



Using water hyacinth (*Eichhornia crassipes*) biomass and humic substances to produce urea-based multi-coated slow release fertilizer

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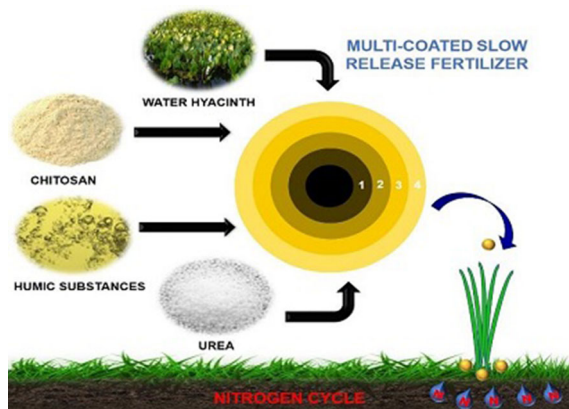
Abstract Urea-based multi-coated slow release fertilizer was produced using water hyacinth, humic substances, and chitosan, with water rich in natural organic matter as a solvent. Elemental analysis showed that the nitrogen content of the fertilizer (FERT) was around 20%. Swelling tests demonstrated the effectiveness of the water hyacinth crosslinker, which reduced the water permeability of the material. Leaching tests showed that FERT released a very low concentration of ammonium (0.82 mg L^{-1}),

compared to the amount released from urea (43.1 mg L^{-1}). No nitrate leaching was observed for FERT, while urea leached 13.1 mg L^{-1} of nitrate. In water and soil, FERT showed maximum releases after 30 and 40 days, respectively, while urea reached maxima in just 2 and 5 days, respectively. The results demonstrated the promising ability of FERT to reduce nitrogen losses, as well as to minimize environmental impacts in the soil–plant–atmosphere system and to improve the efficiency of nitrogen fertilization.

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Graphic abstract



Keywords Urea · Water hyacinth · Natural organic matter · Slow release fertilizer

Introduction

According to the United Nations Food and Agriculture Organization (FAO), in 2050, the global population will reach 9.1 billion inhabitants and food production will need to increase by 70% to meet the demand (FAO 2020). In view of this, the agricultural sector will play an important role in reducing and preventing hunger, with population growth driving the search for strategies to improve soil fertility and increase food production (Araújo et al. 2017).

The use of fertilizers plays a fundamental role in increasing agricultural productivity. According to FAO data, one ton of fertilizer applied to one hectare, according to principles that allow maximum efficiency, enables production equivalent to that of four hectares without fertilization (FAO 2020).

Urea is the nitrogen fertilizer most used in global agriculture, due to its low production cost, high nitrogen content, and ease of application (Trenkel 2010). However, high losses, mainly due to ammonia volatilization and nitrate leaching, make it necessary to develop new technologies that can reduce losses and minimize environmental impacts (Messa and Faez 2020).

A promising option is the development of slow release fertilizers (SRFs), which are produced by coating particles of water-soluble fertilizer with an insoluble material that may be semi-permeable or impermeable with pores. This controls the entry and exit of water, consequently reducing the rate of dissolution of the nutrients contained in the particle core (Azeem et al. 2014).

In this way, urea may be coated with substances that create a physical barrier around the granules, controlling the release of nitrogen according to the nutritional needs of the plant, resulting in longer release of nutrients, compared to conventional urea (Costa et al. 2013).

The addition of extra layers of the fertilizer coating may further delay the release of nutrients. Previous work by our group showed the feasibility of using a coating with chitosan and humic substances in one layer (Araújo et al. 2017). However, a greater number of layers could enable the needs of crops to be met for longer periods, reducing the frequency of application and losses, besides avoiding damage caused by high concentrations of salts (Li and Yang 2004) and the excessive use of nitrogen fertilizers (Yang et al. 2020).

In addition to the use of chitosan and humic substances as growth promoters, studies have reported the use of biomasses such as the water hyacinth plant (*Eichhornia crassipes*) for the production of fertilizers in agriculture (Wolwerton and McDonald 1979), since the plant contains nutrients such as nitrogen and potassium in its composition (Priya et al. 2018).

Water hyacinth can also be used as a chitosan crosslinker, resulting in a biopolymer with increased hydrophobicity, due to the greater quantity of cross-links. This decreases the water permeability of the coating, preventing the nutrients contained within it from being rapidly released into the soil–plant system.

