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# RELATIONSHIP BETWEEN STRUCTURAL PARAMETERS AND WATER ABSORPTION OF BLEACHED SOFTWOOD AND HARDWOOD KRAFT PULPS

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## ABSTRACT

The influence of porosity, relative bonded area and air permeability on water absorption of unbeaten and beaten bleached kraft pulps from different wood species used for tissue paper production was investigated. The water absorption was determined by four different methods such as absorption of water after immersion, initial water absorption, water absorption capacity and saturation rate with water. Linear dependences between water absorption parameters and the structural parameters of individual pulps as well as all tested pulps were obtained. The correlation coefficients obtained within individual bleached kraft pulps were from 0.95 to 1.00 and within the whole group of pulps were from 0.86 to 0.98. Relative bonded area and air permeability were found to be the most suitable parameters for predicting of initial water absorption and saturation rate with water.

KEYWORDS: Bleached kraft pulp, water absorption, porosity, relative bonded area, air permeability, prediction.

## INTRODUCTION

The structural properties of paper play a significant role of its functional properties which are influenced by fibre characteristics. Fibre length, fibre and lumen width, and wall thickness are the key characteristics of fibres. A high coarseness fibre will have improved bulk, porosity, and absorbency, drainage, opacity, low fibre bonding strength and potentially increased picking and linting of papers. Softwood fibres are long with relatively wide lumens, 2.5-4.0 mm long and with coarseness 160-350 $\mu g \cdot m^{-1}$. Hardwood fibres are short, and thick walled, 0.8-1.5 mm long and with coarseness 90-160 $\mu g \cdot m^{-1}$. Within European hardwoods, birch fibres have significantly lower coarseness (80-120 $\mu g \cdot m^{-1}$) and contrary, a higher coarseness is typical for beech (average 138 $\mu g \cdot m^{-1}$) and eucalyptus fibres (145-155 $\mu g \cdot m^{-1}$).

Runkel ratio (RT) is important indices derived from the fibre dimension to determine the sustainability of material for high quality of pulp and paper making and is calculated from wall thickness/fibre lumen width. The fibres with RT < 1 are flexible and collapse more easily and forms paper with higher bonded area. Fibres with RT > 1 are difficult to collapse and the result is more bulky paper with less bonded area.

An important step in papermaking is beating process of pulp fibres. The aim is to apply pressure and shear force to the fibres to obtain paper conformability, bonding capacity and the formation of a bulky or dense structure. The beating process shortens the fibres and increases the specific surface and thus also relative bonded area. Relative bonded area (RBA) which is the fraction of the total available fibre surface that is bonded can be used for predicting the tensile strength. The larger RBA enables more contact of fibres to form fibre network resulting in less porous system. The nitrogen absorption method by the BET (Brunauer 1938) and the light scattering method (Ingmanson and Thode 1979) can be used to measure RBA. By using faster light scattering method, RBA is being evaluated by Eq.1 :

$$RBA = \frac{S_0 - S}{S_0} \times 100 \qquad (\%) \tag{1}$$

where: S - light scattering coefficient for sheet ($m^2 \cdot kg^{-1}$) which was prepared in water and thus the interfibre bonds were formed,

$S_0$ - light scattering coefficient of fibres in unbound states.

One from the method of its determination has been extrapolating data for the light scattering coefficient as a function of tensile strength to zero tensile strength (Ingmanson and Thode 1979). This method of $S_0$ evaluation does not consider the fact that increased beating as well as pressure during paper sheet formation leads to the collapse of lumen and fibre fibrillation which affects the light scattering. There may also be a situation where the two fibre surfaces gets closer than wavelength < 200 nm, and optical but not molecular contact is made (Davison 1980). It results in a lower light scattering which in fact was not caused by the formation of interfibre bond. The presence of fines can also increase the light scattering. At work of Batchelor and Kibblewhite (2006) the light scattering coefficient, corrected for the total surface area of the fibres available for scattering, was plotted against sheet density, and corrected to fibre shape.

