.

Embossing is a forming process that creates a relief pattern on the surface of a material. The material is pressed between two dies using high pressure to leave a replication of the pattern in the material. The use of ultrasonic energy in the process improves the embossing. The material compression is 30% greater compared to conventional embossing of cardboard (Hofmann and Hauptmann 2020). However, the use of ultrasound in the embossing process has several effects, including increased temperature in the middle of the material. Löwe *et al*. (2019) as well as Hofmann and Hauptmann (2020) have shown this heating phenomenon extending from the middle of the cardboard. Temperatures reached

approximately 200 °C to 220 °C, where browning and charring occurred in the inner layers of the cardboard (Löwe *et al*. 2019; Hofmann and Hauptmann 2020).

Further investigations have shown that ultrasound causes physical changes in the fibre network, resulting in changes in the cardboard's mechanical properties. Bending stiffness, compressibility, and splitting strength differed between cardboard samples with and without ultrasonic treatment. These changes in the mechanical properties were largely attributed to the loosening of the fibre network and the evaporation of water (Käppeler *et al*. 2020). Apart from the mechanical changes, it could be assumed that chemical changes or morphological changes within the fibres also occur. When treating fibre suspensions with ultrasound, Jayme and Rosenfeld (1955), Manning and Thompson (2002), Tatsumi *et al*. (2000), and Guo *et al*. (2015) observed increasing fibrillation of the fibres; they used fresh fibre pulp and pulp of used paper. Lieser (1953) described the depolymerisation of cellulose in water by ultrasonic treatment. The depolymerisation is assumed to arise from the friction between fibres and solvent resulting from the ultrasonic waves in the solution (Lieser 1953). In contrast, Laine and Goring (1977) described no shortening of wood fibres in aqueous suspension. Different effects are observed from thermal heating of fibre components and fibres in solution. Not previously explored is the effect within the fibre network of short-duration, high-energy ultrasound. In the aforementioned studies, the ultrasound duration was long (approximately 3 h) with relatively low process temperatures (approximately 60 °C), compared with the present investigations (process time 1 s, process temperatures approximately 125 °C).

Hence, the high temperature increase could affect the fibre composition. The paper fibres consist of three main components: cellulose, hemicellulose, and lignin. Several publications have shown that chemical changes occur in each of these components due to thermal influence. For example, a colour change occurs in cellulose when heated between 120 °C and 180 °C (Lieser 1953; Nikitin 1955; Liu *et al*. 2017). The glass transition point of the amorphous regions of cellulose is between 220 °C and 250 °C (Back *et al*. 1967; Wanske 2010). Temperatures greater than 350 °C result in decomposition of the cellulose (Reh *et al*. 1986; Jonoobi *et al*. 2009). Concerning cardboard and paper materials, there are two phenomena described in the literature. Back *et al*. (1967) and Ensminger (2012) investigated the effect of temperature on the tensile modulus of paper. Changes in the tensile modulus were observed at different temperatures. These were attributed to the relocation of hydrogen bonds and cross-linking reactions of the cellulose (Back *et al*. 1967; Ensminger 2012). Other research has examined temperature's effect on the aging of paper. Cellulose degrades because of cleavage of the 1,4-glycosidic bonds by oxygen. However, heat and ultraviolet light accelerate this process (Bächle 2009).

Hemicellulose is also heat sensitive (Back *et al*. 1967). The glass transition point is between 160 °C and 200 °C, with decomposition at approximately 300 °C. Some studies suggest lower decomposition temperatures (180 °C to 250 °C) (Reh *et al*. 1986; Bächle 2009; Wanske 2010). Furthermore, studies of wood show chemical changes such as the degradation of hydroxyl groups or the splitting of acyl groups (Back *et al*. 1967; Chen *et al*. 2012). Sazanov and Gribanov (2010) suggested that for temperatures between 60 °C and 80 °C, the hydroxyl bond of lignin is destroyed, but this is disputed by other authors.