Crosslinkers possess functional groups that can bind to polymer chains, making them more rigid, due to the creation of cross-links (Maitra and Shukla 2014). Natural substances are preferable to synthetic chemicals for this purpose, since they are (usually) nontoxic, inexpensive, readily available, and do not cause environmental pollution (Rivero et al. 2010). Furthermore, the use of plant biomasses such as water hyacinth can help in reducing environmental problems caused by weed infestations.

Water hyacinth is an invasive aquatic plant, native to Brazil and considered one of the most reproductive

plants in the world, since it can double in size in just six days (Télliez et al. 2008). Consequently, the control of water hyacinth is essential, although the various forms of its control and/or elimination have limitations and are expensive (Shu et al. 2015). Its sustainable use would be much more rational, reinserting it in a productive chain and generating added value.

To the best of our knowledge, there have been no reports in the literature concerning the use of water hyacinth as a crosslinking agent to obtain slow-release nitrogenous fertilizers. Therefore, the aim of this work was to synthesize hybrid spheres with four coating layers, based on urea, chitosan biopolymer, and water hyacinth. These components were dissolved in water with a high content of natural organic matter (NOM), replacing the solvents traditionally used. Evaluation was made of the interactions among the precursors, in order to achieve the controlled release of urea fertilizer, with less environmental damage.

Materials and methods

Biomass and pretreatment

Water hyacinth (*Eichhornia crassipes*) was collected at the Açude Macela reservoir in Itabaiana (Sergipe State, Brazil). For collection, the plants were lifted from the water surface and the roots were removed and discarded. Only the leaves and stems were used, which were dried at room temperature and ground in a knife mill in order to obtain a uniform particle size of 0.85–2.0 mm, facilitating homogenization with the other components of the fertilizer.

Preparation of FERT and spheres of urea, chitosan, and humic substances (UCHS)

Synthesis of the precursor mixture

The fertilizer core was prepared using about 10 g of urea dissolved in 120 mL of water with high NOM content (pH 3.89 and total organic carbon concentration of 23.6 mg L⁻¹), together with 0.5 g of water hyacinth, 4 g of chitosan, and 500 µL of GrowMate Plant (growth regulating hormone and soil fertility enhancer). The pH of the mixture was adjusted to 2.0, keeping the system under mechanical stirring (150 rpm) for 2 h.

Core formation

The mixture was dripped into a 1.0 mol L⁻¹ potassium hydroxide (KOH) solution to form the cores, followed by drying the resulting spheres at room temperature (25 °C).

Immersion in urea solution

After drying, the spheres were immersed in a 2 mol L⁻¹ urea solution for 1 h, in order to increase the percentage of nitrogen in the fertilizer.

Formation of layers

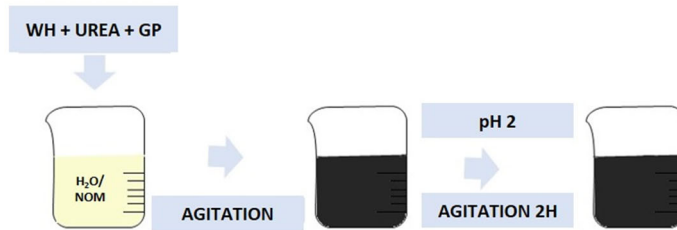
The four layers of the fertilizer were prepared in a manner analogous to the experimental procedure adopted for preparation of the core, maintaining the same proportions of urea and chitosan, while varying the amount of water hyacinth for each layer (1st layer: 0.1 g; 2nd layer: 0.2 g; 3rd layer: 0.3 g; 4th layer: 0.4 g; core: 0.5 g). The amount of water hyacinth increased towards the core, in order to progressively increase the hydrophobic character of the chitosan biopolymer and, consequently, increase the release time, so that the plant would receive fertilizer during all stages of its development, eliminating the need for reapplication. The resulting fertilizer granules were stored at room temperature. The material was named FERT. All the steps of the procedure are shown in Fig. 1.

To evaluate the effectiveness of the water hyacinth crosslinker used in the fertilizer synthesis, spheres composed of urea, chitosan, and humic substances were produced using the procedure described above, maintaining the same proportions, but without water hyacinth. These spheres were named UCHS.