Liquid absorption of paper occurs due to the fact that cellulose molecules contain hydroxyl groups that allow it to form hydrogen bonds with water. At the same time, water is soaked into the pores. The pore volume inside the fibre consists of the lumen volume, small cavities and cracks in the cell wall of the fibres. Intrafibre pore volume consists of the lumen volume and small voids in the fibre cell wall, e. g. cracks between lamellas and still smaller voids within lamellas. The permeability (liquid flow through the fibrous network) mainly involves interfibre pores. In the case of tissue paper, initially the liquid flow or diffusion of vapor into the through interfibre pores occurs, and followed by the diffusion of vapor into the blind pores of the fibres surface. The water can also be absorbed by the cavities on the paper surface. Flexibility and collapsiveness of fibres as well as the surface properties of the fibres and the presence of fine particles, affect the paper permeability. Not only the porosity itself but also the pore size distribution plays an important role in the absorption of liquids. Two methods of pore size distribution measurement were compared, namely capillary porometry and mercury intrinsic porosimetry of cardboard (Gigac et al. 2017). The fine pores content of offset (Gigac et al. 2011), inkjet papers (Gigac et al. 2014, 2016) as well as barrier papers (Gigac et. al. 2018) can be quantified by a measurement of the ultrasound signal intensity during contact of 16% isopropyl alcohol water solution with paper surface. An ideal pore size of high absorptive papers should be a compromise between the presence of large and small pores. During water absorbing, the large pores quickly soaked large amounts of liquid (Milichovský 1995), but gravitational forces act against its remaining inside of the pore. Gradually, the water is transferred to smaller nanopores (Milichovský 1995), where capillary forces enable retain fluid there despite the effects of gravitational forces. Overlaying of two to three layers of paper above itself allows to absorb multiple amount of liquid due to the formation of an interface. The absorbent flowing in porous tissue papers was studied in detail (Beuther et al. 2010).

The aim of the work was to compare the influence of structural parameters on the water absorption of unbeaten and beaten bleached kraft pulps from different hardwood and softwood species and the possibility of predicting water absorptions by these structural parameters was considered.

## MATERIAL AND METHODS

### Bleached kraft pulps

Bleached beech kraft pulp from Bukocel, bleached birch kraft pulps Södra Gold (Birch I) and Botnia Nordic AKI (Birch II), bleached eucalyptus kraft TCF pulps Pontevedra (Eucalyptus I) and Celtejo (Eucalyptus II). Bleached Pine kraft pulp made from young pine wood Södra Black (Pine), bleached nordic Pine kraft pulp with the addition of spruce up to 45% Botnia Nordic pine + JNO (pine-spruce).

### Methods

*Pulp beating and hand sheets preparation*

The bleached softwood and hardwood kraft pulps were beaten to 20, 25 and 30°SR in a laboratory Jokro mill according to ISO 5264-3 (1979). The hand sheets (60 $g{\cdot}m^{-2}$) from unbeaten and beaten pulps were prepared in the sheet former Rapid Köthen according to the standard ISO 5269-2 (2004).

### Analysis

*Apparent density* of paper was evaluated from thickness and basic weight. Thickness was determined according to the standard STN EN ISO 534 (2012) and basic weight according to the standard STN EN ISO 536 (2012). *Porosity (void fraction) ε* was calculating according to literature (Daub et al. 1986). *Relative bonded area (RBA)* was calculated according to Eq. 1 based on the light scattering coefficient S and $S_0$. The light scattering coefficient S ($m^2{\cdot}kg^{-1}$) was measured on Elrepho 2000 colorimeter according to ISO 9416 (2017). The light scattering coefficient $S_0$ was evaluated from the linear dependence between the light scattering coefficient S and the tearing index where the light scattering coefficient $S_0$ was read when the tearing index was extrapolated to zero value. *Air permeability* was measured by method of Gurley according to the standard ISO 5636-5 (2013).

*Water absorption* after immersion for time of 10 s was determined according to the standard ISO 5637 (1989). *Water absorption capacity* was determined by basket-immersion test method according to the standard ISO 12625-8 (2010).

