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Influence of Moisture, Temperature and Bleaching on the Mechanical Properties of Coated Fiber-Based Substrates

Tobias Nienke [1,*], Arno Kwade [2] and Daniel Eggerath [1]

[1] Department of Paper and Packaging Technology, Munich University of Applied Sciences (HM), 80335 Munich, Germany

[2] Institute for Particle Technology, Technische Universität Braunschweig, 38106 Braunschweig, Germany

* Correspondence: tobias.nienke@hm.edu

Abstract: Substrates from fibrous materials are widely used in packaging applications and are produced in high quantities on roll-to-roll production lines. The anisotropic material behavior presents a demanding situation for the process control of the converting step. In this work, the influence of moisture and temperature on the mechanical properties of coated and uncoated fiber-based substrates and the influence of other material properties were investigated. The change of moisture content in relation to the surrounding temperature and relative humidity was investigated for different products. The hygroexpansion in dependence of the humidity is measured in machine and cross direction. The changes in the mechanical properties of the different materials due to changes in moisture content were investigated by tensile testing. The material behavior was highly responsive to the surrounding humidity and, thus, the material's moisture content. A relative humidity between 60% and 70% showed the influence on the material properties most clearly. The presented work showed an influence of the coating on the moisture content for higher grammages. The effect of bleaching was also investigated. Bleaching decreases the potential for moisture absorption and, therefore, influences the moisture content and properties such as hygroexpansion. Coatings influence the hygroexpansion in an anisotropic manner. Coating and bleaching also influence the tensile properties of fiber-based materials. This article is an expanded version of a talk given at the 14th European Coating Symposium, 6–9 September 2021, Brussels, Belgium.

Keywords: fiber; moisture; coated; tensile properties

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1. Introduction

Fiber materials form the basis of many web finishing processes. Tapes and consumer and industrial packaging are the main application areas. Rigidity, moderate market prices and their excellent eco-balance are persuasive sales arguments. On the other hand, the anisotropic material properties [1] present a demanding situation for the process control of the converting process. Changes in the moisture content of fiber-based products are always accompanied by dimensional changes of the material due to swelling of the fibers. This can lead to register inaccuracies in the printing and converting process. If a fiber web is being processed on a coating line, then water-based coating fluids add moisture to the web, the subsequent drying step decreases the contained moisture and re-moistening systems can be used to increase and equalize the moisture content to a specified level before rewinding. The described process steps result in a number of changes of the fiber dimensions [2], while the dwell times at a web velocity of 1000 m/min [3] are only a few seconds.

Coated and finished webs are kept in large rolls. Moisture exchange may occur during storage in climates of higher or lower humidity. For large rolls, this can take from days to months until equilibrium is reached and depends on the material's water vapor transmission rate (WVTR) and potential wrapping [4]. For roll material, the moisture exchange in axial orientation is faster compared to the radial orientation. This results in trough-shaped profiles of moisture inside the rolls [4] and may lead to changes in the material processing along the roll. Changing mechanical properties challenge the converting process, ask for corrections on parameters such as web tension during machine set-up and could potentially increase the risk of web breaks.

This work is an extension of results presented at the European Coating Symposium 2021 in Brussels, Belgium [5]. The influence of moisture and temperature as well as consequential properties from fiber processing steps on the mechanical behavior of coated and uncoated fibrous substrates were investigated. The change of absolute moisture in relation to the surrounding temperature and relative humidity was investigated for different products. The hygroexpansion in dependence of the humidity is measured in machine and cross direction. The changes in the mechanical properties of the different materials due to changes in moisture content were investigated by tensile testing.

The papermaking process orients the fibers to a large extent in the machine direction [6].

Paper is affected by its high sensitivity to variations in environmental conditions. This is mainly due to the high porosity. The relative humidity φ of the surrounding air at any given temperature cannot exceed a maximum value, given by the saturation curve of the Mollier diagram, which gives the maximum vapor pressure at each temperature [7]:

$$\varphi = \frac{p_v}{p_{v,max}} \tag{1}$$

where p_v is the partial vapor pressure and $p_{v,max}$ is the maximum vapor pressure. The absolute moisture content c_{H_2O} is defined in DIN EN ISO 638 [8] as given in Equation (2).

$$c_{H_2O} = \frac{m_0 - m_1}{m_0} \times 100 \tag{2}$$

where m_0 is the sample mass, and m_1 is the mass of the sample after drying until constant mass is reached.

Moisture sorption of fibrous materials results in a change in the material dimensions, which is most pronounced in cross direction (CD). The variation in machine direction (MD) is much smaller [6]. Lindner [9] provided a comprehensive review of the factors affecting hygroexpansion. The literature describes different theories behind the observed behavior.

