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DISPLACEMENT WASHING OF KRAFT PULP COOKED FROM BEECH WOOD

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ABSTRACT

The aim of this work was to investigate the displacement washing of kraft pulp manufactured from beech. The experiments were carried out in a laboratory washing cell. In order to characterise the pulp fibres, the degree of freeness, kappa number, fibre length, as well as the specific volume and specific surface of swollen fibres were determined as well.

The displacement washing process was described by displacement washing curves recorded for alkali lignin as a tracer. Mathematical treatment of the washing curves, obtained as a response to the step input signal, gave the Péclet number characterising the displacement of alkali lignin from the pulp pad. For detailed description of the displacement washing process, further parameters, such as the traditional wash yield, axial dispersion coefficient, as well as the mean residence time and the space time, were also evaluated.

The results obtained for beech pulp were compared with those for softwood pulp published earlier. With respect to different chemical composition and morphological characteristics affecting both concentration of alkali lignin in mother liquor and physical properties of pulp pad, the displacement washing efficiency of hardwood pulp was found to be unambiguously greater in comparison with softwood pulp.

Keywords: beech; displacement washing efficiency; pulp.

INTRODUCTION

The pulp suspension coming from the digester contains both fibres and black liquor containing inorganic chemicals and a large amount of organic substances. The black liquor is removed from the pulp suspension in the subsequent washing and led to the chemical recovery system, where cooking chemicals and energy are recovered. Efficient washing reduces the carry-over of black liquor with the pulp resulting in a decreased consumption of chemicals in bleaching and also in oxygen delignification.

With respect to the practical and theoretical meaning, reasonable attention has been paid to the investigation of the displacement washing of pulp in the last several decades. Experiments were performed on pulp pads formed from fibres manufactured by kraft cooking of pine (GRÄHS 1976), spruce (POIRIER *et al*. 1987, TRINH *et al*. 1987, 1989), a blend of spruce and pine (POTŮČEK 1997, 2003, POTŮČEK, MARHANOVÁ 2002, POTŮČEK, PULCER 2004), and a blend of hardwood and softwood (LEE 1979, 1984). However, up to now the main volume of knowledge concerned the washing of softwood pulps, whereas the washing of hardwood pulps has been explored only marginally. Thus, very little effort has been devoted to the investigation of the influence of chemical composition and morphological characteristics of wood fibres upon the displacement washing efficiency.

In the case of long cylindrical particles such as pulp fibres, the particles are not geometrically similar, since the length-to-width ratio may vary very significantly for hardwood, such as beech, and softwood fibres as demonstrated in Table 1. Therefore, in the case of randomly oriented fibres, an unpredictable labyrinth of the pores of various tortuosity forms a void spaces of the bed. Moreover, higher Runkel ratio fibres are stiffer and less flexible than the lower Runkel ratio fibres.

Tab. 1 Morphological characteristics of selected wood fibres (BLAŽEJ, KRKOŠKA 1989).

wood	fibre length, *L*, mm	fibre width, *W*, µm	wall thickness, *T*, µm	width of lumen, *I*, µm	slenderness index, *L*/*W*	***Runkel ratio**, 2 T/I*
beech	1.05	19.1	6.5	6.1	35	2.10
spruce	3.39	35.0	4.0	27.0	97	0.30
pine	3.38	38.0	4.9	28.2	89	0.35

In our most recent paper (POTŮČEK, MIKLÍK 2010), we focused on the displacement washing of pulp fibres cooked from a blend of hardwoods. In the present paper, the results measured for displacement washing of beech pulp are presented and are compared with those obtained earlier (POTŮČEK, PULCER 2004) for softwood pulp cooked from a blend of spruce and pine.

