J Wood Sci (2011) 57:295–301
DOI 10.1007/s10086-011-1178-x

ORIGINAL ARTICLE

Sheikh Ali Ahmed · Jong In Kim · Kyung Mi Park
Su Kyoung Chun

Ammonium nitrate-impregnated woodchips: a slow-release nitrogen fertilizer for plants

Received: May 31, 2010 / Accepted: January 28, 2011 / Published online: May 25, 2011

Abstract Different types of fertilizers are widely used throughout the world for successful crop production. Chemical fertilizers have some adverse effects on the environment if used indiscriminately and are a major source of soil and water pollution. To minimize environmental pollution, use of slow-release fertilizer (SRF) in agricultural practices is an important and effective method. Different materials have been used so far to formulate SRF, but SRF from wood is a unique technique which reflects a new dimension of wood use. In this aspect, present study was designed to develop a slow-release nitrogen fertilizer using three kinds of woodchips: Japanese red pine (*Pinus densiflora* S. et Z.), eunsasi poplar (*Populus tomentiglandulosa* T. Lee), and konara oak (*Quercus serrata* Thunb.). Fertilizers were prepared from woodchips after full-cell treated with a saturated solution (2140 g/l at 25°C) of ammonium nitrate (NH_4NO_3). The morphology of woodchip fertilizer was investigated by using a field-emission electron microscope (FE-SEM) equipped with an energy-dispersive X-ray (EDX) spectrometer to locate NH_4NO_3 in woodchips. Deposition of nitrogen in the cell lumen was verified by FE-SEM. Deposition inside the cell wall was confirmed by EDX mapping. This study also evaluated the release pattern of nitrogen from impregnated woodchips in distilled water for 768 h and found that nitrogen was released from poplar, pine, and oak in a slow-release pattern. The encapsulated nutrient in the void volume of wood facilitated the slow release. The above findings confirm that woodchip fertilizers can be used as a slow-release nitrogen source for plants.

Key words Slow-release fertilizer · Woodchip fertilizer · Ammonium nitrate · Pressure impregnation

S.A. Ahmed · S.K. Chun (✉)
Department of Forest Biomaterials Engineering, College of Forest and Environmental Sciences, Kangwon National University, Gangwon-do, Chuncheon 200-701, Republic of Korea
Tel. +82-33-250-8326; Fax +82-33-251-8326
e-mail: chun@kangwon.ac.kr

J.I. Kim
Division of Wood Processing, Korea Forest Research Institute, Seoul 130-712, Republic of Korea

K.M. Park
Kangwon National University, Gangwon-do, Chuncheon 200-701, Republic of Korea

Introduction

Fertilizer is one of the most important agricultural inputs in crop production. To ensure efficient crop production, full recognition has been given to the importance of an adequate supply of plant nutrients. However, about 40%–70% of nitrogen, 80%–90% of phosphorus, and 50%–70% of potassium in the fertilizers normally applied are leached out to the environment and cannot be absorbed in plants. This process leads not only to severe economic and resource losses but also to serious environmental pollution.[1] One method of reducing fertilizer nutrient losses involves the use of slow-release fertilizer (SRF). The use of SRF represents an advantageous alternative to traditional soluble fertilizer throughout the growing cycle of crops.[2] It avoids a high salt level in the growing media, improves efficiency of nutrient use, reduces nutrient leaching losses, and lowers labor costs.[2–6] Moreover, application of SRF is a promising management practice that can ensure proper utilization of nutrients, and the economic gains are obvious with SRF technology. If the fertilizer products are formulated and applied properly, SRF could overcome problems normally associated with soluble fertilizer.[7] The gradual release pattern of SRF provides a more consistent and sustained nutrient supply and exhibits better growth performance.[8,9]

Over the past decades, several types of SRF have been formulated from different materials. Formulation of SRF with woodchips represents a new dimension of wood uses. For example, Ahmed and Chun[10] formulated woodchip fertilizer and showed that application of woodchip fertilizer in cultivated cabbage fields increased agricultural productivity relative to soluble fertilizer by minimizing valuable nutrient losses. Principal factors affecting the release of nutrients from slow-release woodchip fertilizer are soil moisture and exposure time. In addition, the factors involved in nutrient release from commercial SRF are fertilizer particle size, soil (substrate) moisture content, pH, and microbial activities.[2,11]

To manufacture woodchip fertilizer, nutrient solutions are impregnated in woodchips using a pressurizing method.[10] Thus, it is clear that nutrient content in woodchip fertilizer is related directly to the permeability of wood species. The permeability of wood varies greatly, with factors such as anatomical features, wood type, moisture content, and the properties of the permeable liquid.[12–14] Furthermore, the permeability of wood is related to the treatment conditions such as the applied pressure and treatment time.[15] Pressure impregnation is an important method for increasing the nutrient content of woodchips. When the nutrient-impregnated woodchips are applied in the field, nutrient from inside the wood void structures enters the soil solution over a long period of time through diffusion and capillary phenomena. Moreover, the woodchip itself can increase the organic matter content of the soil after decomposition.[16,17]

Fertilizer-grade ammonium nitrate normally contains about 34% nitrogen and is popular in many European countries as well as in North America.[18] Although ammonium nitrate contributes greatly to increase crop production, considerable and rapid loss of applied nitrogen has produced air and water pollution. Indeed, the reason for this loss is that ammonium nitrate is relatively prone to leaching and denitrification.[19] Therefore, the present investigation sought to formulate ammonium-nitrate-impregnated SRF using woodchips in order to ensure gradual nutrient release for plants without producing any adverse effects on the environment.

