

RESEARCH

All-Cellulose Composite: Influence of Dissolution Time and Moisture Content of the Starting Material on Wet Strength and Barrier Properties

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ABSTRACT

Packaging is essential for the global transport and storage of goods. However, due to the widespread use of non-biodegradable raw materials, it is a topic of environmental discussions. Paper plays an important role in the packaging sector due to the sustainability of the material, its outstanding flexibility and its high specific strength. But paper also has disadvantages. Paper does not possess wet strength and does not provide barrier properties. These disadvantages have so far been overcome by creating coated paper, paper laminates or through the addition of substances during production. An alternative solution could be All-Cellulose Composites (ACC), which are composites completely made of cellulosic materials.

Within the scope of this research short process times will be tested to determine if it is possible to achieve an increase in wet strength and barrier effect sufficient for packaging application. In addition, it will be investigated whether moist paper can be converted into ACC and to what extent the moisture content influences the resulting properties. The papers that will be converted are produced from bleached kraft pulp fibres (NBSK) on a Rapid Köthen sheet former. The conversion to ACC takes place via an immersion process. NaOH-urea is used as the solvent system, which is cooled to -12.5 °C. The tests show that a treatment period of just a few seconds is sufficient to significantly improve tensile and wet strength. It still needs to be clarified for what kind of technical applications the barrier properties achieved so far are suitable.

KEYWORDS

All-Cellulose Composite, bio-based composite, paper composite, cellulose fiber reinforced composite.

INTRODUCTION

Cellulose-based composite materials have been produced industrially since the middle of the 19th century and marketed, for example, under the name vulcanised fibre or parchment paper [1]. As a consequence of the industrial production of petroleum-based plastics, though, cellulose-based composites have been replaced in many applications. An increasing demand for more sustainable materials to replace petroleum-based plastics in the packaging industry

means that research into cellulose-based composites has intensified again in the last 20 years.

The term ACC was introduced by Nishino in 2004 and has since been used as a term for composite materials consisting entirely of cellulose [2]. The term all-cellulose nanocomposites (ACNC) is also sometimes used in the literature to describe cellulose-based composites that have a non-fibrous reinforcement [3].

ACC have two major advantages compared to biocomposites made from natural fibres and plastic matrices. They are beneficial from an ecological point of view since renewable materials are used as the starting material and ACC is biodegradable. In addition, the fibre–matrix adhesion is better. Huber *et al.* [4] compared the mechanical properties of ACC and biocomposites with a plastic matrix and were able to show that the use of cellulose for the matrix leads to significantly higher mechanical tensile strengths.

For the production of ACC various cellulose-based starting materials and different solvent systems and their influence on different material properties have been investigated. Among others, Dormans and Huber *et al.*, provide a good overview of various solvent systems that have been used for the dissolution of cellulose [4–6]:

- aqueous alkali solution (LiOH, NaOH; enhanced with various additives),
- acidic solutions,
- dimethyl acetamide/lithium chloride (DMAc/LiCl),
- ionic liquids (IL),
- inorganic metal complexes,
- N-methyl-morpholine-N-oxide (NMMO)/water.

For the production of ACC IL, DMAc/LiCl and NaOH/Urea are the most commonly used solvents in the research environment. In industrial production of vulcanised fibre, zinc chloride is normally used [7].

There is also literature detailing different cellulose-containing starting materials for ACC production and their influence on the material properties. The cellulose-based raw materials used include: paper and pulp [8–17], annual plants and agricultural waste [18–20], regenerated cellulose [21–27], and textile waste [28].

The solvent system used and the choice of cellulose-containing starting material have a significant influence on the production conditions. The temperature at which the dissolution process takes place varies from -12.5 °C for NaOH/urea to over 100 °C. The dissolving times also vary greatly from a few seconds up to several hours.

With regard to material properties, improvements in mechanical properties in particular have been published. Comprehensive overviews have been prepared, for example, by Fujisawa *et al.* [29], Baghae and Skrifvars [30]; Huber *et al.* [4] and others. The best mechanical properties were achieved using Bocell fibres, which were unidirectionally aligned before ACC production and transformed with DMAc. A tensile strength of 910 MPa and an Youngs-Modulus of 23 GPa were measured [31].