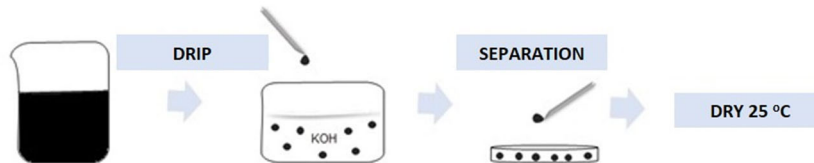
Fourier transform infrared absorption spectroscopy (FTIR)

FTIR spectra of the samples were obtained using a Varian 640 IR instrument, with the samples in the form of KBr tablets (sample:KBr ratio of 1:100). The samples were first dried under vacuum at 50 °C for 24 h. The spectra were acquired by scanning between 4000 and 400 cm⁻¹, at resolution of 4 cm⁻¹, with accumulation of 32 scans for each sample.

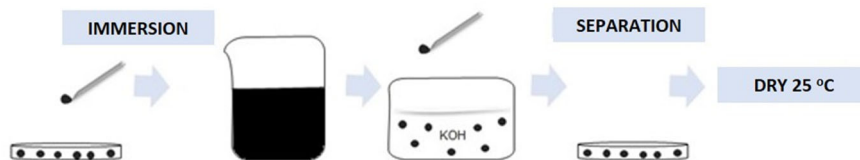
STEP 1: PRODUCTION OF THE PRECURSOR MIXTURE



STEP 2: CORE PRODUCTION



STEP 3: LAYER FORMATION FROM THE CORE



STEP 4: IMMERSION IN THE UREA SOLUTION



Fig. 1 Schematic representation of FERT production

Degree of swelling

Swelling tests were performed by weighing out 0.2 g portions of the spheres and placing them in polyethylene bags to facilitate their removal for weighing during the experiment. They were then immersed in 50 mL of citric acid solution (0.01 mol L^{-1}), to try to simulate the environment around the roots of plants, at pH 5.5, 6.5, and 7.5. After predetermined time intervals (10, 20, 30, 40, 60, and 90 min), the spheres were removed from the solutions and weighed again. The degree of swelling of the samples was calculated according to Eq. (1) (Messa et al. 2016):

$$\text{Degree of swelling (\%)} = \left(\frac{m_u - m_s}{m_u} \right) \times 100 \quad (1)$$

where m_u is the wet mass after immersion and m_s is the initial dry mass of the material.

Elemental analysis

Elemental analysis of the samples employed a CHN628 system (LECO) operating using helium (99.99%) and oxygen (99.99%), with the temperature of the primary oven at $950 \text{ }^\circ\text{C}$ and that of the afterburner at $850 \text{ }^\circ\text{C}$. For the analyses, approximately 50 mg portions of the samples were weighed out onto tin foils.

Release tests in water

The release tests in water were carried out (in triplicate) using 0.2 g portions of FERT and urea, which were placed in polyethylene bags and subsequently immersed in 200 mL of distilled water. After predetermined time intervals (1 h, 2 h, 3 h, 1 day, 2 days, 3 days, 7 days, 10 days, 15 days, 20 days, and 30 days), aliquots of 500 μL were removed and analyzed using a UV–Vis spectrophotometer, in order to assess the release of urea from the materials (Yang et al. 2018). The percentage of urea released during the experiment was calculated according to Eq. (2):

$$\% \text{ Urea release} = \frac{M_n}{M} \times 100\% \quad (2)$$

where M_n is the concentration of urea released on each day of the experiment and M is the final concentration released in the experiment (Yang et al. 2018).

Determination of urea

The urea concentrations were determined by UV–Vis spectroscopy (model UV-1800, Shimadzu), at a wavelength of 421 nm, employing the colorimetric reaction between urea and a solution containing 4% (w/v) 4-(dimethylamino) benzaldehyde and 4% (v/v) sulfuric acid in ethanol (With et al. 1961). The samples were placed in 1 cm path length rectangular quartz cuvettes. The concentrations were obtained from a calibration curve constructed using urea standards, with deionized water used as the blank.

Leaching tests

Leaching tests were performed using approximately 1.4 kg portions of soil in polyvinyl chloride (PVC) tubes, with addition of either 0.56 g of FERT or 0.25 g of conventional urea fertilizer, based on a recommended fertilization dosage of 200 kg N ha⁻¹ (Tedesco et al. 1995). To simulate leaching, a 400 mL volume of water was applied using a beaker, which was equivalent to 50 mm of rain, considering the area of the PVC column (Tedesco et al. 1995). At the end of the leaching, NH_4^+ and NO_3^- were determined using a nitrogen distiller (TE-0364, Tecnal), according to the Kjeldahl method (Jyothi et al. 2018).