*Water penetration dynamics*

The water penetration dynamics were measured by the ultrasound device PDA C.02 (Emtec, Radnor, PA, USA) with a frequency of 2 MHz. The test liquid was water with a surface energy of 72 mJ·m$^{-2}$. Ultrasound signal intensity (USI) change was obtained at 43 ms - 60 s using the SC algorithm. The algorithms for calculating initial water absorption (HYG 1) and saturation rate with water of pulp sheets (HYG 2) were designed from the USI drop in 200 ms and time at minimum value of USI. The resulting values were obtained from the average value on the front and back of the pulp sheet. Fig. 1 is the illustration of the time course of the change in intensity of the ultrasound signal at the interaction of water with the front side of sheet surface from the mixed Pine-spruce pulp. An example of a comparison of the time course of the change in intensity of the ultrasound signal at the interaction of water with the front side of sheet surface from eucalyptus pulp (Eucalyptus II) and the mixed pulp (pine- spruce), beaten to 30°SR at 1 and 10 seconds, is shown in Fig. 1b.

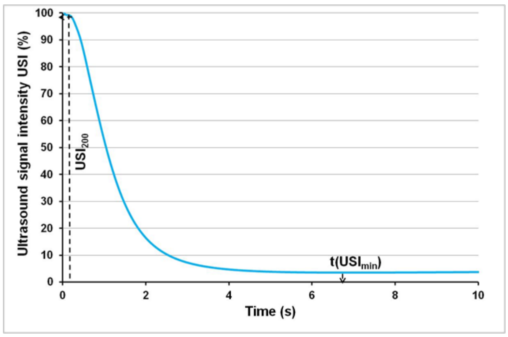


*Fig. 1a: Time course of ultrasound intensity signal in interaction of water with the front side surface of mixed pine-spruce pulp, beaten to 30°SR.*

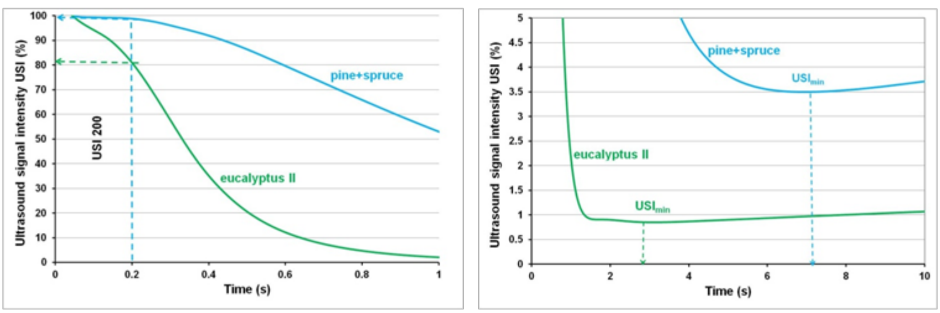


*Fig. 1b: Time course of ultrasound intensity signal in interaction of water with the front side surface of Eucalyptus II pulp and mixed pine-spruce pulp, beaten to 30°SR in time of 1 s (left) and to 10 s (right).*

*Initial water absorption* (HYG 1) was evaluated from the ultrasound signal intensity in time of 200 ms (($USI_{200}$) by the Eq. 2:

$$HYG\ 1 = \frac{100 - USI_{200}}{0.2} \quad (\%) \tag{2}$$

where 0.2 is the constant. The smaller $USI_{200}$ gives the higher initial water absorption.

Saturation rate with water of pulp sheet (HYG 2) was calculated by the Eq. 3:

$$HYG\ 2 = 10 \times \frac{1}{\sqrt{t_{USImin}}} \qquad (s^{-1}) \tag{3}$$

where $t_{USImin}$ is the time when the minimum of the ultrasound intensity signal is reached. The higher HYG 2 gives the higher water absorption rate.