The surface adsorption theory is based on the polarity of functional groups on the internal faces of the fibrous structure. These groups are mostly hydroxyl groups. At first, a monomolecular distribution is assumed during sorption processes. For higher relative humidity, a polymolecular distribution is described. This theory was the foundation for two mathematical models of the moisture content proposed by Brunauer, Emmett, Teller [10] and Guggenheim, Andersen, de Boer [11,12]. The weak point of these models, which will be discussed more in detail below, is their lack of explanation for the drastic increase in moisture content at high relative vapor pressures [13]. The capillary condensation theory is favored for the description of higher relative humidity:

$$ln(\varphi) = \frac{-2\gamma M cos\theta}{\rho r R T} \tag{3}$$

The Kelvin equation (Equation (3)) is used to calculate the amount of filled pores. γ is the surface tension of the liquid, M the molecular weight, θ is the contact angle, ρ is the density, r is the radius of the capillary, R is the gas constant and T is the absolute temperature. The capillary radius is decisive for the amount of moisture uptake.

Capillary condensation plays a minor role below a relative humidity of 80%, but it can explain the large increase in moisture content at high relative humidity (>80%) [13].

For the adsorption of moisture from an initially dry material, a modified version of Equation (3) is more appropriate, where the pores fill in a radial direction [13]:

$$ln\left(\frac{p_0}{p}\right) = \frac{-\gamma M}{\rho r R T} \tag{4}$$

where p_0/p is the relative vapor pressure. The concept of Knudsen diffusion is based on the idea of an increased possibility of adsorption for pores whose size is smaller than the mean free path of the water vapor molecules. They have a probability of colliding with the surrounding walls of the cavities [5].

Voids in the fiber system allow free diffusion. For the simple case of constant difference of concentration, diffusion can be described by Fick's first law [14].

When modeling the moisture transport in a fibrous material, usually vapor transport in the void volume and condensed (bound) water transport in the fiber volume is assumed. The models were proposed by Chatterjee [15] and others. Ramarao provided a review on the previous work [16].

Moisture diffusion models are used to predict deformation behavior such as curling [2]. Different modeling approaches exist. A 3D beam network model was introduced by Motamedian and Kulachenko [17]. Triphasic models combine the intraphase diffusion, seepage and heat conduction during sorption processes [18].

Sorption isotherms of fiber-based substrates usually have a sigmoid shape. A difference between adsorption and desorption, called hysteresis, is usually observed (see Figure 2). This effect has been investigated by numerous researchers. The domain theory, the theory of hydroxyl groups and the swelling theory are result models used to explain the observation [1].

Differences in the papermaking process influence the fiber behavior and sorption capability. Parker has summarized sorption isotherms of different fibrous materials [19]. The processing during the paper making process (e.g., bleaching, beating) also influences the moisture content. The influence of coatings was not the subject of his study. Szewczyk and Glowacki [20] investigated the impact of humidity on energy absorption during paper tensile tests and proposed two methods to calculate the breaking energy as a function of the moisture content. Rhim focused on the effect of moisture content on the tensile properties of paper-based food packaging materials [21]. He observed a decrease in tensile stiffness with increasing moisture for all samples. It was more profound in the cross direction. His explanation for this behavior is the development of hydrogen bonds between hydrophilic hydroxyl groups of cellulose fiber in the paper matrix. Water molecules replace the fiber–fiber interaction and reduce the intermolecular interactions between fibers. Further sorption induces the conformational change of the macromolecular strength of the paper.

1.1. Mathematical Representation of Moisture Content

Numerous approaches have been made to describe sorption isotherms mathematically in the past. Chirife and Iglesias [22] provided a comprehensive review of the past work. The presented work is based on theoretical concepts and semi-empirical models and is fitting of experimental data. The large number of reported equations are mostly mathematically equivalent after rearranging them [19]. The most dominant concepts are the ones proposed by Brunauer, Emmet and Teller (BET) and Guggenheim, Anderson and de Boar (GAB). The BET equation is a two-parameter equation, given in Equation (5), based on the concept of water absorbed onto a surface as a monolayer. The binding energy of the other layers is assumed to be equal to the one of the pure absorbate.

$$c_{H_2O} = \frac{c_{H_2O,0} C\varphi}{(1-\varphi)(1-\varphi+C\varphi)} \tag{5}$$

where $c_{H_2O,0}$ is the monolayer moisture content, and C is a constant related to the net heat of sorption. The BET equation is reported to give a good representation for a relative humidity between 5%–45%. The GAB equation can be used up until 90%–95% relative humidity. It is a three-parameter equation, given in Equation (6), using correction factors for the further layers of water absorption.

$$c_{H_2O} = \frac{c_{H_2O,0} CK\varphi}{(1-K\varphi)(1-K\varphi+CK\varphi)} \tag{6}$$

where K represents the difference in the sorbate's pure liquid state and in the upper states. Sorensen and Hoffman [23] added temperature dependency to the GAB equation and were able to receive a suitable description of the moisture content for temperatures between 2 °C and 25 °C. For a relative humidity higher than 85%, the capillary condensation becomes dominant and, therefore, the precision of the description-based surface adsorption loses precision.

