Forms, Properties and Dissolution of Controlled-Release Nitrogenous Fertilisers

A review is made of the current information concerning the forms, properties and dissolution rates of controlled-release nitrogenous fertilisers. These materials belong to one of the categories: Coated materials, uncoated sparingly water-soluble inorganic and organic compounds. Physical and chemical properties of the commonly-used fertiliser materials are described along with their dissolution characteristics. Similar information about indigenous materials proposed as controlled-release N sources is scarce and is urgently needed to evaluate their comparative efficacy. Future research needs about controlled-release nitrogenous fertilisers are also examined.

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Fertilisers in general and nitrogenous fertilisers in particular have made a major contribution toward agricultural productivity. There is, however, a continuous need to improve the efficiency of N fertiliser use in order to achieve more efficient production of crops and minimise fertiliser-related environmental stresses. Results of several field studies show that only 50-60% of the fertiliser nitrogen is usually recovered by crops plants. The recovery values of applied N for crops such as rice is generally lower than 50%.

Many of the factors which contribute to the poor recovery of N by plants are a result of rapid dissolution of the applied fertiliser materials and release of more mineral N than what is used by the plant or conserved by the soil in available forms. Among the various strategies explored to increase N-use efficiency, development of controlled-released N-carriers is one. Several reviews cover various aspects of controlled release N fertiliser such as chemistry of their manufacturing processes, their reactions in soil and other media and their practical use for food, forage and horticultural crops (1,13,14,15,22,23,28,31). The objective of this paper is to review the present status of research pertaining to the forms, properties and dissolution characteristics of controlled-release nitrogenous fertilis-

ers. The need for future research with regard to the practical use of these materials is also examined.

Forms of Controlled-release Nitrogenous Fertilisers

The following two broad-based approaches to achieve control over dissolution of N have been followed (14):

(i) Preparation of N compounds or materials with sparing or limited water solubility, by chemical reactions e.g. urea-aldehyde condensates, oxamide and metal ammonium phosphate.

(ii) Development of coated soluble-N sources by physical methods in order to retard or control the release of N into the soil solution. For example, sulphur-coated urea, polymer-coated urea, etc.

Several terms used to signify controlled-release are found in the literature: slow-release, controlled availability, slow acting, sustained release, metered release and delayed release. At times, the use of these terms is rather arbitrary and is usually seen relative to the fast N release by soluble N materials, especially prilled urea.

Improved N efficiency can also be

achieved with chemical additions to control soil microbial reactions e.g. by use of nitrification and urease inhibitors, nitrification and urea hydrolysis may be controlled in soil. The fertilisers amended with these chemicals do not strictly fall into the category of controlled-release materials because it is not clear as to how the water solubility and or dissolution of the amended fertilisers is altered by these additives. The literature on urease and nitrification inhibitors is voluminous and has been extremely well reviewed (12, 13, 14, 23, 24, 25).

1. Basic concepts of controlled-release N materials

According to Hauck and Koshino (14) the controlled-release N materials can be classified into four types:

(i) water-soluble N materials containing plant available N where dissolution is controlled by a physical barrier e.g. by a coating;

(ii) N materials of limited water solubility and plant available N forms, e.g. metal ammonium phosphate;