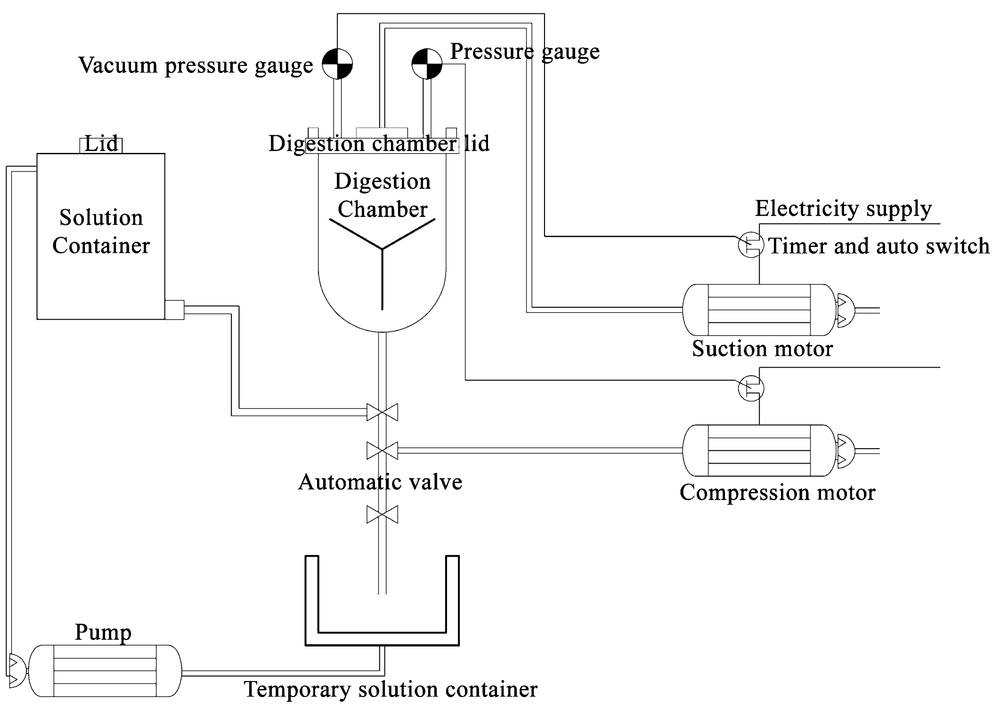
Materials and methods

Woodchip fertilizer preparation

Woodchips were obtained from three wood species: Japanese red pine (*Pinus densiflora* S. et Z.), eunsasi poplar (*Populus tomentiglandulosa* T. Lee), and konara oak (*Quercus serrata* Thunb.). Samples were collected from nonleaning defect-free trees obtained from Jiamri, Sabukmeyon, Chuncheon, Gangwon-do, Republic of Korea (37°58′N, 127°35′E, 290 m above sea level). Small-diameter logs of these three wood species were chipped to an average size of 25 mm × 15 mm × 3 mm. Commercial-grade ammonium nitrate, NH_4NO_3 (Huchems, Korea), was used as the nutrient solution. A large amount of nutrient in the woodchips is required to reduce the bulk volume of woodchip fertilizer applied in the field. Therefore, the woodchips were impregnated with a fully saturated NH_4NO_3 solution (2140 g/l at 25°C). The initial moisture content of the woodchips before impregnation was maintained at 10%–12%. About 500 g of pine, poplar, and oak woodchips were full-cell treated with about 2 l of saturated NH_4NO_3 solution. The treatment schedule was as follows: 15 min vacuum (−82.7 kPa) followed by pressure (1471 kPa) for 60 min at room temperature. Impregnated woodchips were then oven dried in the oven at 60°C for 72 h to fix the NH_4NO_3 solution. Further details about pressure impregnation of liquid can be found in elsewhere.[10,20]

Air-dried woodchips were placed in the digestion chamber of a digester machine (Fig. 1). Vacuum pressure

Fig. 1. Schematic of the digester[20]



