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MINI REVIEW

A BRIEF REVIEW ON MATERIALS USED IN CONTROLLED-RELEASE FERTILISER FORMULATIONS

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ABSTRACT

The excessive use of fertiliser and uncontrolled-release of nutrient into soil environment have caused a significant eutrophication issue and groundwater contamination. These scenarios have necessitated the development of eco-friendly controlled-release fertilisers (CRFs) for agricultural purposes. In recent years, several materials have been developed and tested to control the release of nutrients particularly in agricultural soils. Although, a number of controlled-release fertilisers have shown a great potential to sustain nutrients release, their performance is greatly influenced by the type of soil, plant species and climate. This review focuses on the development of controlled-release fertilisers using different starting materials or precursors. The technique of synthesis, the rate of nutrients release, the characteristics and drawbacks of controlled-release fertilisers developed have also been highlighted in this review. This review is beneficial to scientists in the field of food science, especially innovative agrochemical formulations to improve food production as well as to mitigate the nutrients leaching to groundwater.

KEYWORDS:

Controlled-release, Fertiliser formulations, Nutrient release, Agricultural soil, Sustainable agriculture

INTRODUCTION

Food is nutritious that people, animals and plants needed to maintain life and growth. New challenges focusing food are growing rapidly all over the world, nowadays [1]. Fast solution is calling across the world concerning food production due to the unprecedentedly increasing world population [2]. As estimated by the United Nations, the global population is expected to increase to 9.7 billion in 2050 and 11 billion in 2100 [3]. China and India are the two top most populated countries with world population

percentage of 18.5% and 17.9%, respectively [3]. Undoubtedly, in this context agriculture serves as an important source of essential food crops. In fact, agriculture has been associated with significant economic backbone for many communities around the world, particularly in providing high quality food for public demand [4].

The utilisation of agrochemicals such as pesticide and fertiliser in agriculture to increase crop yield is absolutely unavoidable. Based on a survey conducted by Food and Agriculture Organization of the United Nations (FAO), the total global consumption of pesticides in agriculture was approximately 2.0 million tonnes per year [5]. The major consumer was Europe (45%), followed by the USA (25%) and other countries (25%) [6]. In the context of Asia, the average usage of pesticide was 12.0, 6.6 and 0.5 kg per ha of cropland for Japan, Korea and India, respectively [7].

Statistics data provided by FAO have shown a steady increment in global consumption of agricultural fertiliser from 2013 to 2018. For instance, it was estimated that 182.8 million metric tonnes of agricultural fertilisers were consumed in 2013 while 199.4 million metric tonnes were applied in agricultural activity in 2018, globally [8]. The three main fertilisers used are nitrogen (N), phosphorus pentoxide (P₂O₅) and potassium oxide (K₂O). N fertiliser is the most commonly used in agriculture, followed by P₂O₅ and K₂O [9]. In 2018, the global consumption of N, P₂O₅ and K₂O fertiliser was reported as 119.5, 45.9 and 34 million metric tonnes, respectively [8]. FAO has projected the demand for agricultural fertiliser will continue to increase particularly in South Asia due to the increase in crop prices and favourable weather.

Although it is known that fertiliser is important to maintain soil fertility, increase crop yields and improve harvest quality, in recent years the crop production has facing some challenges in using the suitable nutrients rich fertiliser [10,11]. In agriculture sector, plant growth with a balanced nutrition is a crucial aspect as it influences the plant metabolism



to increase the crop yields [12]. Fageria et al. [13] reported that in the 20th century, the usage of chemical fertilisers has increased 50% of crop productions, worldwide. Furthermore, the unlimited use of chemical fertilisers has not only affected nutrients in the soil, but significantly contributed to 40-75% of nutrients leaching into the environment [14]. This scenario has caused serious environmental effects and health problems to the living community [15]. Moreover, the risk of nitrate leaching may cause the soil acidification and contributed to eutrophication process which is known as a source of groundwater contamination [16].