EXPERIMENTAL

Besides different morphological properties, hardwood and softwood fibres have different chemical composition. For chemical analyses, a small portion of beech chips was grounded to powder, and then passed through a 40-mesh screen as recommended by the relevant Tappi test method T 257 cm-02 (Tappi 2004). The ash content was determined according to Tappi test method T 211 om-02 (Tappi 2004). The solvent extractives content, which is a measure of waxes, fats, resins, oils, tannins and certain other ether-insoluble components, was determined gravimetrically after successive Soxhlet extractions with an ethanol-toluene mixture according to Tappi test method T 204 os-76 (Tappi 2004). The acid-insoluble lignin content was determined by hydrolysing the carbohydrates with 72% sulphuric acid in accordance with Tappi test method T 222 om-02 (Tappi 2004). The holocellulose content was calculated for the total sum to be 100 %. Table 2 summarises chemical composition of beech which belongs among hardwoods, and for spruce, and pine chips used in pulp cooking earlier (POTŮČEK, PULCER 2004).

After handsorting, the middle fraction of beech chips was used to cooking runs conducted on a laboratory digester consisting of six 0.75-L stainless steel autoclaves of 100 g capacity, submerged in an oil bath heated by electric coils. The cooking conditions were as follows: 16 % active alkali on oven-dried wood, 4 : 1 liquor-to-wood ratio, and 170 °C cooking temperature. The temperature regime was as follows: 55 min heating to 135 °C, 25 min dwelling at this temperature, 20 min heating to 170 °C, and then dwelling at cooking temperature. After cooking, the pulp was thoroughly washed with water, screened and dewatered to a consistency of 26 %.

Tab. 2 Chemical composition of selected woods.

Wood	Ash, %	Extractives, %	Acid-insoluble lignin, %	*Holocellulose, %*
beech	0.64	2.64	24.47	*72.25*
spruce	0.41	2.70	30.44	*66.45*
pine	0.25	10.37	29.53	*59.85*

The displacement washing experiments were carried out in a laboratory washing cell. The dewatering ability of unbeaten pulp fibres was characterised by an SR parameter of 14. The swelling capacity of pulp fibres was described by WRV equal to 1.34 g/g. The degree of delignification was expressed in terms of kappa number, equal to 14.1, according to Tappi test method T 236 om-99 (Tappi 2004). Using the Kajjani instrument, the arithmetic average length of hardwood fibres in the wet state was 0.51 mm, while the weighted average length was 0.73

mm. The effective specific surface of hardwood fibres determined according to INGMANSON (1953) had a value of 3.76×10^5 m^{-1}.

Properties of black liquor were as follows: solids content of 15.3 % (of which, the ash made up 37 %, and organic substances 63 %), density 1 071 kg·m^{-3} at 20 °C, and pH value 12.2. The concentration of alkali lignin was measured to be 27 g·dm^{-3}.

A laboratory washing cell described in details earlier (POTŮČEK 1997) with an internal diameter of 36 mm and an adjustable bed height was used. Pulp beds were formed by filtration from a dilute suspension of kraft pulp in black liquor. In all runs, the beds were compressed to a final desired thickness of 30 mm. Then, the consistency, *i.e.*, mass concentration of pulp fibres in the bed, varied within the limits from 10.8 % to 11.5 %. Distilled water at a temperature of 20 to 22 °C was employed as wash liquid.

In order to describe the displacement washing process, the stimulus-response method was chosen. To start the washing run, wash liquid was distributed uniformly through the piston to the top of the bed, approximating a step change in concentration. At the same time, the displaced liquor was collected at different time intervals from the bottom of the bed. In order to measure the time dependence of alkali lignin concentration in the outlet stream, the samples of the liquor discharging the bed were analysed for alkali lignin, by means of Cintra spectrophotometer, operating at a wavelength of 295 nm.

After completing the washing experiment, the volumetric wash liquid flow rate was measured gravimetrically at a pressure drop of 7 kPa to determine permeability and average porosity of pulp bed. Analogous measurements of the volumetric flow rate, however, at various consistencies of the bed were focused on the determination of effective specific volume of the pulp fibres. The effective specific volume of hardwood fibres in the swollen state determined according to INGMANSON (1953) was found to be 3.25×10^{-3} m^3·kg^{-1}.

