DISPLACEMENT WASHING OF KRAFT SPRUCE PULP WITH LOW AND HIGH KAPPA NUMBER

Potůček F., Rahman M. M.

University of Pardubice, Faculty of Chemical Technology, Institute of Chemistry and Technology of Macromolecular Materials, 532 10 Pardubice, Czech Republic
frantisek.potucek@upce.cz

Abstract

The paper deals with the displacement washing of kraft spruce pulp delignified to two kappa number levels. Using the step function input change method, the washing breakthrough curves measured for alkali lignin as a tracer were described by the dispersed plug flow model. Besides the traditional wash yield, the relationships between the axial dispersion coefficient and Reynolds number, as well as between the mean residence time and space time were investigated as well. The results obtained showed that remarkable differences in the wash yield, dispersion coefficient, as well as mean residence time were found for both kappa numbers of kraft spruce pulp.

Key words: displacement washing, kraft spruce pulp, wash yield, dispersed flow model

Introduction

Pulp washing is a crucial unit operation in pulping and bleaching. High production rates must be achieved along with high washing efficiencies while using a minimum quantity of wash water. Poor brownstock washing leads to excessive black liquor and sodium carryover to the bleach plant causing unnecessary chemical consumption and effluents. On the other hand, good washing achieved by the excessive use of wash water increases the demand on the evaporators in the recovery system[1].

The spent liquor in pulp stream includes three parts. The first part in the spaces between fibres, its quantity depends upon the consistency of pulp. The second part in the lumens of cells, its quantity depends upon the size of lumens. The third zone is contained in the walls of the cells.

Brownstock washing can be carried out in four basic processes known as dilution, dewatering, diffusion, and displacement[2]. 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[3–13]. However, evaluation of displacement washing efficiency is very complex and many factors are associated with it. Of course, the effect of the degree of pulp delignification upon the displacement washing efficiency remains obscure so far.

The main objective of the present study was therefore to investigate the displacement washing efficiency for kraft spruce pulps cooked to low and high kappa number.

Experimental

The kraft spruce pulps were cooked under laboratory conditions. Spruce wood mill chips without undesirable components, such as bark, oversized chips, and knots, in accordance with TAPPI Test Method T 265 were undergone batch kraft cooking in a laboratory rotary digester. The pulping conditions were described in detail in our preceding paper[14]. The degree of delignification of pulps cooked by the batch kraft process was expressed in terms of the kappa number of 18.1 and 50.1 in accordance with TAPPI Test Method T 236 om-99. The black liquor was obtained from Mondi Štětí (Czech Republic).

The displacement washing was simulated under laboratory conditions. The stimulus-response experiments, using a step input, were performed in the displacement washing cell consisting of a vertical glass cylinder 110 mm high, having 35 mm inner diameter. The fibre bed occupied the volume between the permeable septum and piston. The experimental apparatus was similar to that used by Lee[4].

For each experiment, the slurry of unbeaten unbleached kraft pulp in black liquor was used. After compressing it to the desired thickness of 30 mm, the consistency, *i. e.*, mass concentration of moisture-free pulp fibres in the bed was maintained within the limits from 127 to 148 kg m^{-3}, the mean value being 130.5 kg m^{-3}. Using a Kajaani analyser, the length of spruce fibres in the wet state was characterised by the weighted average of 2.77 mm and 2.84 mm, as well as the numerical average of 1.91 mm and 2.09 mm for kraft pulps with the kappa number of 18.1 and 50.1, respectively. Estimated coarseness of fibres was about 0.103 and 0.111 mg m^{-1} in the case of 18.1 and 50.1 kappa numbers, respectively. The pulp beds were not mechanically conditioned and were used as prepared.

Concentration of alkali lignin in the black liquor taken from SuperBatch cooking plant was 56 g dm^{-3}. Further properties of sulphate liquor were as follows: solids content of 20.6 %, of which the ash made up 61 %, and organics 39 %, density of 1096 kg m^{-3} at 25 °C, and pH value of 12.6.

Distilled water at a temperature maintained at 25 °C was employed as wash liquid. The superficial wash liquid velocity, based on empty cross-sectional area, varied in the range from 0.052 to 0.25 mm s^{-1}. Samples of the washing effluent leaving the pulp bed were analysed for alkali lignin, using an ultraviolet spectrophotometer Cintra 10e operating at the wavelength of 295 nm. More detailed description of experiments can be found elsewhere[9]. Analogous measurements at various consistencies of the bed were focused on the determination of the effective specific volume and surface of pulp fibres according to Ingmanson[15].

Treatment of Experimental Data

A response to step change in concentration provided time dependences called washing or also breakthrough curves (*cf*. Fig. 1). 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 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.

