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DISPLACEMENT WASHING OF KRAFT PULP AT VARIOUS WASH WATER TEMPERATURE

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ABSTRACT

The aim of the paper was to investigate the influence of the wash liquid temperature on the displacement washing efficiency of unbeaten unbleached kraft coniferous pulp. The simple axially dispersed plug flow model comprising one dimensionless criterion, the Péclet number, was used for displacement washing of black liquor from packed fibre bed using pure wash water with temperature of 10 to 60 °C. The pulp fibre beds were characterised by their hydraulic resistance, average specific resistance, porosity, and equivalent pore diameter, influencing the flow of wash liquid through pulp bed. From breakthrough curves, the wash yield and the Péclet number were evaluated. The results obtained showed that the wash yield is mainly influenced by the Péclet number characterising the time dependence of alkali lignin removal from the pulp bed, however, the effect of the wash water temperature upon the wash yield was not proved in the given temperature range.

Key words: displacement washing, fibre bed resistance, kappa number, kraft pulp, wash yield.

INTRODUCTION

The displacement of a solute from within the pulp fibre pad is affected by several process variables. Besides pad consistency, pad thickness, wash liquid velocity, initial pad liquor concentration, and others, the influence of the wash liquid temperature on the washing efficiency is mentioned by several authors (LEE 1979; HAKAMÄKI, KOVASIN 1985; POIRIER *et al*. 1987; SMITH, DUFFY 1999; TRINH *et al*. 1989).

LEE (1979) reported that displacement efficiency characterised by the sodium-ion yield had an increasing trend with increasing the fibre pad temperature ranging from 20 to 55 °C. For a blend of softwood and hardwood kraft pulps, an efficiency increase depended on the fibre bed consistency. Lower increase in sodium-ion yield of 0.85 to 0.88 was at the pad consistency of 3.5%, while, for the consistency of 17%, an increase of 0.65 to 0.82 was achieved. The greater effect of temperature on washing efficiency at high pad consistency is ascribed to the relative greater portion of stagnant liquor in fibre pads comparing with low consistency when the free liquor occupying the interstitial spaces between fibres prevails.

HAKAMÄKI and KOVASIN (1985) investigated the effect of temperature within the limits of 30 to 90 °C for brownstock washing. Using a drum washer, an increase in washer capacity was observed with increasing the pulp temperature, but the effect of temperature on the washing efficiency was not evaluated.

SMITH and DUFFY (1999) performed the displacement washing of bleached kraft pulp at the black liquor temperature of 1 to 70 °C. The highest sodium-ion yield of 0.692 to 0.713 was recorded for the high black liquor temperature of 70 °C, while a temperature decreasing to 1 °C led to a decrease in the wash yield by 4.5 to 7%.

On the contrary, POIRIER *et al*. (1987) and TRINH *et al*. (1989) who investigated the displacement washing of kraft softwood pulp reported that the temperature changes over the range of 60 to 90 °C had no effect on the displacement washing efficiency determined for both sodium-ion and alkali lignin yields.

Different pulps and experimental techniques belong among several reasons for some contradictions in the results of various authors. Furthermore, it is possible that the effect of temperature on washing efficiency was obscured by interactions with other process variables (TRINH *et al*. 1989). In pulp-liquor pad, the spent pulping liquor is located both in the interstitial spaces between fibres and in the fibre walls. While the free liquor between fibres can be easily removed by displacement (SANTOS, HART 2014), the transport of the liquor from the fibre walls to the liquor flowing through pad pores is influenced by the diffusion of solutes, depending on the driving concentration force and temperature. The leaching of solutes from within fibre walls is relatively slow in comparison with displacement mechanism. Moreover, the diffusion coefficient of lignin in liquids is very low and is influenced markedly with increasing temperature above 70 °C (FAVIS *et al*. 1983).