In paper, fibers establish connections through interfibrillar hydrogen bonds, creating a strong network. While paper exhibits good mechanical strength in a dry state, it easily disintegrates in wet conditions due to the effective breakdown of these bonds. Consequently, various paper applications incorporate wet strength agents to enhance the paper's water resistance. This is achieved through processes such as hydrophobization or the

formation of chemical linkages between fibers [32,16]. Ma *et al.* were able to show that the wet strength of paper is also significantly improved by a treatment with NaOH/urea and the associated conversion to ACC [33].

Fujisawa has discussed various publications on the barrier properties of ACC, in particular regenerated films, and provided a good overview. According to this, such films can exhibit good barrier properties against the migration of gases, particularly at low humidity, and in some cases exceed the barrier properties of commercial plastics [29]. In a study by Tanaka *et al.*, self-produced laboratory sheets from bleached pine kraft pulp were used as starting material. ACC was produced by treatment with the ionic liquid 1-ethyl-3-methylimidazolium and subsequently characterized. According to Tappi T 507cm-99, the ACC produced provides a complete grease barrier [16].

EXPERIMENTAL

Materials/chemicals details

For papermaking, Northern Bleached Softwood Kraft (NBSK, Mercer International Inc., Berlin, Germany) is used. The pulp is treated in a laboratory refiner (Voith LR40, SEL 1.5 J/m, setting 3-1.6-60, 100 kWh/t).

For the solvent system NaOH (99.6 % purity) and urea (99.6 % purity) were used and mixed with deionised water in the weight ratio 7:12:81 (NaOH:urea:H₂O) until a clear solution was obtained. Citric acid (food grade E330) is used to produce a 20 % acid solution with deionised water, which is used for neutralization.

Material synthesis

Isotropic sheets in accordance with ISO 5269-2 (2004) are produced from the refined pulp using a Rapid-Köthen Sheet Former. The laboratory sheets have a basis weight of 100 g/m² (±2 g/m²).

The moisture content of the paper is determined in accordance with ISO 287:2017. In order to set higher moisture contents, the paper is stored in a climate chamber under different conditions. The storage conditions and resulting moisture contents are summarized in **Table 1**.

Table 1. Climate conditions and resulting moisture content in paper.

Climate conditions	105 °C, oven	23 °C, 50 % RH	23 °C, 70 % RH	23 °C, 97 % RH
Moisture content	~0 %	7 %	10 %	20 %

To ensure that the sheets have the desired water content during conversion, they are packed in a plastic bag after climatization and only removed immediately before the conversion process.

The conversion of the paper to ACC is done in a 1-step process, by dipping paper in the solvent system. This manufacturing process is carried out by hand in most studies. In order to increase repeatability, especially for short process times, an automated dipping system was developed. The system consists of an insulated and cooled

area in which an immersion tank with the solvent is located. The solvent system and the surrounding air within this chamber are cooled to -12.5°C . Outside the cooled area there are 2 further dip tanks which are used for neutralization and are filled with 20 % citric acid and deionized water. The transport of the sheets between the different tanks is automated.

During the conversion process, the paper sheets are first dipped in the solvent system for a defined immersion time (1/11/21/31 s). The sheet is then moved out of the solvent tank where it remains inside the cooled area for 2 s so that excess solvent drips off. Next, the sample is transported to the neutralization tank. Leaving the cooled area, where a temperature increase from -12.5°C to room temperature occurs.

The transport times of 9 s are added to the immersion time, resulting in dissolution times of 10/20/30/40 s, whereby an ambient temperature of -12.5°C is only ensured for the first 7/17/27/37 s. For neutralization, the solvent-soaked sheet is first immersed in 20 % citric acid for 1 min. Finally, the ACC is placed in deionised water for 12 h. The ACC is dried in a Rapid Köthen sheet dryer and stored in a standard climate (23°C and 50 % RH) until characterization.