1.2. Industrial Coating of Fiber-Based Substrates

Coatings help tailor the properties of fiber-based substrates. Increased mechanical properties or functional features can be achieved [24]. The influence is, on the one hand, due to the material properties of the coating material, as components can penetrate into the fiber network [25] and interact with cellulose components [26]. Binder levels of coatings [27], pigments, fillers and other additives [28] enlarge solubility and affect diffusion kinetics and equilibrium states.

In addition, the processing steps themselves can also significantly change the properties of the material. Calendering and other pressure and heat intensive steps in the coating process may influence the amount of inter-fiber spaces [29].

Drying during paper production is known to influence the tensile properties of fibrous webs [30]. Dispersion coated material is dried once again and, thus, additional internal stresses and changes of the porous network can be induced.

2. Materials and Methods

2.1. Moisture Content/Sorption Isothermes

Circular samples with an area of 100 cm^2 were conditioned in a climate chamber at different levels of relative humidity between 10% and 90% and temperatures between 10 °C and 60 °C. The absolute moisture content was determined gravimetrically using the oven-drying method [8]. Here, the samples were dried at 105 °C until constant mass was reached. The absolute moisture content was calculated from the mass before and after drying, as described in Equation (2). It was plotted against the relative humidity to create sorption isotherms.

2.2. Hygroexpansion

The measurement of the hygroexpansion was based on the method described in the ISO 8226 [31]. A 25 mm wide stripe of the material was fixed vertically in a device with two clamps, as illustrated in Figure 1. One of the clamps was movable and connected to a micrometer indicator. The free length between the two clamps was adjusted to 100 mm, and the exact value was given by the micrometer indicator. The hygroexpansion can be measured by exposing the loaded device to different humidity conditions in a climate chamber, where the temperature T and the relative humidity φ are controlled constantly. Five samples of every material type were tested for each fiber orientation. The hygroexpansion X is given in relation to the measured sample length at 50% relative humidity, as shown in Equation (7):

$$X = \frac{(l_\varphi - l_0) \cdot 100}{l_0} \tag{7}$$

where l_φ is the measured length at a specific relative humidity, and l_0 is the measured length at 50% relative humidity.

2.3. Tensile Test

Tensile tests according to DIN EN ISO 1924-3 [32] were performed at 100 mm/min using a 2.5 kN tensile tester and using samples with a width of 15 mm conditioned at a relative humidity between 10% and 90%. The samples were clamped with a free clamping length of 100 mm. The breaking force index σ_T^W, the tensile energy absorption index W_T^W and the tensile stiffness index E^W (Equations (9)–(12)) are characteristic values that can be derived from the data recorded during tensile testing. The thickness of fiber-based materials can be changed in calendering processes [6] without necessarily having an effect on the strength of the material. Therefore, the strength is not necessarily directly dependent on the material thickness, and the grammage w is included in the equations known from solid mechanics to consider this. Grammage is defined as the mass per unit area in Equation (8):

$$w = \frac{m}{A} \tag{8}$$

$$\sigma_T^W = \frac{1000 \cdot \overline{F}_T}{b \cdot w} \tag{9}$$

where $\overline{F}_T$ is the average maximum tensile force, b is the sample width of 15 mm and w is the grammage.

$$W_T^b = \frac{1000 \cdot \overline{U}_T}{b \cdot l} \tag{10}$$

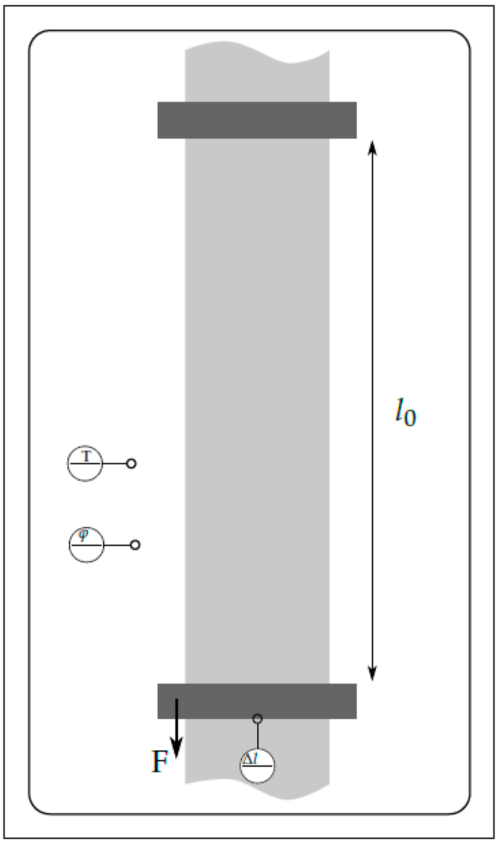

Figure 1. Measurement set-up hygroexpansion.