(iii) materials of limited water solubility which release N in plant available forms during their chemical and microbial
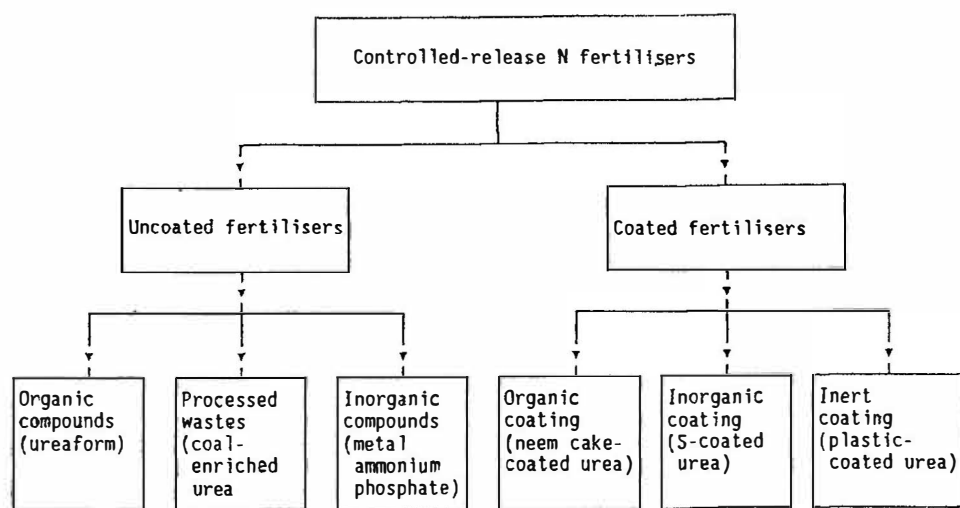
Figure 1 - A general classification of controlled-release nitrogenous fertilisers

decomportion e.g. the ureaforms; and

(iv) soluble or relatively water-soluble N materials which gradually decompose and release mineral N, e.g. guanylurea salts.

All the above forms of controlled-release materials can be covered under coated and uncoated materials and will be discussed under these sub-headings.

A general categorisation of controlled-release N fertilisers is proposed and depicted in **Figure 1**. The proposed classification incorporates the basis of classification used by Hauck and Koshino (14). The proposed classification is more comprehensive and simple. An example of each class of slow-release materials is given in parentheses (**Figure 1**).

2.   Coated-controlled release materials

Coatings, encapsulations, and matrixes have been used to alter the physical characteristics of soluble N materials. The coatings applied can be : (i) impermeable types with tiny pores through which soluble N diffuses; (ii) continuous impermeable coatings that must be decomposed by chemical, microbial or abrasive action before the N is released; and (iii) semipermeable coating through which water diffuses under the internal

solution pressure is sufficient to cause disruption. In a matrix, grains of fertiliser are entrapped in a medium so that diffusion and outward flow of fertiliser solution is impeded by tortuosity of the matrix. Encapsulation can be achieved by using a perforated polythene bag.

Detailed discussion on the use of proposed coating materials in relation to release of N is available in several reviews (10, 15).

3.   Uncoated controlled-release materials

These are sparingly soluble N materials and the release of N is dependent on the rate of dissolution and removal of the solubilised constituent from the fertiliser surface. Dissolution can be controlled by size and compactness of the fertiliser particle.   Larger and harder fertiliser particles dissolve at a slower rate than small and soft particles.

4.   Properties and dissolution characteristics of controlled-release materials

Important physical and chemical properties of several controlled-release materials are discussed under uncoated controlled-release and coated controlled-release subheads. Importance is given to those materials which have found

practical application. Solubility in water provides a good index of dissolution rate of these materials because solubilisation leads to degradation of these compounds following hydrolysis and chemical and biological reactions. In addition, this is the solvent which these materials will encounter when added to soils.

1.   Uncoated controlled-release materials

Numerous chemically prepared organic and inorganic compounds have been proposed as controlled-release materials (1, 13, 14, 15, 16, 19, 22, 28, 30). A comprehensive review of literature containing 1200 references covering the period 1962-1976 shows that among the   most widely-studied, chemically prepared, slow-release materials, the only ones to enjoy any measure of commercial success are the urea-aldehyde addition products. Several waste products including low-grade coals and humates were also covered but they have a limited scope (22).

i) Urea-aldehyde condensation materials

a) Ureaforms : Urea reacts with formaldehyde in the presence of a catalyst to form a mixture of compounds called ureaforms. Ureaforms are white odourless solids which contain about 38% N. According to a definition adopted by the Association of American Fertiliser Control officials ureaform should have at least 35% N, largely insoluble in water but in slowly available form. Water insoluble N should be at least 60% of the total N content of ureaform (15).

