

Isolation of Kraft Black Liquor Lignin



Kraft black liquors acquired from the end of each cook and at other times during the cooks, were isolated by precipitating the lignin from solution by acidification. The black liquors were first filtered through a Whatman #4 filter paper on a Bukner funnel. Approximately 0.5 grams of EDTA-2Na⁺ was added for every 100 ml of black liquor. The liquors were neutralized with 2 M H₂SO₄ until a pH of approximately 6.0 was reached.

The solutions were then stirred vigorously for one hour. The liquors were further acidified to a pH of 3.0 and frozen at -20°C. After thawing the solutions, the precipitates were collected on a medium sintered glass funnel and washed twice with cold water by suspending the precipitates in the water and stirring vigorously at 0°C. The precipitates were collected, air dried, and extracted with pentane for 8 hours in a soxhlet extractor to remove sulfur and other impurities.

The kraft lignin precipitates were further purified by suspending them in a 9:1 dioxane:water solution and stirring for an hour. The solutions were centrifuged and the supernatants collected. The precipitates were washed again with 9:1 dioxane:water and centrifuged. The supernatants were combined and filtered over celite on a medium sintered glass funnel. The dioxane was removed by rotary evaporation and the purified kraft lignins freeze dried. These purified kraft lignins were then used for the subsequent analyses and chemical derivatization.

