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ORIGINAL PAPER

Nutrient release from biodegradable polymer-coated multi-nutrient fertilizer granules in calcareous soils

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Abstract

The use of conventional fertilizers is associated with higher nutrient losses and lesser fertilizer use efficiency; hence, to increase the use efficiency by enhancing the longevity of fertilizer in the soil, coated fertilizers have been developed. The prolonged nutrient release through coated fertilizers may provide continuous plant nutrition, better growth, and improved plant performance. Biodegradable organic acids and amino acids were used as coating materials to control the rate of release of plant available nutrients thereby increasing the nutrient use efficiency. The organic acid and amino acid coated multi-nutrient fertilizer granules (MNFG) were fabricated using citric acid, humic acid, fulvic acid, salicylic acid, and glycine at 3, 5, and 10% of coating concentration by w/v and tested for their nutrient release potentials in calcareous soil. The pH, EC, free $CaCO_3$, and nutrient release from coated fertilizer granules were monitored under controlled conditions, and the results revealed that coating of MNFG with 10% citric acid recorded lesser pH (7.78) and free $CaCO_3$ (7.85%) after the expiry of 45 days of incubation period. The reduction was marked on calcareousness (34.6%) than pH (6.71%). Higher availability of sulfur and micronutrients in calcareous soils was noted with citric acid-coated MNFG granules at 10% where the rate of release was controlled for 45 days. The results demonstrated that the profile of nutrient release from calcareous soil could be controlled by the thickness of coating, and fertilizer nutrient release which fit the crop nutrient requirement in calcareous soils was observed with 10% citric acid coating of MNFG granules.

Keywords Coated fertilizers · Multi-nutrient fertilizer granules · Organic acids and amino acids · Coating concentrations · Calcareous soil · Nutrient release

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Introduction

Soil calcareousness is the major growth limiting factor in many of the arid and semiarid regions, which naturally limits plant growth and yield due to its poor soil organic matter and high pH with lesser availability of essential plant nutrients. The fixation and precipitation of applied macro- and micronutrients in calcareous soils is the major constraint that corresponds to very low use efficiency of applied fertilizer nutrients in the soil due to various chemical reactions like precipitation, fixation, and adsorption even after employing best management practices for crop production (Kassem et al. 2022). The presence of higher $CaCO_3$ in calcareous soils further aggravated the precipitation of applied nutrients into insoluble compounds. Hence, developing suitable management strategies combined with judicious use of nutrients is essential. Increasing the effectiveness of plant nutrient assimilation could be achieved through the application of the multi-nutrient fertilizer which provides many nutrients at a time for plants due to its abundant concentrations (Ni et al. 2012). Research on mixed application of nutrients to calcareous soils is scanty and needs detailed studies (Meena et al. 2022; Shukla et al. 2021).

Among the technological interventions, the use of controlled release fertilizers (CRF) by coating with various polymers to create a physical barrier for controlling the rate of nutrient release is very much essential. The fertilizer may be in the form of granules or pellets that are coated with hydrophobic or partially hydrophilic compounds that restrict the dissolution of fertilizers (Naz and Sulaiman 2016; Gil-Ortiz et al. 2020) and designed to release plant nutrients in a steady manner so as to synchronize the crop demand (Sarkar et al. 2018).CRF is a safer, economical, and more efficient way of administering nutrients to crops. It defeats the disadvantages of traditional fertilizers through constant nutrient delivery, minimal loss, and fertilizer application frequency (Das and Ghosh 2022). A single application of CRF can provide nutrients throughout the crop growing season, thereby minimizing the cost of farming operations (Marcinczyk and Oleszczuk 2022). CRF usage is associated with the improvement of plant growth conditions, such as reduction in stress and specific toxicity resulting from excessive nutrient supply in the root zones (Sempeho et al. 2014). Various coating agents were employed to regulate the nutrient release (Sofyane et al. 2021). Sulfur was tried initially as a coating agent (Azeem et al. 2014) but due to its low wettability, adhesion to the coated core, and sealing resulted in additional expenses to the final product. Moreover, any S remaining in the soil reacts with water thereby acidifying the soil. Subsequent research has focused on polymeric materials derived from petroleum and plastic products (Lligadas et al. 2006). These materials performed better in controlling nutrient release than organic and inorganic ones (Azeem et al. 2014). The resins and thermoplastics were used to produce organic polymers, whereas inorganic coatings were synthesized from sulfur, graphene oxide, gypsum, palygorskite, and many other mineral-based materials (Andelkovic et al. 2018). The fertilizers coated with plastic resin may leave undesired residues of synthetic materials in soil, which can stay in the soil for prolonged periods of time and disrupt soil functions (Ni et al. 2012). The drawback of most of the polymer-coated CRF is the need of organic solvents, which are not readily biodegradable after nutrient release thus creating a new source of soil pollution (Fertahi et al. 2021). To overcome these economic and environmental obstacles, current research focuses shifting toward biopolymers that are biodegradable, biocompatible, and non-toxic and contribute to improvement in soil quality (Azeem et al. 2020; Cerri et al. 2020).

