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DISPLACEMENT WASHING OF SULPHITE AND KRAFT PULPS

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ABSTRACT

The displacement washing of spruce sulphite and kraft pulps was simulated using a laboratory washing cell. Geometrical properties of pulp fibres, as well as intrinsic properties of spent red and black liquors were determined. The sulphite spruce pulp beds showed the intrinsic permeability of 3.0×10^{-12} to 6.7×10^{-12} m^2 and the specific resistance of 1.2×10^9 to 2.3×10^9 m kg^{-1}, while, for the kraft spruce pulp, the former was within the limits of 1.2×10^{-12} to 3.0×10^{-12} m^2 and the latter within the limits of 2.6×10^9 to 6.3×10^9 m kg^{-1}. The displacement washing process was described by the washing curves measured for lignosulphonates and/or alkali lignin. The wash yield determined for sulphite spruce pulp was found to be greater than that for kraft spruce pulp. The relationships between the wash yield, on the one hand, and the Péclet number for each pulp sort and/or two independent dimensionless criteria for all data obtained for both pulp sorts, on the other hand, were expressed by the correlation equations.

Key words: displacement washing; sulphite pulp; kraft pulp; wash yield; Péclet number.

SYMBOLS

a_m	specific surface of fibres based on fibre mass, m^2 kg^{-1}
a_V	specific surface of fibres based on fibre volume, m^{-1}
B	permeability, m^2
D	axial dispersion coefficient, m^2 s^{-1}
d_{eq}	equivalent pore diameter defined by Eq. (4), m
FC	fibre coarseness, mg m^{-1}
h	thickness of pulp bed, m
K	Kozeny constant, dimensionless
l_A	numerical average length of pulp fibres, mm
l_W	weighted average length of pulp fibres, mm
n	number of measurements
ΔP	pressure drop, Pa
Pe	Péclet number based on bed thickness defined by Eq. (5), dimensionless
R	hydraulic resistance of pulp bed, Pa s m^{-1}
Re	Reynolds number based on hydraulic diameter of pore (= $u\ \rho_{WL}$ / (a_V (1–ε) μ)), dimensionless
RW	wash liquor ratio, dimensionless
u	superficial wash liquid velocity, m s^{-1}
$WY_{RW=1}$	wash yield at RW = 1 defined by Eq. (6), dimensionless

Greek letters

α average specific pulp bed resistance defined by Eq. (3), m kg^{-1}

δ mean relative quadratic deviation of wash yield defined as

$$\delta = \left[\frac{1}{n} \sum_{i=1}^{i=n} \left(\frac{WY_{\text{exp}} - WY_{\text{calc}}}{WY_{\text{exp}}} \right)_i^2 \right]^{1/2} \times 100, \%$$

ε average effective bed porosity (void space), dimensionless

μ dynamic viscosity, Pa s

π dimensionless criterion defined by Eq. (9), dimensionless

ρ density of spent liquor, kg m^{-3}

ρ_e exit lignin concentration from bed, kg m^{-3}

ρ_F consistency (mass concentration) of pulp bed, kg m^{-3}

ρ_{WL} density of wash liquid, kg m^{-3}

ρ_0 initial lignin concentration in bed, kg m^{-3}

σ surface tension, N m^{-1}

INTRODUCTION

Sulphite pulping dominated the industry from the late 1800s to the mid-1900s. During the 1920´s, however, the alkaline process became more important, and in the second half of the 20th century the growth of the kraft industry has been rather spectacular (STOCKMAN 1962). The market for sulphite pulp is a small fraction of the kraft pulp's. However, sulphite pulp remains an important commodity for specialty papers, such as tissues and fine quality papers, and a source for non-paper applications such as rayon, cellulose acetate, and cellulose ether derivatives.

The sulphite process uses the acid solutions in the cooking liquor to degrade the lignin bonds between wood fibres (RYDHOLM 1965). The spent sulphite liquor (also called red liquor) contains in soluble form the main part of the non-cellulose components of wood. These components are mainly lignin, present as lignosulfonates, and hemicellulose, present as partially or wholly hydrolysed carbohydrate compounds (SALVESEN, HOGAN 1948).