## RESULTS AND DISCUSSION

### Prediction of water absorption for individual pulps

For evaluation, unbeaten as well as beaten bleached softwood and hardwood kraft pulps which are used in the production of tissue paper for which water absorption is important were selected. The linear relationship between porosity and water absorption after immersion of hand sheets from individual pulps is in Fig. 2a with correlation coefficient 0.968-0.997. The unbeaten eucalyptus pulps and beech pulp have higher water absorption after immersion (378-392%). As opposite, the unbeaten Birch II pulp had significantly lower water absorption after immersion which is result of fibres collapse due to applied pressure during hand sheets formation. Birch fibres with thin wall and low coarseness collapse more readily to give dense bonded hand sheets due to their flexibility and at the same time it gives a less bulky structure resulting in less porosity. After beating, porosity and water absorption after immersion markedly decreased. At the same porosity above 0.62, the highest water absorption after immersion had Eucalyptus II pulp. At porosity values lower than 0.57, the highest absorption after immersion had Birch I pulp. When compared eucalyptus pulps, beech pulp and softwood pulps within range of porosity 0.57-0.67, significantly highest water absorption after immersion of Eucalyptus II pulp was shown. The smaller curve slopes for birch pulps show that the influence of porosity change on water absorption after immersion was less significant. The results showed that the comparison of water absorption after immersion at the same porosity of all seven pulps was possible only within a narrow range of porosity values. The reason is that by beating, the porosity values of individual pulps have been obtained in a narrow range. Thus, we decided to compare the order of pulps beaten to 20°SR in terms of porosity, RBA, air permeability and water absorption. The drainage resistance 20°SR was selected as optimal for water absorption of all tested pulps. At this drainage resistance, the order of pulps considering porosity was: Eucalyptus I = Beech > Eucalyptus II > Pine = Pine-spruce > Birch I = Birch II; while the order of pulps considering water absorption after immersion was: Eucalyptus II > Eucalyptus I > Beech > Birch I > Pine > Pine-spruce > Birch II.

The relationship between RBA and water absorption after immersion is illustrated in Fig. 2b. Water absorption after immersion decreased with RBA increasing as the result of pulp beating. The unbeaten birch pulps had the highest RBA (18.5 and 19.6%) due to higher fibre flexibility with a better tendency to collapse which enables to create larger bonded area. Birch fibres have relatively thin walls 2.0-3.6 μm when compared to that of eucalyptus (4.3-5.0 μm) or beech (3.0-5.5) and fibres are wider. Moreover, average Runkel ratio of birch fibres is below 1 (0.84). The combination of these facts enables birch fibres collapse more easily upon applied pressure at hand sheet formation as well as beating, resulting in more dense paper. As opposite, during beating of eucalyptus and beech fibres with average Runkel ratio 1.75 and 2.13 and higher fibre wall thickness (3.0-5.0 μm), more porous pulp sheet is formed. Similar higher RBA as birch pulps was reached by beating of mixed Pine-spruce pulp. High correlation coefficient 0.982-0.999 of the relationship between RBA and water absorption after immersion (Fig. 2b) was found.

The change of RBA had the smaller effect on water absorption after immersion of birch pulps as seen from the lesser slope of the curve. At the same RBA value below 40%, the Eucalyptus II pulp had the highest water absorption after immersion. On the contrary, the lowest water absorption after immersion of Birch II pulp was obtained up to RBA value of 30%. Above this RBA value, water absorption after immersion of Birch II pulp was the similar to Pine and Beech pulp and of Eucalyptus II pulp was similar to Birch I. At drainage resistance 20°SR, the order of pulps considering RBA was: Eucalyptus I < Beech = Eucalyptus II < Pine < Birch I < Birch II < Pine-spruce. This order is similar to the order of pulps in terms of water absorption after immersion in some extent, with exception of both eucalyptus and beech pulps and differs from the order considering porosity. The correlation coefficients of relationship between RBA and water absorption after immersion of individual pulps were 0.991-0.999.