Characterizations

The tensile test is conducted according to ISO 1924-2 using a Zwick servo-hydraulic testing machine. In deviation from the test specification, the clamping length is 100 mm. The relatively short clamping length is a result of the paper dimensions, constrained by the capacity of the Rapid-Köthen Sheet Former. Due to the different clamping length, the test speed was adjusted to 11.1 mm/min. The samples are secured between pyramid-shaped grips with a contact stress of 5 MPa. The preload is 0.25 N.

The wet strength is tested in accordance with ISO 3781. Before the test, the samples are soaked in deionized water for 6 h. The test is carried out on the same equipment as the tensile tests. Preload, test speed and specimen dimensions are analogous to the tensile test.

Air resistance was measured in units of Gurley seconds using a Messmer-Büchel Parker Print-Surf. A Gurley second is defined, by test method TAPPI/ANSI T 460 om-11, as the time taken for 100 cm^3 to pass through one square inch of paper with a pressure differential of 1.22 kPa.

RESULTS AND DISCUSSION

The results of the tensile strength tests are shown in **Fig. 1**. The values given correspond to the mean values. The error bars indicate the standard deviation.

The conversion from paper to ACC leads to a significant increase in the width-related breaking strength and the Youngs-Modulus. The best results were achieved at dissolution times of 10 s. Here, the width-related breaking strength is 9350 N/m ($\pm 667\text{ N/m}$) and the Youngs-Modulus

5420 MPa ($\pm 212\text{ MPa}$) compared to the reference paper, which only has a width-related breaking strength of 6240 N/m ($\pm 310\text{ N/m}$) and an Youngs-Modulus of 3860 MPa ($\pm 159\text{ MPa}$). An increase in dissolution time tends to lead to lower mechanical strengths. However, no significant differences could be detected between the individual gradations of the dissolution time. For the solvent system and the cellulose materials used, dissolution times of less than 20 s are sufficient to achieve optimum mechanical properties. In contrast to other solvent systems, where significantly longer dissolution times of up to several hours are optimal, the potential for continuous production is demonstrated here.

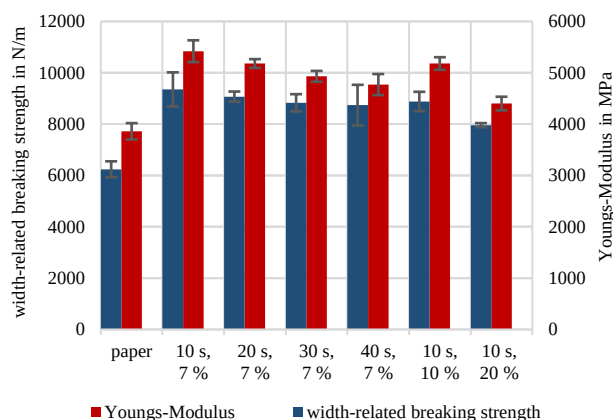


Fig 1. Results of the tensile tests, depending on dissolution time in seconds (s) and moisture content of the paper before converting into ACC in percent (%).

In contrast, the moisture content of the starting material has a much greater influence than the dissolution time. Lower mechanical strengths can already be expected at a moisture content of 10 %. This effect becomes even more significant when the moisture content is increased to 20 %.

The results of the wet strength tests are shown in **Fig. 2**. The wet strength in percent is related to the dry strength from Fig. 1.

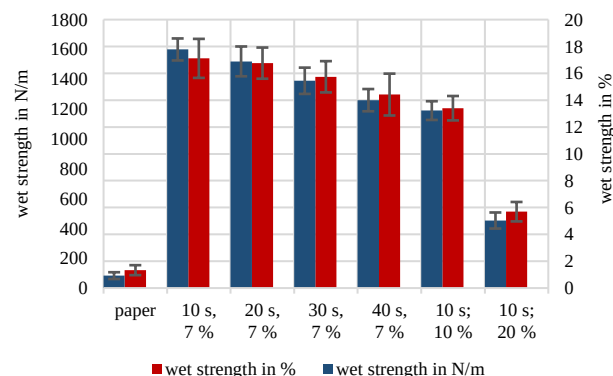


Fig. 2. Results of the wet-strength tests, depending on dissolution time in seconds (s) and moisture content of the paper before converting into ACC in percent (%).