$\overline{U}_T$ is the mean breaking energy, which corresponds to the area underneath the tensile force-elongation curve, and l is the initial free length of the clamped sample.

$$W_T^W = \frac{1000 \cdot W_T^b}{w} \tag{11}$$

$$E^W = \frac{E^b}{w} \tag{12}$$

$$E^b = \frac{\overline{S}_{max} \cdot l}{b} \tag{13}$$

$$S_{max} = \left(\frac{\Delta F}{\Delta \delta}\right)_{max} \tag{14}$$

ΔF is the increase in tensile force, and $\Delta\delta$ is the increase in elongation during tensile testing.

2.4. Materials

The materials used in this work are listed in Table 1. Two different groups of materials were analyzed. Materials 1–4 are based on unbleached, woodfree, sized paper of 50 g/m^2 and 70 g/m^2. The raw paper was compared to a metalized paper with a dispersion top and bottom coating of 3.2 g/m^2 in total. Material 5 and 6 are 70 g/m^2 Kraft papers with a 20 g/m^2 LDPE-coating. The bleached and coated paper was compared to the unbleached coated material. Kraft papers are mostly used for corrugated board and consist of unbleached sulfate pulp, with a high stiffness [6].

Table 1. Specifications of experimental materials.

Material	Type Of Paper	Grammage [g/m^2]	Coating	Coating Weight [g/m^2]
1	unbleached, woodfree, sized, $CaCO_3$-coating	50	dispersion base coating, 20 nm metallization, dispersion top coating	3.2
2	unbleached, woodfree, sized, $CaCO_3$-coating	50	none	-
3	unbleached, woodfree, sized, $CaCO_3$-coating	70	dispersion base coating, 20 nm metallization, dispersion top coating	3.2
4	unbleached, woodfree, sized, $CaCO_3$-coating	70	none	-
5	bleached Kraft paper	70	LDPE	20
6	unbleached Kraft paper	70	LDPE	20

3. Results and Discussion

3.1. Moisture content

The influence of the humidity and temperature on the absolute moisture content c_{H_2O} of metalized and coated paper with a grammage of 50 g/m^2 (Material 1) is shown in Figure 2a. A difference between adsorption and desorption is noticeable. Between 10% and 70%, the moisture increased about 0.5% per 10% increase in relative humidity. Above 70% relative humidity, the increase in moisture content was twice as high. Capillary condensation was the cause for this increase. The curves and absolute values for temperatures between 10 °C and 30 °C were very similar, except for the high relative humidity. Measurements at 40 °C showed a significant difference in the absolute moisture content, which is also illustrated in Figure 3. The equilibrium moisture content was about 10% lower. Wahba and Nashed [33] observed a difference between 20 °C and 30 °C, but there was no difference between 30 °C and 40 °C. The temperature dependent moisture content at different relative humidity for metalized paper with a grammage of 70 g/m^2 (Material 3) is shown in Figure 2b. The absolute moisture content is higher compared to the lower grammage. A behavior comparable to the measurements for 50 g/m^2 in terms of slope and the difference in temperatures can be observed.

Figure 2c shows the sorption isotherms for the metalized paper with 50 g/m^2 and 70 g/m^2 compared to the same materials without coating. The difference is marginal for the grammage of 50 g/m^2. For the higher grammage, the metalized paper's moisture content was about 4% higher compared to the uncoated paper. The difference between both grammages decreased with decreasing relative humidity.