The glass transition point of lignin is between 120 °C and 140 °C (Wanske 2010; Hansen 2015; Vas 2016), depending on the procedure of extraction and on the air humidity (Bayerl and Pichol 1986; Bächle 2009). At greater temperatures between 150 °C and 290 °C, the bonds within the alkyl aryl ether can become separated. At approximately 300 °C, the side chains dissociate from the lignin molecule (Sazanov and Gribanov 2010).

At approximately 370 °C, lignin decomposes due to the breaking of the carbon bonds (Hansen 2015; Vaz, Jr. 2016). The decomposition process also causes a colour change, which was also detected in wood at lower temperatures of approximately 120 °C (Reh *et al*. 1986). This colour change was attributed to the formation of quinoid structures (Chen et al. 2012).

Based on the preliminary studies, the following working hypotheses may be stated. First, the previous studies show the influence of temperature on the single components of the fibre composite. On this basis, it could be assumed that chemical structures of the components change using various temperatures. The investigations were performed on single components or other materials such as wood. It is expected that the effects are transferable to fibre composites. Therefore, the described changes ought to be detectable on the fibre composite. Second, the previous experiments on the changes of fibres using ultrasound show that characteristic values of the single fibres change. Compared to the present study, the examinations differed in the examination medium and the ultrasound duration. Nonetheless, it is assumed that similar effects can be shown when examining the fibre structure.

EXPERIMENTAL

Materials and Methods

For the following studies, conventional industrial cardboard was used. The cardboard consisted of three layers. The outer layers were made from sulfate pulp, with the inner layer from chemi-thermomechanical pulp (CTMP). Therefore, the middle layer contained a higher proportion of lignin. This was checked by colouring with safranin (Käppeler *et al*. 2020). The grammage of the cardboard was 350 g/m^2. For analysing the influence of ultrasound on the cardboard, samples with and without ultrasonic treatment were compared. The embossing process was a discontinuous and gap-controlled system. The samples were prepared with the experimental setup according to Fig. 1. Detailed descriptions of the test equipment are presented by Hofmann and Hauptmann (2020).

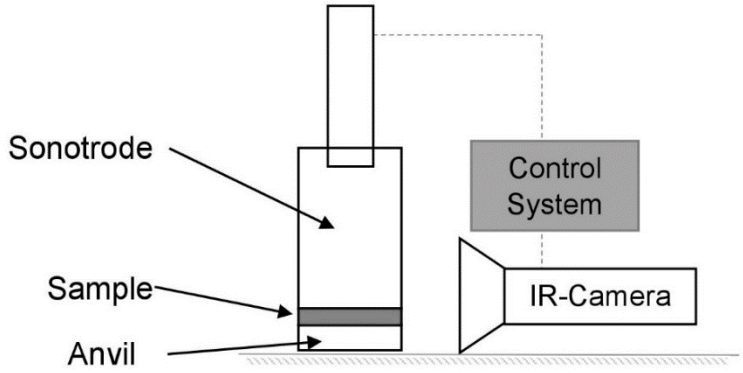

Fig. 1. Experimental setup; IR – infrared

The cardboard sample was placed between the sonotrode and the anvil, and the ultrasound was applied in the material using the sonotrode. After the static pressure was applied to the sample, the ultrasound amplitude was introduced into the material. During the whole process, the gap size was kept constant. The reference samples were embossed

without ultrasound using a universal materials tester (Universalprüfmaschine, ZwickRoell, Ulm, Germany). However, the same pressures were used as those used in the tests with ultrasound.

Depending on the ultrasound parameters, the fibre composite heated up to different temperatures. The temperature was measured in the cross section of the fibre network with a thermographic camera (IR 5320, InfraTec, Dresden, Germany). Preliminary studies showed that the cardboard was prone to scorching when temperatures greater than 140 °C were induced within the material by the ultrasound (Käppeler *et al*. 2020).