The coating of fertilizers using organic acids helps in improving fertilizer use efficiency and reduces the losses in essential plant nutrients. Humic acids and fulvic acids improved the growth of plants as soil conditioners and enhanced the ability of soil to hold more nutrients (Khan et al. 2019; Mohan and Malarvizhi 2020). They promote good soil structure and increase the water holding capacity of the soil and enhance the growth of useful soil organisms thereby increasing the plant performance. Humic acid-coated urea is a good alternative nitrogen source to neem oil-coated urea in the future (Balaganesh et al. 2021). Citric acid is considered as one of the important organic acids that have a huge potential in the production of CRFs (Awadhiya et al. 2016; Mali et al. 2017). It acts as the growth promoting agent, improving photosynthesis and ultimately plant growth, and it reduces the soil pH hence acting as a chelate for positively charged cations in soil solutions (Tusei 2019). Salicylic acid-enriched polymer coating of fertilizers improved the nutrient release in soils and its positive effect depends upon its concentration and environment (Yasmeen et al. 2021). It has many positive impacts on plant growth viz., protein synthesis, nutrient transfer, ion absorption, and gas exchange (Hassoon and Abduljabbar 2019). Amino acids also are of an interesting agent for coating due to their bio-compatibility and higher stability (Aghazadeh et al. 2017). Amine functionalization increases the chance of surface interactions. Glycine could easily attach to the surface of ions through their carbonyl groups and resulted in the controlled release of added nutrients (Dolev et al. 2020). In plants, amino acid act as chelators and significantly improves the nutrient uptake efficiency and accelerated their translocation within the plants (Aslani and Souri 2018).

The quality of coating was important in controlling the nutrient release and was determined by the source and concentrations of polymer used. The thickness, solubility, and biodegradability of coating play an important role in the release of nutrients (Bortoletto-Santos et al. 2019). Previous studies have shown that coatings control the rate of release of nutrients. However, there is a lack of information on the effect of citric acid, humic acid, fulvic acid, salicylic acid, and glycine coatings and their concentrations on nutrient release and recovery in soils (Cruz et al. 2017). Organic acids and amino acids have several positive impacts on soil physicochemical properties and also on plant growth. Hence, the present study aimed to evaluate the rate of nutrients release from the multi-nutrient fertilizer granules coated with different concentrations of organic acids and amino acids in calcareous soils.

Materials and methods

Polymer-coated fertilizer materials

Along with NPK, secondary and micronutrient deficiencies (either single-nutrient or multi-nutrient deficiency) have also been reported in various soils of India. Both macro- and micronutrients play an equally important role in plant metabolism and yield enhancement. Therefore, to maintain the soil fertility and increase the crop productivity, it is imperative to use balanced fertilization (Shukla et al. 2021). Individual fertilizer nutrients application to crops addresses single-nutrient deficiency and involves higher cost also. Under such conditions, application of multi-nutrient fertilizers supplies several plant nutrients simultaneously that are essential for crops and plays an important role in enhancing the crops yield substantially. The granulated multi-nutrient fertilizer (MNFG) was prepared using straight commercial fertilizers of sulfur as well as micronutrients (zinc, iron, copper, and boron). All the fertilizer materials at specified composition based on the weightage of crop demand were physically mixed together in a solid form until they became homogenous. After the addition of a binder, the mixtures were shaken at 250 rpm in a horizontal shaker for 30 min until it gets complete granulation. The granules were sieved to segregate into different sizes and shade dried. Analar grade organic acids and amino acids such as citric acid, humic acid, fulvic acid, salicylic acid, and glycine were used as coating agents. Citric acid, salicylic acid, and glycine were obtained from JK scientific company, Coimbatore, Tamil Nadu. Humic acid and fulvic acid were purchased from Greenlife Biotech Laboratory, Coimbatore, Tamil Nadu. Coatings were performed at 3, 5, and 10% (w/v) concentrations for all the coating materials. The uniform size of MNF granules with an average diameter of 2 mm were coated with various organic acids and amino acids at specified coating concentrations through repetitive spraying after drying. The coated products were dried completely in a hot air oven at 40 °C and tested for their nutrient release and recovery on calcareous soils.

Characterization of multi-nutrient fertilizer granule (MNFG) and experimental soil

The granules of multi-nutrient fertilizer are of regular, spherical shape and white in color with a slight gray shade and lesser crushing strength (soft). The color of the granules changing from light brown to dark brown in citric acid, fulvic acid, and glycine coating; black for humic acid coating; and for salicylic acid coating, it was greenish gray which was majorly associated with the nature of polymers. The granules coated with citric acid and glycine exhibited the highest tensile strength (hard) whereas fulvic acid and salicylic acid coating showed moderate strength (medium hard). On the contrary, the lowest strength (soft) was observed with humic acid-coated MNF. The pH, EC, grain weight, and bulk density of MNFG were 4.04, 6.48 dS m^{-1}, 0.07 g, and 0.80 Mgm^{-3}, respectively. The sulfur content was 17.1% with the iron, zinc, copper, and boron contents of 4692, 869, 725, and 98.1 mg kg^{-1}, respectively. The solubility and moisture content of the granules were observed as 260 g L^{-1} and 2.04%, respectively, in the fabricated MNFG. The physicochemical properties of MNFG were altered significantly by the nature of coating materials and their concentrations. Irrespective of all sources and their concentrations, coating of fertilizer slightly decreased the nutrient contents. Uncoated MNFG recorded the lesser bulk density and EC with higher solubility, moisture content, and pH. The higher bulk density (0.95 Mg m^{-3}), lesser solubility (70 g L^{-1}), and moisture content (0.50%) were recorded in salicylic acid (10%)-coated MNF. Higher EC (8.56 dS·m^{-1}) and lesser pH (2.36) of the MNFG were recorded with citric acid (10%) coating.