Since, for the same amount of wash water, the displacement washing is more effective than washing based on dilution followed by thickening, the displacement washing of kraft pulp was investigated in several papers (GRÄHS 1976; LEE 1979; POIRIER *et al*. 1987, 1988; POTŮČEK 1997; POTŮČEK, MARHANOVÁ 2000; POTŮČEK, MIKLÍK 2010; POTŮČEK, HÁJKOVÁ 2015; TRINH *et al*. 1987, 1989). But the research of the displacement washing for sulphite pulps remains obscure. Only INGMANSON (1953) investigated the filtration resistance for packed beds of bleached sulphite pulp. The main objective of the present study was therefore to investigate the displacement washing efficiency for displacement of spent red liquor from the pad of sulphite pulp. The results obtained were compared with those for kraft pulp delignified to low kappa number.

EXPERIMENTAL PART

The sulphite spruce pulp and spent red liquor produced by Mondi Ybbstaler Zellstoff GmbH (Austria) were used for displacement washing experiments. The kappa number of sulphite pulp determined according to the Tappi Test Method T 236 om-99 was 12.5.

The kraft spruce pulp was cooked under laboratory conditions. Spruce wood mill chips without undesirable components, such as bark, oversized chips, and knots, were undergone batch kraft cooking in a laboratory rotary digester comprising six autoclaves of 750 cm^3 capacity, immersed in an oil bath. The pulping conditions were described in detail in our preceding paper (RAHMAN, POTŮČEK 2017). The degree of delignification of pulp cooked by the batch kraft process was expressed by the kappa number of 18.1. The black liquor was obtained from Mondi Štětí (Czech Republic).

Using the Kajaani FS-100 instrument, the distribution of the fibre length was also measured for sulphite and kraft pulps. The length of fibres in the wet state was characterized by the weighted average, l_W, as well as the numerical average, l_A. The fibre coarseness, *FC*, was evaluated as well. The effective specific surface of fibres based on volume, a_V, and on fibre mass, a_m, for both pulps tested was determined by the method according to INGMANSON (1953). The pulp fibre characteristics and physical properties of spent liquors are summarized in Tables 1 and 2, respectively.

Tab. 1 Fibre length, coarseness, and specific surface for sulphite and kraft pulps.

Pulp	Kappa no.	l_W mm	l_A mm	*FC* mg m^{-1}	$a_V \times 10^{-5}$ m^{-1}	a_m m^2 kg^{-1}
Sulphite	12.5	2.46	1.24	0.279	2.08	675
Kraft	18.1	2.77	1.91	0.103	1.51	584

Tab. 2 Intrinsic properties (density, ρ, dynamic viscosity, μ, and surface tension, σ) at 25 °C, solids content, and lignin concentration, ρ_0, for red and black liquors.

Liquor	pH	ρ kg m^{-3}	μ mPa s	σ mN m^{-1}	Solids mass %	ρ_0 kg m^{-3}
Red	2.2	1059	1.30	55.64	13.6	76.7
Black	12.6	1096	1.79	39.95	20.6	56.0

The density, ρ, dynamic viscosity, μ, and surface tension, σ, of the both spent liquors were measured by the pycnometric method, using a capillary viscometer, and stalagmometric method, respectively. Solids content of both spent liquors was determined according to the Tappi Test Method T 211 om-02. It is worth mentioning that the red liquor solids comprise 89.3 mass % of organics and only 10.7 mass % of ash, while the black liquor solids 39.2 mass % of organics and 60.8 mass % of inorganics (ash). The initial lignin concentrations, ρ_0, *i. e.*, lignosulphonates concentration of the red liquor and alkali lignin concentration of the black liquor, were measured using an ultraviolet spectrophotometer Cintra 10e at a wavelength of 260 nm and 295 nm, respectively.

Displacement washing experiments 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 volume between the permeable septum and a piston, covered with 45 mesh screens to prevent fibre loss from the bed.

Pulp beds were formed from a dilute suspension of unbeaten unbleached sulphite and/or kraft pulps in red and/or black liquor, respectively. After compressing to desire thickness of 30 mm, the consistency, *i. e.*, mass concentration of moisture-free pulp fibres in the bed varied within the limits from 120 to 149 kg m^{-3}. The pulp beds were not mechanically conditioned and were used as formed.