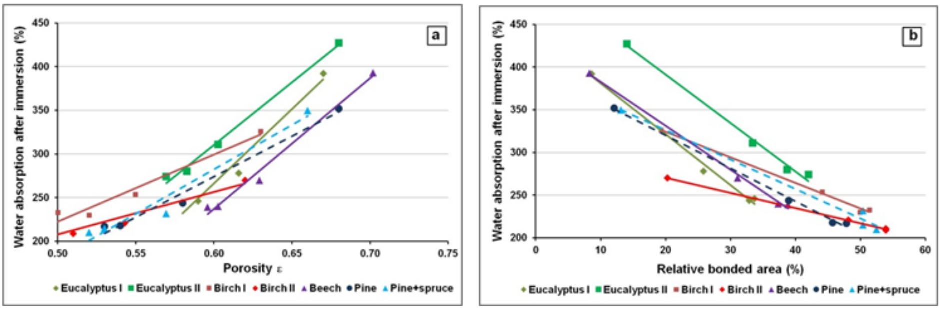


*Fig. 2: The relationship between water absorption after immersion of individual pulps and a) porosity; b) relative bonded area (RBA).*

Permeability of pulp sheets was evaluated by air permeability resistance method. Fig. 3 presents the relationship between water absorption after immersion and air permeability with correlation coefficients 0.980-0.999. Air permeability decreased with beating in accordance with porosity decreasing. The birch pulps had very low air permeability up to values of 75-82 μm.Pa$^{-1}$·s$^{-1}$. It was possible to compare the water absorption after immersion of all pulps in only a narrow range of air permeability. Water absorption after immersion of the Birch I pulp increased more with air permeability as other pulps. When compared the both eucalyptus and softwood pulps and beech, where higher air permeability was obtained, Eucalyptus II had significantly higher water absorption after immersion.

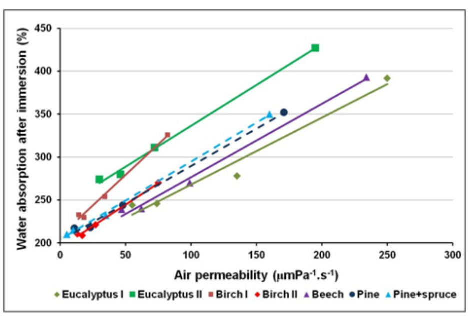


*Fig. 3: The relationship between water absorption after immersion and air permeability of individual pulps.*

At air permeability value of about 70 μm·Pa$^{-1}$·s$^{-1}$, the beaten Eucalyptus II pulp and unbeaten Birch II pulp had the similar water absorption after immersion (326%). The order of pulps at 20°SR considering air permeability was: Eucalyptus I > Beech > Eucalyptus II > Pine > Birch I > Birch II > Pine-spruce and this order was similar to the order of relative bonded area.

The relationship between porosity and water absorption capacity is presented in Fig. 4a. With porosity increasing, water absorption capacity increased, the correlation coefficients were in the range of 0.968-1.0. The porous unbeaten Eucalyptus I, Eucalyptus II, Beech and Pine pulps had higher water absorption capacity (430-443%) when compared to Pine-spruce pulp (410%) and birch pulps (302 and 374%). As all pulps can be compared only within a narrow range porosity of 0.57-0.62, in this case, the highest water absorption capacity at the same porosity had the Birch I pulp. At the same porosity 0.68, highest water absorption capacity had Eucalyptus I pulp. There was less steepness of the graph curves of both birch pulps, which again points to the fact that the influence of porosity on the water absorption capacity was not as significant as it was with other pulps. The water absorption capacity of beech pulp was the lowest within whole range of porosity.

Fig. 4b illustrates the relationship between RBA and water absorption capacity of individual pulps. With increasing of RBA, water absorption capacity decreased, the correlation coefficients were high (0.974-0.998). At the same RBA value below 40%, Eucalyptus II pulp had only slightly higher water absorption capacity when compared to other pulps and Birch II pulp had the lowest value. At the same values of RBA below 33%, Eucalyptus I, Pine and Beech pulps had similar water absorption capacity which declined more rapidly with RBA increasing. The influence of RBA change of the birch pulps on the water absorption capacity was less pronounced. At 20°SR, the order of pulps considering water absorption capacity was: Eucalyptus II > Eucalyptus I > Beech > Pine > Birch I = Pine-spruce > Birch II and was similar to the order of water absorption after immersion.