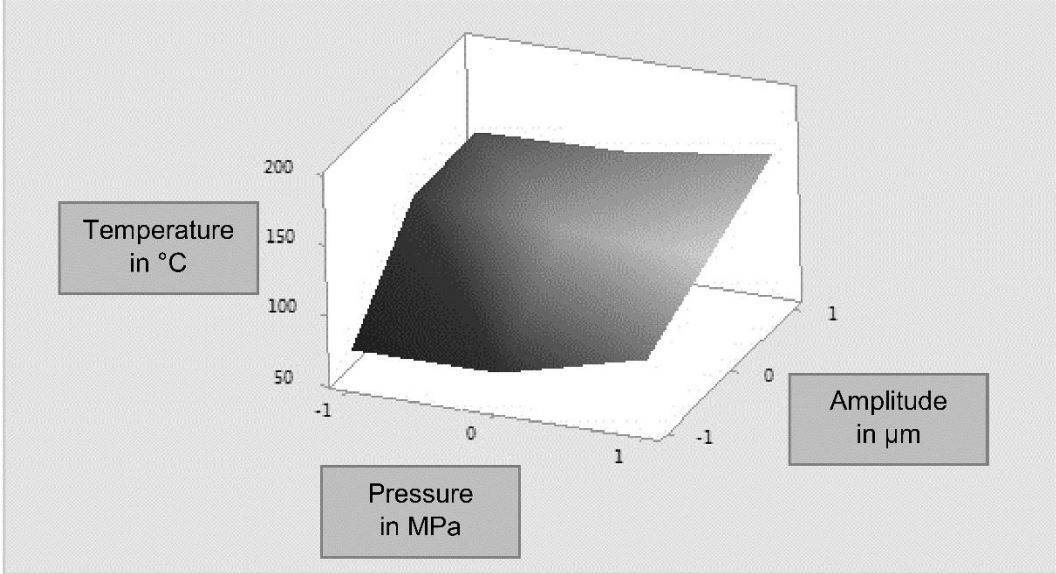

Fig. 2. Response surface plot of the reached temperature using different pressures and ultrasound amplitudes

Therefore, the ultrasound parameters were set for temperatures less than 140 °C. The ultrasound duration and contact pressure were set to 600 ms and 1 MPa, respectively. The amplitude varied from 25 µm to a maximum of 37 µm to induce different temperatures. Figure 2 shows the response surface plot of the reached temperatures using different pressures and ultrasound amplitudes. Without browning the samples, a maximum temperature of 124 °C was reached with the chosen parameters. Table 1 summarizes the parameter combinations and their resulting temperatures. For the following experiments, the greatest temperature level was chosen to detect the major changes. It may be expected that changes that did not occur within the samples of the greatest temperature also would not exist within the samples of lower temperature.

Table 1. Experimental Settings

Setting	Level			
	T_{Ref}	$T1_{US}$	$T2_{US}$	$T3_{US}$
Pressure (MPa)	1	1	1	1
Amplitude (µm)	-	25	31	37
Measured Temperature (°C)	RT	68	95	124
Confidence Interval (p = 0.975) (°C)	-	2	2	3

RT – standard atmospheric conditions, 23 °C, and 50% air humidity

The samples were analysed by infrared spectroscopy with attenuated total reflection (ATR-IR spectroscopy), Raman spectroscopy, and single fibre analysis. The spectroscopic methods were performed to investigate possible chemical changes of functional groups within the single fibre components of cellulose, hemicellulose, and lignin result from the ultrasonic energy and the resultant temperature induced. The single fibre analysis allowed observation of changes in fibre properties such as fibre length, kink, or fibrillation.