To investigate the displacement washing process, the stimulus-response method was chosen. Distilled water at the temperature of 25 °C employed as wash liquid was distributed uniformly through the piston to the top of bed at the start of the washing experiment, approximating a step change in 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. Samples of the washing effluent leaving the pulp bed were analysed for lignosulphonates and/or alkali lignin spectrophotometrically. Displacement washing experiments with pulp 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 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 according to INGMANSON (1953).

Treatment of experimental data

A pulp mat formed on a cylinder´s surface of the rotary washer or on a travelling screen of the belt washer may be classified as unconsolidated porous medium with randomly oriented fibres with the central cavity called lumen. In displacement washing stage, the volumetric flow rate of a wash liquid flowing linearly through porous medium is directly proportional to the driving force, pressure drop, ΔP, and inversely to the hydraulic resistance. The superficial wash liquid velocity defined as $u = V/A$ where V is the volumetric flow rate and A is the cross-sectional area of the pulp mat may be expressed by Darcy´s law (TILTON 1997) in the form

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

which is usually valid for creeping flow where the Reynolds number as defined for porous medium in the Symbols section is less than one (BIRD 1968; GREENKORN 1981; SAMPSON, KROPHOLLER 1996). Then, the hydraulic resistance of pulp mat to the flow of the liquid is expressed as $R = \mu h/B$ where μ is the liquid viscosity, h is the mat thickness, and B is the intrinsic permeability of the pulp mat and is a function only of the pore structure.

The intrinsic permeability may be expressed in terms of the specific surface of pulp fibres, a_V, and the void space of the pulp mat, ε, as

$$B = \frac{\varepsilon^3}{(1-\varepsilon)^2 a_V^2 K} \tag{2}$$

where 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 mat thickness, has an average value of 5.55 for randomly packed fibre beds (INGMANSON 1953).

Then the average specific mat resistance defined as $\alpha = (B \rho_F)^{-1}$ (KROTSCHECK 2006) may be written in the form

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

where ρ_F is the consistency of the pulp mat.

The size of pores available for the wash liquid flow through the pulp mat can be expressed as an equivalent diameter, d_{eq}, defined as 4 times the hydraulic radius given as the ratio of the cross-sectional area of the pore to the wetted perimeter of the pore (MCCABE *et al*. 2001). Hence, for the case of pulp fibre mat, the equivalent pore dimeter may be determined from the following relationship

$$d_{eq} = \frac{4\varepsilon}{(1-\varepsilon) a_V} \tag{4}$$

The hydraulic resistance and specific resistance of pulp bed along with the intrinsic permeability and porosity rank among macroscopic or bulk properties.

In order to characterize non-ideal flow patterns within packed bed, a dispersion model drawn on the analogy between mixing in actual flow and a diffusional process can be used (LEVENSPIEL 1962).

Step function input signal has been widely used in the analysis of the flow through a packed bed of solid particles. A response to a step change in concentration, called washing or breakthrough curve, is the dependence of the dimensionless concentration of tracer in the outlet stream, expressed as a ratio of the exit concentration to the initial tracer concentration, ρ_e/ρ_0, against the wash liquor ratio, RW, 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.

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) for a given system in unsteady state in the following form

$$Pe = \frac{h u}{D \varepsilon} \tag{5}$$

where D is the longitudinal dispersion coefficient and ε is the average effective porosity of packed bed (LINDSAY 1994). The evaluation of the Péclet number from the breakthrough curves was described in detail in the previous paper (POTŮČEK 1997).

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

$$WY_{RW=1} = \frac{\int_{RW=0}^{RW=1} \frac{\rho_e}{\rho_0} \mathrm{d}(RW)}{\int_{RW=0}^{RW\to\infty} \frac{\rho_e}{\rho_0} \mathrm{d}(RW)} \tag{6}$$

Thus, the traditional 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 bed properties

The permeability specifically refers to the ability for water to drain through a pulp fibre bed. The permeability of a pulp bed was evaluated from the Darcy´s law (Eq. (1)) which holds true in the creeping (streamline) flow regime when the Reynolds number is less than unity. The Reynolds number based on the hydraulic pore diameter (defined by Eq. (4)) varied within the interval of 0.66×10^{-3} to 2.6×10^{-3}, indicating that all experiments were conducted in the creeping flow regime.

