Review

Methods for Determining Nitrogen Release from Controlled-release Fertilizers Used for Vegetable Production

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ADDITIONAL INDEX WORDS. incubation, column, pouch method, leaching, enhanced efficiency

SUMMARY. The purpose of this article is to review nitrogen (N) controlled-release fertilizer (CRF) research methods used to measure nutrient release from CRFs. If CRF-N release patterns match vegetable crop needs, crop N uptake may become more efficient, thus resulting in similar or greater yields, reduced fertilizer N needs, and reduced environmental N losses. Three methods categories to estimate N release are: laboratory; growth chamber, greenhouse, or both; and field methods. Laboratory methods include a standard and accelerated temperature-controlled incubation methods (TCIMs); methods incubate CRF using selected time periods, temperatures, and/or sampling methods. Accelerated TCIMs, in contrast to the standard method, allow for shorter incubation periods. Growth chamber and greenhouse methods, including column and plastic bag studies, may be used to test new CRF products in conditions similar to particular vegetable production systems. However, the column method predicts N release from CRFs more effectively than the plastic bag method because of ammonia volatilization and lower N recovery rates associated with the bag method. Both field methods, pot-in-pot and pouch methods, are viable vegetable research options. The pouch method measures N remaining in the CRF prill and the pot-in-pot method measures N released from the CRF, thus each method can be applied to different research objectives. Nitrogen released during incubation may be measured using methods such as total Kjeldahl N (TKN), prill weight loss, combustion, colorimetric, or ion-specific electrodes. The prill weight loss method is the least expensive but can only be used with urea CRF. Thus, the CRF-N source(s) and research objectives will determine the appropriate N analysis method. More research needs to be completed on correlations of field and laboratory CRF extractions. Field release methods should be considered the most reliable indicator of CRF-N performance until a laboratory method reliably predicts CRF-N expected field response.

Enhanced efficiency fertilizers (EEF) are a group of fertilizers that reduce the risk of nutrient loss to the environment and

subsequently increase fertilizer use efficiency (Slater, 2010). This increase

may be accomplished through maintaining nutrients in the root zone by physical barriers (coating), reduced solubility, or retaining nutrients in a less leachable form (Trenkel, 2010). There are three subgroups of EEFs with different characteristics for horticultural production systems. Slow-release fertilizers (SRFs) contain N in a less-soluble, plant-unavailable form that usually need to be microbially degraded into plant available N. Stabilized fertilizers are a group of fertilizers that have a chemical inhibitor to either stop the oxidation of ammonium (NH_4^+) to nitrate (NO_3^-) by bacteria or to slow the enzymatic transformation of urea to NH_4^+ (Trenkel, 1997). Controlled-release fertilizers, the last subgroup of EEFs, are urea, ammonium nitrate, potassium nitrate, or other soluble fertilizer materials coated with a polymer (polyethylene and ethylene-vinyl-acetate or thermoplastics), resin (a subgroup of polymers and refers as alkyd-type resins and polyurethane-like coatings), sulfur, or a hybrid of sulfur-coated urea (SCU) coated with a polymer or resin. These coated materials release nutrients in water at a predictable rate when used at the manufacturer specified temperature (e.g., 25 °C) (Trenkel, 2010). The European Committee for Standardization's (2002) method determines nutrient release time based on 75% nutrient release from CRFs.

The European Union has developed both standard and accelerated laboratory procedures for measuring N release from CRFs; however, researchers in the United States are still developing a universal test for CRFs and SRFs for commerce purposes (European Committee for Standardization, 2002; Sartain et al., 2004). Growth chamber and greenhouse methods are used to evaluate or compare how CRFs will act in a particular controlled environment (Broschat and Moore, 2007; Huett and Gogel, 2000). Lastly, field methods are used to measure N release in commercial

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Units

To convert U.S. to SI,
multiply by

U.S. unit

SI unit

To convert SI to U.S.,
multiply by

29.5735

fl oz

mL

0.0338

2.54

inch(es)

cm

0.3937

645.1600

inch²

mm²

0.0016

28.3495

oz

g

0.0353

(°F - 32) ÷ 1.8

°F

°C

(1.8 × °C) + 32

vegetable field conditions (Simonne and Hutchinson, 2005). Each research method has its own advantages and disadvantages (Engelsjord et al., 1996; Sartain et al., 2004; Simonne and Hutchinson, 2005). Therefore, the objective of this publication is to describe and summarize laboratory, growth chamber, greenhouse, and field methods currently used to measure CRF-N release on vegetable production using the research literature.