As with the tensile tests in the dry state, the conversion to ACC leads to a significant increase in wet strength. The best values were achieved with a dissolution time of 10 s. A width-related wet strength of 1600 N/m (± 73.8) was measured. This corresponds to a wet strength of 17.1 % (± 1.5 %) compared to the initial dry strength or about 16 Nm/g. Longer dissolution times tend to lead to lower wet strengths, but a clear correlation between wet strength and dissolution time cannot be shown. As with the tensile tests, even a slightly higher moisture content leads to a significantly lower wet strength. The best wet strengths obtained are comparable with the results from other publications, e.g. by Tanaka *et al.* [16] who achieved a wet strength of 18.23 Nm/g for ACC produced from paper.

The results of the Gurley measurements are shown in Fig. 3.

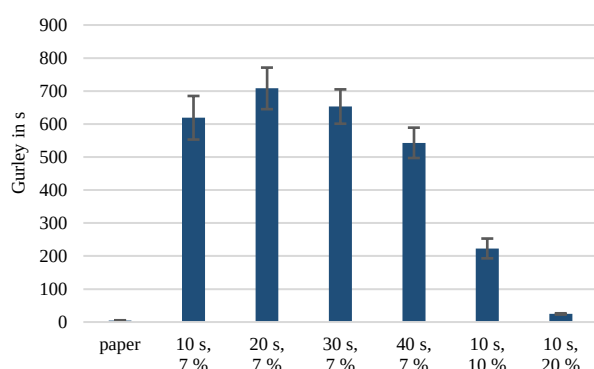


Fig. 3. Results of the Gurley-Test, depending on dissolution time in seconds (s) and moisture content of the paper before converting into ACC in percent (%).

Again, the conversion process leads to a significant improvement in properties. The best results were achieved for a solution time of 20 s and amount to 708 s (± 63 s). In comparison, paper only shows a value of 5.4 s. With longer solution times, the air barrier appears to decrease again. The exact reasons for this cannot yet be clarified. It was expected that the barrier effect would further increase with longer dissolution times, as a homogeneous material structure with fewer pore spaces is to be expected as a result of the progressive transformation. In other publications, a significantly lower air permeability was obtained [16,34,35]. One possible reason for this could be the porosity of the starting material.

CONCLUSION

In this work ACC were prepared by immersion of paper in a cooled solvent system containing NaOH. The paper was produced from NBSK using a Rapid Köthen Sheet Former. NaOH/urea (7 %/12 %) was used as the solvent system,

which was cooled to -12.5 °C. ACC production was carried out by varying the solution time and the moisture content of the paper before conversion.

It was shown that the conversion to ACC results in an improvement in the tensile strength, the Youngs-Modulus, the wet strength, and the air barrier. The best results were achieved with a moisture content of 7 % and dissolution times of 10 s to 20 s. For industrial production, this means that complete drying appears to be necessary. Selective dissolution during paper production in a moist state is possible, but leads to smaller improvements in the properties of the resulting ACC.

The achieved wet strength, which is already sufficient for various applications, is particularly promising. With regard to industrial production, the comparatively short process times compared to other studies are also advantageous. For certain applications in the packaging sector and for disposable tableware, ACC therefore shows great potential for reducing the use of petroleum-based materials and offering a fully bio-based, conditionally recyclable and biodegradable alternative. Considering that 39 % of the plastic produced was used for packaging products in Europe in 2023, there are opportunities for quantitatively relevant savings and for avoiding the potential generation of plastic waste and microplastics [36]. However, it must be mentioned that a neutralisation process can currently still be problematic for industrial implementation and that more research is needed in this area.

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CONFLICTS OF INTEREST

There are no conflicts to declare.

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GRAPHICAL ABSTRACT

The influence of moisture content of the raw paper and the dissolution time on tensile strength, wet strength and air resistance are investigated. The results show that that even short dissolution times of 10 seconds lead to a significant increase in tensile strength, wet strength and air resistance. In addition, it is shown that an increased moisture content in the starting paper has a negative effect on the material properties of the resulting ACC.