The N activity index for ureaforms is an empirical index of water insoluble N available for nitrification in six months.

$$\text{Activity index} = \frac{\%CWIN - \%HWIN}{\%CWN} \times 100$$

Where, CWIN is cold water insoluble N at $25 \pm 2°C$ and HWIN is fraction of N insoluble in hot (98-100°C) aqueous solu-

tion of a phosphate buffer such as KH_2PO_4 or K_2HPO_4 (pH 7.5).

An activity index of 40 or more is considered satisfactory for use as fertiliser. Ureaform is primarily a mixture of methylene urea polymers. It also contains small amount of free urea. A ureaform product with an activity index of 35 has cold water insoluble N of 28% and a moisture content of 3% (3).

The specification for commercial ureaform are (15) :

Total N : 38.0% (minimum)
CWIN : 27.0% (minimum)
Activity index : 40 (minimum)

For products of different activity indexes the release of N would vary greatly. For example, a cold water soluble fraction, which contain 2-4, average number of urea per molecule would release all the N during a few weeks time. While the fraction insoluble in cold but soluble in hot water may take several months to release the N. The fraction of N insoluble in hot water is considered extremely slow-release and the release of N may take 1-2 years (15).

b) Urea - Z : The reaction of urea with acetaldehyde gives a mixture of compounds consisting primarily of ethylene diurea and 2-ethylene-3-urea. Small amount of free urea and ethylol urea is also present. The mixed product, known as urea-Z, contains 33-38% N, of which about 15% is water soluble (14). Urea-Z has a solubility in water of about 0.4%.

c) Crotonylidene diurea (CDU) : It is a condensation product of urea-acetaldehyde or urea-crotonaldehyde in the presence of an acid catalyst and is a ring structured compound, CDU with two side-chain nitrogen atoms. The N content of CDU is about 32% and water soluble or hydrolyzable N compounds constitute less than 3%. Its solubility in water is 0.6g/l at 20°C and increases to 2.2g/l at 50°C. Its solubility is higher in dilute sulphuric acid. The release of N from CDU is also influenced by its size of granules. The release of N is reported to be considerably slower under waterlogged conditions than under upland soil conditions (23).

d) Isobutylidene diurea (IBDU) : IBDU is the major reaction product of urea and isobutyraldehyde under acid catalysed conditions. It is a white non-hygroscopic compound containing 32.2% N with low solubility in water. The solubility in water ranges from 0.1-0.01% depending on pH and temperature, which control both solubility and hydrolysis of IBDU. Fertiliser grade IBDU contains about 30% N. Because of its low solubility in water, the effectiveness of IBDU is greatly influenced by its particle size. Particle size is indeed the key factor in controlling the rate of N release from IBDU.

ii) Other urea-aldehyde condensation materials

Several other urea-aldehyde condensation materials have been proposed as controlled-release fertilisers e.g. glycoluril, a condensation product of urea with glyoxal is a ring compound containing 39.4% N. It has a water solubility of only 0.2% at 30°C. Similarly, difurfurylidene diurea, a condensation product of urea with futural has 25% N with very low water solubility (14, 28).

iii) Organic compounds

a) Triazines : Several triazines have been proposed as controlled-release N materials, the important ones being cyanuric acid, ammeline, ammelide and melamine. Triazines are polymerised products of urea formed in the presence of ammonia at high temperature. These have a six-numbered ring containing alternating carbon and nitrogen atoms. One or all hydroxyl groups attached to the three carbon atoms can be replaced with amino groups to form a family of compounds — cyanuric acid, ammeline, ammelide, and melamine — identical in structure except for the replacement of zero, one, two, or all hydroxyle groups, respectively. The corresponding N contents of cyanuric acid, ammeline, ammelide and melamine are 32.4, 43.7, 49.4 and 66.5%. They have very low rates of dissolution in water. This coupled with their high N content and relative ease of production make them as potential controlled-release N fertilisers (14, 20).

