



## **Development of Slow-Release Fertilizer from Animal Origin Wastes: Sustainable Organic Agricultural Perspective**

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### **Abstract**

Improved crop quality and quantity is the main target of soil fertilization in conventional agriculture. However, in organic agriculture, adding nutrients to the soil is the threshold replenishment of nutrients lost through every crop. Mineral fertilizers provide essential macro and microelements to plants for their active growth and development. Unfortunately, these kinds of fertilizers easily run off from plant vicinity because of frequent watering and rain. This leads to the recurrent application of fertilizers, which causes severe environmental problems. To resolve these issues many researchers developed different slow-release fertilizers, which can provide nutrients to plants for an extended period. However, slow-release fertilizers are much more expensive than normal mineral fertilizers. The present work attempts to develop new slow-release fertilizer using mariculture waste mussel shell and cow urine. Significant macronutrient adsorption was observed in the shell saturation experiment and the leaching experiment proves that shell powder after nutrient impregnation with urine had a slow nutrient-releasing capacity. The formation of struvite crystals is another highlight of this study, which provides a long-term nutrient supply in the soil. BET analysis confirms the nutrient scrubbing and releasing ability of shell powder. Independently cow urine and mussels shells are used in agriculture for different purposes. Mussel shells are regularly used to reduce soil acidity and cow urine is applied to plants as a cheap nitrogen supplement in a diluted manner. However, cow urine components quickly run off from the plant root proximity. Nutrient impregnated shell powder has both characters of mussel shell powder and cow urine and it is truly formed from animal origin waste products. Because of no usage of synthetic chemicals, new slow-release fertilizer couldn't make any environmental problems.



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## Introduction

Plant nutrition significantly impacts on cropping systems, environmental sustainability, and human and other animals' health and well-being. Minerals are a vital requirement for a plant's growth, metabolism, and completion of its life cycle. Mainly, plants require 14 elements for their development and they are categorized into macroelements and microelements. Six mineral elements, such as nitrogen (N), phosphorous (P), potassium (K), calcium (Ca), magnesium (Mg), and sulphur (S) are included in the macroelements category and are required in large quantities. Among macroelements, nitrogen (N), phosphorous (P), and potassium (K) are considered primary macronutrients and calcium (Ca), magnesium (Mg), and sulphur (S) are secondary macronutrients. At the same time, microelements are required in small quantities, which include chlorine (Cl), boron (B), iron (Fe), manganese (Mn), copper (Cu), zinc (Zn), nickel (Ni), and molybdenum (Mo).<sup>1</sup> Plants require optimal mineral elements for good quantity and quality crop production. When the concentration of mineral elements increases exponentially harms plant growth and crop yield. Humans and other animals acquired essential minerals elements from plants through the food chain.<sup>2</sup>

The plants experience nutritional imbalance when they grow in areas with low phytoavailability. Such deficiencies are resolved by the use of fertilizers, through which they acquire essential elements. The invention of the energy-intensive Haber-Bosch process leads to synthetic fertilizers massive production. NPK is a widely used chemical fertilizer, and nitrogen is one of the essential elements for plant growth, whereas its excess accumulation causes detrimental environmental problems. N-fertilizer synthesis is one of the reasons for the production of greenhouse gaseous and N and P fertilizers contribute to the eutrophication process.<sup>3,4</sup> Similarly, excess rock phosphate and rock sulphate utilisation for fertilizer synthesis causes their extermination within the next 25-100 years. The chemical fertilizers' efficiency was decreasing over time and excessive use causes serious soil degradation, nitrogen leaching, soil compaction, reduction in soil organic matter, and loss of soil carbon.<sup>5</sup> Hence in the future,

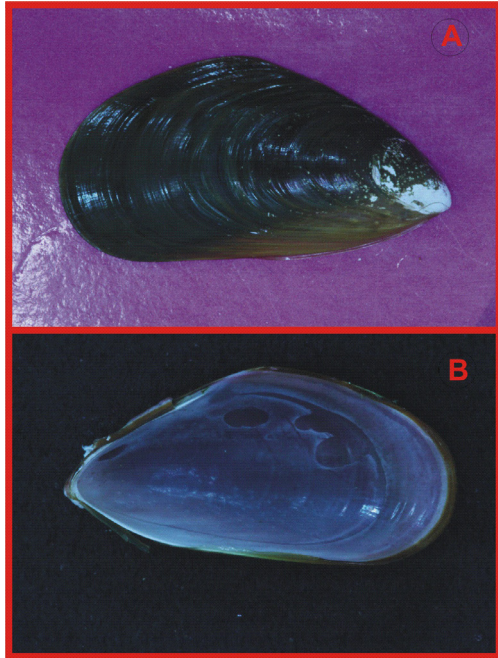
sustainable fertilizer management is required in the field of crop production.<sup>2,6</sup>