The displacement washing curve area (Fig. 1) is directly proportional to the amount of alkali lignin removed from the bed. Quality of the displacement washing can be characterised by the wash yield. The traditional displacement wash yield, $WY_{\mathrm{RW=1}}$, 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

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

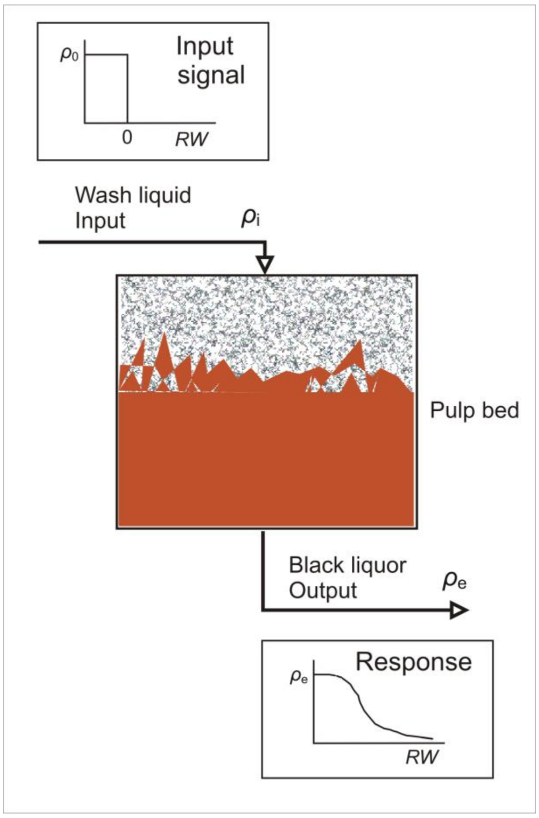

Figure 1. Illustration of displacement washing

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

$$Pe = \frac{h\,u}{D\,\varepsilon} \quad . \qquad (2)$$

The determination of the Péclet number from the residence time distribution measured for displacement washing can be found in the previous paper[9].

In order to predict flow pattern in a pore of given geometry, the Reynolds number characterising the ratio of inertial forces to viscous forces within a fluid has wide applications. For wash liquid flowing through an individual pore in a packed bed, the Reynolds number can be written in the form

$$Re = \frac{d_m u\,\rho}{\varepsilon\,\mu} \qquad (3)$$

where $d_m = 4\ \varepsilon/(a_v\ (1\text{-}\varepsilon))$ is the hydraulic mean diameter defined as four times the cross-sectional area divided by the wetted perimeter of the pore.

The average porosity of pulp bed, ε, was evaluated on the basis of permeability determined experimentally. Depending on the consistency of pulp bed, the permeability varied within the limits of 0.66×10^{-12} to 3.0×10^{-12} m^2. For comparison, the permeability of the bed of glass beads was found to be 9×10^{-11} m^2. More details can be found in the previous paper[16].

Two time parameters enable to compare the holding times of tracer displaced from the porous bed and of wash liquid used as the displacing medium. While the mean residence time of a solute chosen as the tracer for describing breakthrough curve is defined as

$$t_m = \int_{t=0}^{t\to\infty} \frac{\rho_e}{\rho_0}\, dt \qquad (4)$$

the space time, τ, characterising the holding time of wash liquid in the bed is defined as the void volume of the bed divided by the volumetric flow rate of the wash liquid and can be expressed by the following equation

$$\tau = \frac{\varepsilon h}{u} \qquad (5)$$

Results and Discussion

Wash yield

The influence of the Péclet number on the wash yield for kraft spruce pulp is shown in Fig. 2. In spite of the scatter in the data, it is evident that the wash yield increases with increasing the Péclet number. Similarly, as for kraft softwood[9,10] and hardwood[11,12] pulps, as well as for soda rapeseed pulp[13], the experimental points are located below the curve derived for the packed bed of non-porous particles by Brenner[17]. 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 case of packed bed of compressible porous particles in the swollen state, like 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 within fibre walls towards the wash liquid.

Based on our own data measured for the kraft spruce pulp with the kappa number of 18.1, the following equation

$$WY_{RW=1} = 0.656\, Pe^{0.0885} \qquad (6)$$

was derived for the quantitative evaluation of the effect of wash liquid dispersion on the wash yield The suitability of Eq. (6) was evaluated on basis of the mean relative quadratic deviation of the wash yield which was 0.6 %. Values of the Péclet number varied in the range of 10.2 to 19.9. Since the values of regression coefficients, evaluated by the least square method, represent an estimate of the real values, the 95 % confidence intervals were also calculated for the coefficient of 0.656 ± 0.0087 and for the power of the Péclet number of 0.0885 ± 0.0047.