Permeability data measured for beds from sulphite and kraft pulps after complete washing are illustrated in Fig. 1. Higher permeability for sulphite pulp can be ascribed to the higher effective porosity (LINDSAY 1994) ranged from 0.54 to 0.61 with the average of 0.58, while, for pulp beds formed from kraft pulp, the effective bed porosity varied from 0.38 to 0.47 with the average of 0.42. It is worth pointing out that, for sulphite and kraft pulp beds having a consistency in the range of 120 to 143 kg m^{-3} with the average of 133 kg m^{-3} and of 125 to 149 kg m^{-3} with the average of 130 kg m^{-3}, respectively, the consistency effect upon the permeability was not remarkable.

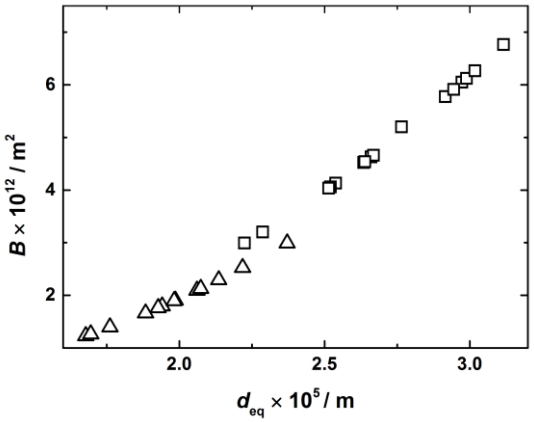

Fig. 1 Dependence of permeability on equivalent pore diameter for wash water at 25 °C. Pulp fibre bed: sulphite (□), kraft (△).

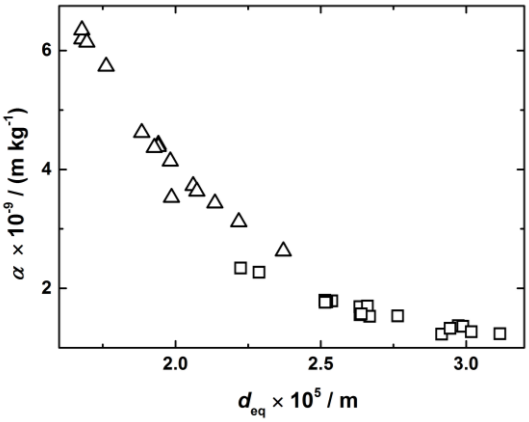

Fig. 2 Dependence of specific bed resistance on equivalent pore diameter for wash water (25 °C) flowing through sulphite (□) and kraft (△) pulp bed.

Permeability data are consistent with the specific bed resistance obtained for both pulps tested in this work (*cf*. Fig. 2). Higher specific bed resistance found for kraft pulp corresponds to lower pore diameter and lower effective porosity compared with the sulphite pulp. The equivalent pore diameter defined by Eq. (4) has the same order of magnitude as spruce bleached sulphite pulp fibre width of 31 μm (BLAŽEJ, KRKOŠKA 1989). It means that the pulp fibre beds tested in our work, when the consistency was between 120 to 149 kg m^{-3}, can be characterised as a porous medium where the average pore diameter and fibre width are the same order of magnitude.

It should be noted that, at the consistency range studied in the present work, the permeability properties measured for spruce sulphite and kraft pulps are comparable with those of 3×10^{-12} to 8×10^{-12} m^2 reported by RAINEY *et al*. (2009) for bagasse pulp. For coniferous bleached sulphite pulp at the pressure drop of 7 kPa, when the specific surface of pulp fibres had a value of 886 m^2 kg^{-1} (*cf*. Table 1), INGMANSON (1953) reports the specific resistance of 0.847×10^9 m kg^{-1}, slightly lower in comparison with unbleached sulphite pulp tested in our work.