A number of strategies have been implemented in order to mitigate the negative environmental impacts of fertiliser application, particularly through increment in fertiliser efficiency [1, 17]. Several efforts were made to increase the fertiliser efficiency such as improving fertiliser application techniques, precision of fertilisation, fertigation-fertilisation via irrigation systems and the use of eco-friendly fertilisers [4, 18]. Currently, controlled-release (CR) technology has been applied in various field as an alternative approach with potential for solving the environmental problems caused by using conventional chemical fertiliser to increase the production of crop yield [7,19]. The purpose of CR fertilisers are: (a) to control the uptake of the nutrients; (b) to allow the release of the nutrients to the specific area; and (c) to maintain the solubility of the nutrients within the optimum rate in a specified time duration [20,21]. CR fertilisers are designed to provide plant nutrients in a way which either: (a) slow its availability for plant uptake from the soil, or (b) reducing its high solubility into the soil and water surface [22, 23]. In many cases, CR fertilisers are normally coated with environmental friendly materials. These materials can be degraded in soil and converted into carbon dioxide, water, methane inorganic compounds and microbial biomass [21, 24]. This review mainly discusses materials including zeolite, urea, layered double hydroxide, carbon, biochar and wastes that are used in formulations of controlled-release fertilisers. The mechanism and processes involved in retaining nutrients release are also discussed.

MATERIALS USED FOR CONTROLLED-RELEASE FERTILISER

Zeolite. Zeolites are crystalline minerals with microporous aluminosilicates containing small pore structures known as 'honey-comb' that consist of tetrahedral TO_4 units, where T is Si or Al. Zeolites can also be derived with the molecular formula of $M^{n+}_{x/n} \cdot AlO_2 \cdot x SiO_2 \cdot yH_2O$ where M is counter ion, n is counter ion valence, x is silicon/aluminum ratio and y refers to content of hydrate water [25]. In industry, zeolites with well-defined micropore holes are called as "molecular sieves". Zeolite molecular sieves have

excellent properties to remove water and other molecules easily. Owing to their unique characteristics, they are widely used in industrial applications since more than 60 years, particularly as effective adsorbents for separating and eliminating impurities in gas or liquid phase [26].

According to Teixeira et al. [27] zeolites are classified as aluminosilicate crystalline minerals with a tetrahedral framework structure which contains oxygen, aluminium and silicon atoms while water and cations are surrounded in the zeolite porous framework. It has been estimated that there are nearly 50 types of zeolites with different physico-chemical properties. Some of commercial zeolites are A, beta, mordenite, Y and ZSM-5. In recent years, there is a great interest in scientific community to synthesis micropores, mesoporous and macroporous zeolites for various applications such as catalyst, fertiliser, water treatment and petrochemical industry. Furthermore, the 3-dimensional framework of zeolite is similar to 'honey-comb' which consists of open micropores interconnected with cages and tunnels. This unique properties enable zeolite molecular sieves to adsorb water and can undergo ion exchange process. As discussed by Tsintskaladze et al. [28], the larger the tunnels and cages, the easier cations which are known as guest ions can pass through the framework. In recent years, several innovative strategies have been made on zeolite with the aim to control nutrient release.

The mechanism of nutrients release from a zeolite-based fertiliser known as Greenfeed Slow Release Fertiliser (GSRF), which was produced in Malaysia, has been investigated by Wea et al. [29]. Several fundamental aspects such as nutrients release behaviour, pH balancer and water retainer in soil were studied. In their study, the release of nutrients from soil used for banana and paddy growth which previously received 16 g per piece of compressed and pelletised GSRF, was based on gradient difference between the soil and fertiliser. Furthermore, the nutrients release from GSRF was discussed to involve three main processes, namely: (1) rehydration, (2) reactivation, and (3) equivalence. The mechanisms of nutrients release for aforementioned processes are given in Table 1. Overall, the use of GSRF was reported to successfully retain the nutrients in a longer period in the plant and improve the pH level of the soil.

Meanwhile, Lateef et al. [30] assessed the feasibility of zeolite nanocomposite (ZNC) slow release fertiliser to retain nutrients in soil. The ZNC slow release fertiliser was developed by using a two-step approach, namely: (1) synthesis of nanozeolite (NZ) using simple co-precipitation method, and (2) impregnation of nine nutrients from salt of each nutrient. They impregnated ZNC with six macro (N, P, K, S, Ca and Mg) and three micro (Cu, Zn and Fe) nutrients for plant growth. Based on a 14-day slow re-



lease study which was conducted in soil environment, they found that Mg^{2+} exhibited the highest release rate (nearly 50%) as compared to other nutrients. Table 2 presents the steps entailed in developing ZNC slow release fertiliser.