The soil used in the study was collected from the farmer's field at Coimbatore district in Tamil Nadu, India (11°16′07.1″ N, 77°06′27.9″ E). The soil was processed, sieved through a 2-mm sieve, and air dried and stored for further use. It was sandy clay loam in texture with a pH of 8.34 and belongs to Periyanayakenpalayam soil series (Typic ustrochrepts). It was moderately calcareous in nature with a free $CaCO_3$ content of 12%. The electrical conductivity (0.22 dS·m^{-1}) and available nitrogen (252 kg ha^{-1}) were low in status, medium in available phosphorus (13.4 kg ha^{-1}), and high in available potassium (521 kg ha^{-1}). The secondary nutrients were sufficient in availability, and the values were 312, 148, and 13 mg kg^{-1} for calcium, magnesium, and sulfur, respectively. As regards the micronutrients, the soil was sufficient in available Cu (0.84 mg kg^{-1}) and Mn (3.09 mg kg^{-1}) while deficient in available Fe (5.22 mg kg^{-1}), Zn (0.72 mg kg^{-1}), and B (0.37 mg kg^{-1}).

Experimental details

An incubation experiment was conducted with two main factors including coated multi-nutrient fertilizer granules (citric acid, humic acid, fulvic acid, salicylic acid, and glycine) at four different concentrations (0, 3, 5, and 10%) and replicated thrice in the Department of Soil Science and Agricultural Chemistry, Tamil Nadu Agricultural University, Coimbatore. A hundred gram of processed experimental soil was placed in 250-ml plastic bottles and applied with the coated multi-nutrient fertilizer granules at 12.5 kg ha^{-1} which was fixed as the optimum dose for multi-nutrient application based on previous research works. Water holding capacity was determined as outlined by Viji et al. (2012). The soil was saturated to field capacity with double distilled water based on weight loss and restored to field capacity by the addition of required double distilled water at a two-day intervals. Destructive sampling was carried out on 7, 15, 30, and 45 days after incubation, dried, homogenized, and analyzed for basic soil properties and available nutrients as per standard procedures.

Measurement of basic soil properties and available nutrients

The pH and electrical conductivity in 1: 2.5 soil–water suspension was determined by using digital pH meter. The EC was measured using conductivity meter and expressed in dS·m^{-1} (Jackson 1973). The free $CaCO_3$ content (%) in the soil was estimated by the rapid titration method (Piper 1966). The amount of soil available sulfur was estimated using 0.15% calcium chloride extractant and expressed in mg·kg^{-1}(Williams and Steinbergs 1959). Available soil micronutrients were determined using 0.005 M DTPA extractant by atomic absorption spectrophotometry (model: GBC Avanta PM) as described by Lindsay and Norwell (1978). Water-soluble boron in the soil was determined by azomethine-H method using UV spectrophotometer at a wavelength of 420 nm (Berger and Truog 1939).

Statistical analysis

Data recorded during incubation was analyzed statistically under factorial experiment based on a completely randomized block design with three factors, viz., coating materials, concentrations, and days with three replications (Snedecor and Cochran 1967) in SPSS 16.0 software. Valid conciliations were drawn after determining significant differences between treatments, and the mean comparisons were made using Fisher's least significant difference (LSD) test at $P = 0.05$. The critical difference was calculated to compare the treatment means.

Results and discussion

Soil reaction (pH)

Data showed that pH was significantly affected by coating materials and their concentrations, as increasing coating concentration resulted in decreasing pH irrespective of all polymers with the overall mean of 7.99 to 8.30. Higher reduction in soil pH (7.78) was noted with the application of 10% citric acid-coated MNF granules followed by 10% fulvic acid-coated MNFG (7.85). Lesser reduction in soil pH was recorded in soil treated with uncoated MNF granules (8.27) even after 45 days of incubation period which was shown in Table 1. Increasing coating concentrations of polymer brought significant changes in reducing the soil pH and higher coating concentration (10%) recorded greater pH reduction. The order of higher pH reduction with polymers was as follows: citric acid (6.71%) > fulvic acid (5.88%) > humic acid (5.28%) > salicylic acid (4.20%) > glycine (3.36%) regardless of their coating concentration. The effect of sources and coating concentrations was significant in influencing the soil pH whereas the interaction between the sources, coating contractions, and incubation time was found non-significant.

Table 1 Effect of various sources and coating concentrations of organic acids and amino acids on soil pH

Sources	7th day					15th day				
	Coating concentrations (%)					Coating concentrations (%)				
	0	3	5	10	Mean	0	3	5	10	Mean
Citric acid	8.32^{ae}	8.24^{abe}	8.21^{abce}	8.18^{ace}	8.24	8.28^{abe}	8.21^{abe}	8.16^{abce}	8.10^{abce}	8.32
Humic acid	8.33^{ac}	8.28^{abc}	8.25^{abc}	8.22^{ac}	8.27	8.28^{abc}	8.25^{abc}	8.22^{abc}	8.18^{abc}	8.33
Fulvic acid	8.34^{ad}	8.26^{abd}	8.23^{abcd}	8.20^{acd}	8.26	8.31^{abd}	8.25^{abd}	8.19^{abcd}	8.15^{abcd}	8.34
Salicylic acid	8.31^{a}	8.30^{ab}	8.26^{abc}	8.22^{ac}	8.27	8.29^{ab}	8.26^{ab}	8.23^{abc}	8.19^{abc}	8.31
Glycine	8.29^{ab}	8.31^{ab}	8.28^{abc}	8.25^{abc}	8.28	8.27^{ab}	8.27^{ab}	8.25^{abc}	8.21^{abc}	8.29
Mean	8.32	8.28	8.25	8.21	8.26	8.29	8.25	8.21	8.17	8.32
	30th day					**45th day**				
Citric acid	8.26^{be}	8.12^{be}	8.08^{be}	7.90 ^{be}	8.09	8.24^{ce}	8.01^{ce}	7.96^{ce}	7.78^{ce}	8.26
Humic acid	8.24^{bc}	8.18^{bc}	8.15^{bc}	8.09^{bc}	8.17	8.20^{c}	8.11^{c}	8.03^{c}	7.90^{c}	8.24
Fulvic acid	8.29^{bd}	8.15^{bd}	8.10^{bd}	8.06^{bd}	8.15	8.27 ^{cd}	8.08 ^{cd}	8.00 ^{cd}	7.85 ^{cd}	8.29
Salicylic acid	8.26^{ab}	8.20^{ab}	8.17 ^{ab}	8.12^{ab}	8.19	8.22^{ac}	8.15^{ac}	8.07^{ac}	7.99^{ac}	8.26
Glycine	8.25^{b}	8.22^{b}	8.19^{b}	8.15^{b}	8.20	8.20^{bc}	8.17^{bc}	8.10^{bc}	8.06^{bc}	8.25
Mean	8.26	8.17	8.14	8.06	8.16	8.23	8.10	8.03	7.92	8.07
		S	C	D	$S \times C$	$S \times D$	$C \times D$	$S \times C \times D$		
SEd		0.04	0.04	0.03	0.08	0.08	0.07	0.01		
CD ($P = 0.05$)		0.08	0.07	0.07	NS	NS	NS	NS		