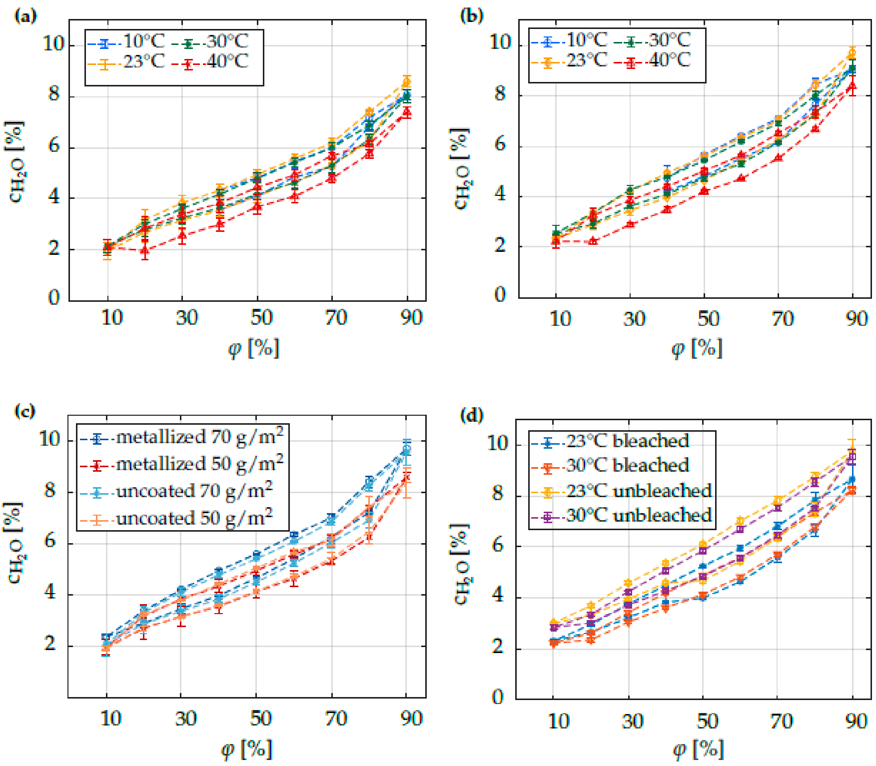

Figure 2. Sorption isotherms of 50 g/m^2 (**a**) and 70 g/m^2 (**b**) metalized paper at different temperatures, the metalized paper compared to the uncoated paper (**c**) and the bleached compared to the unbleached Kraft paper at 23 °C and 30 °C (**d**).

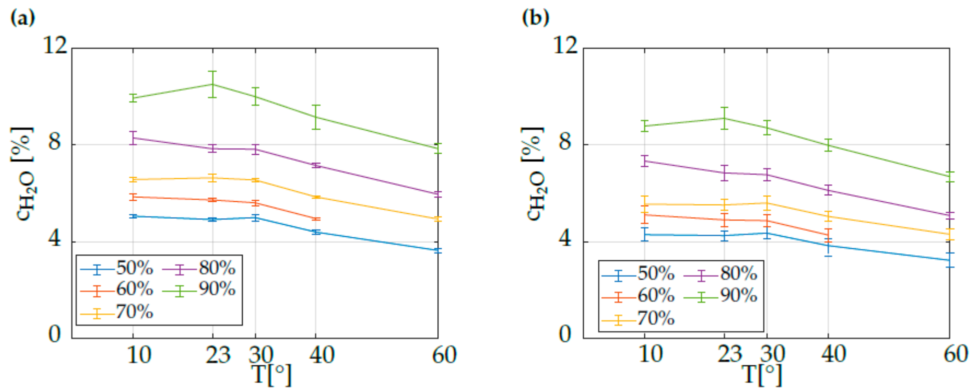

Figure 3. Moisture content during adsorption in dependence of the temperature for different relative humidity for 70 g/m^2 (**a**) and 50 g/m^2 metalized paper (**b**).

The moisture content of the unbleached Kraft paper with LDPE coating was higher than the unbleached equivalent. Figure 2d shows this for 23 °C and 30 °C. Similar observations on bleached fiber material were mentioned by Parker [19]. The difference between samples at 23 °C and 30 °C was more noticeable for the Kraft paper, as it was for materials 1–4. However, the same trend can be observed for the increasing temperature and the decrease in the moisture content c_{H_2O} in the material.

3.2. Hygroexpansion

Figure 4a shows the dimensional changes of Materials 1 and 3 and their uncoated equivalent due to sorption. Decreasing relative humidity caused a reduction in the size of the specimens. This hygroexpansion was much higher in the cross direction. This effect is well-known in the literature and is based on the micro fibrils inside the fibers, which are oriented in the length direction of the fiber. This way, they prevent shrinkage in this direction during moisture removal or swelling during moisture intake. A similar amount of dimensional change in the machine direction is observed between 10% and 50% and between 50% and 90% relative humidity. For 90% relative humidity, the dimensional change is 1.5 times the size of the change at 10% relative humidity. The almost linear relation to the relative humidity was also observed by other researchers [34–36]. The coated material shows about the same dimensional change in MD and 5%–22% more in CD compared to the raw material.