Release tests in soil

Amounts of 0.3 g of FERT or 0.25 g of urea were added to 200 mL plastic cups containing 25 g of soil, based on the nitrogen content of each material and a recommended fertilization dosage of 200 kg N ha⁻¹, followed by addition of a further 25 g of the same soil to cover the fertilizer. A 40 mL volume of distilled water was added to each cup, followed by incubation at ambient temperature for different periods (1, 3, 5, 7, 10, 15, 20, 25, 30, and 40 days) (Tedesco et al. 1995). Throughout the experiment, the moisture content of the soil was maintained at 30% by the addition of distilled water. After incubation, the remaining FERT and urea were removed and the amount of nitrogen released in the soil was determined by the Kjeldahl method (Jyothi et al. 2018). The amounts of nitrogen released in the forms of ammonium (NH_4^+) and nitrate (NO_3^-), expressed in mg kg⁻¹, were calculated using Eq. (3):

$$\text{mg kg}^{-1} = (\text{mL H}^+ \text{ sample} - \text{mL H}^+ \text{ blank}) \times 70 \times 2.5/5 \text{ g} \quad (3)$$

Physical–chemical parameters of the soil

The soil used in the experiment was classified as a type 1 medium sandy loam. Its physical–chemical characteristics are shown in Table 1.

Results and discussion

Elemental analysis

Table 2 presents the results for the elemental analysis of carbon, hydrogen, and nitrogen in FERT, chitosan, urea, and water hyacinth. It should be noted that although the main functions of the chitosan and water hyacinth were to act as a coating and a polymer crosslinker, respectively, during synthesis of the fertilizer, both were also additional sources of nitrogen.

The nitrogen content of FERT was approximately 20%, indicating its potential for large-scale application, since the content was similar to that of commercial ammonium sulfate ((NH_4)₂SO₄, with 21% N and 24% S), one of the main sources of nitrogen used in

Table 1 Physical–chemical characterization of the soil

Characteristics	Result	Unit	Method	
Organic matter	1.4	dag/kg	Silva (2009)	
Magnesium	0.41	cmol _c dm ⁻³	Silva (2009)	
Potassium	0.13	cmol _c dm ⁻³	Silva (2009)	
Aluminum ^a	0.11	cmol _c dm ⁻³	Silva (2009)	
Calcium ^a	1.56	cmol _c dm ⁻³	Silva (2009)	
Phosphorus ^a	3.10	mg dm ⁻³	Silva (2009)	
pH in water ^a	5.11	–	Silva (2009)	
CEC (T)	3.97	cmol _c dm ⁻³	Silva (2009)	
PES	1.28	%	Silva (2009)	
CEC (T): Potential cation exchange capacity	Base saturation	54.2	%	Silva (2009)
PES: Percentage of exchangeable sodium	Hydrogen + aluminum (potential acidity)	1.82	cmol _c dm ⁻³	Silva (2009)
	Sum of exchangeable bases (SB)	2.15	cmol _c dm ⁻³	Silva (2009)

^aTest carried out as established by the Brazilian Network of Laboratories (RBLE)

Table 2 Carbon, hydrogen, and nitrogen contents of urea, water hyacinth, chitosan, and FERT

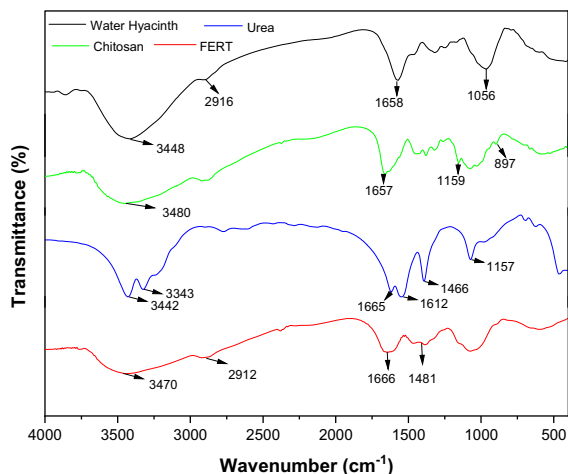
Sample	%C	%H	%N
Urea	20.20	6.06	45.70
Chitosan	27.7	6.30	9.70
Water hyacinth	26.32	6.82	3.57
FERT	26.77	7.08	19.91

global agriculture (Barbosa Filho et al. 2004). However, different to (NH₄)₂SO₄, the fertilizer produced in this work could release nutrients slowly, reducing losses, the costs associated with reapplications, and environmental problems such as soil acidification, leaching, volatilization, and release of greenhouse gases (Ku et al. 2017).