For kraft spruce pulp with the kappa number of 50.1, when the Péclet number varied in the interval from 6.3 to 16.5, the correlation was derived in the form:

$$WY_{RW=1} = 0.700\, Pe^{0.0681} \qquad (7)$$

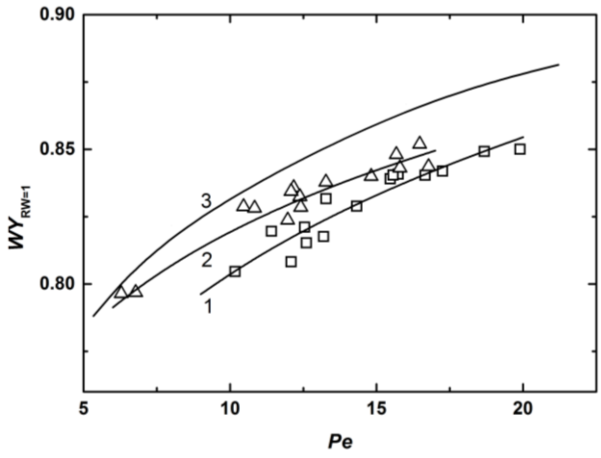

Figure 2. Displacement wash yield as a function of the Péclet number for kraft pulp cooked to kappa number of 18.1 (□) and of 50.1 (△). 1 Eq. (6), 2 Eq. (7), 3 non-porous particles according to Brenner[17]

with the mean relative quadratic deviation of 0.7 %. The 95 % confidence intervals for the coefficient and the power of the Péclet number were 0.700 ± 0.0064 and 0.0681 ± 0.0034, respectively. It was confirmed again

that the wash yield depends upon the Péclet number in a small degree, similarly as for kraft hardwood[11,12], soda rapeseed[13] pulps, and non-porous particles[17].
In order to express the effect of the delignification degree of kraft spruce pulps tested in our work, mathematical treatment of the all experimental data gave the following correlation equation

$$WY_{\mathrm{RW}=1} = 0.656\, Pe^{0.0741}\, \kappa^{0.0128} \qquad (8)$$

with the mean relative deviation of 0.6 %. The 95 % confidence intervals for the coefficient and for the power of the Péclet number and the kappa number were (0.652; 0.661), (0.0722; 0.0759), and (0.0119; 0.0137), 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 –317 for correlation equation (8), while the values of –157 and –151 were found for Eqs. (6) and (7), respectively. One can suppose that the lower Akaike information criterion is, the more suitable statistical model is.

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. (2)), containing the dispersion coefficient, *D*.

Figure 3 is a plot of the axial dispersion coefficient against the Reynolds number defined by Eq. (3), showing our results for kraft spruce pulps with different kappa number. In spite of the scatter in the results, the dependence of the dispersion coefficient on the Reynolds number shows an increasing trend for both types of pulp bed in a good agreement with the authors[18,19]. Comparing the values of dispersion coefficient measured for softwood[9,10] and hardwood[11,12] pulps, 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.

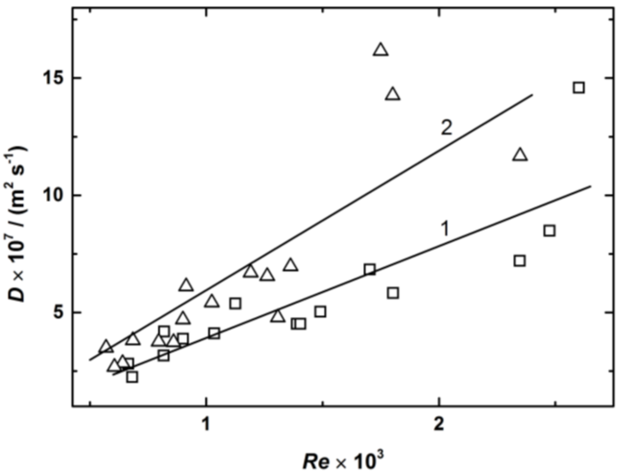

Figure 3. Effect of Reynolds number on axial dispersion coefficient for kraft pulp cooked to kappa number of 18.1 (□) and of 50.1 (△). 1 Eq. (9), 2 Eq. (10)

From the results shown in Fig. 3, it follows that a linear dependence of the dispersion coefficient on the Reynolds number can be assumed. The correlations between *D* in $m^2\ s^{-1}$ and *Re* in the forms

$$D = 0.391 \times 10^{-3}\, Re \qquad (9)$$

with a correlation coefficient of 0.87 for spruce pulp having the kappa number of 18.1, and

$$D = 0.595 \times 10^{-3}\, Re \qquad (10)$$

with a correlation coefficient of 0.85 for spruce pulp having the kappa number of 50.1 were evaluated.
The linear function between the dispersion coefficient and the Reynolds number obtained for kraft spruce pulp studied in the present work is in accordance with the results reported earlier for softwood pulp[9,10], as well as for pulp from a blend of hardwoods[11]. It should be noted that the Reynolds number based on the hydraulic pore diameter (defined by Eq. (3)) varied within the interval of 0.57×10^{-3} to 2.6×10^{-3}, indicating that all experiments were conducted in the creeping flow regime.