Spectroscopic analysis

Both ATR-IR spectroscopy and Raman spectroscopy could give information about different functional groups and their possible changes within the fibre network. Each component of the fibre has a characteristic vibration at a certain wavelength. For the analysis with ATR-IR spectroscopy (Spectrum One FTIR spectrometer, PerkinElmer, Waltham, MA, USA), the layers of the compound were split off to analyse only the lignin-containing middle layer. The sample was pressed with a stamp onto the measurement crystal. The spectra were taken with 16 measurement replications with each sample to generate one spectrum. The possible change of the spectra was visually analysed. For Raman spectroscopy, a confocal Raman microscope (Raman imaging microscope alpha300 R+, WITec Wissenschaftliche Instrumente und Technologie GmbH, Ulm, Germany) was used to focus the middle layer of the cardboard. The thickness of the middle layer and its localization were checked by colouring the cross section of the cardboard with safranin. Safranin only colours fibres that contain lignin with a deep red colour (Wanner 2004; Käppeler *et al*. 2020) The spectrum was taken with a 5-mW infrared laser (λ = 785 nm, 0.5 s integration time, and 1000 accumulations). The dark spectra were subtracted. Spectra were processed with a median filter for smoothing.

Single fibre analysis

For the single fibre analysis, the samples were soaked in water for 24 h, and the layers were manually separated.

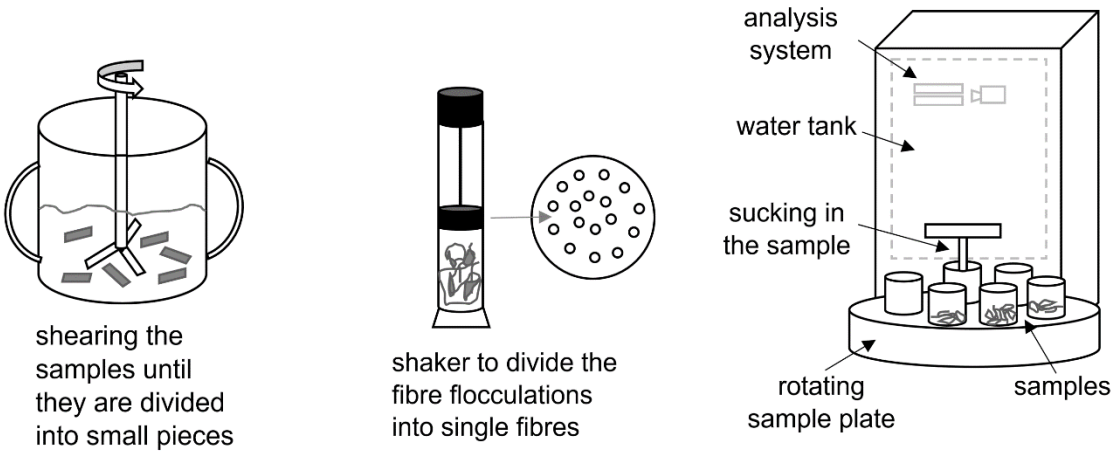

Fig. 3. Experimental setup for analysing the single fibres

The three layers were placed into separate beakers containing water and sheared until the samples were divided into small pieces (Noram disintegrator, Lorentzen & Wettre, Kista, Sweden). The suspensions were added into smaller shakers to divide the fibre

flocculation into single fibres. Remaining larger groups of flocculated fibres were removed with forceps. The resulting emulsion was analysed with a Fiber Tester Plus (Lorentzen & Wettre, Kista, Sweden) to measure parameters such as fibre length and fibre width. The experimental setup is shown in Fig. 3.

RESULTS AND DISCUSSION

Spectroscopic Analyses

Figure 4 shows the staggered spectra from ATR-IR spectroscopy and Raman spectroscopy for one sample with and one sample without ultrasound influence during the embossing process. The sample $T3_{US}$ was measured and compared to the sample without ultrasonic treatment according to the working hypothesis that the single components of the fibre network should have changed. Remarkably, the fibre composite showed a browning effect within temperatures of approximately 140 °C. The degradation temperature of the single components is stated to be even higher. Therefore, it could be assumed that the effects described in the literature could probably have taken place at temperatures less than the measured temperature of 140 °C. It is also possible that there were areas with stronger heating of the fibre composite than the measurable mean temperature and therefore the degradation took place. These possibilities should be analysed in further investigations. However, as the first working hypothesis says that structural changes possibly occurred, the change of the intensities of different wavelengths could indicate a chemical change in the fibre composition.