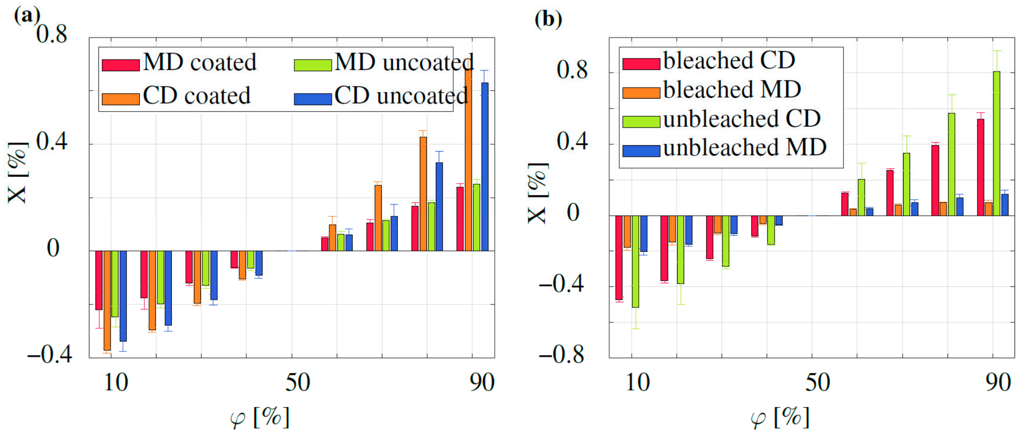

Figure 4. Hygroexpansion of metalized/coated paper compared to raw paper (**a**) and Kraft paper (**b**) in machine direction (MD) and cross direction (CD).

The dimensional changes based on moisture sorption for Material 5 and 6 are presented in Figure 4b. A significantly higher expansion in CD was also observed for these materials. The bleached fiber (Material 5) showed less expansion. Bleaching decreases the potential for moisture absorption and results in less fiber swelling [18].

3.3. Tensile Stiffness

In general, less elongation and higher tensile stiffness are measured on samples stressed in the machine direction, as shown in Figure 5. Samples conditioned in high relative humidity break at lower loads and have a higher elongation before reaching the breaking point.

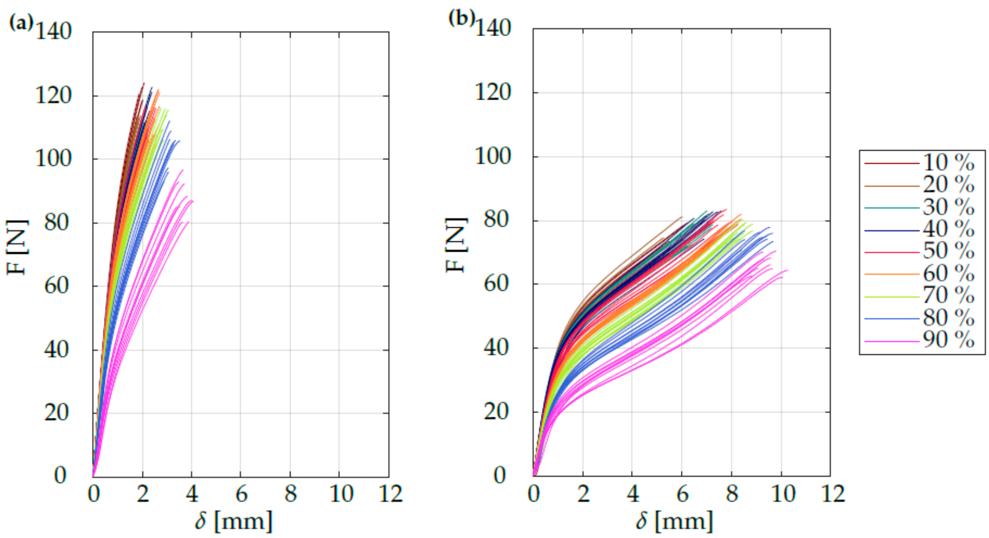

Figure 5. Tensile stiffness of bleached Kraft paper at different relative humidity in MD (**a**) and CD (**b**).

The detailed results of the tensile test for the different materials are shown in Figure 6. Figure 6a,b shows the tensile energy absorption index W_T^W in the machine direction

(Figure 6a) and the cross direction (Figure 6b). In the MD, the absorption index of the uncoated material with 70 g/m^2 was higher compared to the coated product. This difference is small for lower grammage. The tensile energy absorption index of the coated Kraft papers increased with the increasing moisture content in both directions. It was also lower for the other materials in the MD at low relative humidity, but the dependency on the humidity decreased for relative humidity greater than 50%. In the CD, no significant influence on the uncoated material or the metalized paper was observed for relative humidity smaller than 70%. For higher relative humidity, the tensile energy absorption index decreased.