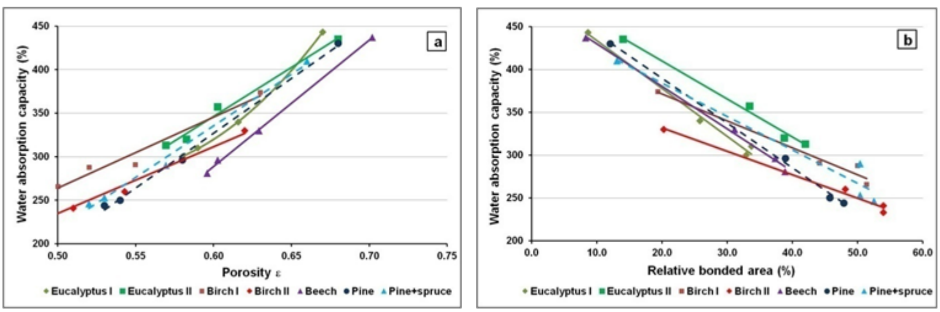


*Fig. 4: The relationship between water absorption capacity of individual pulps and a) porosity; b) relative bonded area (RBA).*

Fig. 5 shows the relationship between air permeability and water absorption capacity of individual pulps. Water absorption capacity increased with air permeability increasing, correlation coefficients were in the range of 0.982-0.999. The course of the water absorption capacity change depending on the air permeability of the Eucalyptus I and Beech pulp was identical. At lower air permeability (approximately 50 μm·Pa$^{-1}$·s$^{-1}$), water absorption capacity of Eucalyptus II and Birch I pulp is higher when compared to other pulps similarly as was the highest water absorption after immersion (Fig. 3). The more markedly trend of water absorption capacity increasing with air permeability rising was observed for both types of birch. The order of air permeability increasing as well as RBA decreasing of pulps at 20°SR was different as the order of water absorption capacity and water absorption after immersion increasing when compared eucalyptus pulps and beech pulp.

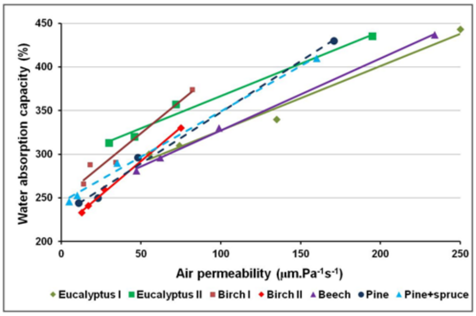


*Fig. 5: The relationship between water absorption capacity and air permeability of individual pulps.*

Fig. 6a shows the relationship between initial water absorption and porosity of individual pulps, where correlation coefficients in the range of 0.961-1.0 were reached. The initial water absorption rapidly increased with porosity increasing. When unbeaten pulps are compared, both eucalyptus pulps and beech pulp showed highest initial water absorption (327-393%) due to high porosity, similarly as highest water absorption after immersion and water absorption capacity. Unbeaten Birch II pulp had the lowest initial water absorption (278%) which had low porosity in comparison to other unbeaten pulps. After beating, initial water absorption decreased rapidly. At the same porosity within range 0.52-0.62, initial water absorption of Birch I pulp was significantly higher in comparison to other pulps. The lowest initial water absorption had Beech pulp within whole range of porosity and also Pine pulp at the porosity 0.65 and more.

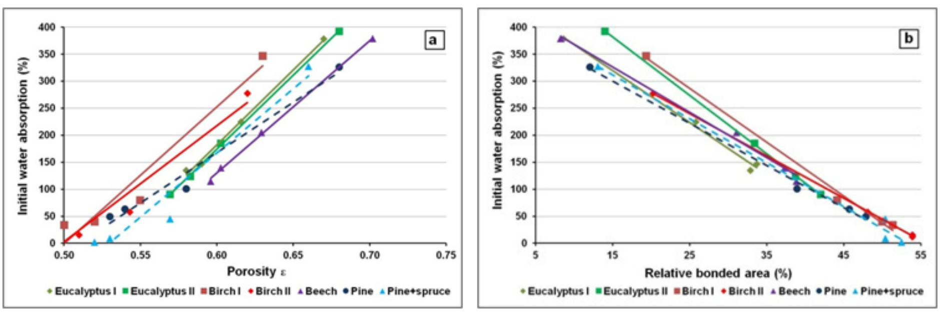


*Fig. 6: The relationship between initial water absorption of individual pulps and a) porosity; b) relative bonded area (RBA).*