Tsintskaladze et al. [28] fused clinoptilolite natural zeolite using ammonium dihydrophosphate ($NH_4H_2PO_4$). During the fusion process, water molecules were desorbed out while ammonium dihydrophosphate molecules were occupied the nanostructure of clinoptilolite. Based on Fourier transform infrared (FTIR) analysis, they confirmed the appearance of P-O-P bonds at wavenumber 1145 cm^{-1} , which represented the phosphate ion bond. They designed six culture tests using ash gray soil with low humus content, and used garlic and onion as plant receptors. The application of clinoptilolite and $NH_4H_2PO_4$ mixture was reported to increase the yield of garlic and onion by 25-30 % and 12-13 %, respectively.

In another study, Li et al. [31] modified a natural zeolite using ammonium chloride, monoammonium orthophosphate and potassium sulphate as an effort to produce a slow release fertiliser known as Eco-Zeolite. The natural zeolite was impregnated with 1 mol/L of nitrogen and potassium at the ratio of 2:1. They also studied the influence of nitrogen and potassium release from Eco-Zeolite slow release fertiliser on the growth and quality of spinach. They conducted a greenhouse study using brown soil (pH 5.27), which contained nitrogen (0.96 g/kg), phosphorous (0.46 g/kg) and potassium (30.66 g/kg). After 6 weeks of greenhouse study, Eco-Zeolite produced a higher yield of spinach with 11 g per plant as compared to 1.24 g per plant for control. Additionally, Eco-Zeolite was reported did not reduce the content of nitrate and oxalate in spinach.

On the other hand, Flores et al. [32] studied the release of potassium from potassic zeolite slow release fertiliser which was derived from Brazilian coal ash. They treated Brazilian coal ash using a conventional hydrothermal method set at $150\text{ }^\circ\text{C}$, for 24 h and 5 mol/L KOH as a solvent. The potassium release from potassic zeolite was evaluated under several experimental conditions, namely: (1) concentration and mass of KOH, (2) mass of coal fly ash, (3) volume of water, (4) temperature, and (5) time. They observed a similar release pattern of potassium for both control and treatment sets. Although they have accomplished a number of important experiments and analyses, they recommended researcher to consider several other soil properties such as ion-exchange, porosity, water retention or nutrient release. As a matter of fact, from soil chemistry perspective it is imperative to study the influence of those properties on nutrients release and impacts on plant growth.

Urea. Urea is a popular synthetic nitrogen fertiliser, worldwide. It has been widely used in agriculture to sustain soil fertility and increase crop yield [33]. A statistics data on production and international trade provided by International Fertilizer Association revealed urea accounts for more than 70% of global fertiliser application [34]. The nitrogen content in urea is high (about 46%) and it is readily converted to ammonia in the soil [35]. Urea can be applied alone to the soil or sprayed on foliage, as well as in the form of mixture with other compound. For example, it can be incorporated with formaldehyde to produce methylene-urea fertilisers, which release nitrogen continuously and uniformly at a slower rate [36].

TABLE 1
Mechanisms of nutrients release in GSRF.

Process	Mechanism description
Rehydration	Movement of soil moisture to the surface of GSRF
Reactivation	Conversion of nutrients into ionic form
Equivalence	Release of nutrients through transfusion

TABLE 2
Processes involved in production of ZNC slow release fertiliser.

Steps	Simple co-precipitation method to synthesis NZ	Simple impregnation method to impregnated the nutrients
1	Precipitation	Suspension
2	Re-concentration	Vacuum
3	Filtration	Filtration
4	Drying	Drying
5	Annealing	Grind

Although urea is comparatively inexpensive as compared to other synthetic nitrogen fertilisers, it tends to release its high nitrogen content into the soil and water through some processes like soil volatilisation, nutrients leaching and groundwater contamination [37]. It is known that only a small amount of nitrogen is taken up by plants for growth, while the remaining nitrogen nutrient is either leaching into the soil surface, causing eutrophication in aquatic systems, or NO_3^- ions runoff into groundwater, changing the pH of the soil [34]. Eutrophication is defined as the enrichment of water by nutrients causing an accelerated growth of algae and higher forms of plant life to produce an undesirable disturbance to the balance of organisms present in the water and to the quality of the water concerned [38].