Fourier transform infrared spectroscopy (FTIR) analyses

The infrared spectra of FERT, urea, water hyacinth, and chitosan are shown in Fig. 2. Bands in common at around 3440 cm⁻¹ could be attributed to stretching of the O–H bonds of alcohols, phenols, carboxylic acids, and adsorbed water (Sundari and Ramesh 2012). Bands at around 1660 cm⁻¹ were due to deformation of the bonds of C=O groups of aromatics (Kammoun et al. 2013).

In the water hyacinth spectrum, a band at 934 cm⁻¹ corresponded to bending of the C–H bonds of lignin, hemicellulose, and cellulose, characterizing the

**Fig. 2** Infrared spectra of FERT, water hyacinth, chitosan, and urea

material as a lignocellulosic biomass that could be used for the production of renewable fuels and chemicals (Yi et al. 2020).

In the urea spectrum, bands at 3442 and 3393 cm⁻¹ corresponded to asymmetric and symmetrical stretching of the N–H bond, characteristic of primary amides such as urea (Pavia et al. 2010). The bands present in the urea spectrum were in agreement with those reported by Chan-Chan et al. (2017).

In the chitosan spectrum, a band at 1657 cm⁻¹ corresponding to C=O deformation indicated that the chitosan retained acetyl groups, since it was obtained by means of partial deacetylation of chitin (Anand et al. 2014). All the bands in the chitosan spectrum were similar to those described by Lucas et al. (2020).

In the FERT spectrum, bands similar to those presented by water hyacinth (at 2912 cm^{-1}), urea (at 1481 cm^{-1}), and chitosan (at 3470 cm^{-1}) were present, with some modifications that possibly resulted from interactions with urea, since the interaction with other components can cause conformational changes in the polymeric structure of chitosan (Staroszczyk et al. 2014). The FERT spectrum showed decreases of the bands related to urea, due to the interactions with other compounds during the formation process. Hence, the interactions between the components, as indicated by the spectra, evidenced the formation of the desired material.

Degree of swelling

The degree of swelling is related to the rate of absorption or permeability to solvents, being influenced by the chemical resistance of the polymeric network of the material (Gehrke and Harsh 1991). The swelling experiments were conducted at pH 5.5, 6.5, and 7.5, with the aim of covering the pH range in which the main nutrients are available to plants (Broch and Ranno 2010).

The results (Fig. 3) showed that the UCHS spheres exhibited a much higher degree of swelling than FERT, at all pH values. This could be explained by the addition of the crosslinker in the synthesis of FERT, providing the material with a greater quantity of joined chains and hindering the entry of solvent (Bispo et al. 2010). In the soil, this would be an important feature,

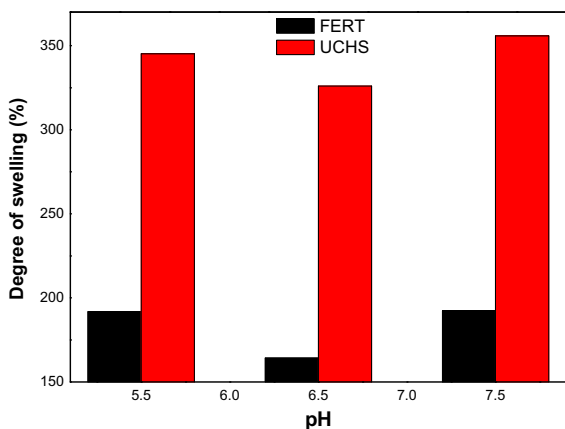


Fig. 3 Degree of swelling of the FERT and UCHS spheres, as a function of pH. Conditions: 0.2 g of the materials; 0.01 mol L^{-1} citric acid solution at $25\text{ }^{\circ}\text{C}$

since it would ensure slow penetration of water into the fertilizer, consequently releasing nutrients to the plants in a controlled manner.