b) Guanylurea : Guanylurea is formed by polymerising cyanamide with dicyandiamide and then hydrolysing dicyandiamide to guanylurea. Nitrate, phosphate and sulphate salts of guanylurea have been proposed as slow-release N sources. These contain 37.1, 27.8 and 37.0% N, respectively (14). Guanylurea is decomposed mainly by soil microbial activity and N release is affected by soil and environmental factors. The material decomposes more rapidly in waterlogged soils especially when the soil is reduced (Eh below 0 mV). Guanylurea is strongly adsorbed on soil colloids and thus its use finds favour under flooded conditions despite its appreciable solubility in water (16).

c) Oxamide : It is the diamide of oxalic acid, and is a white, non hygroscopic compound, which contains 31.8% N. Oxamide is sparingly soluble in water (0.4 g/l at 7.5°C). The rate of N release from oxamide is reported to be similar to that from IBDU and depends on particle size (34). However, oxamide is decomposed by microbial activity in the soil and factors such as pH, temperature and moisture affect N release.

iv) Other organic compounds

Among several organic compounds proposed and evaluated as controlled-release N fertilisers, thiourea and dicyandiamide have been reported to have slow-release properties (14, 23). Both these compounds are toxic to plants when used at higher rates to supply enough N. However, these have nitrification inhibitory property and are used as amendments to fertilisers to delay nitrification, where the application rates are much lower (14, 23, 24, 25). Hexamethylene-

tetraamine (hexamine) proposed as a slow-release N compound was found to release mineral N at a rate only slightly slower than ammonium salts (5).

Recently, a urea-sulphur product has been proposed as a slow-release fertiliser. The product is a hard granule, virtually dustfree with a guaranteed analysis of 30-0-0-20 S. This material has uniform granule size and is also recommended for blending with other fertilisers (2). Elemental S in the fertiliser is absorbed by plants only when oxidised to sulphate form. This product behaves like an organic N source and differs considerably from sulphur coated urea. There is no coating on the urea molecule and N release is controlled by biochemical processes involving urea hydrolysis and S oxidation.

2. Processed organic waste products

Several waste materials ranging from crop residues such as bagasse, non-edible oilseed cakes, molasses, saw dust, seaweed, seed hulls to wastes which as enriched-coal, dried blood, wool and leather scrap have been processed by heating, oxidation, nitration, ammonification and other processes to raise their N content for use as slow-release N materials (14, 16, 21, 24, 32).

A product prepared from oxidative ammoniation of coal has been proposed as a slow-release N material (6). The product contains 20% N two-thirds of which is slowly available. In another study it was found that the mineralisation of enriched coal (14-22% total N) was very low and after 40 days of incubation only 15% of the N was released (23). Similar coal-enriched materials have been prepared and proposed as controlled-release N sources in Germany, India, Japan, Russia, South Africa and USA (1, 13, 14).

In India several oilseed cakes have been used as slow-release N sources to supplement N from chemical fertilisers because they alone are low (3-8% N) in N content

(24). Infact, recently these materials have been used as coatings for preparing slow-release urea-based fertilisers (30, 31).

Ammoniated vermiculite has been proposed as an efficient controlled-release N source for a variety of crops (27). The conditions under which the clay is ammoniated control the nature and N content of the product.

The greater need for efficient waste disposal would stimulate further development of processed organic wastes as slow-release nutrient sources. But from an economic point of view these materials must primarily be considered as end products for the waste disposal system rather than as competitive fertiliser materials (14).

3. Uncoated incorganic compounds

Several metal ammonium phosphates with the general formula, $MeNH_4PO_4.XH_2O$ where Me is a divalent metal have been studied. These materials have limited water solubility. Magnesium ammonium phosphate is the most common of these compounds and metal ammonium phosphates containing Mn, Zn, Fe (ferrous), Cu and Co as the divalent metal are also available. All these have N content varying from 6.9 to 8.3%. The nitrification of metal ammonium phosphate controls the availability of its N to plants. The rate of nitrification varies with granule size. The larger the granules, the slower the rate of nitrification in soil (14, 23).