Time parameters

Further parameters that seem to be useful tools suitable for describing the displacement washing mechanisms are time parameters defined by Eqs. (4) and (5).

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[9,10]. 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 relationships between the mean residence time and the space time for kraft spruce pulp may be expressed as follows

$$t_m = 1.94\,\tau \qquad (11)$$

with a correlation coefficient of 0.96 for the kappa number of 18.1, and

$$t_m = 1.64\,\tau \qquad (12)$$

with a correlation coefficient of 0.98 for the kappa number of 50.1.

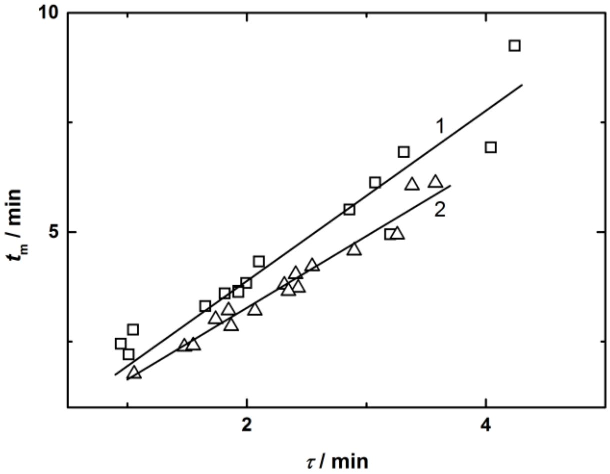

Figure 4. Mean residence time as a function of space time for kraft pulp cooked to kappa number of 18.1 (□) and of 50.1 (△). 1 Eq. (11), 2 Eq. (12)

Conclusions

The preliminary results obtained for spruce kraft pulp with the kappa numbers of 18.1 and 50.1 enabled that some conclusions valid within the framework of our study can be made.
(i) The wash yield measured for kraft spruce pulp with the kappa number of 50.1 was greater in comparison with that for the kappa number of 18.1.
(ii) One of possible reasons may be lower rate of leaching influencing the mean residence time of alkali lignin in the case of kraft spruce pulp with low kappa number. The mean residence time for kraft spruce pulp with lower kappa number was found to be greater comparing to that for pulp with kappa number of 50.1.
(iii) The axial dispersion coefficient increases with increasing the Reynolds number. Greater values of the axial dispersion coefficient were achieved for kraft spruce pulp with higher kappa number.
(iv) The dispersion of alkali lignin in the pulp bed is induced particularly by the mechanical or geometric dispersion resulting from the fluctuations of the local wash liquid velocity caused by fibres random configuration and also by molecular diffusion at the interface between the miscible fluids, namely wash liquid and black liquor. The results obtained showed that the pulp fibre bed formation is a critical step influencing alkali lignin dispersion during displacement washing. Even if the experimental conditions during pulp bed formation are strictly identical, the difference in geometry, that is, in average pore size and in pore size distribution must be taken into account.

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Symbols

a_v	specific surface of pulp fibres, m^{-1}
D	axial dispersion coefficient, $m^2\,s^{-1}$
d_m	hydraulic mean diameter (= $4\,\varepsilon/(a_v\,(1-\varepsilon))$), m
h	thickness of pulp bed, m
Pe	Péclet number based on bed thickness defined by Eq. (2), dimensionless
Re	Reynolds number (= $u\,\rho/(a_v\,\mu\,(1-\varepsilon))$), dimensionless
RW	wash liquor ratio, dimensionless
t	time, min
t_m	mean residence time defined by Eq. (4), min
u	wash liquid superficial velocity, $m\,s^{-1}$
$WY_{RW=1}$	wash yield at RW = 1 defined by Eq. (1), dimensionless

Greek letters

ε	average porosity of packed bed, dimensionless
κ	kappa number, dimensionless
μ	wash liquid viscosity, Pa s
ρ	wash liquid density, kg m^{-3}
ρ_e	exit solute (in our case alkali lignin) concentration from bed, kg m^{-3}
ρ_i	solute concentration in wash liquid, kg m^{-3}
ρ_0	initial solute (in our case alkali lignin) concentration in bed at $t = 0$, kg m^{-3}
τ	space time defined by Eq. (5), min

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