RESULTS AND DISCUSSION

Washing curves

A response to step change in concentration provided time dependences called washing or also breakthrough curves. In order to compare displacement washing process for various wash liquid velocity, the washing curves were plotted as the dependence of dimensionless concentration of a tracer, in our case alkali lignin, in the outlet stream expressed as ρ_e/ρ_0, against to the wash liquor ratio, *RW*, defined as the mass of wash liquid passed through the bed at that time divided by the mass of liquor originally present in the bed.

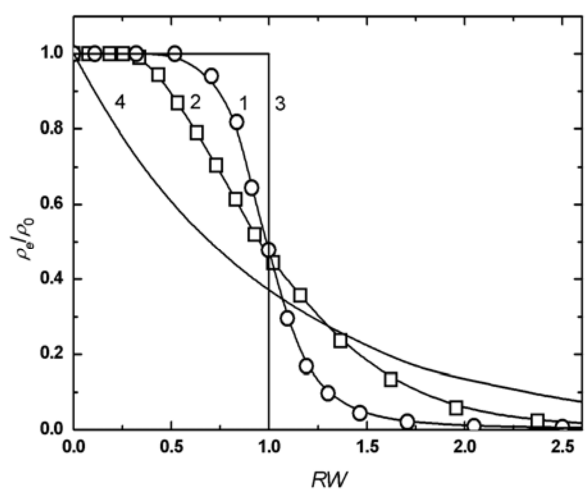

Fig. 1 Typical displacement washing curves: 1 – beech pulp, 2 – softwood pulp (POTŮČEK, PULCER 2004), 3 – plug flow, 4 – perfectly mixed flow

A typical breakthrough curve measured for beech pulp is shown in Fig. 1. The first portions of the liquor discharged from the pulp bed are of the same concentration as the mother liquor. Then, the concentration of a solute in the outlet stream drops very rapidly. In this washing period, it can be supposed that the major part of the mother liquor in interparticle voids is removed and replaced by the wash liquid. At the end of the washing, the solute adsorbed on the surface of the fibres, as well as the solute inside the fibre walls is being transferred by diffusion to the wash liquid surrounding the fibres. In this last period, the leaching operation prevails over displacement mechanism.

For comparison, a breakthrough curve measured for displacement of black liquor from pulp bed formed from softwood pulp fibres cooked from a blend of spruce and pine (POTŮČEK, PULCER 2004) is also demonstrated in Fig. 1. Our results showed that both systems differ markedly from one another. From Fig. 1 follows that the breakthrough curve for beech pulp fibres, as well as for a blend of hardwoods (POTŮČEK, MIKLÍK 2010) approaches that of plug flow. The flat profile of the breakthrough curve obtained for softwood pulp fibres having a kappa number of 32 is probably the result of the nature of pulp bed which can be characterised as an unmovable packing dumped randomly into the washing cell, along with black liquor occurring both in the void spaces and inside porous, compressible particles. A comparison of physical characteristics of pulp bed for hardwood and softwood fibres is given in Table 3. The ratio of the weighted to arithmetic average length, which is a measure of the polydispersity of fibre length, was 1.4, and 2.3 for hardwood and softwood fibres, respectively. With respect to larger polydispersity the greater axial dispersion in the case of softwood pulp can be achieved.

Tab. 3 Comparison of physical characteristics of beech and softwood pulp bed.

pulp	bed consistency, %	average bed porosity	specific surface of wet fibres, $m^2 \cdot kg^{-1}$	arithmetic average length of fibres, mm	weighted average length of fibres, mm
beech	10.8 – 11.5	0.47 – 0.61	1 220	0.51	0.73
softwood*	11.8 – 13.9	0.64 – 0.67	794	1.46	3.35

* POTŮČEK, PULCER 2004

The shape of the washing curve can be characterised in terms of the dimensionless Péclet number, derived from the mass balance of the tracer for a given system in unsteady state, in the following form

$$Pe = \frac{hu}{D\varepsilon} \quad . \tag{1}$$

The Péclet number obtained on the basis of breakthrough curve characterises not only hydrodynamic conditions in the bed, but also, particularly, the ratio of the convective to the diffusive solute transport mechanisms. In accordance with the shape of washing curves, the Péclet number evaluated for hardwood pulp beds varied in the limits of 25 to 41, while the values of the Péclet number obtained for softwood pulp beds were in the range of 7 to 16.