Trinh et al. [39] investigated multi-diffusion mechanism of controlled-release Agrium coated urea fertiliser. They developed 2-D geometry controlled release fertiliser using urea by Finite element method (FEM) and studied the nitrogen release. The nitrogen release by multi-diffusion mechanism was explained to involve 4 phases, namely: (1) core urea absorbed water but no nutrients release, (2) urea start to release nutrients when reached surface between particle and soil, (3) nitrogen release became constant, and (4) core urea completely dissolved. Furthermore, by using Cosmol Multiphysics software they have estimated that the released nitrogen will reach the surface of coated urea fertiliser in 1.7 mins. Improvements are being carried out by the research team in order to prolong the time for released nitrogen to reach the surface of coated fertiliser.

Meanwhile, Ibrahim et al. [40] have modified melted urea using bentonite and organic polymer based on Chinese Patent Specification (CN201110003090.6) in order to produce a novel slow release fertiliser known as S-urea. Melted urea had been modified using three steps, which involved: (1) moulding, (2) recrystallization, and (3) drying. Moreover, they designed a static release model to investigate nutrients release from urea into water using Peppas and double-exponent kinetic equations. Based on comparison study, they reported that common urea (control) released nutrients in 30 mins while S-urea incorporated with 1 % bentonite released nutrients in 240 mins. They also noted that the release of nutrients from S-urea did not follow the Higuchi kinetic model.

In another study, Li et al. [41] compared the mechanisms of leaching and soil surface volatilisation on nitrogen loss from three polymers coated urea in soil. They produced three controlled-release urea (CRU) fertilisers, which was based on three polymers coated urea namely: (1) polyurethane (CRU-1), (2) degradable polymer (CRU-2), and (3) water-based polymer coated with urea (CRU-3). They performed two field experiments using Alfisol soil in or-

der to determine volatilisation of ammonia and nitrogen release. The volatilisation of ammonia was studied using a vented-chamber method, while nitrogen release was assessed using buried bag-weight method. They reported that during a 75-day interval, CRU-1, CRU-2 and CRU-3 released 70-80 % of total nitrogen. The volatilisation rate exhibited a decrement trend and was explained due to the effects of air temperature and precipitation, which have occurred during rice growing period.

Moreover, Tian et al. [42] designed a 4-year completely randomised block field experiment to study nitrogen release behaviour in soil from controlled-release urea fertiliser. Table 3 lists several methods used to study the nutrients content in soil. The field experiment was conducted in three replicates and seven treatments, labelled as: (1) 50 % CRU in 100 kg/ha, (2) 100 % CRU in 200 kg/ha, (3) 150 % CRU in 300 kg/ha, (4) 50 % urea in 100 kg/ha, (5), 100 % urea in 200 kg/ha, (6) 150 % urea in 300 kg/ha, and (7) CK as control without urea. The total nitrogen content was reported to increase in 0-20 cm soil layer for all treatments, but they obtained no significant difference of nitrogen content in 40-100 cm soil layer treated with CRU.

Yamamoto et al. [43] developed a novel matrix nanocomposite which was known as intelligent nitrogen-loaded slow release fertiliser using exfoliation of montmorillonite as a plasticiser. Intelligent nanocomposite nitrogen-loaded SRF was prepared using MMT which was exfoliated in nanoscale using a simple cold plastic high-productive extrusion method and underwent in situ polymerisation using paraformaldehyde. They used several steps to prepare urea matrix nanocomposite, which involved: (1) pre-mixing, (2) extrusion, (3) shaping, and (4) curing. The nitrogen release in water from urea nanocomposite SRF was studied using Kjeldahl method in Noporo soil. Based on statistical data analyses, they reported that at a higher temperature and a greater degree of polymerisation, less NO_3^- was released from urea nanocomposite SRF.