S, sources of polymers; *C*, coating concentrations; *D*, days; *NS*, non-significant

Free $CaCO_3$

The changes in soil free $CaCO_3$ content was depicted in Table 2 which was significantly decreased by the application of various polymer-coated MNFG. A linear decrease in free $CaCO_3$ content was observed with increasing coating concentration of all the organic acid and amino acid polymers. Though the reduction in free $CaCO_3$ was common, the magnitude of reduction varied with nature of polymers and the interaction between polymers, their concentrations, and days after incubation. Results revealed that the application of uncoated MNF granules recorded higher soil free $CaCO_3$ content (11.0%) with lesser reduction (8.3%). Increasing coating concentrations of polymers reduced the free $CaCO_3$; content substantially and higher reduction were observed with 10% coating. The order of reduction was as follows: citric acid (34.6%) > fulvic acid (32.9%) > humic acid (29.1%) > salicylic acid (21.6%) > glycine (26.3%, Table 2). Citric acid coated at 10% concentration effectively reduced the soil pH and free $CaCO_3$ content in calcareous soil which was in agreement with the reports of Kpombleko and Tabatabai (2003) who stated that tricarboxylic acids (citric acid) are efficient in reducing soil pH and free $CaCO_3$ than monocarboxylic acids (salicylic acid). Significant reduction in soil pH and free $CaCO_3$ with time intervals was also registered and ascribed to acidic pH of coating materials and supply of sulfate ions from the added fertilizer sources (Dey et al. 2019).

Electrical conductivity (EC)

The EC is indicative of fertilizer's soluble salts, which slightly increased owing to the increasing coating concentration of all the organic acids and amino acids. Compared to the control, effect of organic acids and amino acids and their coating concentrations on the electrical conductivity was marked and the magnitude of the increase was higher with 10% coating concentration (Table 3). Initially, the electrical conductivity of soil was about 0.22 $dS{\cdot}m^{-1}$, which increased gradually with time, reaching a maximum electrical conductivity of 0.80 $dS{\cdot}m^{-1}$ at the end of incubation period. The greater increase in EC was noted in soil applied with 10% fulvic acid-coated MNF granules followed by MNFG coated with 10% humic acid (0.64 $dS{\cdot}m^{-1}$). The lesser electrical conductivity was observed with uncoated MNFG after 45 days of incubation (0.34 $dS{\cdot}m^{-1}$). The interaction between the coating concentration of polymers and EC was highly significant, which implies that EC of calcareous soil was highly influenced and increased with increasing levels of coating concentrations which were in agreement with the findings of Nunes et al. (2021). The present findings also indicated that an increment in fulvic and humic acid concentrations accompanies the increase in soil EC (Gumus and Seker 2015). Higher electrical conductivity (> 2 $dS{\cdot}m^{-1}$) hinders nutrient uptake by increasing the osmotic pressure of the nutrient solution (Ding et al. 2018).

Table 2 Effect of various sources and coating concentrations of organic acids and amino acids on free $CaCO_3$ (%) content in soil