Therefore, it seems that the displacement washing efficiency can be mainly affected by a longitudinal dispersion in the pulp fibre pad. Viscosity and density differences of displacing and displaced liquids can lead to the formation of fingering or channelling (NUNGE, GILL 1969; LEE 1984; POTŮČEK 2003). It can be supposed that channelling between dissimilar miscible liquids during displacement in a pad of wood pulp fibres occurs primarily as a result of preferential penetration of the wash liquid in regions of the porous structure having a higher permeability.

The objective of this paper was to investigate the effect of wash liquid temperature ranging of 10 to 60 °C on the displacement wash yield of alkali lignin. The influences of wash water temperature upon some characteristics affecting hydraulic resistance of the static pulp bed and mobility of wash water were evaluated as well.

EXPERIMENTAL PART

Displacement washing runs simulated under the laboratory conditions were performed in a cylindrical glass cell with inside diameter of 35 mm under constant pulp bed height of 30 mm. The fibre pulp bed occupied the space between the permeable septum and a piston, covered with 45 mesh screens (sieve opening of 0.354 mm, nominal wire diameter of 0.224 mm) to prevent fibre losses from the bed.

Pulp beds were formed from a dilute suspension of unbeaten unbleached kraft pulp in black liquor. Properties of black liquor were as follows: solids content of 21.4 % (of which ash presented 64 % and organic substances 36 %), density of 1097 kg m^{-3} at 22 °C, pH value of 12.0, and alkali lignin concentration 56 g dm^{-3}. Kraft softwood pulp was cooked industrially from a blend of spruce and pine. In accordance with ČSN ISO 302 the degree of delignification of kraft pulp was characterised by the kappa number of 24.9. Using the Kajaani instrument, the length of pulp fibres in the wet state was expressed in terms of the weighted and numerical averages, equal to 2.19 mm and 1.22 mm, respectively. The fibre coarseness had a value of 0.167 mg m^{-1}.

A sample of the wet kraft pulp with a consistency of about 42 % was disintegrated manually and then gently mixed with 100 cm^3 of the black liquor for approximately 20 min.

The 4% pulp suspension was poured into the washing cell and pulp pad was allowed to form by gravity drainage. The pad was compressed to a thickness of 30 mm and a consistency of 128 to 134 kg m^{-3}, with an average of 130 kg m^{-3}. The pulp beds were not mechanically conditioned and were used as formed.

Distilled water was used as wash liquid for all runs. The temperature of wash liquid prior to entering the washing cell was maintained at six different levels within the limits of 10 to 60 °C. To investigate the displacement washing process, the stimulus-response method was chosen. Distilled water was distributed uniformly through the piston to the top of bed at the start of the washing experiment, approximating a step change in alkali lignin concentration. At the same time the displaced liquor was collected at atmospheric pressure from the bottom of the bed through the septum. The washing effluent was sampled at different time intervals until the effluent was colourless. Using an ultraviolet spectrophotometer operating at a wavelength of 295 nm, samples of the washing effluent leaving the pulp bed were analysed for alkali lignin. At all wash liquid temperatures, washing runs were once replicated. Displacement washing experiments with pulp fibres including washing equipment were described in detail in the preceding paper (POTŮČEK 1997).

After completing the washing run, the volumetric flow rate of wash liquid was measured gravimetrically at the pressure drop of 7 kPa to determine a permeability and average effective porosity of the pulp bed. Analogous measurements at various consistencies of the bed were focused on the determination of the effective specific surface of pulp fibres based on fibre mass, a_{m}, and on fibre volume, a_{V}, according to INGMANSON (1953). The evaluation of the specific fibre surfaces was described in detail earlier (POTŮČEK 1997; POTŮČEK, MARHANOVÁ 1998).