Harris (2009) reported that polymers produced with crosslinkers generally exhibit lower degrees of swelling, compared to those synthesized without crosslinking agents. Therefore, this test can also be used to indirectly assess crosslinking, since swelling depends on the degree of crosslinking of the material (Gehrke and Harsh 1991).

It can be seen from Fig. 3 that the degree of swelling was influenced by the pH, with both materials showing higher degrees of swelling at pH 5.5 and 7.5. It is possible that at pH 6.5, there was the presence of a high density of groups with positive charges, due to the protonation of amino groups in the main chain of the chitosan, which hindered the absorption of solvent by the materials (Vaarum et al. 2005).

However, the amount of solvent absorbed by the material is also influenced by the hydrophilicity of the polymer (Oliveira et al. 2003). Here, the hydrophobic character of the polymer used to coat the fertilizer was increased by the crosslinking along the layers, causing the interaction of water with the polymer to be reduced, consequently decreasing its degree of swelling. This confirmed the effectiveness of the water hyacinth used as a crosslinker, with the further benefit that this biomass, considered an environmental liability, also provided an additional source of plant nutrients (Priya et al. 2018).

Leaching tests

Table 3 shows the concentrations and standard deviations for ammonium (NH_4^+), nitrate (NO_3^-), and total nitrogen in the soil leachates obtained after application of FERT and the conventional urea fertilizer. For all the treatments, there was greater leaching of NH_4^+ , compared to NO_3^- , probably due to the short duration of the experiment, which was not sufficient for oxidation of all the ammonium to nitrate. Rogeri et al. (2015) reported that in soil, almost all the NH_4^+ is converted to NO_3^- in up to 40 days.

The greater leaching of ammonium may also have been due to the low cation exchange capacity (CEC) of the soil (3.97 cmol dm^{-3} ; Table 1), which could have contributed to the low retention of this ion in the soil exchange complex, facilitating its leaching (Araújo 2004). The pH of the soil used in the experiment (pH

Table 3 Concentrations and standard deviations for ammonium, nitrate, and total nitrogen, obtained from the leaching of an argisol from the rural campus of the Federal University of

Sergipe, fertilized with doses of urea and FERT equivalent to 200 kg h⁻¹. Conditions: 0.25 g of urea, 0.56 g of FERT, 1.4 kg of soil, and 400 mL of water. Experiment duration: 2 h

Treatment	NH ₄ ⁺ (mg L ⁻¹)	NO ₃ ⁻ (mg L ⁻¹)	Total nitrogen (mg L ⁻¹)
Urea	41.3 ± 0.074	13.1 ± 0.31	54.4
FERT	0.82 ± 0.02	Nd ^a	0.82

^aNd = not detected

5.11) may also have contributed to ammonium leaching, since ammonium ions predominate over nitrate at acidic pH (Silva et al. 1999).

The urea treatment resulted in substantial leaching of ammonium and nitrate (Table 3), due to the high solubility of urea and its rapid hydrolysis by the urease enzyme, with conversion into other chemical forms that are more liable to losses (Pereira et al. 2017). A much smaller amount of ammonium was leached from the FERT treatment, and there was no nitrate leaching. This demonstrated the potential of the material for application in the field, since it managed to eliminate one of the main forms of losses resulting from the application of urea, which can account for 25–70% of the applied urea (Ernani et al. 2002). Furthermore, the use of FERT could help to minimize environmental damage associated with the contamination of ground and surface waters by high concentrations of nitrate (Chowrady 2005).

Release tests in water and soil

The release rates of FERT and the conventional urea fertilizer in distilled water and soil are shown in Fig. 4a, b, respectively. In water, urea rapidly reached equilibrium, due to its high solubility, with 75% release in the first 24 h and maximum release on day 7. In the case of FERT, equilibrium was only reached on day 30, showing that the material released the nutrient much more slowly, compared to the conventional urea fertilizer, which is the most important characteristic of coated fertilizers (Sofyane et al. 2020). On the first day, there was almost no release of FERT, while increases occurred from day 2 onwards, with leveling off after day 20.

Shaviv (2001) reported that the release of nutrients from fertilizers presents a pattern, with an initial phase of almost no release, moving to a second phase of faster release (linear phase), followed by a third and

final phase, when the nutrient is fully available. The release kinetics observed for FERT would allow nutrients to be supplied to crops in synchrony with their needs, reducing both the frequency of applications and the possibility of environmental damage. As pointed out by Ureña-Amate et al. (2011), the indiscriminate use of current conventional nitrogen fertilizers can not only lead to the contamination of surface water and groundwater, but also to the emission of gases such as nitrous oxide into the atmosphere, contributing to climate change.