Researches in the development of high-potential, environment-friendly fertilizers are very essential in the agriculture sector.<sup>7</sup> A group of fertilizers is called slow-release fertilizers (Eg: Urea formaldehyde (UF), Nitroform (UF derivative), methylene urea (MU)); they can hang on the soil after their application and is available for a significantly longer time. As a result, they didn't quickly wash out from soil through rain and irrigation; hence it reduces eutrophication effects.<sup>8</sup> Slow-release fertilizers are safer, able to decrease soil toxicity, cause less damage to plant roots and also reduce the frequency of fertilization.<sup>9</sup> Thermoplastic resins such as polyolefin, polyvinylidene chloride, and co-polymers are used as a coating material for the synthesis of slow-release fertilizers because they cannot degrade easily.<sup>10</sup> Natural coating materials are more environmentally friendly and safer; hence researchers are now focused on this field. Mussel shell and cow urine are organic waste products, but they are rich in micro and macronutrients. Nowadays, mussels shell waste accumulation creates a high level of pollution. In this scenario, natural, slow-release fertiliser synthesis using mussel shells is considered an environment-friendly waste disposal method. The present study mainly focused on synthesising mineral-rich, natural, slow-release fertiliser using waste materials such as mussel shells and cow urine.

## Materials and Methods

### Sample Collection

The raw *Perna perna* mussel shells wastes were collected from hotels and markets located 5 km around Kovalam, Thiruvananthapuram, Kerala, India (8.3988° N, 76.9820° E). Shells were thoroughly washed and removed debris attached to the shell surface. After proper drying, shells were crushed using an iron rod and powered in the mixer grinder. Then the powder was sieved in a brass frame sieve (Standard laboratory test sieves) with a 45-micron mesh size. Cow urine was collected from cattle farm without contamination and filtered urine was used for the study.



**Fig. 1: Shell of *Perna perna*, A: Dorsal region, B: Ventral region**

#### Shell saturation Experiment

In this experiment, shell powder remained constant and on every alternate day, urine changed. For this, 75 ml fresh cow urine was added to a 25 g mussel shell containing a beaker and given one hour of gentle stirring in a closed container. After stirring, the mixture retains without disturbance. On alternative days, urine in the beaker was removed and added fresh urine followed by one hour of gentle stirring. This procedure continues for 10 days. Every alternate day, 1 g of shell powder was washed with distilled water for removing excess urine content, dried thoroughly, and used for further analysis.

#### Urine Filtration Experiment

In this experiment, 25 g shell powder was added to 75 ml fresh cow urine and continuously stirred for one hour. In this setup, urine was constant, and shell powder was removed every alternate day. The experiment was conducted for 10 days, and every alternate day the removed shell powder was washed with distilled water to remove excess urine content and dried thoroughly and store for further analysis.

#### Shell leaching Experiment

For this experiment, 25 g nutrient-impregnated shell powder was mixed with 75 ml distilled water, and the

setup was kept for 30 days to measure the leaching of elements. Then, every 10 days 1 g shell powder was taken from the setup, dried and concentrations of elements were measured.

#### XRF Analysis

Elemental concentration variation in the shell saturation experiment and urine leaching experiment was calculated by X-Ray fluorescence spectroscopy (Spectro-Ametek, SPECTRO XEPOS-XRF). We analysed the concentration variations of macro elements such as P, K, Ca, S, and Mg and microelements such as Fe, Zn, Cu, Mn, Mo, Cl, and Ni.

#### BET Analysis

The specific surface area and pore size distribution of normal mussel shell, nutrient impregnated shell powder, and 30 days nutrient leached shell powder was measured by BET (Brunauer–Emmett–Teller) analyser (BELSORP-max) with  $N_2$  as the analysis gas.