Treatment of experimental data

Pulp fibre bed characteristics

Generally, the superficial velocity of wash liquid, *u*, flowing through a porous medium, such as the pulp fibre bed, directly proportional to the driving force, pressure difference Δ*P*, and indirectly proportional of the hydraulic resistance, *R*, is given by Darcy´s law in the form

$$u = B \frac{\Delta P}{\mu h} \tag{1}$$

which holds in the streamline flow regime (BIRD *et al*. 1968). Then, the hydraulic resistance of pulp bed to the flow of the liquid is expressed as

$$R = \frac{\mu h}{B} \tag{2}$$

where μ is the liquid viscosity, *h* is the bed thickness, and *B* is the average permeability which expresses a measure of the liquid conductivity through the porous bed.

By assuming that the pore network consists of many distinct, continuous, and regular channels, the permeability can be expressed using the well-known classical macroscopic Kozeny-Carman equation in the form

$$B = \frac{\varepsilon^3}{(1-\varepsilon)^2 \, a_{\mathrm{V}}^2 \, K} \tag{3}$$

where ε is the average effective bed porosity defined as a ratio of the volume of the pore space open to flow to the volume of the porous pulp bed (LINDSAY 1994), and a_{V} is the specific surface of fibres. The Kozeny constant, *K*, depending only upon the shape of pores and the ratio of the tortuous length that liquid traverses in passing through the bed to the actual bed thickness has an average value of 5.55 for randomly packed fibre beds (INGMANSON 1953; LINDSAY 1994).

If the mass of fibres in the bed $m_F = h\,A\,\rho_F$ where A is the cross-sectional area and ρ_F is the consistency of pulp, then the average specific bed resistance, $\alpha = (B\,\rho_F)^{-1}$ (HERWIJN *et al.* 1995), may be written as

$$\alpha = \frac{(1-\varepsilon)^2\, a_V^2\, K}{\varepsilon^3\, \rho_F} \tag{4}$$

The average specific bed resistance, α, is a measure of the resistance offered by the fibre bed to the flow of wash liquid.

If the Kozeny-Carman equation is applicable for the flow through porous bed (MCCABE *et al.* 2001), the equivalent pore diameter

$$d_{eq} = 8.2\sqrt{\frac{u\,\mu\,h}{\varepsilon\,\Delta P}} \tag{5}$$

can be calculated in the streamline flow regime, knowing the superficial wash liquid velocity, u, and the pressure drop across the pulp bed, ΔP.

Washing breakthrough curves

The shape of the washing curve can be characterised in terms of the dimensionless Péclet number derived from the mass balance of the tracer (POTŮČEK 2001), in our case alkali lignin, for a given system in unsteady state in the following form

$$Pe = \frac{h\,u}{D\varepsilon} \tag{6}$$

where h is the thickness of the pulp bed, u is the wash liquid superficial velocity, D is the longitudinal dispersion coefficient, and ε is the average effective porosity of packed bed. Evaluation of the Péclet number from the breakthrough curves was described in detail in the previous papers (POTŮČEK 1997, 2001). In short, after converting the washing curve obtained as a response to the step input signal to a normalised dependence of dimensionless exit solute concentration upon the dimensionless time, one may reduce it by differentiation to the corresponding response to the pulse input signal which can be characterised by its variance (LEVENSPIEL 1962). For closed system of finite length, the relationship between the variance and the Péclet number was derived by LAAN (1957).

The displacement washing curve area is directly proportional to the amount of alkali lignin removed from the pulp bed. The traditional wash yield, $WY_{RW=1}$, can be expressed as

$$WY_{RW=1} = \frac{\displaystyle\int_{RW=0}^{RW=1} \frac{\rho_e}{\rho_0}\, d(RW)}{\displaystyle\int_{RW=0}^{RW\to\infty} \frac{\rho_e}{\rho_0}\, d(RW)} \tag{7}$$

where RW is the wash liquor ratio, defined as the mass of wash liquid passed through the bed to the given time divided by the mass of mother liquor originally present in the bed, ρ_e is the exit lignin concentration and ρ_0 is the initial lignin concentration in the bed at $RW = 0$. Thus, the wash yield is defined as the amount of solute washed out at $RW = 1$ divided by the total amount of solute removed from the pulp bed during the washing run.