Laboratory methods

Laboratory methods allow for CRF incubation in controlled environmental conditions compared with field conditions. These methods may be used to compare CRFs and to quickly screen CRFs when an accelerated method is used. These methods may be used to predict laboratory release but will not predict field release when used alone. There are two types of methods based on release time. The so-called standard method incubates CRF for specified nutrient release time or until a threshold amount of nutrients (e.g., 75%) are released (Dai et al., 2008; Du et al., 2006; European Committee for Standardization, 2002). The accelerated method incubates CRF for a shorter time (e.g., 74 h) at a higher temperature than the standard methods (Dai et al., 2008; European Committee for Standardization, 2002; Sartain et al., 2004). There are variations in both TCIMs, each designed to test CRF using selected time periods, temperatures, and/or sample collection methods.

TEMPERATURE-CONTROLLED INCUBATION METHOD—STANDARD. The standard TCIM incubates a beaker containing CRF and water [i.e., 12.5:250, 5:33.3, and 1:50 (grams of CRF:milliliters of water)] at a constant temperature of 25 °C (Dai et al., 2008; Ko et al., 1996; Shaviv, 2001). Incubation times are based on manufacturer-stated release length (4-month release), or based on research objectives such as measuring release until 100% of the urea is released (Dai et al., 2008; Ko et al., 1996). Typically, CRF remain static during incubation (Du et al., 2006; Lamont et al., 1987; Perez-Garcia et al., 2007). However, the European standard method incubates CRFs with stirring at 25 °C.

TEMPERATURE-CONTROLLED INCUBATION METHOD—ACCELERATED. For commercial applications, the

standard TCIM produces reliable results, but requires extended incubation periods to achieve the 75% nutrient release requirements, thus accelerated methods have been developed (Dai et al., 2008). Sartain et al. (2004) and Medina et al. (2009) described a 74-h accelerated TCIM, which was correlated to CRF incubated in columns. Sartain et al.'s (2004) method uses jacketed chromatography columns that consisted of hollow glass tubes surrounded by an integrated water jacket where the sample can be placed in the inside and water controls the temperature. The method uses four separate extractions per sample with temperatures increasing from 25 to 60 °C and time to obtain a release curve. Most methods use water as the extracting solution, while Sartain et al. (2004) use dilute citric acid (0.2 N). Sartain et al. (2004) and Medina et al. (2009) found that the accelerated method could successfully predict N release from a variety of column-incubated SRF and CRF products. Dai et al. (2008) used five separatory funnels to incubate trincote, resin-coated CRF at 50 to 90 °C and continuously leached (250 mL per 15 min) CRF samples for 6 h. The results of the high temperature incubations were compared with a standard TCIM at 25 °C and found that 80 °C was the optimal temperature partially due to reduced coating integrity at 90 °C (Dai et al., 2008). Accelerated TCIMs have the advantage of reducing the time and labor cost compared with the standard TCIM, but neither predict field release.

Growth chamber and greenhouse methods

Growth chamber and greenhouse methods may be used to test CRF products in conditions more similar to a particular production system compared with laboratory methods (Abraham and Rajasekharan Pillai, 1996; Broschat, 1996; Broschat and Moore, 2007; Sato and Morgan, 2008).