4. Coated fertilisers

Coated fertilisers with controlled-release N property are prepared by physically coating with semi-permeable membranes or inert materials on to otherwise soluble fertiliser granules. Soil temperature is the most important factor that greatly influences the release of N from coated materials, and generally, a 10°C increase in temperature doubles the rate of release.

i) Sulphur-coated urea : Sulphur-coated urea, first developed by TVA, is the most common slow-release N source. Sulphur-coated urea contains 36-38% N. The release of N from sulphur-coated urea is controlled by the thickness and uniformity fo the coating, any microbiocide added, temperature and time of contact with the soil. Sulphur-coated urea has dissolution rate in water ranging from 17.5%-35.0% in 7 days and a rate of 1% or less thereafter for periods up to 100 days (32). The rate of N release from sulphur-coated urea was reported to be about 1% per day after the first day (8,17). The release of N is slightly higher in soil than in water, with greater release in acidic pH soils than in alkaline pH soils (28).

Moisture regime greatly affects the mineralisation of sulphur-coated urea. Prasad et al (23) reported a slower mineralisation under waterlogged conditions than under field capacity moisture. In some soils, reduction of the outer layer of sulphur to sulphide led to the formation of a precipitate of ferrous sulphide around the urea pellet, which sealed the pellet against the action of soil urease. Sulphur-coatings thus appear to be more stable in waterlogged than in aerated soil systems (14). Sharma (28) discussed results from literature and stated that N release from sulphur-coated urea is much higher in moist than in flooded soil and supports the views expressed by others (14, 23).

ii) Fertilisers coated with inert materials : Several water-resistant coatings or membranes have been employed to cover soluble N fertiliser granules with the aim of achieving controlled-release of N. Among the range of coating materials, prominent ones are polyethylene, acrylic acetate, chlorinated rubber, polypropylene, polyvinylidene chloride, polyvinyl alcohol, polyester, polycarbonate and polymide (9,10,22,23). Several resins especially thermoplastic resins, waxes, paraffins, gums, tars and asphaltic substances have also been tried. Fujita et al (10) and Fujii and Yazawa (9) have made excellent

reviews on the use of different coating materials for the development of controlled-release materials.

Fujita et al (10) found that among the several resins tested thermoplastic resins were found to be the most suitable coating material. Polyolefin resin and polyvinylidene chloride resin were found to have the least water vapour permeability (1-2 g/m^2/24h) while materials such as cellulose, polyvinyl alcohol and polyamide had high degree of water permeability (> 100 g/m^2/24h).

Important physical and chemical properties and dissolution of some coated fertiliser materials are discussed below:

iii) Polyolefin-coated urea (POCU) : POCU (40% N) is a controlled-release fertiliser whose N dissolution rate is temperature-dependent. The release of urea is partly due to a concentration gradient but largely due to dissolution into the absorbed water and diffusion through voids in the coating film (11). The dissolution of POCU at higher temperature is characterised initially by a zero-order reaction followed by a first-order reaction, while at lower temperature, dissolution follows a zero-order reaction. The dissolutin rate of POCU under field conditions was predicted using soil or air temperature. Cumulative N-release gradually increased with time after application and reached about 80% of the total N content after 125 days (11). Cumulative N release (CNR) with time between 10 and 30°C was described by a quadratic equation in the general form CNR = a + b (DAF) + c (DAF)^2, where a, b, and c are constants and DAF is days after application of the fertiliser.

iv) Reactive layer coated urea (RLCU) : The material is prepared by coating urea with a mixture of diisocyanate and polyol in the presence of a catalyst. The hard, durable layer that results on the granule imparts the slow-release character to the product. The release of N from the product increase with increasing temperature and decreas-

ing coating thickness but was unaffected by pH or addition of arbon sources to increase microbial activity (7). Based on the urea particle size and coating thickness, the 7-day release rate of the product in water ranged from 20 to 73% at 38°C.