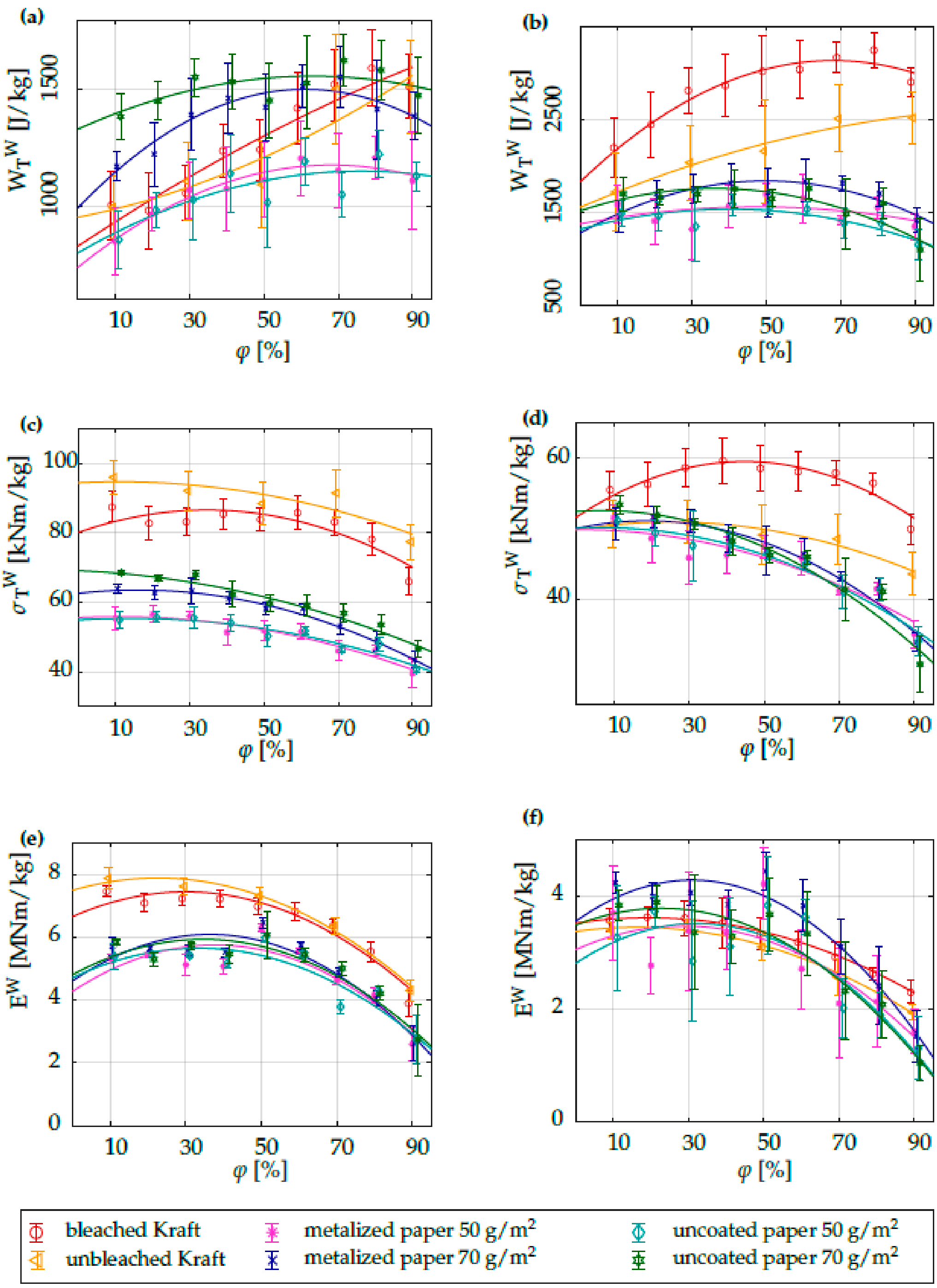

Figure 6. Tensile energy absorption index (**a**,**b**), breaking force index (**c**,**d**) and tensile stiffness index at different relative humidity in the machine direction (**a**,**c**,**e**) and the cross direction (**b**,**d**,**f**).

The breaking force index ${\sigma_T}^W$ decreased for relative humidity >70%. Coated material in the machine direction had a lower breaking force index (Figure 6c) compared to the uncoated material. This is probably due to the number of fillers and pigments in the coating. Bleaching decreases the breaking force in the MD slightly. In the cross direction, ${\sigma_T}^W$ also decreased for higher relative humidity, but the difference between the coated and uncoated material was less pronounced. Bleached Kraft paper had a higher breaking force index, with a maximum for medium relative humidity.