RESULTS AND DISCUSSION

Pulp fibre bed characteristics

The change in the wash water temperature influenced the wash water mobility, defined as the permeability to water viscosity ratio, B/μ (Fig. 1). The wash water mobility had an increasing trend with increasing the temperature which had a substantial impact on the wash water viscosity. As would be expected, the hydraulic bed resistance defined by Eq. (2) decreased with increasing the wash water temperature (*cf*. Fig. 2). The decreasing trend of the hydraulic bed resistance was due to a decrease in the wash water viscosity with increasing the temperature. It must be stressed also that all displacement washing runs were carried out under the streamline flow regime when the Reynolds number of the wash water (defined in Symbols) varied in the range of 8.5×10^{-3} to 3.4×10^{-2}. These results also confirmed that, for given pressure difference of 7 kPa, the volumetric flow rate of wash liquid through pulp fibre bed increases with increasing the temperature.

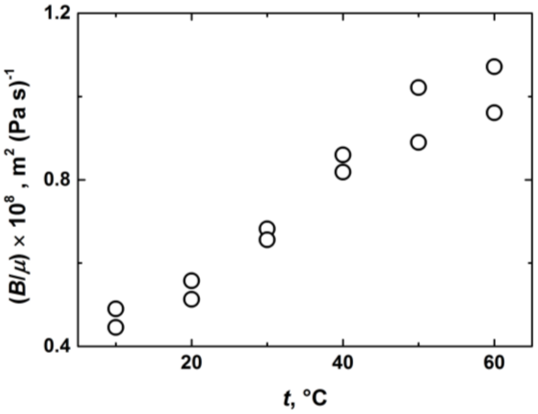

Fig. 1 Influence of wash water temperature on the mobility of wash water.

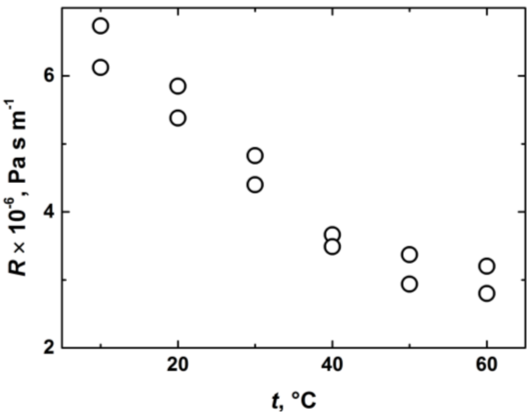

Fig. 2 Influence of wash water temperature on the hydraulic pulp bed resistance.

In contrast to the hydraulic bed resistance influenced by the wash liquid viscosity and the bed permeability (Fig. 2), the specific bed resistance seems to be almost independent upon the wash water temperature (*cf*. Fig. 3). Most of the values of the average specific resistance lies within the limits of 1.3×10^{9} to 1.6×10^{9} m kg^{-1}. A relatively great difference in the average specific bed resistance obtained for 50 and 60 °C can be ascribed to the formation of pulp beds from fibre suspension. Pulp beds formed from 4% suspension comprising relatively long fibres of spruce and pine having the tendency to form bundles of multiple fibres together appeared to be quite flocculated. With respect to this fact, the permeability ranging of 4.2×10^{-12} to 5.6×10^{-12} m^2 was achieved at the wash water temperature of 50 and 60 °C. Owing to the differences in local porosity and pore size distribution, the pulp bed can be characterised as non-homogeneous and stochastic system. In spite of this fact, our results agree well with those reported by Ingmanson (1953) who found that the temperature in the range of 10 to 40 °C had no effect on the specific bed resistance. For unbeaten coniferous bleached sulphite pulp at water temperature of 30 °C, Ingmanson (1953) reported the average specific bed resistance of 0.81×10^{9} m kg^{-1} at the pressure drop of 7 kPa.