Layered double hydroxide (LDH). Layered Double Hydroxides (LDHs) are 2-D synthetic nanomaterials and anionic clay composed of 'brucite-like' cationic layers compound with general formula of $[\text{M}^{2+}_{1-x}\text{M}^{3+}_x(\text{OH})_2]^{x+}[\text{A}^{n-}]_{x/n} \cdot m\text{H}_2\text{O}$, where $\text{M}^{2+}/\text{M}^{3+}$ is divalent/trivalent metal cation, A^{n-} is interlayer anion, n - is charge on interlayer ion, m is the number of water molecules located in the interlayer space, and, x and y are fraction constants [44]. Magnesium-aluminium (Mg-Al) and nickel-iron (Ni-Fe) are two examples of LDH exciting family members. Research using LDHs has received great attention from scientists particularly in controlled-release field mainly due to anion exchange between the interlayer and antimicrobial activity [45].



TABLE 3
Method used to determine nutrients in soil.

Nutrient	Method
Organic substance	Wet oxidation with $K_2Cr_2O_7$ and concentrated H_2SO_4
Nitrogen	Kjeldahl digestion
Phosphorus	Olsen
Potassium	CH_3COONH_4 extraction

Bernardo et al. [46] loaded phosphorus into a hydrotalcite-like LDH to produce a smart controlled-release fertiliser. They proposed a simple methodology named as “memory effect”, which was based on adsorption of phosphate nutrient into LDH. They investigated the release of phosphate in both water and soil environments. The development of smart controlled-release fertiliser involved 7 main steps, namely: (1) thermal treatment, (2) reconstruction in pure water, (3) equilibration, (4) agitation, (5) centrifugation, (6) re-suspension in water, and (7) lyophilization. They reported that phosphate-loaded LDH released 90% of phosphate in water within 5 min, while 40-50% in the soil.

On the other hand, Everaert et al. [47] have synthesised different P-exchanged LDHs slow release fertilisers which contained Mg^{2+} and Al^{3+} as metal cations using simple co-precipitation method. They studied the release of phosphate from P-LDHs in soil using zero sink system in 2 mM $NaHCO_3$ at pH 8.3. The phosphate released from Anion Exchange Membrane (AEMs) in LDHs was reported to be in the range of 30 h in Mg-Al (2:1) to 41 h in Mg-Al (3:1), which was slower than in soluble form of phosphate. Furthermore, they designed a pot trial experiment to investigate the phosphate uptake by barley using acidic soil with pH 4.3 and calcareous soil with pH 7.0. Total shoot phosphate uptake in acidic soil increased by 830 mg P/kg, while 1000 mg P/kg in calcareous soil.

In another study, Benício et al. [48] intercalated Layered Double Hydroxide (LDH) with phosphate which containing nitrate ions between the layers using ion-exchange method known as LDH-phosphate controlled-release fertiliser. They used LDH as a nanostructured material because every positive layer with octahedral shape where metal cations located in the middle and hydroxyl anions are at edges can reduce the contact of phosphate ion with soil. They synthesised LDH- NO_3 using co-precipitation method, followed by anion exchange method using K_2HPO_4 to produce LDH-P. Based on the kinetics data, the release of P was reported to be best fitted to bimodal release behaviour. In addition, they noted that the fastest released step in LDH-P started at 60 min, while the commercial fertiliser triple superphosphate (TSP) started after 60 min of in vitro release study.

Besides, López-Rayó et al. [49] assessed the potential of novel 2-D nanostructured Zn-doped Mg-Fe-LDHs zinc controlled-release fertiliser in plant uptake. They accommodated zinc in LDHs layer

mainly to stimulate the activity of 300 enzymes in plants and therefore reduce the pH of rhizosphere. They prepared Zn-doped Mg-Fe-LDH- NO_3 and Zn-doped Mg-Fe-LDH- CO_3 at constant pH value using simple co-precipitation method. They also conducted a greenhouse plant experiment to study the zinc uptake by barley using quartz sand and calcareous soil. Overall, the plant with Zn-doped Mg-Fe-LDHs released 45% of the total zinc content, while concentration of zinc was reported to be 2- and 9.5-fold higher than controls (plants received no Zn-doped Mg-Fe-LDHs treatment).

