Methodologies to Characterize Nutrient Release of Slow and Controlled Release Fertilizers for Regulatory and Formulation and Implementation Applications

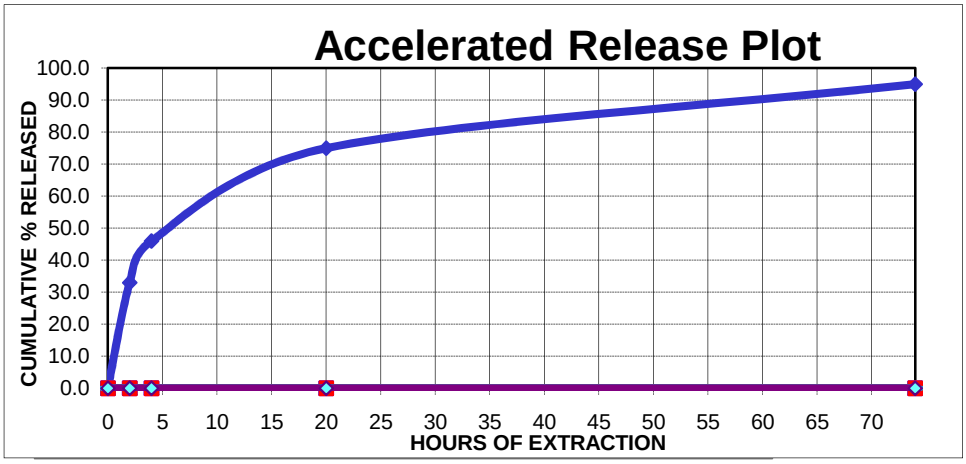
W. Hall, Mosaic, Mulberry, FL; L.C. Medina, Monsanto, St. Louis, MO; J.B. Sartain University of Florida, Gainesville, FL; T Obreza, University of Florida, Gainesville, FL

Many slow and controlled -release fertilizers include a nutrient release claim for a specific time period. These claims include remarks such as – "will release in a single growing season", "80% of the nutrients will release within 200 days at XX temperature". Currently no validated methodology exists to verify such longevity claims; most are based on company or product specific internal testing. The Association of American Plant Food Control Officials (AAPFCO) has worked to address this gap in analytical methodology and claim verification. In response, a short-term (74 hr.) extraction method was developed to assess nutrient release under accelerated laboratory conditions. Additionally, a long-term soil incubation method was developed to characterize nutrient release at ambient temperatures with a biologically-active soil environment. Both methodologies have been optimized by evaluating the effect of critical variables such as temperature and time of extraction on nutrient release characteristics. Ruggedness and suitability of the ambient soil incubation (soil sample size, incubation temperature, and soil type) and laboratory extraction methods (sample size, extraction temperature, and extraction time) will be discussed. Correlation of the two methods is critical to demonstrate the ability of the accelerated method to assess nutrient release in a controlled laboratory environment, yet in a timely (4 to 7 days) manner. Manuscripts for this work have been submitted and accepted for publication. Work is underway to validate both accelerated and ambient soil methods through AOAC international. Additionally the accelerated method is proposed as a new work item through ISO TC-134. This approach will provide an official method to verify nutrient release claims of slow and controlled release fertilizers.

Accelerated Method

A representative unground sample is exposed to increasingly aggressive extractions. A 30 g sample is suspended in a jacketed chromatography column. Temperature controlled water is circulated around the sample in the outer column as extraction solution (0.2% citric acid) is pumped through the inner column and sample. By raising temperature and increasing extraction time as extractions proceed each extraction is designed to isolate nutrients that become available over time. Data collected by analysis of each extract is used to develop information specific to the cumulative percent of total nutrient released.