The tensile stiffness index E^W (Figure 6e,f) decreased for relative humidity > 60% in both directions. No influence of bleaching and coating was observed in MD. The tensile stiffness in the cross direction was slightly higher for bleached material. The deviation of the measurement data for the lower grammage coated and uncoated paper (Materials 1 and 2) was quite high. Therefore, it is difficult to make a statement. The coated material had a higher tensile stiffness index compared to the uncoated material. This influence was less pronounced for relative humidity above 70%, where the influence of the coating became smaller. This dependence on the relative humidity leads to the conclusion that the coating does not contribute to the strength of the material itself. Its main influence is the water vapor barrier.

4. Conclusions

The influence of temperature, moisture and bleaching on the mechanical properties of industrially coated fiber-based substrates was investigated. The material behavior was highly related to the surrounding humidity and, thus, the material's moisture content.

A relative humidity larger than 60%–70% had a strong impact on the mechanical properties of the investigated materials. Roll-to-roll processes, which rely on high web tension for the material transport, should avoid these. The resulting moisture content for these conditions increases the risk of web breaks. Long cycles of preprocessing in suitable humidity would be the strategy to process these rolls [37], which is time-consuming and therefore costly. The known hysteresis effect was observed while investigating the influence of humidity and temperature. Temperatures between 10 °C and 30 °C do not result in significant differences in absolute moisture, except for a high relative humidity above 70%. At 40 °C, the moisture content decreased by a factor of 0.9. This should be taken into account when storing material in warehouses.

The presented work showed an influence of the coating process on the moisture content for higher grammage. Other processing steps in paper-making also highly influence the material properties. The effect of bleaching on substrates for coating products was taken into account for this investigation. The bleaching of fibrous material decreased the potential for moisture absorption and, therefore, influenced the moisture content and properties such as hygroexpansion. This material behavior is not restricted by the coating. It was also seen for the coated Kraft paper investigated in this work. Moisture contents about 1.15 times lower were observed for bleached materials. Therefore, the hygroexpansion was up to 1.6 times higher for unbleached materials. This difference became smaller for conditions around 50% relative humidity.

Coatings influenced the hygroexpansion in an anisotropic manner. In the cross direction, up to 1.27 times larger expansion was observed for coated materials compared to uncoated materials. In the machine direction, the hygroexpansion was up to 1.1 times higher for the uncoated material. Therefore, it was highly recommended to evaluate the hygroexpansion for different materials used in industrial converting processes individually. Register errors or other web handling difficulties can otherwise be the consequences.

Coating and bleaching also influenced the tensile properties of fiber-based materials. The tensile energy absorption was about 1.18 times higher for uncoated materials in the machine direction. This effect was not observed for lower grammage. The breaking force index decreased by a factor of 0.8 in the machine direction for coated materials. Bleaching decreased the breaking force in the machine direction around a factor of 0.9. The tensile stiffness index increased by a factor of 1.12 in cross direction for coated materials. The influence of bleaching on the tensile stiffness index was minimal.

In the next step, the presented laboratory measurements at steady-state conditions should be transferred to industrial trials to investigate transient effects during moisture changes during production. A more detailed analysis of the viscoelastic properties of the different components using dynamic mechanical analysis (DMA), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) could also be of interest for future research. Furthermore, higher grammages and different coatings could be analyzed in future work.

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Abbreviations

The following abbreviations are used in this manuscript:

b	width [mm]
c_{H_2O}	moisture content [%]
$c_{H_2O,0}$	monolayer moisture content [%]
C	constant related to the net heat of sorption
E^b	tensile stiffness [kN/m]
E^W	tensile stiffness index [MNm/kg]
ΔF	increase in tensile force [N]
$\overline{F}_T$	mean breaking Force [N]
l_φ	length at equilibrium for given relative humidity [mm]
l_0	length at 50% relative humidity [mm]
K	constant
m_0	mass [g]
m_1	mass after drying [g]
M	molecular weight [g/mol]
p_v	vapor pressure [Pa]
$p_{v,max}$	maximum vapor pressure [Pa]
p_0/p	relative vapor pressure [-]
r	radius [m]
R	gas constant [J/(mol K]
S_{max}	maximum slope of tensile force over elongation [N/mm]
T	temperature [K]
$\overline{U}_T$	mean breaking energy [mJ]
w	grammage, mass per unit area [g/m^2]
W_T^b	tensile energy absorption [J/m^2]
W_T^W	tensile energy absorption index [J/kg]
X	hygroexpansion [%]
γ	surface tension [N/m]
$\Delta\delta$	increase in elongation [mm]

θ	contact angle [°]
ρ	density [kg/m^3]
σ_T^W	breaking force index [kNm/kg]
φ	relative humidity [%]
BET	Brunauer–Emmett–Teller equation
CD	cross direction
GAB	Guggenheim–Andersen–de Boer equation
LDPE	low density polyethylene
MD	machine direction

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