The displacement washing curve area is directly proportional to the amount of alkali lignin removed from the bed. It must be emphasised that the washing experiments were finished at the wash liquor ratio equal to about 7 when the lignin concentration in the exit stream was lower than one thousandth of the initial lignin concentration in the fibre bed.

Wash yield

Quality of the displacement washing can be characterised by the wash yield. The traditional displacement wash yield, $WY_{RW=1}$, is defined as the amount of solute washed out at the wash liquor ratio equal to unity divided by the total amount of solute present in the bed at time equal to zero. This yield may be expressed as

$$WY_{RW=1} = \frac{\int_{RW=0}^{RW=1} \frac{\rho_e}{\rho_0} d(RW)}{\int_{RW=0}^{RW\to\infty} \frac{\rho_e}{\rho_0} d(RW)} \quad . \tag{2}$$

The influence of the Péclet number on the wash yield is shown in Fig. 2. In spite of the scatter in the data, in the case of beech pulp fibres covering a range of the Péclet number from 25 to 41, it is evident that the wash yield slightly increases with increasing Péclet number. The experimental points are located below the curve derived for the packed bed of non-porous particles by BRENNER (1962). In contrast to the packed bed of non-porous particles, when the washing process is reduced to the displacement mechanism and interfacial mixing between the displaced and displacing fluids, leaching may play a significant role in the case of compressible porous fibres in the swollen state.

Comparing the hardwood and softwood pulp fibres, the greater values of the wash yield were achieved for beech pulp, similarly as with a blend of hardwoods (POTŮČEK, MIKLÍK 2010). Presumably, lower values of the Péclet number covering the range from 7 to 16 measured for softwood pulp fibre bed (POTŮČEK, PULCER 2004) represent more bridgings between particles and greater variations in local voidages which promote the channelling phenomenon. Lower values of the wash yield reported for softwood pulp (POTŮČEK, PULCER 2004) can be also attributed to the high initial alkali lignin concentration of 48 kg·m^{-3} in contrast to 27 kg·m^{-3} in the case of beech pulp in the present paper. Besides the degree of delignification, the initial alkali lignin concentration is affected by lignin content in the wood fibres as it is demonstrated in Table 2. As previously reported (POTŮČEK 1997), the displacement wash yield decreases with increasing initial lignin concentration in the mother liquor. As for the values of the wash yield obtained in our work for the initial lignin concentration of 27 kg·m^{-3}, TRINH *et al.* (1989) reported the wash yield varying from 0.84 to 0.87 for initial lignin concentration of 25 kg·m^{-3} in the bed of softwood pulp fibres.