For given pulp consistency, the average specific bed resistance is influenced by the average effective bed porosity and the specific surface of fibres. The dependence of the average effective bed porosity, calculated from Eq. (3) on the basis of permeability measurements after displacement washing, upon the wash water temperature is shown in

Fig. 4. The influence of the wash water temperature upon the specific surface of pulp fibres is illustrated in Fig. 5. For comparison, for unbeaten coniferous bleached sulphite pulp at a water temperature of 30 °C, INGMANSON (1953) reported the specific fibre surface of 886 $m^2 kg^{-1}$.

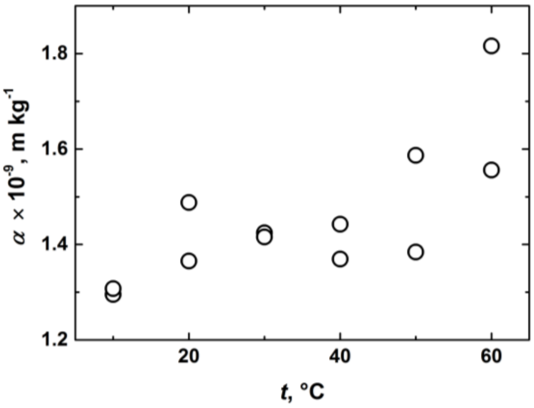

Fig. 3 Influence of wash water temperature on the average specific pulp fibre resistance.

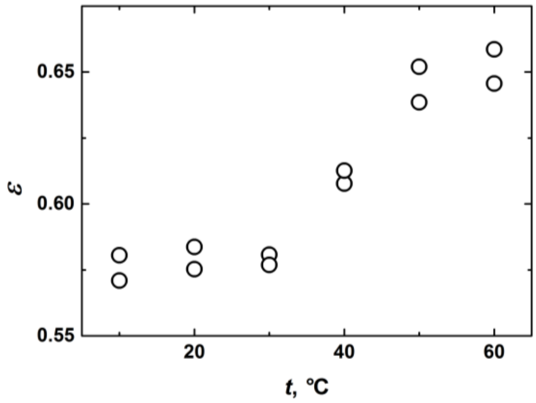

Fig. 4 Influence of wash water temperature on the average effective porosity.

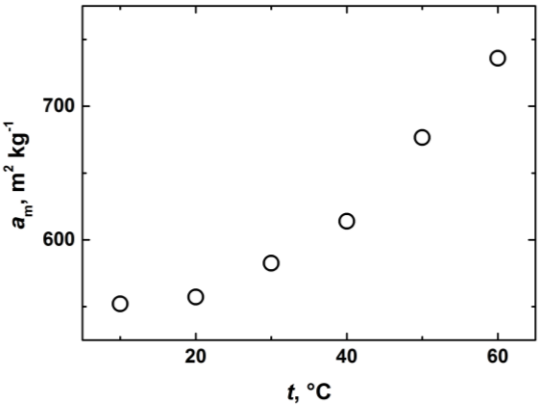

Fig. 5 Influence of wash water temperature on the specific surface of pulp fibres.

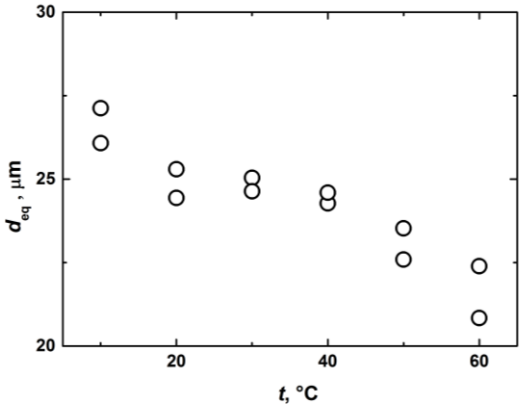

Fig. 6 Influence of wash water temperature on the equivalent pore diameter.