Sources	7th day					15th day				
	Coating concentrations (%)					Coating concentrations (%)				
	0	3	5	10	Mean	0	3	5	10	Mean
Citric acid	11.5^{ae}	10.7^{abe}	10.4^{ace}	10.0^{ade}	10.7	11.6^{abe}	10.4^{be}	10.2^{cbe}	9.35^{bde}	10.4
Humic acid	11.5^{ac}	11.0^{abc}	10.8^{ac}	10.5^{acd}	11.0	11.2^{abc}	10.8^{bc}	10.3^{cb}	10.0^{bcd}	10.6
Fulvic acid	11.7^{ad}	10.9^{abd}	10.6^{acd}	10.2^{ad}	10.9	11.5^{abd}	10.3^{bd}	10.1^{bcd}	9.81^{bd}	10.4
Salicylic acid	12.0^{a}	11.4^{ab}	11.0^{ac}	10.9^{ad}	11.3	11.7^{ab}	11.1^{ab}	10.7^{abc}	10.3^{abd}	11.0
Glycine	11.9^{ab}	11.2^{ab}	11.0^{abc}	10.7^{abd}	11.2	11.6^{ab}	11.0^{b}	10.7^{bc}	10.1^{bd}	10.9
Mean	11.7	11.0	10.8	10.5	11.0	11.5	10.7	10.4	9.90	11.7
	30th day					45th day				
Citric acid	11.0^{ace}	9.81^{bce}	9.56^{ce}	8.54^{cde}	9.70	10.8^{ade}	9.10^{bde}	8.37^{cde}	7.85^{de}	9.00
Humic acid	11.1^{ac}	10.2^{bc}	9.89^{c}	9.45^{cd}	10.2	10.6^{adc}	9.74^{bcd}	9.37 cd	8.51^{cd}	9.60
Fulvic acid	11.2^{acd}	9.91^{bcd}	9.63^{cd}	9.03^{cd}	9.90	10.7^{ad}	9.53^{bd}	8.94 cd	8.05^{d}	9.30
Salicylic acid	11.4^{ac}	10.8^{abc}	10.6^{ac}	9.82^{acd}	10.7	11.0^{ad}	10.6^{abd}	10.2^{adc}	9.41^{ad}	10.3
Glycine	11.3^{abc}	10.5^{bc}	10.3^{bc}	9.53^{bcd}	10.4	10.9^{abd}	9.93^{bd}	9.61^{bdc}	8.84^{bd}	9.80
Mean	11.2	10.2	10.0	9.30	10.2	10.8	9.80	9.30	8.50	9.60
		S	*C*	*D*	*S*×*C*	*S*×*D*	*C*×*D*	*S*×*C*×*D*		
SEd		0.05	0.05	0.05	0.11	0.12	0.10	0.23		
CD (*P* = 0.05)		0.11	0.10	0.10	0.23	0.23	0.20	0.46		

S, sources of polymers; *C*, coating concentrations, *D*, days

Table 3 Effect of various sources and coating concentrations of organic acids and amino acids on soil electrical conductivity (dS·m^{-1})

Sources	7th day					15th day				
	Coating concentrations (%)					Coating concentrations (%)				
	0	3	5	10	Mean	0	3	5	10	Mean
Citric acid	0.24^{cd}	0.32^{cd}	0.35^{bcd}	0.39^{acd}	0.33	0.25^{cd}	0.36^{c}	0.40^{bc}	0.43^{ac}	0.35
Humic acid	0.25^{bd}	0.35^{bcd}	0.39^{bd}	0.43^{abd}	0.36	0.31^{bcd}	0.39^{bc}	0.45^{bc}	0.50^{abc}	0.41
Fulvic acid	0.27^{d}	0.37^{acd}	0.45^{abd}	0.50^{ad}	0.40	0.29^{acd}	0.41^{ac}	0.53^{abc}	0.62^{ac}	0.46
Salicylic acid	0.23^{de}	0.29^{cde}	0.3^{bde}	0.34^{ade}	0.29	0.26^{cde}	0.32^{ce}	0.35^{bce}	0.39^{ace}	0.33
Glycine	0.26^{d}	0.30^{cd}	0.33^{bd}	0.37^{ad}	0.32	0.33^{cd}	0.34^{cd}	0.38^{bcd}	0.41^{acd}	0.37
Mean	0.25	0.33	0.37	0.41	0.34	0.29	0.36	0.42	0.47	0.38
	30th day					**45th day**				
Citric acid	0.32^{bcd}	0.47^{bc}	0.49^{bc}	0.54^{abc}	0.46	0.34^{acd}	0.50^{ac}	0.55^{abc}	0.60^{ac}	0.50
Humic acid	0.33^{bd}	0.48^{bc}	0.53^{b}	0.59^{ab}	0.48	0.36^{abd}	0.52^{abc}	0.58^{ab}	0.64^{ab}	0.53
Fulvic acid	0.35^{abd}	0.56^{abc}	0.62^{ab}	0.74^{ab}	0.57	0.38^{ad}	0.60^{ac}	0.68^{ab}	0.80^{a}	0.62
Salicylic acid	0.34^{bde}	0.38^{bce}	0.40^{be}	0.46^{abe}	0.40	0.35^{ade}	0.41^{ace}	0.44^{abe}	0.50^{ae}	0.43
Glycine	0.36^{bd}	0.40^{bcd}	0.45^{bd}	0.50^{abd}	0.43	0.37^{ad}	0.45^{acd}	0.49^{abd}	0.55^{ad}	0.47
Mean	0.36	0.40	0.45	0.50	0.43	0.37	0.45	0.49	0.55	0.47
		S	C	D	$S\times C$	$S\times D$	$C\times D$	$S\times C\times D$		
SEd		0.002	0.002	0.002	0.004	0.004	0.004	0.009		
CD ($P=0.05$)		0.004	0.003	0.003	0.008	0.007	0.008	0.017		

S, sources of polymers; *C*, coating concentrations; *D*, days; *NS*, non-significant

Nutrient release and recovery

Sulfur (S)

The S release from coated MNF granules was in a controlled manner throughout the incubation period than the uncoated MNF granules. Effect of coating agents on S release was distinct during different incubation intervals. From the results, it was observed that MNF granules coated with citric acid at 10% registered higher S availability (18.2 mg kg^{-1}) with 40% increase after 45 days of incubation which was followed by 10% humic acid-coated MNF granules (17.3 mg kg^{-1}) where the release was 33.1%. At the end of incubation, salicylic acid-coated MNFG granules at 3% recorded lesser amount of S availability (13.6 mg kg^{-1}) followed by 5% salicylic acid-coated MNFG (14.0 mg kg^{-1}). The hydrophobicity of salicylic acid resulted in lesser nutrient release in soil. Sulfur availability helps in the formation of plant proteins and is essential for the formation of chlorophyll, maintaining cell integrity and membrane permeability, which activates many enzymes and plays a vital role in the nutrition of oil seed crops as a constituent of S-containing amino acids. The interaction between the sources, coating concentrations, and incubation time intervals are highly significant. Cumulative S release from various concentrations of organic acids and amino acid-coated MNFG was shown in Figs. 1a and 2a. During the 45 days incubation experiment, higher release of S was accompanied with: the following citric acid > fulvic acid > humic acid > glycine > salicylic acid.