A 100-day incubation experiment was designed by Everaert et al. [50] in order to assess phosphorus release between three types of fertiliser, namely: (1) P-exchange Mg-Al LDHs powdered (slow release fertiliser), (2) struvite, and (3) mono-ammonium phosphate (MAP) in Kingaroy, Monarto and Streaky Bay soil. The incubation experiment was set up by adding individual soil into a petri dish (diameter 5.5 cm) with each of LDHs powder, struvite and MAP was placed in the centre of the petri dish. The samples were incubated for 100 days at 25 °C. As a result, they obtained a higher availability of P from LDH powder in Kingaroy soil as compared to other fertiliser and soil. This observation was related to soil acidity. They explained that in such acidic soil, LDHs powder was partially dissolved and released metal cations and OH⁻ anions. Moreover, they designed a pot experiment and the phosphorus uptake in MAP was found to be higher than LDHs powder and struvite, which indicated a slower release of phosphorus in LDHs and struvite.

Carbon. Due to their unique 2-D structure with single layer, carbon-based materials such as graphene oxide have been used in agrochemicals industry. Graphene-based materials offer a number of excellent and attractive properties such as large surface area (more than 2500 m²/g), high loading capacity, high zeta potential and contain high density of oxygen based functional groups that are crucial to formulate controlled-release fertiliser [51]. The presence of oxygen based functional groups such as carbonyl, hydroxyl, carboxyl and epoxy on the surface and edge of graphene oxide will favour loading mechanism for micronutrients such as copper and zinc. Moreover, a higher number of oxygen based functional groups will allow a greater interaction between graphene oxide and polymer to form covalent and noncovalent bonding [52].

Zhang et al. [53] modified mesoporous carbon

nanoparticles (MCN) and loaded selenite to produce Se controlled-release fertiliser. They observed a new peak at 63.0 eV of X-ray photoelectron spectroscopy (XPS) spectrum, which confirmed the presence of Se in MCN-Se fertiliser. Furthermore, loading capacity of Se into carbon nanosystem CRFs was investigated with 1 mM, 2 mM and 5 mM of phosphate and carbonate solutions. Meanwhile, the release of Se content from MCN was assessed in a 28-day leaching experiment using vegetables as plant receptors. They reported that the application of MCN-Se fertiliser has significantly increased the height and size of leaves of the vegetable plants.

However, Zhang et al. [54] have developed a controlled-release fertiliser without application of organic solvents and toxic initiators, which was mainly based on reduced graphene oxide (r-GO) film. The formulation involved encapsulation of KNO_3 pellets (r-GO-coated KNO_3) using ion-mediated thermal reduction method. Atomic force microscopy (AFM) cross-sectional images showed that the thickness of r-GO-loaded KNO_3 was ranging from 0.5 to 0.9 nm. They compared the release behaviour of potassium from r-GO-loaded KNO_3 with uncoated KNO_3 . The results at the initial stage have demonstrated that 34.5% of potassium ions from r-GO-loaded KNO_3 were released in water between 0 to 7 hr, while only 1 hour was recorded for uncoated KNO_3 .

In a similar study, An et al. [55] have developed a graphene based controlled-release fertiliser in sandwich structure which was loaded with KNO_3 granules using organic solvent styrene-butyl acrylate-methyl methacrylate latex through copolymerisation process followed by fluidised bed. They chose graphene oxide as a starting 2-D nanomaterial mainly due to large amount of oxygen with a single-atom thickness. Furthermore, the release of potassium was calculated from cumulative release percentage between loaded and unloaded graphene oxide. They reported that the loaded graphene oxide CRF has successfully extended the release of potassium to 38 days, as compared to 24 days for unloaded graphene oxide CRF. Based on this finding, they concluded that the presence of graphene oxide was the key factor in controlling the release the potassium into the soil environment.

Biochar. Biochar is a multifunctional carbon-based materials known as charcoal which produced by waste materials like animal wastes, industrial

wastes and wood residues using pyrolysis process at low temperature and low amount of oxygen. Biochars have been regarded as low-cost materials and have been used in agriculture due to their pore structure and large specific surface area [56]. It is interesting to note that, the application of biochars as soil fertilisers and soil amendment has increased tremendously in recent years. Some studies have applied biochars in acidic and alkaline soils, and had highlighted several advantages such as increased soil fertility, improved ions exchange capacity and enhanced ability to retain nutrients in soil [57]. Overall, biochars will not only able to improve soil properties, but also to increase the crop yield.