An increase in the average effective porosity with increasing the wash water temperature could be associated with a drop of the thickness of liquid layer immobilised on the fibre surface (POTŮČEK, PULCER 2006). Then, with decreasing thickness of stagnant liquid layer the effective surface of pulp fibres should be increased (*cf.* Fig. 4 and 5) since the effective specific volume of fibres involves the volume of fibres in the water-swollen state, including the volume of the liquid immobilised on their surface. However, in case that the specific fibre surface increases with decreasing the thickness of the stagnant liquid layer, the pore diameter should be increased. Of course in our case, when an increase in the specific fibre surface and in the effective bed porosity is accompanied by a decrease in the equivalent pore diameter (*cf.* Fig. 6), the possible explanation of these facts based on accessibility of small pores to the wash water flow seems to be plausible. A decrease of the equivalent pore diameter with increasing the wash liquid temperature may be attributed to an increase of the number of pores opened to flow when the small pores become accessible to the wash liquid flow.

It is interesting that the equivalent pore diameter is of the same order of magnitude as the coniferous fibre width (BLAŽEJ, KRKOŠKA 1989). However, it should be emphasized that, in this work, as well as in the preceding paper (POTŮČEK, PULCER 2006), the specific fibre surface were determined for water suspensions where the degree of swelling can be lower, comparing with alkaline solutions such as black liquor. Also, much of the pore space in pulp fibre bed, *e. g.*, many lumens, micropores in the cell wall, and dead-end pores opened only at one end, may be inaccessible to wash water flow in the presence of given pressure gradient.

Wash yield

A response to a step change in concentration, called washing or breakthrough curve, was measured as the time dependence of the lignin concentration in the stream leaving the pulp fibre bed. In order to normalise the response record, the washing curves were plotted as the dependence of the dimensionless concentration of alkali lignin in the outlet stream, expressed as a ratio of the exit concentration to the initial lignin concentration, ρ_e/ρ_0, against the wash liquor ratio, RW.

Breakthrough curves measured for the wash water temperature of 20 and 60 °C are shown in Fig. 7. It should be also noted that the displacement washing runs were finished at the wash liquor ratio between 6 and 7 when the lignin concentration in output stream was less than one thousandth of the initial lignin concentration in the pulp bed. However, for a better optical comparison, the experimental points connected by means of the cubic spline method are illustrated only for $RW < 3$ in Fig. 7. From the dimensionless concentration profile of alkali lignin in the exit stream, it is obvious that the displacement of lignin was non-ideal. The pulp bed consisted of compressible porous fibres where geometrical similarity does not exist. Moreover, the formation of a pulp bed in a washing cell influences the shape of the breakthrough curves. Even if the experimental conditions were strictly identical, the fibre bed was always different with respect pore size distribution. Due to inhomogeneities of the fibre bed, the different local porosities influenced significantly the flow of wash liquid through the bed. Hence, for example, at the wash water temperature of 20 and 60 °C, the washing process illustrated in Fig. 7 was characterised by the Péclet numbers of 5.5 and 11.1, respectively.

Influence of the Péclet number on the wash yield for various wash water temperatures is shown in Fig. 8. For comparison, the dependence of $WY_{RW=1}$ *vs*. Pe calculated by BRENNER (1962) for unmovable bed of non-porous particles is illustrated as well. From the results it is obvious that the change in the wash water temperature had no unambiguous effect on the wash yield. However, in the majority of runs, the wash yield was found to be greater than 0.8.

In spite of the scatter in the data, it is evident that the wash yield increases with increasing the Péclet number. However, the experimental points are located below the curve derived for the packed bed of non-porous particles by BRENNER (1962). The reason is that, for packed bed of non-porous particles, the washing process is reduced to the displacement mechanism accompanied by interfacial mixing between displaced and displacing fluids. However, in the case of a packed bed of compressible porous particles in the swollen state, such as pulp fibres, the leaching may play a significant role mainly in the spaces of the pulp bed in which the inter-particle pores were filled up with the wash liquid and the concentration driving force enables the transfer of alkali lignin macromolecules from the fibre walls towards the wash liquid.