The nutrient release from uncoated MNFG in the beginning was obviously higher at 7 days and later get decreased whereas, the MNF granules coated at 3% concentration with different polymers provided controlled release of S up to15 days. This could be due to the reactions between soil constituents through various processes like precipitation, adsorption, and sorption (Kassem et al. 2022). The release of S from granules coated at 5% was maintained throughout the incubation period up to 30 days. Coating at 10% concentration could be a turning point of the effect on nutrient release which showed greater potential for 45 days. Therefore, lesser coating concentrations had less benefit in controlling nutrient release for a longer period of time, which could result in insufficient nutrient supply in the latter period of plant growth. Depending on the thickness of polymer coating, the rate of nutrient release was adjusted in both aquatic and soil environments (Jia et al. 2013). As expected, increasing thickness of the coating delayed the release of S and the rate of release was much slower throughout the incubation period (Cruz et al. 2017).

Citric acid is a weak acid and is expected to have a short lifetime due to its utilization by microbes as carbon source which might be the reason for observing higher S release in soil applied with citric acid-coated MNF (Medina et al. 2014). The water permeability and solubility of polymers determine their rate of hydrolysis (Islam et al. 2015). The hydrophobic nature and poor solubility in water (less than 2 g L^{-1}) could be the reason for lesser S release from salicylic acid-coated MNF granules. The water absorption of

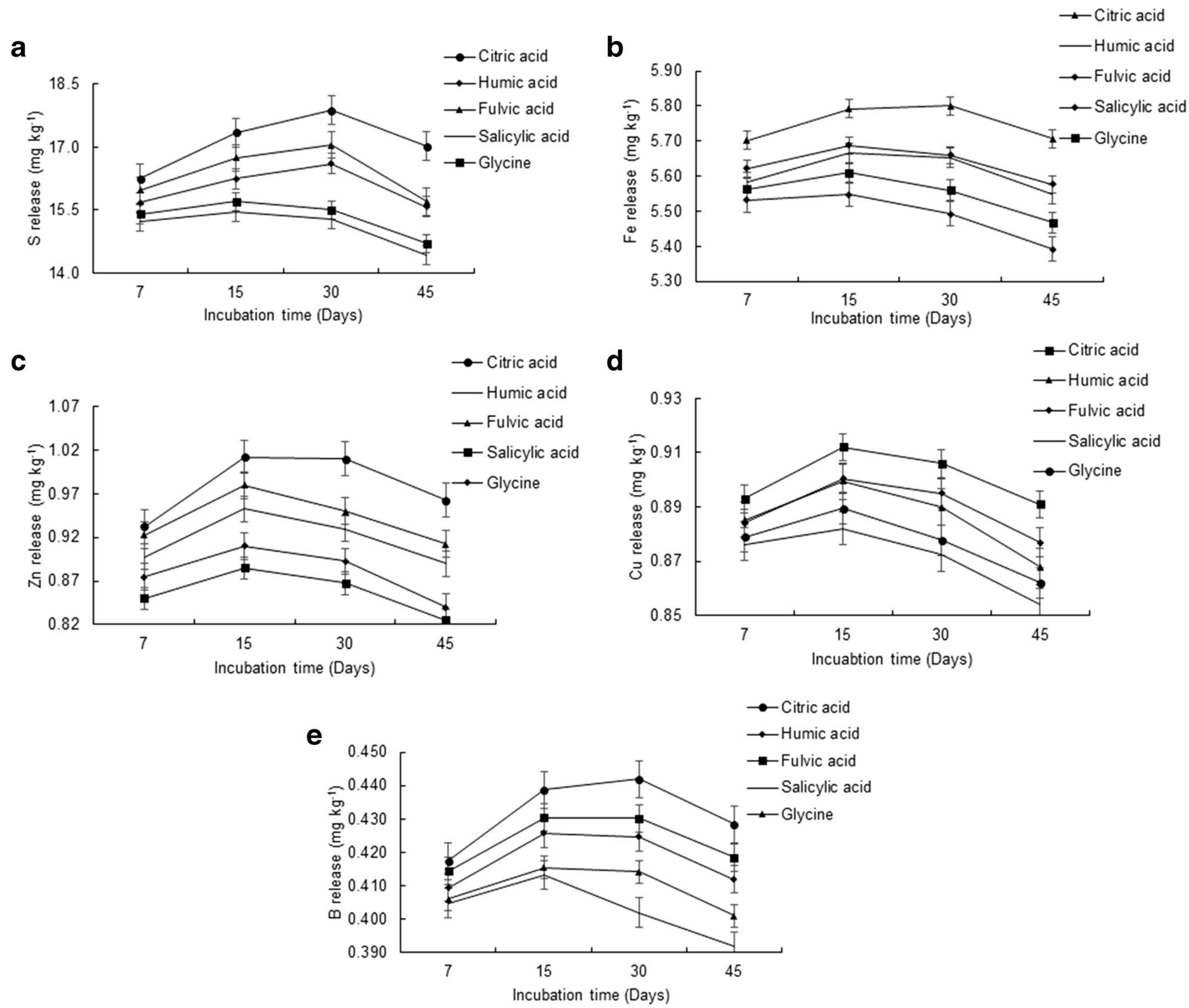

Fig. 1 Effect of various sources of organic acids and amino acids coated multi-nutrient fertilizer granules (MNFG) on nutrient release in soil ((**a**) sulfur, (**b**) iron, (**c**) zinc, (**d**) copper, and (**e**) boron; error bars indicate significance level at 5%)