COLUMN STUDIES. Column studies use columns that measure 30 cm long and 5 to 7.5 cm in diameter (Broschat, 1996; Broschat and Moore, 2007; Huett and Gogel, 2000; Sartain et al., 2004). One end of the column, which is considered the bottom, is fitted with mesh or gauze, then placed in a funnel, reservoir, or capped (the

cap contains a luer fitting for drainage). The columns are positioned vertically and filled with media and the top capped. Sartain et al. (2004) included an ammonia (NH₃) trap in the top of the column to capture volatilized NH₃. A 5-g sample of CRF can be placed 1 to 5 cm below the media surface. The standard column method uses hydrochloric acid washed sand to reduce the likelihood of nutrient retention by the media (Broschat, 1996; Broschat and Moore, 2007; Huett and Gogel, 2000). Engelsjord et al. (1996) used peat to fill the columns; however, peat mineralization affected N release results. Sartain et al. (2004) filled columns with 95% uncoated quartz sand and 5% surface layer (sand) for biological activity to convert urea to nitrate. Columns are leached at different frequencies and volumes of water depending on the goal of the research and the size of the column. Huett and Gogel (2000) and Broschat and Moore (2007) leached columns three times per week using 80 and 50 mL of water, respectively. Leachate was collected once per week for 53 weeks (Huett and Gogel, 2000). Conversely, Broschat and Moore (2007) collected leachate weekly until all leachate nutrient concentrations were less than 3 ppm; a resin-coated fertilizer had 100% nitrate release and ≈10% iron release in 64 and 40 weeks before leachate concentrations fell below 3 ppm. Medina et al. (2008, 2009) leached columns with 500 mL citric acid (0.1 N) at increasing intervals from 7 to 270 d and 7 to 180 d. Incubation temperatures in growth chambers or greenhouses should match field soil temperatures or the manufacturers' specified temperature if testing manufacturers' claims.

PLASTIC BAG METHOD. The plastic bag method uses plastic zipper bags filled with 100 to 250 g of soil and a sample of CRF incubated at room temperature (Cahill et al., 2010; Sartain et al., 2004). Cahill et al. (2010) found high standard error of the means for polymer-coated urea, phosphate-coated urea, granular urea, and urea ammonium nitrate incubated using this method. Sartain et al. (2004) reported a strong smell of NH₃ when the bags were opened and a maximum N recovery rate of 60% from biosolids, SCU, urea-formaldehyde, isobutylidene diurea, and urea. For these reasons, the plastic bag method will be a poor research tool to use in CRF-N

research making the column method the preferred greenhouse and growth chamber method.

Field methods

Field methods can be used to determine how CRFs will release under actual field conditions. The field method should subject the CRF to an environment similar to CRFs applied in vegetable production systems (Wilson et al., 2009). Ideally, CRF-N release matches crop N uptake and releases N throughout the entire vegetable production cycle (Lammel, 2005). Nitrogen release should be measured throughout the entire crop cycle or until 75% of the N is released or recovered (Trenkel, 1997).

POUCH METHOD. The pouch method uses pouches made of fiberglass mesh screen that allows movement of moisture to the CRF prill. For the release curves to accurately reflect environmental conditions, the pouch materials must not interfere with water movement to CRF prills. Polymer-coated urea incubated in polypropylene mesh pouches with 1.2-mm² openings had significantly greater N release than pouches constructed from weed block material with 0.07-mm² openings (Wilson et al., 2009). Pouch dimensions range from 2 × 2.4 to 5 × 5 inches with CRF sample sizes ranging from 1.3 to 5 g N (Gandeza et al., 1991; Haase et al., 2007; Jacobs et al., 2003; Medina, 2006; Wilson et al., 2009; Zvomuya et al., 2003). Soil can be included into the pouch with the CRF sample (Broschat, 2005; Gandeza et al., 1991). Pouch placement in the field should follow growers' production practices such as buried under vegetable beds with plastic mulch or in open potato hills (Broschat, 2005;

Medina et al., 2008, 2009; Wilson et al., 2009; Zvomuya et al., 2003). Pouches can be collected at predetermined times during the vegetable production cycle and remaining N in the CRF can be determined. Medina et al. (2008) found that CRFs performed differently in citrus groves with different row orientation (north to south vs. east to west) because of the different wetting and drying patterns found in the groves. Differences due to grove-row orientation show that the pouch method allows CRF prills to be subjected to real field environments. This method measures N remaining in CRF prills, thus pouch studies can be used when CRF behavior is monitored (Simonne and Hutchinson, 2005).