v) Gypsum coated urea : The product is developed by coating urea with gypsum. The hard and durable shell that develops, imparts a controlled-release N character to the product. The product contains about 35% N and the coating starts disintegrating 3-4 days after application (4).

vi) Neem (Azadirachta indicata) cake coated urea : A product containing 38% N has been developed by ICI (India) but no other details of the product are available. It appears difficult to produce neem-cake coated urea with a N content higher than 38% N.

vii) Neem extract-coated urea : A concentrated extract from neem seeds has been developed in India and is commercially marketed as NIMIN. The extract contains about 5% neem bitters, mainly triterpenes. The coating agent comprises 1% by weight of urea and causes only a small reduction in the N content of the coated urea. The neem extract is insoluble in water but is soluble in organic solvents such as benzene and xylene. The extract is self-adhesive and no external adhesives are required. Significant inhibition of soil nitrification was achieved upto 30 days or longer with the product as compared to untreated urea (33). Another report using incubated soils showed that the neem extract-coated urea had a significantly slower rate of nitrification upto 2 weeks as compared to ordinary urea (18).

viii) Lac-coated urea (LCU) : The Shellac Export Promotion Council in India has developed a urea-based controlled-release N fertiliser. The method involves coating of urea granules with shellac, a refined form of lac. The product contains about 34% N (26). Incubation studies with soil showed that LCU had weak slow-release property as compared

to IBDU, CDU and ureaform fertilisers as most of its urea (95% of 200 mg N/kg soil) was hydrolysed by first week in three soils (pH 5.4, 6.0 and 8.3) under waterlogged or at 80% WHC moisture and at room temperature (28-33°C). However, about 8% of IBDU and 59% of CDU nitrogen was released after first week of incubation under similar conditions of temperature and soil moisture. It was concluded that LCU showed little controlled-release property and behaved like uncoated urea (26).

Conclusions and Future Research Needs

Controlled-release nitrogenous fertilisers can be used to advantage especially in situations where fast release rate of mineral N from conventional fertilisers leads to inefficient use of N and its losses. Materials with a range of dissolution characteristics can be further developed simply by changing particle size or coating thickness and by suitably choosing coatings with differing controlled-release characteristics and by adding microbiocides.

A lot of work has been done under laboratory and greenhouse conditions and there is an obvious need to evaluate promising materials under field conditions and under model field conditions for simulating crop response to N. Clearly, a better understanding between N release characteristics and plant needs is needed to choose ideal slow-release fertilisers suitable for different crops and cropping systems.

However, recent research by Japanese workers (9, 10, 11) shows that the release of N from polyolefin - coated urea, controlled by temperature, can be predicted under field conditions using either soil or air temperature. Cumulative N release during a cropping season is controlled by cumulative soil or air temperature factor. The results from these studies are encouraging because they would have significant implications on N supply to plants, fertiliser N use efficiency and N related environmental pollution.

Basic data on the dissolution characteristics and other properties of most indigenously processed materials need to be generated to supplement field data as well as to further modify their N release characteristics. Research is especially needed to developing coatings for urea that are resistant to degradation, light in weight and thin so that N content of the product is not lower than 40%. Heavier coatings reduce the N content of the coated fertilisers and thus introduce hidden costs associated with them during bagging, storage, handling, transport and application costs per unit of N.

Suitable controlled-release N materials are needed for cropping systems whre N-use efficiency is low for example under rice culture and in high rainfall areas with coarse-textured soils.

There is need to evaluate mixtures of promising uncoated controlled-release N sources and coated materials under laboratory, greenhouse and field conditions to develop materials suitable for cropping systems involving short and long-duration crops.

Finally, for controlled-release materials to be attractive for practical agriculture, they need to be not only technologically sound and agronomically effective but also their use should be economically attractive and practically feasible. Unless the extra cost associated with them is offset by higher crops yields as a result of improved N efficiency, their use would remain confined to speciality crops, glasshouse plants, lawns, golf courses and ornamentals.

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