coated polymers depends on number of hydrophilic groups and elasticity of the polymer networks (Liang et al. 2007). The porosity (Silva et al. 2012), thickness of coating layer (Lubkowski 2014), and composition of polymer mainly controls the nutrients diffusion coefficient from the granule to soil which contributes to delayed release of coated fertilizers (Hanafi et al. 2000; Ahmad et al. 2015).

Micronutrients

The coated MNF granules released Fe, Zn, Cu, and B into soil slowly in a very less amounts than the uncoated MNFG. Coating of MNF granules with 10% concentration showed distinctly slower release of nutrients as compared to 5% and 3% coating concentrations. The data pertinent to 0.005 M DTPA extractable micronutrient status in the soil treated with coated MNFG was influenced by the sources and coating concentrations of polymers which was presented in Figs. 1b–e and 2b–e. Maximum availability of Fe (5.84 mg kg^{-1}), Zn (1.01 mg kg^{-1}), Cu (0.917 mg kg^{-1}), and B (0.442 mg kg^{-1}) was observed with the addition of MNF granules coated with 10% citric acid coating which was followed by 10% humic acid-coated MNFG (5.71, 0.95, 0.899, and 0.427 mg kg^{-1}, respectively, at the end of incubation period). The release percentage of Fe, Zn, Cu, and B were 11.8, 40.2, 9.16, and 19.4% with the application of 10% citric acid-coated MNFG whereas 9.38, 31.9, 7.02, and 15.4% in humic acid (10%)-coated MNFG, respectively. The lesser amount of available Fe (5.25 mg kg^{-1}), Zn (0.75 mg kg^{-1}), Cu (0.839 mg kg^{-1}), and B (0.379 mg kg^{-1}) was noted with 3% salicylic acid-coated

