Polarographic Determination of Lignosulfonic Acid in Sulfite Spent Liquor

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Determination of lignosulfonic acid in sulfite spent liquor is very troublesome, because in sulfite spent liquor there are many impurities which behave very similarly to lignosulfonic acid in its physical and chemical behavior.

The present communication deals with the rapid and accurate determination of lignosulfonic acid in sulfite spent liquor with polarographic method. Lignosulfonic acid does not show any d. c. polarographic waves. It was found, however, that lignosulfonic acid combines quantitatively with polarographically active methylene blue to give polarographically inactive form, leaving excess methylene blue active.

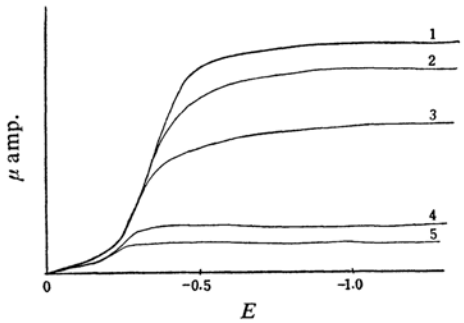

Fig. 1. Polarogram of methylene blue with lignosulfonate.
KCl: 0.5 N. Methylene blue: 0.00125 mol.
1 Lignosulfonate: 0%
2 Lignosulfonate: 0.01%
3 Lignosulfonate: 0.02%
4 Lignosulfonate: 0.05%
5 Lignosulfonate: 0.06%

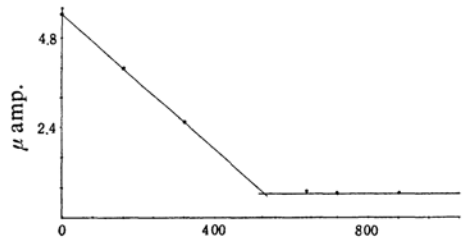

Fig. 2. The relation of wave-height to lignosulfonate concentration.
KCl: 0.5 N, Methylene blue: 0.00125 mol., Sodium lignosulfonate: 0~900 g./mol. M.B.

The polarograms of methylene blue and the combination of methylene blue and purified lignosulfonic acid are shown in Fig. 1. The wave height of methylene blue is depressed linearly by addition of lignosulfonic acid and after a certain point it becomes constant as shown in Fig. 2. This equivalent point was 530 g. of lignosulfonic acid per 1 mol. of methylene blue, being in good agreement with the sulfonic acid equivalence of lignosulfonic acid. Diluted and neutralized sulfite spent liquor, which contained 12.0% of lignosulfonic acid by β-naphthylamine method and 11.4% by methoxyl method, was analyzed by this method, and it was found that 11.6% of lignosulfonic acid was contained.

In order to simplify this method, the amperometric titration of methylene blue by sulfite spent liquor was carried out at −0.8 V. of mercury electrode potential. 11.6% of lignosulfonic acid was found again in the sulfite spent liquor.

These results show that in sulfite spent liquor only lignosulfonic acid combines quantitatively with methylene blue and is precipitated in polarographically inactive form. This method is very useful for the rapid and accurate determination of lignosulfonic acid in sulfite spent liquor.

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