Displacement washing

A typical example of washing curves measured for sulphite and kraft pulps is illustrated in Fig. 3, as the dependence of the dimensionless exit concentration of the lignin, lignosulphonates and alkali lignin, respectively, against the wash liquor ratio. At the beginning of a displacement, the first portions discharged from the bed are fully as concentrated as was the mother liquor. As soon as the first portion of wash liquid passes through the pulp bed, the concentration of lignin drops very rapidly. However, a decrease in lignin concentration starts at lower wash liquor ratio for sulphite fibre bed comparing to kraft pulp fibres. The dimensionless exit lignin concentration at $RW = 1$ ranged from 0.254 to 0.389 with the average of 0.347 for sulphite pulp, while, for kraft pulp, it varied from 0.416 to 0.499 with the average of 0.459. The reason for this can be higher permeability, as well as lower specific bed resistance and greater equivalent pore diameter in the pulp bed formed from sulphite pulp fibres.

Of course, at the wash liquor ratio greater than approximately 1.5 the exit lignosulphonates concentration is greater than that of alkali lignin. In the last period for the wash liquor ratio above 1.5, only remains of spent liquor are removed from inside narrow pores and fibre walls. With respect to higher fibre coarseness of sulphite fibres (*cf*. Table 1), the leaching referring to the desorption and diffusion lignosulphonates from within the fibres is more intensive for sulphite pulp compared to kraft pulp. It should be nevertheless

stressed that the formation of a pulp bed in a washing cell influences the shape of the washing curves. Even if the experimental conditions are strictly identical, the each pulp fibre bed is an original porous medium with respect to various local variations in pore size and their spatial configuration (MAURET, RENAUD 2002). Owing to a tortuosity of pores, the wash liquid is forced to take a longer path than the bed thickness is.

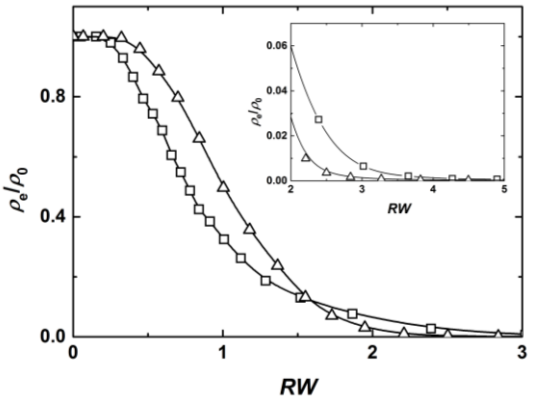

Fig. 3 Typical breakthrough washing curves: sulphite pulp *Pe* = 6.6 (□) and kraft pulp *Pe* = 12.1 (△).

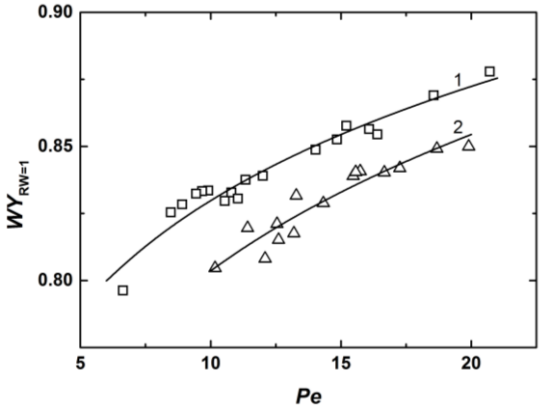

Fig. 4 Displacement wash yield as a function of the Péclet number for sulphite (□) and kraft (△) pulps. Eq. (7) (line 1), Eq. (8) (line 2).

Influence of the Péclet number (Eq. (5)) on the wash yield (Eq. (6)) for sulphite and kraft pulps is shown in Fig. 4. In spite of the scatter in the data, it is evident that the wash yield increases with increasing the Péclet number ranging from 6.6 to 20.7 in agreement with the previous papers (POTŮČEK 1997; POTŮČEK, MARHANOVÁ 2000; POTŮČEK, HÁJKOVÁ 2015, 2016). The higher values of the wash yield obtained for sulphite pulp can be ascribed to the higher average porosity of the pulp bed in comparison with kraft pulp bed as mentioned above. The removal of lignin from the pulp bed is accomplished *via* combined displacement and diffusion processes occurring during leaching of lignin from within the fibres. Our results reveal that the higher average effective porosity is, the larger amount of the spent liquor is available to displacement, having a positive impact on the washing efficiency, mainly in the case of short time contact between pulp fibres and wash liquid.

