ABSTRACT

PROCESS DESIGN FOR THE PRODUCTION OF MALEIC ACID HYDRAZIDE FOR WEED CONTROL

by

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In the investigation the effects of excess maleic anhydride, hydrazine hydrate-solvent reagent addition time, volume of reaction mass after concentration by heating at 100 °C, mixing of reactants, and the solvent selected were studied for the heterogeneous reaction of maleic anhydride and hydrazine hydrate. A reaction time of 12 minutes was employed with the ratio of solvent to reactants held constant at 75 weight per cent throughout the tests. Solvents employed in the investigation included ethanol, methanol, isopropanol, glacial acetic acid, water, hydrachloric acid, and benzene. Atmospheric drying tests at 25 to 88 °C were employed on hydrazide slurries of free moisture content from 1.258 to 1.515 pounds of water per pound of hydrazide. Centrifuge tests at 2000 to 4700 revolutions per minute and a rotary filtration test under a 10 inch vacuum were employed on 13 weight per cent hydrazide slurries. Hydrazide filtrate evaporation tests at 100 °C

were performed on samples of 18 to 1715 milliliters to determine the approximate hydrazide content in the slurries.

Field applications of 0.10 to 0.22 weight per cent hydrazide solutions in water were made on "wild" varieties of briers, bermuda grass, johnson grass, milkweed, red pine, ragweed, and honey locust in the Blacksburg, Virginia, area from May to August, 1956.

The yield of maleic acid hydrazide was increased from 42.6 to 67.0 per cent when the maleic anhydride excess was increased to 20 per cent in the reaction. The optimum addition time for the ethanol-hydrazine hydrate reagent to the maleic anhydride was found to be 3.8 seconds, while the optimum volume of reaction mass after concentration by heating at 100 °C was 10 to 15 milliliters for the non-agitated reactions. Agitation of the reaction mass and the solvent chosen were determined to increase the yield of the hydrazide. The optimum drying temperature and time for the drying of the hydrazide slurries were determined to be 88 °C and 75 minutes, respectively. Rotary vacuum filtration of the hydrazide slurries was determined to produce a cake free moisture of 1.33 pounds of water per pound of hydrazide as com-

pared with 1.38 for the centrifuge test at 4700 revolutions per minute. The hydrazide content of the filtrate samples was determined to be approximately 10 to 15 per cent.

Field applications on "wild" plots indicated that 40 to 80 per cent control of briers, bermuda grass, ragweed, johnson grass, and red pine could be achieved from one application of 0.10 to 0.22 weight per cent hydrazide solutions in early spring. On milkweed and honey locust growth, the spraying solution would not adhere to the leaf.

A total fixed plus working capital of $1,151,740 was determined to be necessary to build a plant for the production of 242 tons of 95.5 per cent pure maleic acid hydrazide per year. On this basis, a selling price of $3.00 per pound ($0.05 per gallon) would yield a 13.7 per cent return as new earnings on total fixed plus working capital.