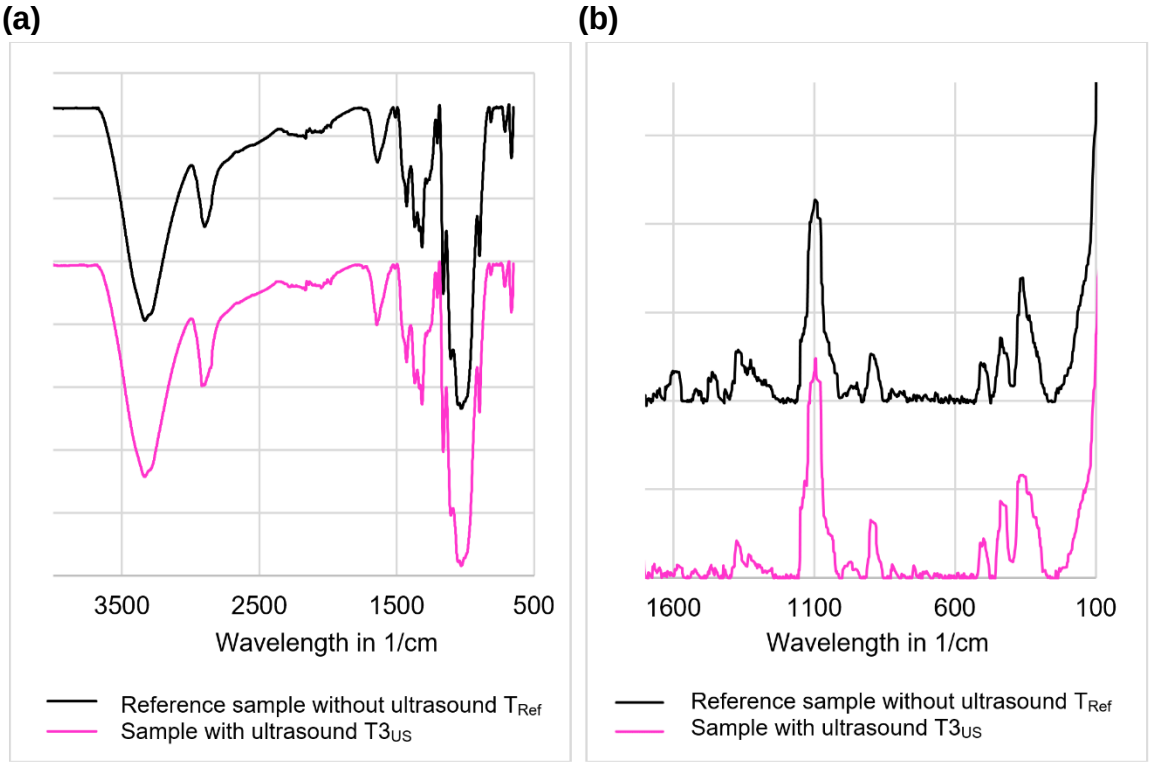

Fig. 4. Spectra of (a) ATR-IR spectroscopy and (b) Raman spectroscopy of cardboard samples with ($T3_{US}$) and without (T_{Ref}) ultrasound treatment

The differences between the spectra of the samples with and without ultrasonic treatment were not reproducible. The spectra of different samples were not consistent in their relative peak intensity, although the spectra were based on replicated measurements. Comparing the spectra of different samples before and after the embossing process in a difference spectrum it can be seen, that there were no changes in the intensity at different wavelengths. This result can have different reasons. First, it could be assumed that no chemical changes occurred during the short-term ultrasonic-assisted embossing process. Second, the changes from these methods were too small to be detected or just localized in a few spots in the fibre network. The temperature seemed to have no remarkable effect, comparable with the aging process of paper. In this phenomenon, cellulose degrades because of cleavage of the 1,4-glycosidic bonds by oxygen, which is accelerated by ultraviolet light (Bächle 2009). This change was not detectable. Furthermore, no colour change was observed. This was described in previous studies by heating up cellulose and lignin (Nikitin 1955; Back *et al*. 1967; Lieser *et al*. 1953; Reh *et al*. 1986). These effects were not noticeable. The colour change of wood was attributed to the formation of quinoid structures (Chen *et al*. 2012). However, in this study, the spectra did not change.