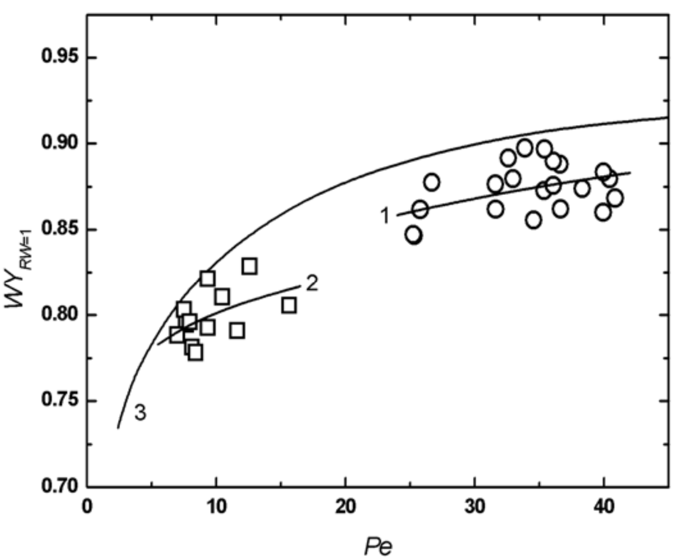

Fig. 2 Displacement wash yield as a function of the Péclet number: ○ beech pulp, □ softwood pulp (POTŮČEK, PULCER 2004), 1 – Eq. (3), 2 – Eq. (4), 3 – Eq. (5)

Hence, our results proved that the chemical composition of the wood material has a considerable influence on pulp washing. The chemical composition of beech, spruce, and pine differs from one another. However, the differences of chemical composition between softwood and beech are clearly evident. The highest Klason lignin content appears in spruce and is comparable with that of pine, while beech evidences the lowest lignin content. Generally, hardwood has lower lignin content, which is advantageous for delignification and for following washing for lower lignin concentration in the mother liquor.

On the basis of our own data measured for beech pulp bed, the effect of the Péclet number on the wash yield can also be expressed by the following correlation equation

$$WY_{RW=1} = 0.732\ Pe^{0.0503} \tag{3}$$

with a mean relative quadratic deviation equal to 1.4 %. Since the values of regression coefficients, which were evaluated by the least square method, represent an estimate of the real values, the 95 % confidence intervals were calculated as well. They are (0.709; 0.753) and (0.0416; 0.0591) for the coefficient and the power of the Péclet number, respectively. For the qualitative evaluation of the effect of the Péclet number on the wash yield, the following correlation equation

$$WY_{RW=1} = 0.73\ Pe^{0.039} \tag{4}$$

was developed for softwood pulp beds in the previous work (POTŮČEK, PULCER 2004) when softwood pulp was cooked industrially from a mixture of spruce and pine in a 4 : 1 mass ratio.

For comparison with the correlations given by Eqs (3), and (4), the theoretical wash yield evaluated by BRENNER (1962) for displacement of a solute from a packed bed of non-porous particles was expressed as a function of the Péclet number in the form

$$WY_{RW=1} = 0.736\ Pe^{0.0575} \tag{5}$$

for the Péclet number ranging from 3 to 80. In accordance with preceding papers (POTŮČEK 1997, 2003, POTŮČEK, MARHANOVÁ 2002, POTŮČEK, PULCER 2004), our results obtained for beech pulp bed confirmed again that the wash yield increases with the increase of the Péclet number, but the power of the Péclet number is below 0.1 for porous particles, as well as for non-porous ones.

Dispersion coefficient

In porous media, dispersion is created by both the microscopic differences in velocity which exist in the interstices between fibres and by large-scale or macroscopic effects such as channelling. The dispersion of lignin in packed beds with random packing was characterised by a dimensionless group, the Péclet number (Eq. (1)), containing the dispersion coefficient, D.

Figure 3 is a plot of the axial dispersion coefficient against the wash liquid superficial velocity, showing our results for beech and softwood pulps. In spite of the scatter in the results, the dependence of the dispersion coefficient on the wash liquid superficial velocity shows an increasing trend for both types of pulp bed in a good agreement with the papers (SHERMAN 1964, MAURET, RENAUD 2002). Comparing the values of dispersion coefficient measured for beech and softwood pulps (POTŮČEK, PULCER 2004), it seems that the difference in geometry, that is, in average pore size and in pore size distribution, resulted in higher values of the dispersion coefficient reached for softwood material.

From the results shown in Fig. 3, it follows that a linear dependence of the dispersion coefficient on the wash liquid superficial velocity can be assumed. The correlation between D in $m^2 \cdot s^{-1}$ and u in $m \cdot s^{-1}$ was derived in the forms

$$D = 1.54 \times 10^{-3} u \tag{6}$$

with a correlation coefficient of 0.91 for beech pulp fibres.