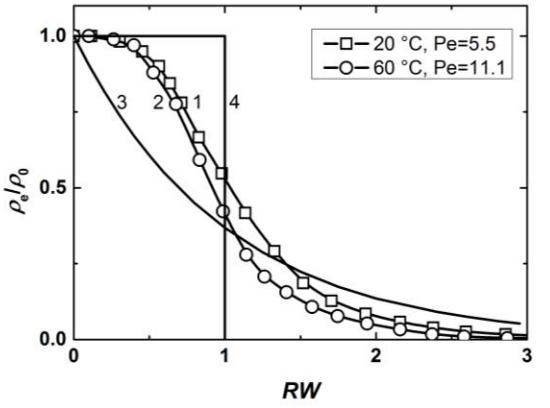

Fig. 7 Comparison of breakthrough curves measured at wash water temperature of 20 °C (1) and 60 °C (2) with theoretical responses for ideal mixing vessel (3) and plug flow (4).

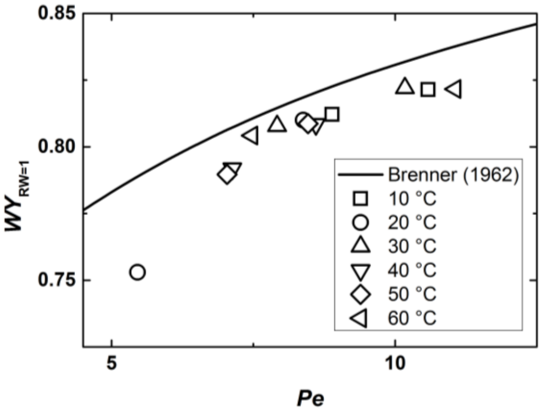

Fig. 8 Wash yield as a function of the Péclet number for various wash water temperatures.

It showed that the displacement washing efficiency expressed by the wash yield depends mainly upon the dispersion of the wash liquid inside pulp bed whereas the effect of the wash liquid temperature was negligible (*cf*. Fig. 8). Thus, based on our own data measured for the kraft pulp bed, the following equation was derived for the quantitative evaluation of the effect of the Péclet number on the wash yield

$$WY_{\mathrm{RW=1}} = 0.629\ Pe^{0.116} \tag{8}$$

with a mean relative deviation of 0.75 %, 95% confidence limits of the coefficient (0.619; 0.640) and of the power of the Péclet number (0.108; 0.124). The Akaike information criterion was found to be –140. In accordance with our preceding papers (Potůček, Miklík 2011; Potůček, Rahman 2018), our results confirmed unambiguously that the wash yield increases with the increase of the Péclet number.

For comparison with the correlation given by Eq. (8), the theoretical wash yield of the displacement in a packed bed of non-porous particles calculated according to Brenner (1962) was expressed as a function of the Péclet number in the form

$$WY_{\mathrm{RW=1}} = 0.688\, Pe^{0,0823} \tag{9}$$

for the Péclet number within the range from 3.2 to 24.

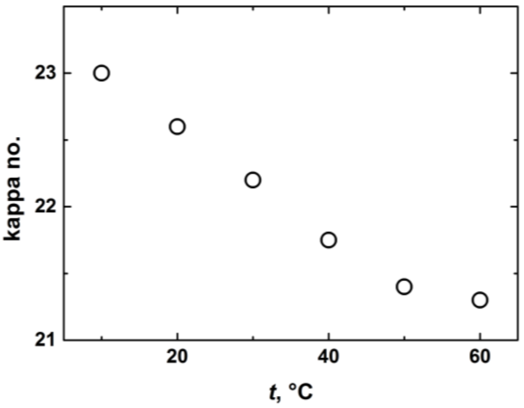

Fig. 9 Influence of wash water temperature on the kappa number of washed pulp.