Single fibre analysis

Figure 5 shows the single fibres within the analysis process. The single fibre analysis characterised fibres in terms of length, width, form, fibrillation, and kinks by analysing images of the single fibres. Changes of these terms can show the effects of pressure and heating during the ultrasonic-assisted embossing process. The different characteristic values are shown in Fig. 5.

The average fibre length describes the length of the external contour of the fibre (contour fibre length), while the average width of the fibre describes the middle fibre width of the fibre. Fines are defined as particles smaller than 0.2 mm. The value of fines describes the relationship between the length of the fines and the total length of the fibres.

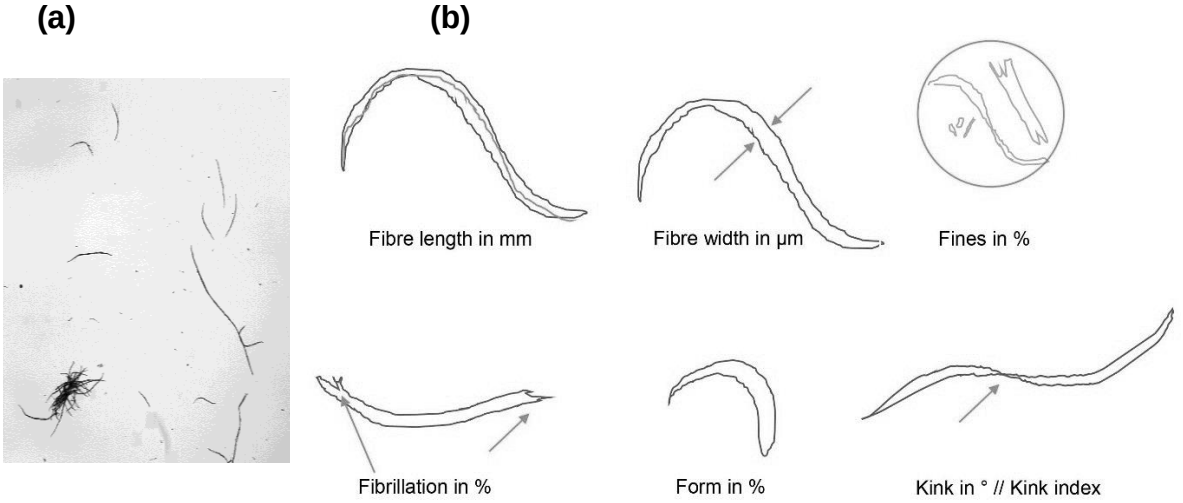

Fig. 5. (a) Single fibres taken *via* single fibre measurement device and (b) their characteristic values

The fibrillation describes the relationship between the perimeters of the fibre with and without fibrils. The form describes the curvature of the fibre—straight fibres have a form value of 100%. The kink index is related to the average angle of the fibres. The

average angle describes the arithmetic mean of the angles, while the kink index weights the angles.

The percentage changes of the single fibre measurement values of the samples with ultrasound ($T3_{US}$) compared to the reference samples without ultrasound are shown in Fig. 6 with double-sided confidence intervals (p = 0.975). The measurement values were measured for the middle layer and the surface of the cardboard. Most of the measurements showed no significant changes to the fibres from the ultrasound treatment. Only the fibre length on the surface of the samples seemed to increase. It could be assumed that this could be due to the effect of smoothing the surface as well. Wanske (2010) showed such an effect in his investigation. That goes together with the decreasing kink index (Fig. 6). It could also be assumed that the fibres get squashed and therefor they show a higher fibre length. This finding has to be analyzed further more.