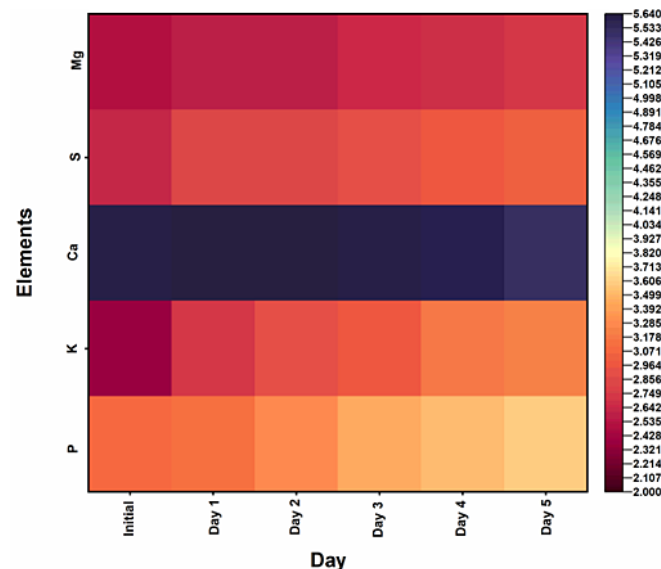
#### Results and Discussion

Synthesis of natural slow-release fertilizers is a highly considerable aspect of research that diminishes the adverse effects of chemical fertilizers. Natural slow-release fertilizers provide a steady supply of essential elements to plants over a course of time. Runoff of elements from plants' proximity is the major reason for nutrient scarcity in plants; hence the idea of slow release or controlled release fertilizer was developed. Cow urine and mussel shell powder are waste products but they are rich with essential elements necessary for plant growth. In the shell saturation experiment, except for calcium and magnesium, all other macronutrient (P, K, Ca, S) concentrations were significantly increased and the results presented as graph (Figure 2) which was plotted using log values of concentration (ppm). Calcium concentration was exponentially higher in the mussel shell; hence calcium was leached into the urine. Macronutrients are vital to plant nutrients found in every plant cell and are involved in cellular function and plant growth. They act as activators of many enzymes, are involved in metabolism, photosynthesis, and cellular multiplication, and play a critical role in the formation of the best quality and quantity yield from crops, etc.<sup>11</sup> The interactions between nutrients are also needed for plant development. For example, sufficient potassium

supply promotes N metabolism and facilitates the synthesis of amino acids and proteins.<sup>12</sup> Hence, an insufficient supply of a single nutrient adversely affects the activities of others.

Among macronutrients N, P, and K are the primary macronutrients. Nitrogen is an indispensable and unavoidable element that utilizes plants in the form of  $\text{NO}_3^{2-}$  and  $\text{NH}_4^+$ . Nitrogen is an essential constituent of protein and chlorophyll synthesis, hence optimal nitrogen concentration increases photosynthesis, leaf area production, and leaf duration. These factors are directly linked with crop production. Phosphorous is mainly involved in plant growth, which is involved in proper plant maturation, helping to withstand

harsh environmental conditions, cell division, seed development, etc.<sup>13</sup> Similarly, potassium is involved mainly in the physiological processes of plants including nutrient transport, nitrogen metabolism, carbon assimilation, etc. which determine crop quality.<sup>11</sup> The secondary macronutrients are sulphur, magnesium, and calcium. Sulphur is mainly involved in synthesising sulfur-containing amino acids and is the critical component of many prosthetic groups. In plants, calcium acts as a structural component and is involved in cellular signalling and magnesium is the key component of chlorophyll. Hence the necessity of macronutrients in plants is vital and its deficiencies directly affect the quality and quantity of crop production.<sup>14</sup>



**Fig. 2: Macronutrients concentration in shell saturation experiment**

A significant concentration difference was not observed in the case of micronutrients (Table 1). Shell powder had more concentration of micronutrients than cow urine. The requirement and accumulation of micronutrients were traces, but they are essential for the better growth of plants. They play various physical and metabolic functions including pollen germination, photosynthesis, chlorophyll synthesis, and various hormonal and enzyme activities.<sup>15</sup> In plants, zinc is an important micronutrient that mainly regulates several enzyme activities including the activity of alcohol dehydrogenase, carbonic