With RBA increasing, the initial water absorption decreased (Fig. 6b). The correlation coefficients between RBA and initial water absorption were in the range of 0.995-1.0. At the same RBA within values of 20-40%, the Birch I pulp had the highest initial water absorption. When compared pulps at 20°SR, the order of pulps considering initial water absorption was: Eucalyptus I > Beech > Eucalyptus II > Pine > Birch I> Birch II > Pine-spruce. This order is the same as in the case of RBA as well as air permeability but differs from the porosity order.

The high initial water absorption (379 and 392%) of unbeaten eucalyptus pulps and beech pulp is due to high porosity. These results are in accordance to high air permeability (195, 234 and 250 μm·Pa$^{-1}$·s$^{-1}$) as shown in Fig. 7. On the other hand, high initial water absorption had also unbeaten Birch I pulp (347%) with very low air permeability. The correlation coefficients between initial water absorption and air permeability of the pulps were within 0.990-1.0. There has been a very rapid decline of initial water absorption with a change of air permeability of the Birch I and Birch II pulp. The orders of pulps at 20°SR considering initial water absorption and air permeability were the same.

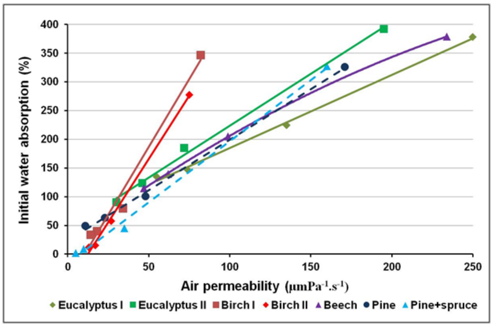


*Fig. 7: Relationship between initial water absorption and air permeability of individual pulps.*

The relationship between porosity and saturation rate with water of individual pulps with correlation coefficients in the range of 0.965-0.998 is shown in Fig. 8a. Among the unbeaten pulps, the highest saturation rate with water had Eucalyptus I (12.7 s$^{-1}$) and Beech (12.8 s$^{-1}$) pulps, which is related to their high porosity (0.66 and 0.70). This result differs from measurement of water absorption after immersion, water absorption capacity and initial water absorption where the highest values were obtained for unbeaten Eucalyptus II pulp. At the same porosity values below 0.62, the highest saturation rate with water of Birch I pulp was achieved. When compared the pulps from eucalyptus, beech and softwood within porosity values above 0.62, the highest saturation rate with water was achieved for Eucalyptus I pulp. It is evident from Fig. 8a that the increase of porosity had a more pronounced effect on the saturation rate with water of Eucalyptus I and Beech pulp.

The saturation rate with water of Eucalyptus I and Beech pulp changed more rapidly with RBA than of other pulps (Fig. 8b). The correlation coefficients of the relationship between saturation rate with water and RBA for individual pulps were within the range of 0.986-1.0.

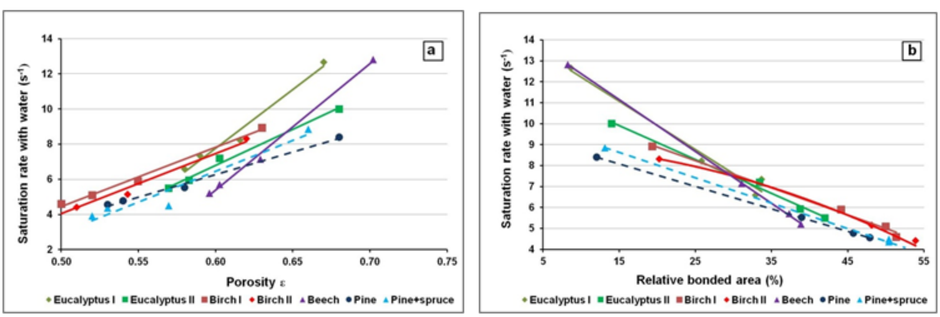


*Fig. 8: The relationship between saturation rate with water of individual pulps and a) porosity; b) relative bonded area (RBA).*

At higher ranges of RBA, saturation rate with water of individual pulps differed minimally (4.5-5.5 $s^{-1}$). The order of pulps at 20°SR considering saturation rate with water was: Eucalyptus I > Eucalyptus II = Beech > Birch I > Pine > Birch II > Pine-spruce and is similar to the order of RBA and air permeability.

