SHORT COMMUNICATIONS

On the Extraction of Residual Lignin in the Unbleached Sulfite Pulp

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(Received March 4, 1958)

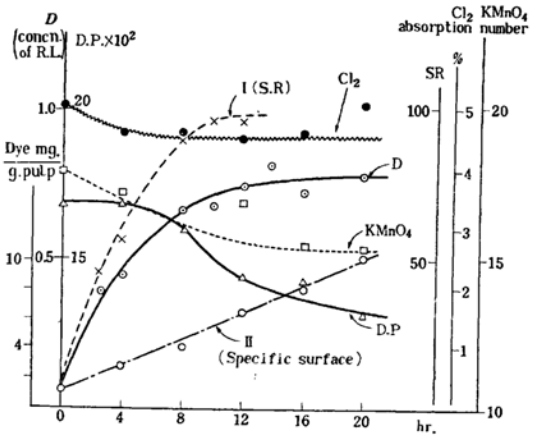
Generally lignin can not be removed completely by ordinary pulping process. In order to elucidate the bleaching mechanism, it is desirable to make clear the nature of the residual lignin (R.L.) in the unbleached pulp. Mainly because of the difficulties of separation of this lignin, almost nothing is known about it. As the reasons for this difficulty, one or several of the following facts will be considered in the case of sulpfite process. Residual lignin 1) is not sulfonated sufficiently, 2) is combined to carbohydrate firmly, 3) goes into solution with difficulty, because of morphological reasons of fiber, and 4) is condensed to other lignin unit during the digestion.

We have found that the resolution of residual lignin is effected with drastic beating in water, which causes fibrillation of the fiber and is expected to diminish morphological hindrance of the dissolution of R.L.

Commercial unbleached sulfite pulp (air dry, 540 g.) prepared from spruce (*Picea jezoensis*) was suspended in water (21 l) and beaten with NIAGARA-TYPE 1 to 1/2 pound BEATER (TAPPI-STANDARD T200m-45) for 20 hr.

Fig. 1 shows the course of treatment.

Curve I shows the increase of freeness. Freeness levels off at about SR 96 after 12 hr. no further increase being observed. Specific surface (curve II) measured with



dye (Benzo Fast Scarlet 4BS) adsorption[1], increased linearly, and this shows that the fibrillation was progressing yet after beating 20 hr. On the other hand, the curves representing the permanganate number and the chlorine absorption of the beaten pulp have a slightly downward tendency.

The dissolution of R.L., measured by the ultraviolet absorption spectra of the filtrate of the beaten pulp which was diluted three times with water, reached maximum at 12 hr. After 8 hr., the D.P. of pulp began to decrease slowly. In this treatment the amount of the dissolved lignosulfonic acid does not exceed one-fifth of the total amount of R.L. Further dissolution of the remaining lignosulfonic acid in the beaten pulp is now under investigation.

The sulfonation degree of this lignin thus obtained is as low as that of the so-called "low-sulfonated lignosulfonic acid." Analytical data of R.L., low-sulfonated lignosulfonic acid (LSLA)[2] and ordinary lignosulfonic acid (L.S.A.)[3] are compared in the following table.

R.L.	$C_9H_{9.43}O_{3.90}(OCH_3)_{0.88}$
	$(SO_3Ba/2)_{0.27}S^{neut}_{0.038}\ B^{excess}_{a_{0.014}}$
L.S.L.A.	$C_9H_{9.80}O_{3.76}(OCH_3)_{1.05}$
	$(SO_3Ba/2)_{0.23}S^{neut}_{0.03}\ B^{excess}_{a_{0.028}}$
L.S.A.	$C_9H_{10.7}O_{3.2}(OCH_3)_{1.05}$
	$(SO_3Ba/2)_{0.51}S^{neut}_{0.08}\ B^{excess}_{a_{0.04}}$

The amount of the total phenolic hydroxyl group of R.L. measured by the G.A.-Erdtman's method[4] is 0.17~0.21 per methoxyl, being the same with that of L.S.A. In the case of R.L. the phenolic hydroxyl group belonging to type I[5] is only 20% of the total, the remaining 80% being expected to be type II.

Type I Type II

Along with the dissolution of R.L., some amount of hemicellulose goes also into solution.

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2) H. Mikawa, K. Sato, T. Takasaki and K. Ebisawa: unpublished data.
3) H. Mikawa, K. Sato, T. Takasaki and K. Ebisawa: This Bulletin **28** 653 (1955).
4) G. A. Erdtman: *Svensk Papperstidn* **56** (1953) 287.
5) H. Mikawa, K. Sato, T. Takasaki and K. Ebisawa: This Bulletin **29** 245 (1956).