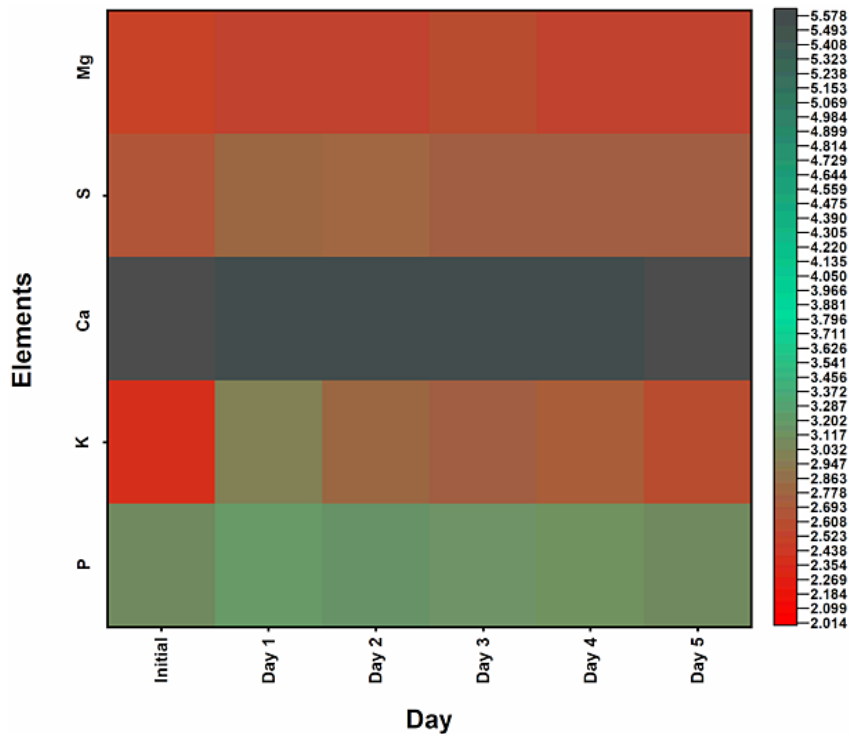
anhydrase, superoxide dismutase and also it is part of some enzyme structures such as Alkaline Phosphatase, phosphatides lipase, Carboxy peptidase, RNA polymerase, Dehydrogenase and Aldolase.<sup>16,17,18</sup> Similarly, Fe, Mn and Cl are involved in photosynthesis and carbohydrate synthesis and Cu is involved in protein and chlorophyll synthesis. The major function of Mo is nitrogen fixation and Ni is nitrogen metabolism, hence its deficiency causes stunted growth and chlorosis.<sup>15</sup> In the case of the urine filtration experiment significant nutrient scrubbing was not observed (Figure 3 and Table 2).

Macronutrient concentration was significantly lower in the urine filtration experiment than shell saturation experiment. Similarly, in the case of micronutrients, nickel was absent in all samples from the urine

filtration experiment. Hence the shell powder from the shell saturation experiment was richer with macro and microelements.

**Table 1: micronutrients concentration in shell saturation experiment**

Sample	Fe (ppm)	Zn (ppm)	Cu (ppm)	Mn (ppm)	Mo (ppm)	Cl (ppm)	Ni (ppm)
Raw cow urine	0.68± 0.02	0.52 ± 0.02	0.413 ± 0.15	3.64 ± 0.25	0.27 ± 0.029	1170.33 ± 250.52	0.62 ± 0.006
Raw shell powder	56.76 ± 9.37	1.58 ± 0.23	1.77 ± 0.09	0.085 ± 0	0.64± 0.09	287.42± 18.67	0.085 ± 0
S1	56.59± 2.1	1.33± 0.35	1.50± 0.15	0.085 ± 0	0.58± 0.14	227.79± 4.42	0.085 0
S2	58.43 ± 9.9	1.44± 0.3	1.66± 0.26	0.085 ± 0	0.53± 0.16	239.96± 1.7	2.91±0.29
S3	60.01 ± 3.45	1.47 ± 0.41	1.74 ± 0.17	0.085 ± 0	0.71 ± 0.17	289 ± 5.89	2.86 ± 0.15
S4	60.24± 1.04	1.56± 0.2	1.81± 0.11	0.085 ± 0	0.74± 0.04	284.32± 1.04	2.90± 0.14
S5	60.45± 3.17	1.62± 0.71	1.92± 0.3	0.085 ± 0	0.68± 0.11	295.17± 2.47	2.91± 0.2



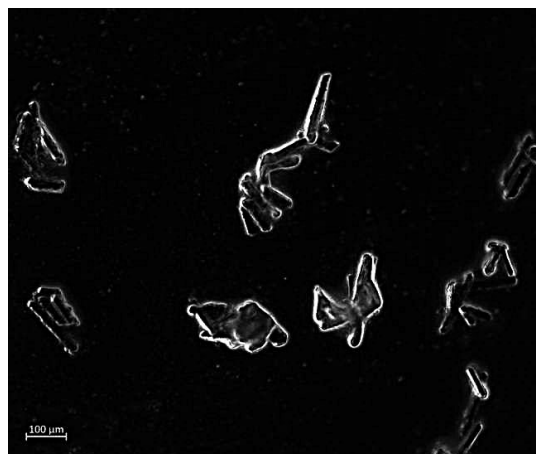
**Fig. 3: Macronutrients concentration in urine filtration experiment**