Example accelerated release data display –

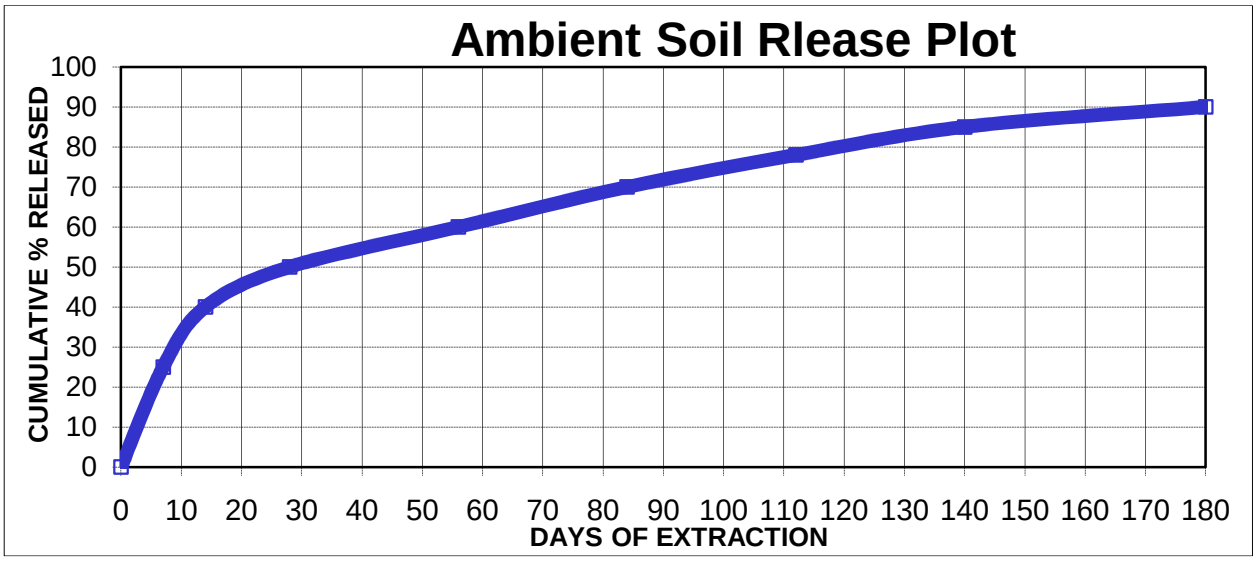


Extraction sequence - Day 1 - Extraction #1- 2 hrs. @25°C; extraction #2- 2 hours @ 50°C; extraction #3- 20 hrs. @ 55°C; day 2 - extraction #4 - 50 hrs. @ 60°C; day 4 – extraction #5 - 94 hours @ 60°C - complete extraction 4, begin extraction #5; day 7 - complete extraction 5.

Ambient Soil Method

A soil incubation-column leaching technique determine long-term nitrogen (N) release patterns of slow and controlled release fertilizers. The leaching column technique is based on the aerobic incubation of a sand/soil/fertilizer mixture exposed to intermittent leaching during a 180 day period. These mixtures included: 1710g of non-coated quartz sand; 90g of the surface (0 to 15 cm) layer of a typical soil from Florida such as Arredondo fine sand; and the equivalent of 450mg N from the material under evaluation. Components were mixed, placed in lysimeters, and incubated for 180 days. The incubation lysimeters were constructed from cylindrical PVC pipe measuring 30 cm long by 7.5 cm diameter. Mixtures were retained in the lysimeters by glass wool mesh placed at the bottom. The sand/soil/fertilizer mixture was initially moistened to 10% moisture content by adding 180 mL of water. An ammonia trap consisting of 20 mL of 0.2 M H2SO4 contained in a 50 mL beaker was placed in the head space of the lysimeter. This solution was replaced and analyzed for NH4-N by titration every 7 days to determine volatile-N. The columns were incubated at ambient temperature (averaging 21°C) in the laboratory. Nine leachate fractions were collected at 7, 14, 28, 42, 56, 84, 112, 140 and 180 days from inception using one pore volume of 0.01% citric acid (500 mL). The use of citric acid aids in pH control (minimize volatilization) and provides carbon to support microbial activity. The columns were leached by gravity and excess solution was removed under vacuum for 2 min. The vacuum after leaching and gaseous interchange through the connected at the bottom cap was sufficient to maintain the system in aerobic conditions; thus ensuring microbial activity and limited denitrification losses. At each leaching, the leachate volume was recorded and an aliquot was frozen for analysis of total N. The weight of total N recovered in the leachate was determined by multiplying N concentration by leachate volume. At each sampling interval, the total N released versus time was determined by adding total N in the leachate with the volatile-N.

Example ambient soil release data display –



The two methods produce data that can be used to predict the outcome of the other, thus providing a way of comparing the accelerated data to the ambient soil data. This provides a tool for regulators to assess longevity claims as well as for producers and users to assess potential best fit products.

W. L. Hall, Product Stewardship Specialist, Mosaic Fertilizer LLC, 13830 Circa Crossing Drive, Lithia, FL 33547 Phone:813-500-6670, email: bill.hall@mosaicco.com; L.C. Medina, Monsanto, Phone: 314-335-7574, email: carolina.medina@monsanto.com; J.B. Sartain, Soil and Water Science Dept., 414 Newell Hall, Univ. of Florida, Gainesville, FL 32611; ; T.A. Obreza, Interim Associate Dean for Extension, Univ. of Florida/IFAS, 1038 McCarty Hall, P O Box 110210 Gainesville, FL 32611-0210, Phone: 352-392-1761.