The release behaviors of FERT and urea in soil were also evaluated. As shown in Fig. 4b, the rate of urea release in soil rapidly reached equilibrium in the first days. The process was slightly slower, compared to the release in distilled water, which could be explained by the fact that the fertilizer was not in an environment of extreme conditions (such as in water), so its diffusion rate was reduced (Shen et al. 2020).

For FERT, release equilibrium was also reached after a longer time (40 days), demonstrating that the layered coating was able to effectively control the rate of urea release in the soil, over a period of time 4 times longer than observed for conventional urea. These results provided further evidence of the potential of the material for use on a large scale, replacing the conventional fertilizers currently employed.

Cui et al. (2020) produced slow release fertilizers with coatings of one or two layers of natural rubber. It was found that nitrogen release equilibrium was reached in 20 days for materials coated with one layer, and in 30 days for materials with a double layer of coating, showing that the addition of extra layers can extend the duration of nutrient release.

The observed release rate for FERT was very low in the first 24 h, with 73% release reached after 30 days. This was in compliance with the release standards for slow release fertilizers (Trenkel 2010), where the release should be less than 15% in the first 24 h and

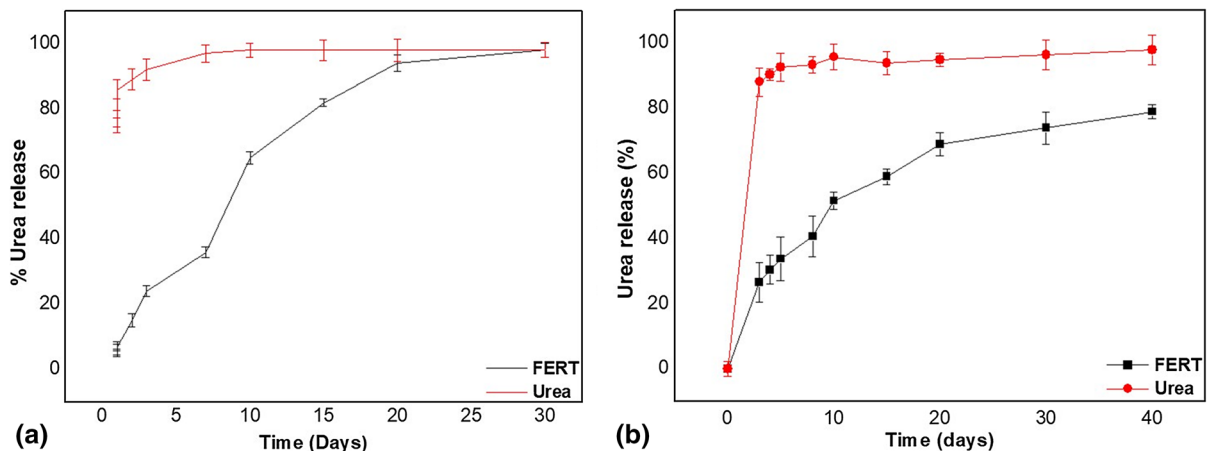


Fig. 4 (a) Percentage releases (with standard deviations) of FERT and conventional urea fertilizer in distilled water. Conditions: 0.2 g of FERT and urea; 200 mL of distilled water

less than 75% after 30 days. Therefore, the results obtained for FERT showed the promise of this novel fertilizer for application in crops.

The findings of this work indicated that the multilayer fertilizer coated with chitosan and cross-linked using water hyacinth has excellent potential to increase the efficiency of nitrogen fertilization and reduce the frequency of applications. It could assist in reducing nitrogen losses in the soil–plant–atmosphere system, implying lower environmental impacts, with better alignment between nutrient availability and the requirements of plants.

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Availability of data and materials Not applicable.

Code availability Not applicable.

at 25 °C. (b) Percentage releases (with standard deviations) of FERT and conventional urea fertilizer in soil. Conditions: 0.3 g of FERT and 0.25 g of urea; 50 g of soil at 25 °C

Compliance with ethical standards

Conflict of interest The authors declare that there are no conflicts of interest associated with the work presented.

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