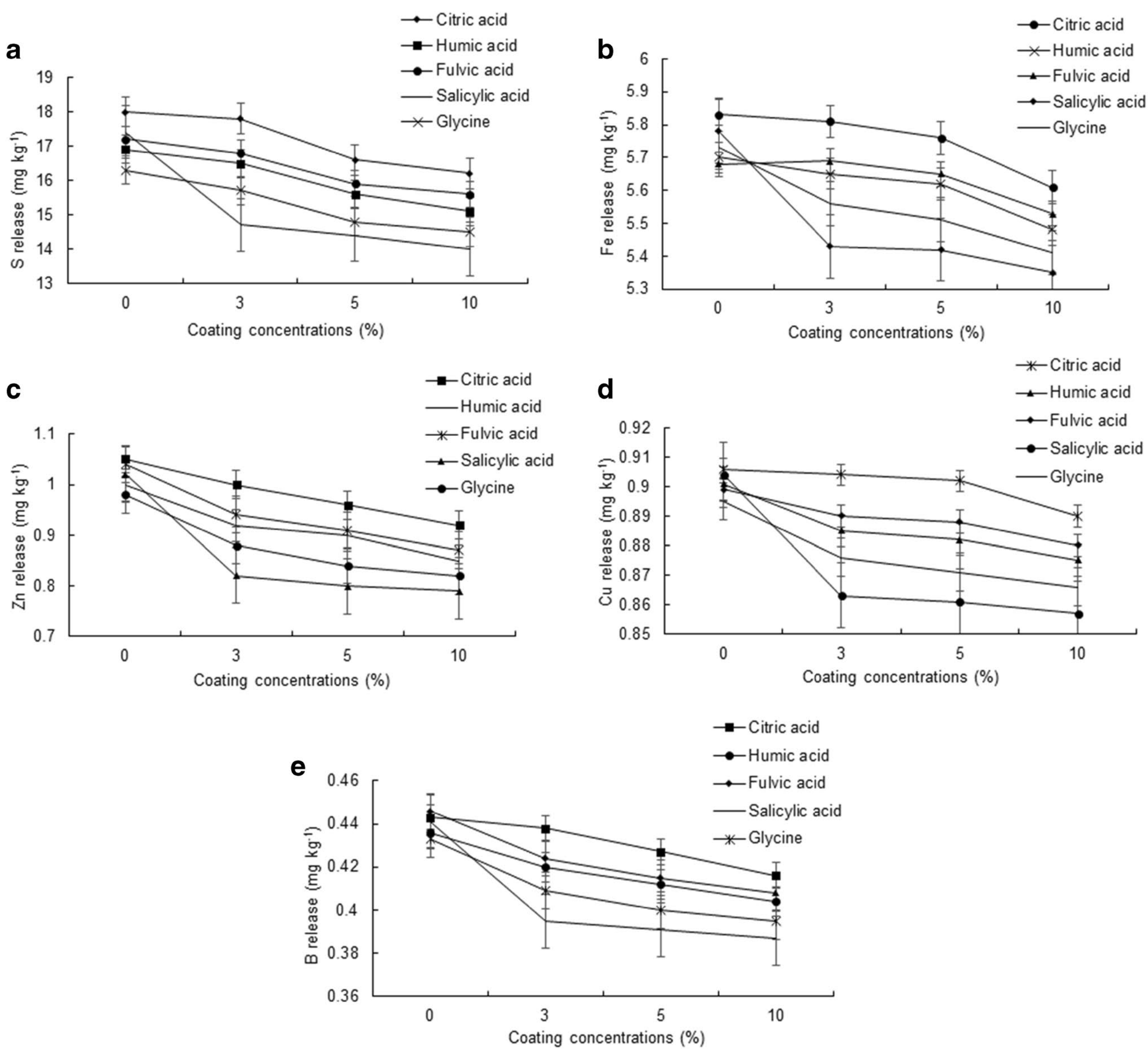

Fig. 2 Effect of various coating concentrations of organic acids and amino acids coated multi-nutrient fertilizer granules (MNFG) on nutrient release in soil ((**a**) sulfur, (**b**) iron, (**c**) zinc, (**d**) copper, and (**e**) boron; error bars indicate significance level at 5%)

MNFG after 45 days. The cumulative micronutrient release from various concentrations of organic acids and amino acid-coated MNFG was depicted in Figs. 1b–e and 2b–e. It was evident that after 45 days of incubation Fe, Zn, Cu, and B release was significantly higher in soil applied with citric acid-coated MNFG, followed by fulvic acid > humic acid > glycine > salicylic acid-coated MNFG.

The soil treated with uncoated MNFG showed higher micronutrients content in the initial days of incubation due to rapid solubilization and release. However, from 7 days onwards, the micronutrient contents decreased due to their reactions with soil matrix. The controlled release of MNFG provided by the coating concentration limits the volume of soil in contact with coated granules and therefore provided greater control of micronutrient release (Abedi-Koupai et al. 2012). The poor control of nutrient release up to 15 days was noted with 3% coating compared to the granules with higher polymer contents during the incubation which indicates that the thicker the coating layer probably had lower interconnected porosity and better nutrient release. With the increase in coating thickness, diffusion increases which lead to increase in the diffusion coefficient of nutrient release and vice versa (Azeem et al. 2020). According to Ibrahim et al. (2014), higher concentration used for the MNFG coating extended the release of nutrients in soil during the course of incubation period.

Soil fertilization with micronutrients reacts with soil components and reduce their bioavailability by forming insoluble complexes. On the other hand, often dissolved too quickly and then washed off to deeper layers, making them unaccessible to plants. The release rate of micronutrients from the coated granules was significantly lesser than the uncoated granules which establish the slow release of newly synthesized coated fertilizer granules and are likely to reduce nutrient fixation in soil and maintain a higher amount of bio-available fraction in soil solution, thus supplying nutrients over a larger temporal frame. The coated granules released lesser amount of nutrients presumably because of the reduction in effective surface area available for contact between the core of coated granules and soil (Sarkar et al. 2018). This might be the reason for coated granules to slow down the movement of nutrients into the soil, and the results are in agreement with the findings of Wu et al. (2008) and Jia et al. (2013). Feng et al. (2015) suggested that biodegradable coatings surrounding the fertilizer granules reduce nutrient losses and contribute to the improvement in soil nutrient availability.

The release of nutrients from fertilizer granules could be related to the hydrophilicity or hydrophobicity of the coated material (Islam et al. 2015). When the coating material is hydrophobic, the affinity between the layer and water is weak which prevents the penetration of more water into the fertilizer core and decreases the dissolution (Jarosiewicz and Tomaszewska 2003). This might be the reason for lesser nutrient release from salicylic acid-coated multi-nutrient fertilizer granules. The molecular characteristics of humic and fulvic acid with an intermediate hydrophobicity provided a more gradual release of nutrients over time as compared to citric acid coated MNFG where the release was higher (Teixeira et al. 2016).

Conclusion

Controlled release and consistent availability of sulfur and micronutrients in the soil over time are the desirable characteristics of coated fertilizers. In this study, coating of multi-nutrient fertilizer granules with biodegradable polymers like organic acids and amino acids was shown to be promising for the slow release and availability of nutrients in the soil. The study revealed a linear reduction in pH and free $CaCO_3$ throughout the incubation period from 7 to 45 days. The MNFG coated with 10% citric acid showed greater reduction in pH (6.71%) and free $CaCO_3$ (34.6%). Higher availability of S and micronutrients was noted with MNFG coated with citric acid at 10% where the rate of release was controlled up to 45 days. The rate of nutrient release in the soil was controlled by the thickness of coating and 3% coating concentrations provided control release of nutrients up to 15 days only. However, increasing the coating concentration enabled greater release of nutrients in soil, 5% coating for 30 days and 10% showing potential for more than 45 days. The coating of MNF granules using organic acids and amino acids at 3, 5, and 10% concentrations controlled the nutrient release up to 15, 30, and 45 days, respectively, and the time of nutrient release was directly proportional to the thickness of the coating layer whereas the quantity of nutrient release was inversely proportional to coating thickness. The results indicated that MNFG coated with citric acid at 10% concentration was found to be the better source for the controlled release of fertilizer nutrients that fit the crop nutrient requirement in calcareous soils.

As a future perspective, to understand the kinetics of nutrient release and mobility of the MNFG in the soil, a series of laboratory and column experiments needs to be carried out. Further, the degradability over a period of time needs to be worked out and validated with field experiments to know the efficiency of organic acids and amino acid-coated fertilizers in improving crop nutrition and nutrient use efficiency of crops on calcareous soils.

Data availability The authors declare no data sharing as the research data cannot be shared.

Declarations

Conflict of interests The authors declared that no competing interests exist among them.

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