There are some unique characteristics of biochar such as the ability to increase water-loading capacity and soil porosity, and to reduce soil density. Consequently, for this reason Chen et al. [58] have developed a novel controlled-release nitrogen fertiliser based on biochar waterborne copolymers. They prepared biochar based CRFs from maize straw, rice straw and forest litter, which were blended with polyvinyl alcohol (PVA) and polyvinylpyrrolidone (PVP) copolymers using cross-linking method. They investigated the nitrogen release in soil by using soil column leaching method. They reported that on the 22nd day of soil column leaching study, the rice straw biochar showed an excellent controlled-release behaviour of which 65.28% of nitrogen was determined. This scenario was related to lack hydrophilic-OH bonds of rice straw biochar.

In a similar interest, Kim et al. [59] have combined biochar and CRFs application to reduce methane emission and increase rice production in a paddy field. They used a commercial biochar known as rice chaff (*Oryza sativa*) with a diameter of 5 mm, which was produced by pyrolysis method at 600 °C for 20 min. The rice chaff was reported to contain carbon (45.4%), nitrogen (0.57%), potassium (1.245%) and phosphorus (0.079%). The combination of biochar and CRFs was successfully increased the rice yield and reduced the methane emissions to 5.45 Mg/ha and 8,916 mg/m² CH₄ as compared to 5.16 Mg/ha and 13,858 mg/m² CH₄ without biochar application. This success was discussed due to the improvements in soil porosity, soil aeration and content of oxygen through methane oxidation by methanotroph from biochar. Furthermore, they suggested to clarify the effect of combined application of biochar and CRFs using microbial activity and community base structure in the future studies.

TABLE 4
Extractable P content in three types of biochar.

Biochar	Extractable P content (mg/kg)	Total P content (%)
Peanut straw	1918	28
Rice straw	2172	70
Rape straw	700	40



TABLE 5
Influence of temperature on production of biochar.

Parameter	Influence on biochar				
Temperature increases	Increases		Decreases		
	pH	Surface area	Electrical conductivity	Cation exchange capacity	Yield

TABLE 6
Properties of commercial pine-hardwood biochar.

Properties	Values
pH	6.5
Exchange Capacity	283 mmhos/cm
Carbon	74.2 %
Ash	21.4 %
Nitrogen	0.59 %
Phosphorus	0.25 %
Potassium	0.35 %
Micronutrients	< 5 mm/kg

Jing et al. [60] have carried out a greenhouse pot experiment to investigate the growth of soybean plant in an alkaline calcareous soil and the release of phosphorus nutrient using biochar controlled-release phosphorus fertiliser. They produced different size of biochar CRFs from rice straw, peanut straw and rape straw under pyrolysis at 500 °C for 3 hr and extractable phosphorus from 0.5 M NaHCO₃ using molybdenum-blue colorimetry. Table 4 presents extractable phosphorus content in three types of biochar used by Jing et al. [60]. From the pot experiment, the biomass of soybean was reported to increase by 24% and the phosphorus content in the soil was increased from 4.6 mg/kg to 11.3mg/kg following the addition of biochar CRFs. Rice straw biochar with 0.149 mm particle size was reported to successfully extract 1,259 mg/kg P, which was the highest level as compared to peanut straw and rape straw biochars.

Meanwhile, Cheng et al. [56] have developed four types of CRFs based on wheat straw biochar, namely B₂₅₀, B₃₅₀, B₄₅₀, and B₅₅₀. These biochars were prepared separately at 250, 350, 450 and 550 °C under pyrolysis for 1 hr and the surface properties were analysed using adsorption-desorption of nitrogen gas according to Brunauer-Emmett-teller (BET) method. They conducted leaching experiment and analysed the N content in the leachate using colorimetric method, the P content with Murphy and Riley method, while the Na, K and Ca content was determined using flame photometer. Table 5 presents the influence of temperature during pyrolysis on the production of biochar.