**Table 2: Micronutrients concentration in urine filtration experiment**

Sample	Fe (ppm)	Zn (ppm)	Cu (ppm)	Mn (ppm)	Mo (ppm)	Cl (ppm)	Ni (ppm)
Raw cow urine	0.68± 0.02	0.52 ± 0.02	0.413 ± 0.15	3.64 ± 0.25	0.27 ± 0.029	1170.33 ± 250.52	0.62 ± 0.006
Raw shell powder	56.76 ± 9.37	1.58 ± 0.23	1.77 ± 0.09	0.085 ± 0	0.64± 0.09	287.42± 18.67	0.085 ± 0
US1	46.23± 5.31	1.66± 0.22	1.56± 0.04	0.085 ± 0	0.51± 0.01	314.21± 10.41	ND
US2	43.59± 3.14	1.59± 0.07	1.47± 0.57	0.085 ± 0	0.6± 0.12	297± 9.34	ND
US3	36.51± 1.64	1.53± 0.62	1.61± 0.1	0.085 ± 0	0.59± 0.04	305± 4.12	ND
US4	44.78± 4.28	1.6± 0.3	1.59± 0.07	0.085 ± 0	0.58± 0.11	284± 8.54	ND
US5	45.52± 2.98	1.54± 0.24	1.64± 0.74	0.085 ± 0	0.67± 0.74	297±5	ND

The formation of struvite crystals was an important highlight of these experiments (Figure 3). Struvites are transparent crystals formed of equimolar concentrations of magnesium, phosphorous, and ammonium with the formula  $\text{NH}_4\text{MgPO}_4 \cdot 6\text{H}_2\text{O}$ . Struvites are less soluble crystals; hence they can provide a continuous supply of magnesium, phosphorous, and nitrogen to the soil over a period of time. Cow urine is rich in potassium and ammonium, whereas magnesium concentration was limited. However, shell powder contains a sufficient quantity of magnesium, hence, when cow urine mix with shell powder, the magnesium from the shell act as an additional source for struvite formation. In the study of Liu *et al.* (2013)<sup>19</sup> seawater and brine were used as additional magnesium sources for the struvite synthesis from urine. pH is another limiting factor for struvite formation. Struvites are formed only in alkaline conditions. Cow urine and shell powder have alkaline nature; hence they facilitate struvite synthesis.<sup>20</sup> In the past decade, many studies have been conducted to analyse the efficiency of struvite as fertilizers. Because of the slow-release nature of struvite could provide a long-term source of macroelements such as ammonium, phosphorous,

and magnesium to plants.<sup>26</sup> Struvites are mainly used for phosphate recovery because phosphate rocks and non-renewable geological reserves are diminishing and hence they use as a major phosphate source in plants. The studies of Johnston and Richards (2003),<sup>21</sup> Antonini *et al.* (2012),<sup>22</sup> and Talboys *et al.* (2016)<sup>23</sup> revealed that struvite could be used as a sustainable alternative to conventional P-based fertilizer.