The influence of air permeability on the change of saturation rate with water of the pulp sheets is shown in Fig. 9 and correlation coefficients from 0.991 to 1.0 were achieved. High saturation rate with water of unbeaten Eucalyptus I and Beech pulps corresponds to the higher air permeability resistance. By beating, air permeability of the pulps decreased and change of air permeability had more obvious effect on saturation rate with water of both birch pulps and beech pulp. The order of the pulps at 20°SR in the terms of air permeability decreasing similarly as relative bonded area increasing corresponded with the order of saturation rate with water decreasing.

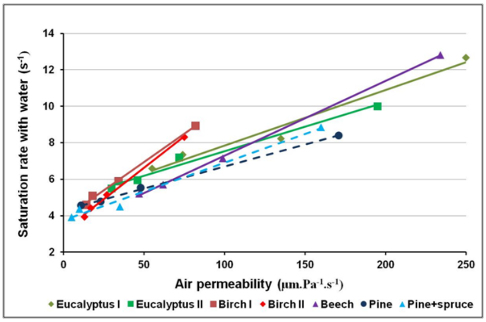


*Fig. 9: The relationship between saturation rate with water of individual pulps and air permeability.*

## Prediction of water absorption for all pulps

A good correlation between the water absorption parameters and the structural parameters for individual pulps was found. Therefore, there was an effort to find out whether it is possible to obtain a good correlation also for all pulps. In Tab. 1, the correlation coefficient between structural parameters as porosity (ε) and RBA, air permeability and water absorption parameters of all pulps is shown.

*Tab. 1: Correlation coefficient between the water absorption parameters and the structural parameters for all pulps.*

| Properties | Water absorption after immersion (%) | Water absorption capacity (%) | Initial water absorption (%) | Saturation rate with water ($s^{-1}$) |
|---|---|---|---|---|
| Porosity | 0.892 | 0.940 | 0.949 | 0.897 |
| RBA (%) | 0.906 | 0.949 | 0.986 | 0.946 |
| Air permeability ($\mu m \cdot Pa^{-1} \cdot s^{-1}$) | 0.923 | 0.939 | 0.920 | 0.952 |

The linear relationship was found and the correlation coefficients of pulps ranged from 0.892 to 0.986 which were lower than for individual pulps. The best correlation was obtained between RBA and initial water absorption.

## CONCLUSIONS

For the tissue paper production, the selection of pulps as well as conditions of technology is important. Linear relationship between structural and water absorptions parameters showed a different slope of curves for individual pulps.

The influence of porosity and relative bonded area on the saturation rate with water and water absorption after immersion was more pronounced for Eucalyptus I and Beech pulp.

Increasing of relative bonded area and decreasing of porosity of both birch pulps resulted only in a moderate decrease of water absorption capacity compared to other pulps.

Increasing of air permeability had a more pronounced impact on initial water absorption, water absorption capacity and saturation rate with water of both birch pulps and on water absorption after immersion of Birch I pulp.

A higher correlation between the water absorption and structural parameters was found for individual pulps in comparison to all tested pulps. The order of the individual pulps at drainage resistance 20°SR in terms of initial water absorption and saturation rate with water decreasing is fully consistent with order of air permeability decreasing and relative bonded area increasing. The order of pulps in terms of decreasing of water absorption after immersion and water absorption capacity was partially in accordance with air permeability decreasing and relative bonded area increasing. This accordance was obtained at comparison of birch and softwood pulps, excluding beech and eucalyptus pulps. The results show that the relative bonded area and air permeability could be used to predict primarily initial water absorption and saturation rate with water, measured by the ultrasound method.

## ACKNOWLEDGMENTS

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