Moreover, Pfister et al. [61] have evaluated the effects of application of commercial pine-hardwood biochar based CRF on the growth of 72 sunflower plants using a randomised block design. Table 6 lists the properties of commercial pine-hardwood biochar, which was produced from pyrolysis. Furthermore, they used Tukey's HSD test to study the effect of biochar based CRFs using eight types of parameters, while the nutrients release from the leaf of sunflower was studied using EPA Method 3052 followed by ICP-OES. Table 7 presents the parameters that they studied and the effects of application of biochar based CRFs on sunflower growth in a greenhouse study.

Wastes. Waste materials derived from agricultural and industrial by-products can be alternative sources of micronutrients such as zinc and copper for plant growth. As these nutrients can be obtained from waste materials, the conversion of waste materials into fertiliser has been regarded as a green strategy to reduce the amount of wastes worldwide [62]. In fact, this technique offers relatively a lower production cost than conventional fertiliser production. Food wastes are normally consist of portions of food which are not edible such as fruit and vegetable peels, fish bones and egg shells that can be converted into organic fertilisers. This is an eco-friendly technique of which, food wastes will be broken down through anaerobic process and it reduces the release of methane gas from spoiled food.

Navizaga et al. [63] derived 'zinc micronutrient' controlled-release fertiliser from dairy wastewater (Zn-WWT) and they assessed the effectiveness of Zn-WWT in a pot experiment. The effects of Zn-WWT application on the growth and nutrient uptake in biomass of 'SL *Enormo* FAO' maize and spring wheat 'Triso' was compared against ZnSO₄, ZnO and Zn-EDTA fertilisers. Table 8 shows the steps entailed in the synthesis of Zn-WWT controlled-release fertiliser. Based on elemental composition analysis, Zn-WWT contains Zn (7.75%), N (4.06%), P (5.38%), K (0.1%), Ca (6.37%), Mg (0.31%), S (0.09%) and C (35.45%). Subsequently, Zn-WWT was reported to increase the dry mass of spring wheat by 8.0% while did not affect the dry mass of maize. They also noted that the content of Ca, Mg and P in the dry mass of spring wheat was significantly changed, while N and K were not affected by Zn-WWT treatment.



TABLE 7
Effects of biochar-based CRFs on eight parameters of sunflower growth.

Parameters	Effects
Plant height	Increased from 112.3 to 124.5 cm
Chlorophyll content	29.2-32.7
Biomass yield	Increased
Feedstock energy	Not affected
Ash content	Not affected
Tissue nutrients content	Copper decreased
Soil moisture	Increased
Soil pH	Decreased

In another studies, Schneider et al. [27] encapsulated yerba mate (*Ilex paraguariensis*) waste with sodium alginate in a capsule form with 8 to 9 mm of nitrogen based controlled-release fertiliser. They chose yerba mate waste as a raw material mainly due to its large size of leaf portion and small size of stem fragments. Yerba mate waste fertiliser was encapsulated with sodium alginate using ionic gelation and gelling bath method. They carried out nitrogen release study using a soil with pH 7.50. Based on final water absorption point, they observed a curious findings whereby at low yerba mate content used in the capsule formulation, the hygroscopicity of controlled-release fertiliser was increased.

CONCLUSIONS

The cost of CRFs is probably the greatest hurdle limiting its implementation in agricultural sector. This creates great opportunities to scientists to formulate cost effective CRFs with innovative strategies. Several aspects must be considered such as the availability and cost of raw materials, operational cost, effectiveness and applicability. Although results from preliminary and comprehensive laboratory experiments are important, the effectiveness and applicability of CRFs should be evaluated in situ on agricultural field. This is crucial as it will provide an insight into the outcomes from real application, particularly the nutrient uptake by various plant species and mitigation of nutrients leaching to groundwater system. In many cases, researchers are normally developed and assessed the effectiveness of CRFs in laboratory (ex situ) only. This is a major drawback as it failed to convince the potential consumers. Overall, the acceptance of CRFs technology in agriculture will be also dependent upon the consistent demonstration of the agronomic and environmental advantages, as well innovation and technological advances which reduce the cost of CRFs production and improve the nutrient release characteristics to match the dynamic needs of crop growth.

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PAGE 1

PAGE 2

PAGE 3

PAGE 4

PAGE 5

PAGE 6

PAGE 7

PAGE 8

PAGE 9

PAGE 10

PAGE 11

PAGE 12