Since the alkali lignin is transported from inside the fibre walls into free wash liquid by diffusion mechanism under assumption that the driving force exists, it may be expected that the greater wash liquor temperature will have a positive impact on alkali lignin leaching from the pulp fibres. Our displacement washing results showed that the kappa number of washed pulp was lower than kappa number of unwashed pulp, which was 24.9, and decreased with increasing the wash water temperature (*cf*. Fig. 9). Of course, a decrease in the kappa number can be caused not only by the change of residual lignin amount but also of other components, such as extractives, hexeneuronic acids, and some chemical structures with double bonds and/or carbonyl groups (ALA-KAILA *et al*. 2003). Nevertheless, higher efficiency of leaching of black liquor components did not have a significant influence upon the wash yield.

CONCLUSION

Displacement washing of pulp fibres is influenced by a number of phenomena occurring in porous medium, such as pore size distribution, different local porosity of the bed, geometrical properties of fibres including their swelling, and others. The pulp fibre bed ranks therefore among non-homogeneous and stochastic systems. The shape of washing curve is strongly influenced by a highly complex network of pores and its tail also by leaching of a solute from within fibre walls into the wash liquid. In spite of these facts, several conclusions valid within the framework of our study can be drawn.

The results obtained for the wash water temperature range of 10 to 60 °C showed that

(i) with increasing wash water temperature the hydraulic bed resistance, as well as the equivalent pore diameter decrease, on the contrary, the average effective bed porosity, as well as the specific surface of fibres and wash water mobility show an increasing trend even if, at given wash liquid temperature, a relatively great scattering for pulp bed parameters must be taken into account with respect to the quality of bed formation inducing anomalous dispersion in this porous medium;

(ii) the average specific bed resistance seems to be independent on the wash water temperature mainly in the range of 20 to 50 °C. On the other hand, an increase in pulp bed temperature resulting in a drop of liquid viscosity will increase the wash water flowrate and thus the washer capacity;

(iii) the efficiency of displacement washing is dependent primarily on the inhomogeneity of the pulp fibre bed, having an impact on the shape of the breakthrough curves recording concentration of solute removed from the pulp bed. The data obtained for pulp fibre beds at various wash liquid temperature were well fitted by the correlation between the wash yield and the Péclet number (Eq. (8)) and error did not exceed 0.75 %;

(iv) the influence of molecular transport processes, depending on the temperature, upon the washing efficiency was not markedly evident in the range of the wash water temperatures investigated in this work. However, the kappa number of washed pulp decreased with increasing the wash water temperature, indicating that the higher temperature had a favourable impact on lignin leaching rate from the fibre walls.

SYMBOLS

a_m	specific surface of fibres based on fibre mass, $\mathrm{m^2\ kg^{-1}}$
a_V	specific surface of fibres based on volume, defined as a ratio of specific surface (in $\mathrm{m^2\ kg^{-1}}$) and specific volume (in $\mathrm{m^3\ kg^{-1}}$), $\mathrm{m^{-1}}$
B	permeability, $\mathrm{m^2}$

D	longitudinal dispersion coefficient, $m^2\ s^{-1}$
d_{eq}	equivalent diameter of pores defined by Eq. (5), m
h	bed thickness, m
K	Kozeny constant
ΔP	pressure difference, Pa
Pe	Péclet number defined by Eq. (6)
R	hydraulic bed resistance defined by Eq. (2), $Pa\ s\ m^{-1}$
Re	Reynolds number ($= 4u\rho_{WL}/(a_V(1-\varepsilon)\mu)$)
RW	wash liquor ratio
t	wash liquid temperature, °C
u	superficial wash liquid velocity, $m\ s^{-1}$
$WY_{RW=1}$	wash yield defined by Eq. (7)

Greek letters

α	average specific bed resistance defined by Eq. (4), $m\ kg^{-1}$
ε	average bed porosity
μ	liquid viscosity, Pa s
ρ_e	exit lignin concentration, $kg\ m^{-3}$
ρ_F	consistency of pulp, $kg\ m^{-3}$
ρ_{WL}	density of wash liquid, $kg\ m^{-3}$
ρ_0	initial lignin concentration, $kg\ m^{-3}$

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