The lack of change of the fibrillation observed in this study was contrary to the findings of Tatsumi *et al*. (2000), Manning and Thompson (2002), Guo *et al*. (2015), and Jayme and Rosenfeld (1955). However, the ultrasonic energy was introduced to fibres held in a solvent suspension. Against the second working hypothesis, it can be assumed that the effects on the fibres would be different in a suspension than in a solid network such as cardboard.

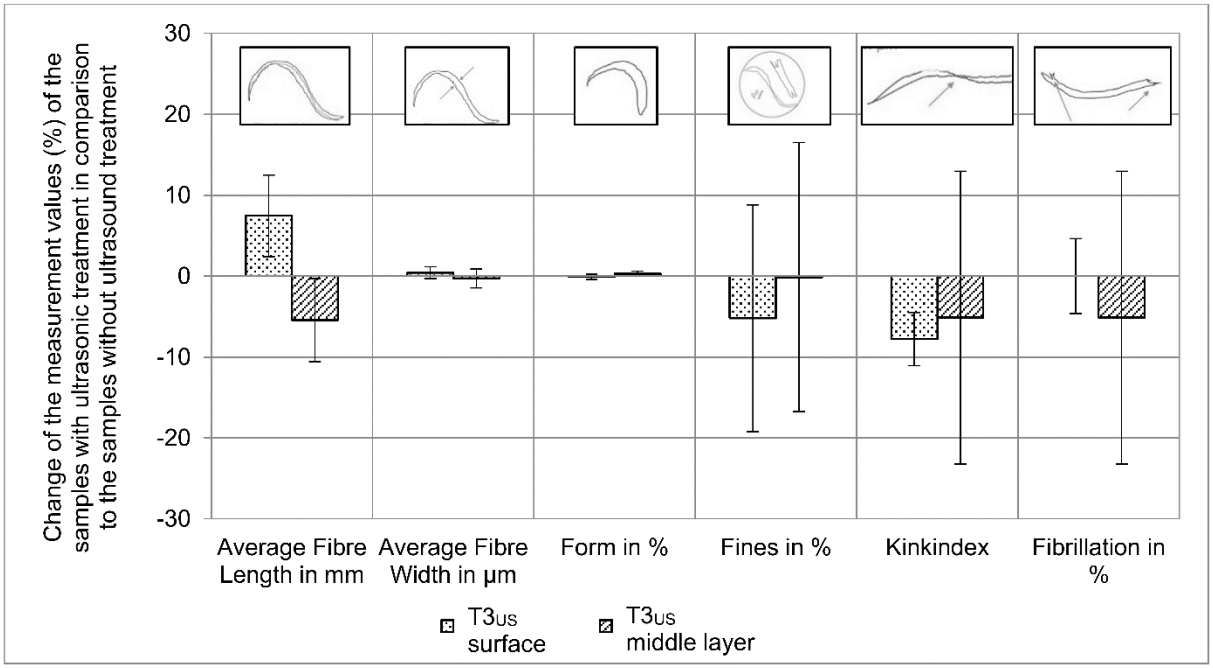

Fig. 6. Changes of the single fibre measurement values in the middle and outer layers of cardboard samples with ultrasonic treatment ($T3_{US}$), compared to cardboard samples without ultrasonic treatment

The distributions of fibre lengths are shown in Fig. 7 for the outer layers and the middle layer of the cardboard. The fibre lengths were well distributed and showed no significant changes between the samples with and without ultrasound treatment.