The linear function between the dispersion coefficient and the wash liquid superficial velocity obtained for beech pulp studied in the present work is in accordance with the results reported earlier for softwood pulp (POTŮČEK 1997, 2003, POTŮČEK, MARHANOVÁ 2002, POTŮČEK, PULCER 2004), as well as for pulp from a blend of hardwoods (POTŮČEK, MIKLÍK 2010). For comparison, the correlation in the form

$$D = 5.23 \times 10^{-3} u \qquad (7)$$

was developed for softwood pulp fibres (POTŮČEK, PULCER 2004). As mentioned above, the dispersion coefficient of alkali lignin, which is a measure of the rate at which a material will spread axially in the system, increases with increasing wash liquid superficial velocity.

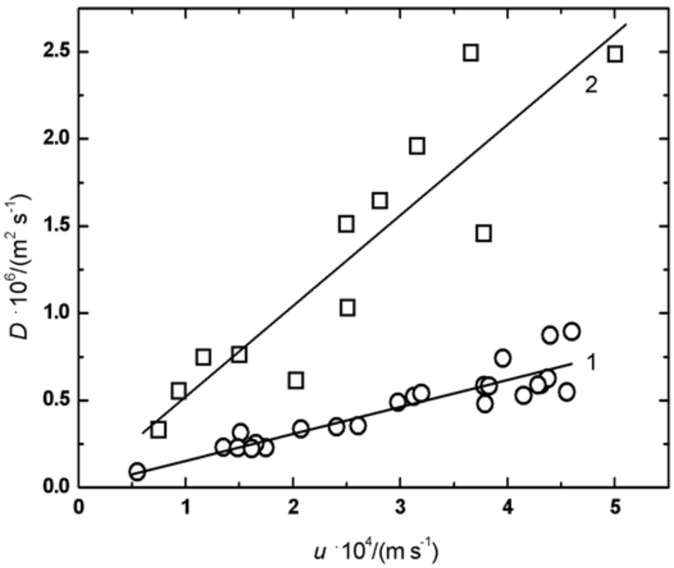

Fig. 3 Effect of superficial wash liquid velocity on axial dispersion coefficient: ○ beech pulp, □ softwood pulp (POTŮČEK, PULCER 2004), 1 – Eq. (6), 2 – Eq. (7).

Time parameters

Further parameters that seem to be useful tools suitable for describing the displacement washing mechanisms are time parameters. In addition, the existence of leaching in the pulp bed has an impact on the relation between the mean residence time of a solute defined by the following equation

$$t_m = \int_{t=0}^{t \to \infty} \frac{\rho_e}{\rho_0} dt \qquad (8)$$

and the space time, which is defined as the void volume of the bed divided by the volumetric flow rate of wash liquid. After rearranging it can be expressed as

$$\tau = \frac{\varepsilon h}{u} \; . \qquad (9)$$

The space time characterises a holding time of wash liquid and is usually equal to the mean residence time of the tracer without sorption on the surface of particles in the bed (POTŮČEK 1997, 2003, POTŮČEK, MARHANOVÁ 2002). For pulp fibres, however, when the leaching of a solute from the fibres exists, the mean residence time is always higher than the space time, as it follows from Fig. 4. From our experimental data, the relationship between the mean residence time and the space time for beech pulp may be expressed as follows

$$t_m = 1.47\, \tau \qquad (10)$$

with a correlation coefficient of 0.96. The correlation of data reported in the previous work

(POTŮČEK, PULCER 2004) is given by the following equation

$$t_m = 1.29\,\tau \tag{11}$$

derived for softwood fibres with a correlation coefficient of 0.95.