**Fig. 4: Struvite crystals**



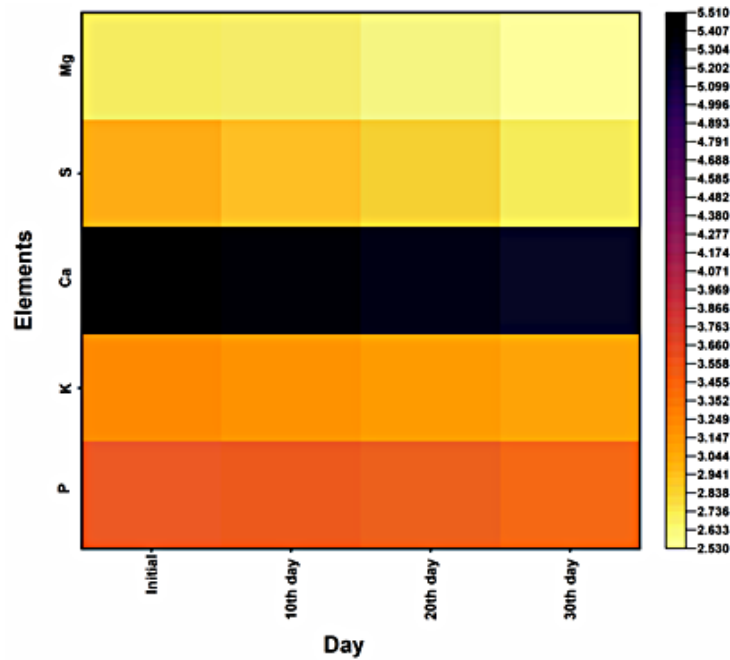


Fig. 5: Macroelements concentration after leaching

Table 3: Microelements concentration after leaching

Sample	Fe (ppm)	Zn (ppm)	Cu (ppm)	Mn (ppm)	Mo (ppm)	Cl (ppm)	Ni (ppm)
Initial	60.45± 3.17	1.62±0. 71	1.92±0. .3	0.085 ± 0	0.68± 0.11	295.17± 2.47	2.91± 0.2
10 <sup>th</sup> day	49.31±2. 41	0.77±0. 01	0.89±0. 04	0.085 ± 0	0.14±0. 005	164.34± 2.11	1.03±0 .07
20 <sup>th</sup> day	36.87±2. 22	0.54±0. 04	0.41± 0.01	0.085 ± 0	0.085 ± 0	106.4±3. 74	0.45±0 .01
30 <sup>th</sup> day	29.45±4. 14	0.37±0. 02	0.085 ± 0	0.085 ± 0	0.085 ± 0	56.45± 1.89	0.085 ± 0

Table 4: Bet analysis

Measures	Normal shell	Nutrient immobilized shell	Nutrient leached shell
Surface area	0.70858 m <sup>2</sup> /g	0.37814 m <sup>2</sup> /g	2.2811 m <sup>2</sup> /g
Pore size	1.26 nm	1.26 nm	1.74 nm
Pore volume	0.2246 m <sup>3</sup> /g	0.17186 m <sup>3</sup> /g	1.0682 m <sup>3</sup> /g

Slow-release fertilizer development reduces drainage of fertilizer from soil and also environmental pollution. The leaching experiment results (Figure 4 and Table 3) revealed that more than half

of the macroelements were retained in the shell after 30 days of continuous leaching. These results suggest that nutrient-impregnated shell powder can use as an excellent slow-release fertilizer. Nutrient

impregnation and leaching were confirmed by BET analysis (Table 4), and the results highlighted that after nutrient impregnation, the surface area and pore volume of the shell decreased because nutrients used the space. However, surface area, pore size, and pore volume increase after leaching, because the nutrient-withdrawn area expands the surface. Many plants are intolerant to soil acidity and which causes reduced crop yield. Liming materials helps to increase the soil's pH, facilitating excellent activity and composition of microbial organisms. Microbes play a critical role in the transformation and cycling of organic matter.<sup>20</sup> Traditionally, calcium hydroxide is used as a liming agent, but its production is more cost effective and power-consuming. Similarly, slow-release fertilizer production is relatively expensive.<sup>24</sup> Carbon base, slow-release fertilizers are very efficient for nutrient management because they have high porosity, surface area, and abundance of the functional group which helps to absorb nutrients and they are cost-effective.<sup>25</sup> The present work concept was the production of slow-release fertilizer from waste materials; hence it is a highly efficient, cost-effective, eco-friendly fertilizer and acts as the best liming material.

### Conclusion

Several environmental hazards and economic losses widely occur due to excessive use of chemical fertilizers, unbalanced fertilization, and improper fertilization methods. Conventional mineral fertilizers are readily soluble in soil and they easily run out after rain leading to an increased risk of eutrophication. Hence, slow-release fertilizers have been developed to meet the sustainable nutrient availability to plants,

increase yield and quality, maintain economic balance, and create environment-friendly agriculture. Mussel shells and cow urine are rich in minerals, but their independent application could not critical impact on soil fertility. Besides, mussel shell accumulation makes severe environmental pollution. Recycling waste mussel shells in the form of slow-release fertilizer becomes a pollution control strategy. The formation of struvite crystals provides a more slow-release nature to fertilizer and gives the regular flow of macronutrients such as nitrogen, phosphorous, and magnesium. The present slow-release fertilizer reduces environmental issues created by conventional mineral fertilizers. This new mineral-rich natural fertiliser has four-way action, (1) it reduces soil acidity, (2) provides micro and macronutrients, (3) gives long-term nutrient supply because of slow-release action, (4) Eco-friendly waste disposal of a mussel shell.

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### Conflicts of Interest

The authors declare no conflict of interest.

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