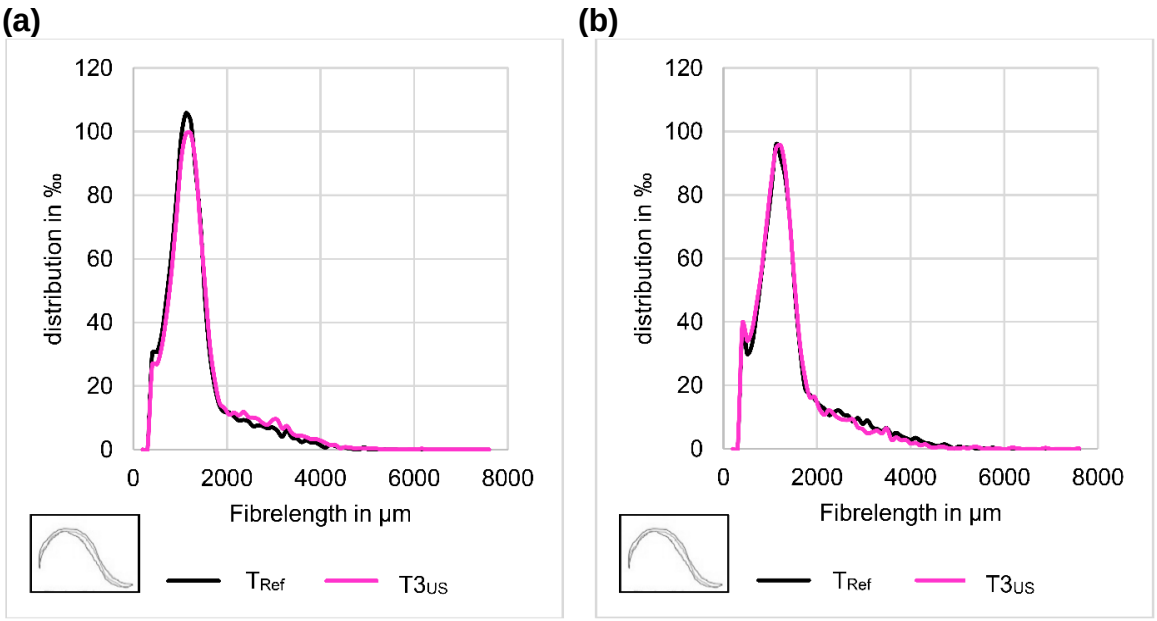

Fig. 7. Fibre lengths of cardboard samples with ($T3_{US}$) and without (T_{Ref}) ultrasound treatment in (a) the outer layers and (b) the middle layer

Consequently, temperature showed no influence on the fibre length. Therefore, unlike the results of Laine and Goring (1977), in this case the ultrasonic treatment did not result in a shortening of the fibres. The presumption of the shortening of the fibres cannot be supported by these measurement results. Thus, this result evidences, among other things, that the results of the previous investigations on fibres in solution cannot be transferred to dry fibre networks.

The research was performed to investigate the impact of heating by ultrasonic treatment while embossing. Therefore, comparable embossed cardboard samples with and without ultrasonic treatment were analysed with spectroscopic methods and fibre analysis. Chemical changes in the fibre composition and structure were expected. It was assumed that the individual components (cellulose, hemicellulose, and lignin) would chemically change due to the thermal influence. In other studies, the influence of heat on the individual substances was examined. The results in this study showed no significant changes in the chemical composition. However, the results showed that the previous results cannot be transferred to the effects of short-term and strong heating of the fibre structure by ultrasound. The findings of the experiments on ultrasound treatment of a fibre suspension could not be transferred to the fibre structure either. The results showed no noticeable changes in the fibre geometric properties. These findings suggest that the effects of ultrasound on the fibre characteristics observed by others for fibres in a suspension were not transferrable to fibres in a solid network such as paper or cardboard.

The findings are advantageous for an application such as embossing, because shortening of fibres, changes in functional groups, or chemical decomposition of the single components (such as cellulose or hemicellulose) could result in a weakening of the structure and negative effects on other properties of the cardboard. Nevertheless, greater material compression was observed during ultrasound embossing. This study could not show that the reason for the different material behaviour is chemical or morphological change in the material. In further investigations, it would have to be examined whether the mechanical loading leads to mechanical conditioning of the material and whether this is

the reason for showing a different material behaviour compared to the conventional embossing process.

CONCLUSIONS

1. The ultrasonically induced heat (124 °C within 1 s of ultrasound duration) in the material showed no influence on the chemical composition of the single components.
2. The ultrasonic embossing process showed no huge influence on the fibre geometry.
3. The greater precision with simultaneously decreased force by using ultrasound in the embossing process seems to have causes other than chemical or morphological changes.

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