Nevertheless, the wash yields obtained for sulphite and kraft pulps cooked from spruce wood was found to be lower in comparison with those achieved for kraft hardwood pulp in the previous paper (POTŮČEK, MIKLÍK 2010, 2011) where the wash yields within the limits of 0.873 to 0.914 were measured. Visual observations showed that the bed formed from hardwood pulp fibres was more homogeneous in contrast to the bed formed from softwood pulp fibres in which channelling due to local inhomogeneities is often noticeable. Hence, the Péclet number determined for hardwood pulp beds changed between 25.3 and 50.4.

In order to evaluate the effect of the pulp bed structure on the washing efficiency, the wash yield defined by Eq. (6) may be correlated as a function of the Péclet number only. Based on our own data, the following equations were derived:

$$WY_{\mathrm{RW=1}} = 0.703 Pe^{0.0721} \tag{7}$$

for sulphite pulp, and

$$WY_{\mathrm{RW=1}} = 0.656 Pe^{0.0885} \tag{8}$$

for kraft pulp.

The accuracy of Eqs. (7) and (8) was judged on the basis of the mean relative deviation of the wash yield, δ (defined in Symbol) which was 0.58 % and 0.56 %, respectively. Since the values of regression coefficients in Eqs. (7) and (8) represent an estimate of the real values, the 95% confidence intervals were also calculated for the coefficient of (0.698; 0.707) and (0.647; 0.664), respectively, and for the power of the Péclet number of (0.0696; 0.0745) and (0.0837; 0.0932), respectively.

From the dimensionless concentration profile of the tracer, lignosulphonates and alkali lignin, in the output stream (*cf.* Fig. 3), it is obvious that movements of the wash liquid is not identical for both sorts of pulp undergone to displacement washing. In miscible displacement in porous media like a pulp fibre bed, the wash liquid is forced to flow through a porous bed to displace the mother liquor from the bed. In unconsolidated porous medium with randomly oriented fibres, local variations in the liquid flow direction and in the liquid flow velocity occur. The properties of pulp fibres, as well as of the mother liquor can contribute to the mechanical dispersion influencing the displacement washing process.

In order to express the effect of the different properties of sulphite and kraft pulp fibres and of red and black liquors upon the displacement washing efficiency, the specific surface, a_V, of pulp fibres along with the dynamic viscosity, μ, density, ρ, and surface tension, σ, of spent liquors were grouped into the following dimensionless criterion

$$\pi = \frac{a_V \mu^2}{\rho \sigma} \tag{9}$$

Mathematical treatment of the experimental data showed that two independent dimensionless groups, the Péclet number, *Pe*, and criterion π, derived on the basis of dimensional analysis, are sufficient to description of our results obtained for the sulphite and kraft pulps. For the values of the Péclet number and those of the criterion π varied in the range of 6.6 to 20.7 and of 0.006 to 0.011, respectively, the correlation equation in the form

$$WY_{RW=1} = 0.556 Pe^{0.0761} \pi^{-0.0438} \tag{10}$$

was derived with the mean relative deviation of 0.57 %. The 95% confidence intervals for the coefficient and for the power of the Péclet number and the criterion π were (0.551; 0.561), (0.0746; 0.0776), and (–0.0451; –0.0425), respectively. It should be noted that the Akaike information criterion as an estimator of the relative quality of statistical models for given set of data has a value of –348 for correlation equation (10), while the values of –192 and –157 were found for Eqs. (7) and (8), respectively. One can suppose that the lower Akaike information criterion is, the more suitable statistical model is.

CONCLUSION

Packed bed of pulp fibres is a very tangled system with randomly oriented porous, compressible particles with different size and a central cavity known as lumen. Whether the wash liquid flows through lumens during the displacement washing is not known so far.

In spite of these facts, the results obtained showed that the washing curves measured for sulphite spruce pulp differ from those measured for kraft spruce pulp having the greater specific resistance and lower intrinsic permeability compared to sulphite pulp. The displacement wash yield for spruce sulphite and kraft pulps showed an increasing trend with increasing the Péclet number in agreement with the results obtained for softwood and

hardwood pulp fibres in the previous papers. However, the wash yield obtained for sulphite pulp was greater than that for kraft spruce pulp at given interval of the Péclet numbers ranging of 6.6 to 20.7. Finally, one can conclude from our displacement washing results that the kraft pulp seems to be more difficultly washable in comparison with sulphite pulp.

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