POT-IN-POT METHOD. The pot-in-pot method consists of two 8-inch pots nested together separated by a 0.75-inch spacer. The interior pots with screened drain holes were filled with soil and a 4.8 to 6.2 g CRF sample. Covered pots were buried in a potato hill with 1 inch of the bottom pot above the soil surface (Simonne and Hutchinson, 2005). Incubated pots were leached with water at prearranged dates, and the following day leachate volumes were collected and measured. The pot-in-pot method and the column method measure N released from the CRF rather than N remaining in the prills. Measuring released N takes into consideration soil microbial activity on the N, thus being representative of plant available N (Simonne and Hutchinson, 2005). The project research objectives will determine the importance of measuring N released in leachate, while requiring additional labor, or measuring N remaining in the CRF prill. Important factors to consider are that environmental field conditions can be

highly variable, and CRFs are temperature dependent, therefore field studies must include all growing seasons and multiple years (Fraisie et al., 2010). For vegetable production, both CRF field methods can be viable methods for measuring CRF-N successfully.

Correlations between methods

Controlled release fertilizer nutrient release differs in free water, water saturated sand, and sand at field capacity (Du et al., 2006). Thus, TCIMs without correlation can offer only restricted practical use for commercial vegetable production because the results will not reflect nutrient release obtained under field conditions. Sartain et al. (2004) compared the accelerated TCIM extraction of polymer SCU with a column extraction at room temperature and found a positive *R*² of 0.90 suggesting that the accelerated TCIM may be able to predict N release from column incubations accurately. A correlation between the TCIM and a field method has not been done. In field conditions, there are several factors to consider such as release time, temperature, moisture, placement, rate, and cultural practices making the correlation difficult to achieve (Sartain et al., 2004). These complex effects and their interactions present under field conditions added variability compared with that found with the TCIM methods.

Procedure to measure N

With all CRF research methods (laboratory, growth chamber, greenhouse, and field methods), N concentration in leachate or in the CRF prills needs to be measured after incubation. Methods to measure N include TKN (Gandeza et al., 1991; Greenberg et al., 1985; Haase et al., 2007;

Table 1. Nitrogen (N) determination method to use with different incubation methods, N species, and controlled-release fertilizer N source.

N determination method	CRF incubation methods ^z	N species measured ^y	CRF-N source ^y
Total Kjeldahl N	L, G, F	organic-N, NH ₄ -N, and NO ₃ -N with modification	NH ₄ -N, NO ₃ -N, and urea
Prill weight loss	F (pouch)	None—measures change in mass	Urea only
Combustion	L, G, F	NH ₄ -N, NO ₃ -N, and urea-N	NH ₄ -N, NO ₃ -N, and urea
Colorimetric	L, G, F	NO ₃ -N or NH ₄ -N	NH ₄ -N, NO ₃ -N, and urea
Ion-specific electrodes	L, G, F	NH ₄ -N, NO ₃ -N, and urea, but it must be transformed with urease	NH ₄ -N, NO ₃ -N, and urea

^zL = laboratory, G = growth chamber and greenhouse, F = field methods.

^yNH₄-N = ammonium-N, NO₃-N = nitrate-N.

Table 2. Cost of laboratory analysis for nitrogen (N) content remaining in controlled-release fertilizer prills in a field trial consisting of 6 replications, 5 treatments, and 11 sampling dates (Medina et al., 2008).

N determination method ^z	Cost/unit (\$) ^y	Total cost (\$) ^x
Total Kjeldahl N (TKN)	8.25–13.20	2,723–4,356
Prill weight loss	0.17–0.20	56–66
Combustion	7.5–11.4	2,475–3,762
Colorimetric (NO ₃ -N or NH ₄ -N)	6.00–10.00	1,980–3,300 (NO ₃ -N)
	7.00–10.00	2,310–3,300 (NH ₄ -N)
	11.6–12.5	3,828–4,125 (both) ^w
Ion-specific electrode	0.25–0.33	83–110

^zNO₃-N = nitrate-N, NH₄-N = ammonium-N.