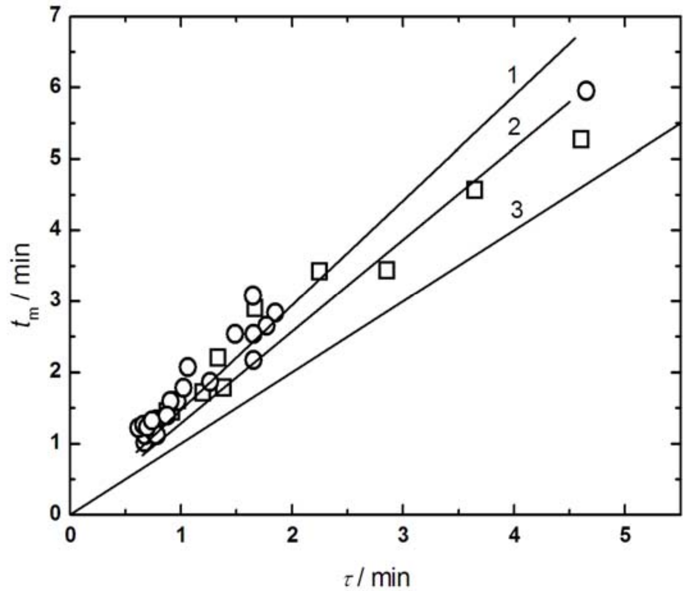

Fig. 4 Mean residence time as a function of space time: ○ beech pulp, □ softwood pulp (POTŮČEK, PULCER 2004), 1 – Eq. (10), 2 – Eq. (11), 3 – $t_m = \tau$.

Although softwood pulp had slightly higher consistency (POTŮČEK, PULCER 2004), the beds formed from shorter beech fibres and/or longer softwood pulp fibres have a porosity of 0.47 to 0.61 with an average of 0.56, and of 0.64 to 0.67, respectively, depending mainly upon the given pulp bed consistency. Lower values of porosity in beech pulp beds are in accordance with their lower permeability of 0.5×10^{-12} m^2 to 1.9×10^{-12} m^2, while, in the case of softwood pulp beds (POTŮČEK, PULCER 2004), their permeability varied within the range from 4.7×10^{-12} m^2 to 6.6×10^{-12} m^2. Similarly, at overall pressure drop of 7 kPa and the bed thickness of 30 mm, the average bed resistance including washing cell was 2.6×10^{10} kg·m^{-4}·s^{-1} and 0.6×10^{10} kg·m^{-4}·s^{-1} for beech and softwood fibres, respectively. According to the previous paper (POTŮČEK, PULCER 2004), the space time is directly proportional to the void volume of the pulp bed, while the mean residence time is directly proportional to the total volume of the spent liquor present in the bed. Hence, the greater difference between the space time and the mean residence time in the case of hardwood fibres may be ascribed to lower values of porosity in beech pulp fibres in comparison with softwood fibres.

In conclusion, the results obtained for beech pulp fibres are in a good agreement with those for a blend of hardwoods (POTŮČEK, MIKLÍK 2010). Visual observations showed that the bed formed from hardwood pulp fibres is more homogeneous in contrast to the bed formed from softwood pulp fibres in which channeling due to local inhomogeneities is often noticeable. The differences in the morphological properties and in chemical composition of hardwood and softwood fibres may also cause their different displacement washing efficiency, in the case of hardwood, positively influenced by lower lignin content in the mother liquor.

SYMBOLS

D axial dispersion coefficient, m^2·s^{-1}
h thickness of bed, m

Pe Péclet number defined by Eq. (1)
RW wash liquor ratio
t time from start of experiment, s
t_m mean residence time defined by Eq. (8), s
u wash liquid superficial velocity, $m \cdot s^{-1}$
$WY_{RW=1}$ wash yield at $RW = 1$ defined by Eq. (2)
Greek letters
ε average porosity of packed bed
ρ_e exit solute (in our case lignin) concentration from bed, $kg \cdot m^{-3}$
ρ_0 initial solute (in our case lignin) concentration in bed at $t = 0$, $kg \cdot m^{-3}$
τ space time, s

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