^yLaboratory method unit cost for TKN, combustion, and colorimetric are a range of prices given on analytical laboratories websites (University of Minnesota Research Analytical Laboratory, St. Paul; Cornell Nutrient Analysis Laboratory, Ithaca, NY; University of California—Davis Analytical Laboratory, Davis, CA; and Pennsylvania State University Agricultural Analytical Services Laboratory, University Park) where the applicable laboratory tests are performed. Unit cost for prill weight loss and ion-specific electrode methods are based on a laboratory technician measuring 50 to 60 and 30 to 40 samples per hour, respectively, at an hourly rate of \$10/h and that the laboratory equipment is available (D.E. Lucas, personal communication).

^xTotal costs estimates, collected in Jan. 2012, do not include shipping and do not include drying time, which may be necessary for some methods, with inconsequential labor costs to evaluate dryness.

^wThis cost represents the price set by laboratories when both NO₃-N and NH₄-N are measured.

Zvomuya et al., 2003), prill weight loss (Salman et al., 1989; Savant et al., 1982), combustion (Wilson et al., 2009), colorimetrically with an auto-analyzer (Pack et al., 2006), and ion-specific electrodes to measure NH₄⁺ or NO₃ (Broschat, 2005) (Table 1). The standard and most popular method, TKN, is a time consuming laboratory procedure, which includes concentrated sulfuric acid and sodium hydroxide. All CRF-N sources and research methodologies may use TKN. Prill weight loss is a quick procedure where the mass of dried-incubated prills is subtracted from the original dry prill mass. Unfortunately, this method may only be used with pouch-incubated urea CRF. Each type of ion [e.g., NH₄⁺, NO₃⁻, potassium (K⁺), urea, etc.] diffuses out of the CRF prill at a different rate; therefore, it cannot be assumed that the ion ratio inside the incubated CRF prill and the nonincubated CRF prill are equal. For example, a potassium nitrate fertilizer is composed of 50% K⁺ ions and 50% NO₃⁻ ions. Nitrate releases more quickly than K⁺, thus K⁺ will represent a larger portion of the nutrients in the prill near the end of a trial (Broschat and Moore, 2007). Combustion and colorimetric N determination with an autoanalyzer use a solution, so both methods may be used with any of the CRF research methods or N sources. Ion-specific electrodes may be used to measure N in leachate and solubilized (homogenized) CRF prills; however, free urea cannot be measured using these electrodes unless the urease

enzyme is added and the solution is incubated (Guilbault et al., 1969). Wilson et al. (2009) compared prill weight loss to combustion methods and found that both were equally reliable methods for measuring N release.

Laboratory analysis of N remaining in CRF prills or in leachate varies in cost. Table 2 shows laboratory costs for N analysis from a pouch incubation trial consisting of 6 replications, 5 treatments, and 11 sampling dates (Medina et al., 2008). Prill weight loss costs the least per sample to measure N, but this method can only be used with pouch-incubated CRF urea. Ion-specific electrodes are the next most inexpensive method, but each electrode only measures one N species, therefore more than one electrode must be used to measure total inorganic N. Both organic-N and NH₄-N may be measured using TKN. Total Kjeldahl N costs more to conduct than the prill weight loss or the ion-specific electrode method. Total Kjeldahl N does not measure NO₃-N, but modified methods are available to measure NO₃-N along with NH₄-N and organic-N (Latimer, 2010). Selection of a laboratory that uses the modified method will reduce the number of analytical tests required to measure total fertilizer N. Colorimetric measurements for NO₃-N or NH₄-N need separate analyses. Using both colorimetric N analyses would be more expensive than all methods but TKN. The combustion method costs around the same amount as TKN; neither method provides N species

information like colorimetric analysis. Since, the prill weight loss method and the combustion method are equally acceptable, it would be fiscally responsible to use prill weight loss to determine N release, when CRF type allows. Depending on the amount of information needed regarding N species, multiple methods can be used to measure N released for CRF on vegetable production systems.

The accelerated TCIM is preferred when compared with the standard TCIM method because of the savings on time and labor costs. Column studies can be used to test new CRFs before going to the field from controlled environments, but column studies can be time consuming with associated high cost. Field methods will be the preferred research tools by vegetable growers until the accelerated TCIM has been correlated and calibrated to field studies with a positive crop response, thus determining a CRF's suitability for vegetable production in a shorter amount of time.

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