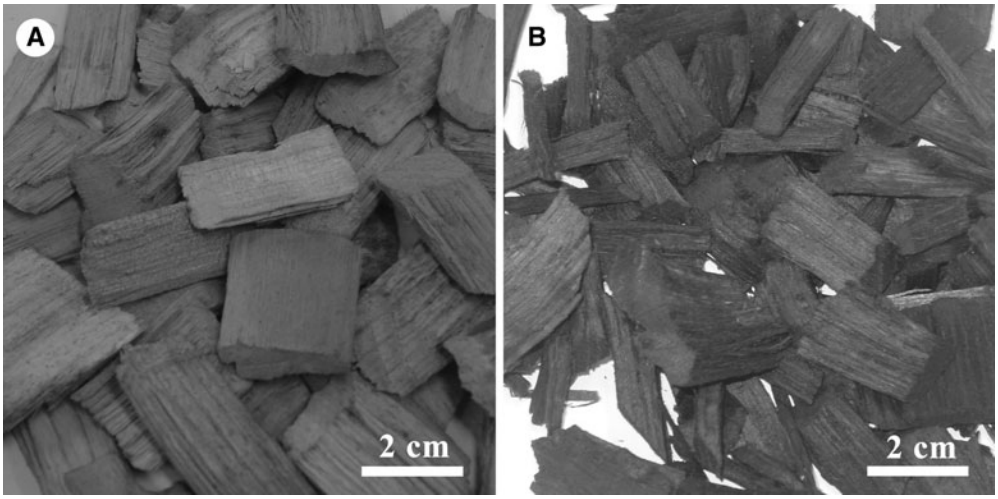


Fig. 2. Oak woodchips before (**A**) and after (**B**) impregnation with NH_4NO_3 solution (N fertilizer)

was applied to remove the air from within the cells of the woodchips. Nutrient solution was introduced to soak the woodchip inside the digestion chamber. When woodchips were fully immersed in nutrient solution, pressure was applied to enhance maximum penetration of nutrient solution into the woodchips. After applying continuous pressure for 60 min, nutrient solution was drained from the digestion chamber, and the treated woodchips were dried. The woodchip fertilizer is ready for use after drying (Fig. 2).

Field-emission scanning electron microscopy

The deposition of nutrient in woodchip fertilizer was observed by FE-SEM (Hitachi S-4300, Japan) equipped with an energy dispersive X-ray spectrometer (EDX, EMAX 6853-H, Horiba EPS, Japan). The FE-SEM was operated at 15 kV for EDX analysis and mapping. Sample blocks 2 mm × 2 mm × 1 mm were used for FE-SEM observation. For SEM-EDX mapping, very thin cross-sectional slices (15–20 μm) were used. After vacuum drying, samples were adhered onto aluminum stubs with double-sided tape and coated with platinum (Pt) by using an ion sputter apparatus (Hitachi E-1010, Japan). The recording times for EDX analysis and mapping were 5 and 60 min, respectively.

Estimation of nutrient content in woodchip fertilizer

Solid woodchip fertilizers were Wiley-milled using a 1-mm (40-mesh) screen. After wet digestion by H_2O_2-H_2SO_4, total Nitrogen (T-N) was estimated by the Kjeldahl method.[21] The content of Ca, Fe, Cu, Zn, Cd, Cr, and Ni was determined with an inductively coupled plasma spectrometer, ICP (LEEMAN PS950, Leeman Labs, USA).[21]

Slow-release test of woodchip fertilizer

Woodchip fertilizer weighing of 1.5 g each was immersed in 1000 ml distilled water. The mixture was then incubated at room temperature. The release test for each species was replicated three times. The supernatant solutions were sampled (1 ml) at predetermined time intervals (24, 48, 96, 192, 384, and 768 h) and replaced by fresh distilled water. N as the sum of ammonium and nitrate in the samples was estimated by an ion chromatograph (DIONEX DX-320, USA) equipped with an AS11-HC column (4 × 250 mm, anion exchange) and a CS 12A column (4 × 250 mm, cation exchange). Suppressed conductivity detection was used. An Ultra (4-mm) anion self-regenerating suppressor (ASRS) and an Ultra (4-mm) cation self-regenerating suppressor (CSRS) from Dionex were utilized for the anion and cation separations, respectively. Ion chromatographic conditions were as follows: eluent, 30 mM KOH for anions; 18 mM H_2SO_4 for cations; flow rate, 1 ml/min; injection volume, 25 μl; temperature, 25°C. The nitrogen release for different soaking times from three woodchip fertilizers was analyzed by using one-way ANOVA. When significant differences occurred ($P \leq 0.05$), the Duncan significant difference post hoc test was run to distinguish the species effects on nitrogen release (SPSS, Version 12.0.1, 2003).

Results and discussion

Nutrient composition

The content of chemical components of prepared nitrogen (N) SRFs using woodchip is shown in Table 1. Of the three kinds of woodchip fertilizer tested, poplar woodchip retained the highest amount of N, whereas oak retained the lowest amount. Poplar and pine woodchip fertilizers retained 56% and 38% more N than did oak woodchip fertilizer. Toxic metals such as Cr, Cd, Ni, and Zn were not detected, and the content of Cu was considerably lower than the limits set in the standard for Korean Fertilizer Regulation.[22] Some trace elements, including Ca, Fe, and Cu, were found in the woodchip fertilizers. Accordingly, woodchip fertilizer can supply some secondary and micronutrients in addition to N.

Table 1. Chemical properties of slow-release nitrogen fertilizer made from different woodchips

Element	Treated woodchip			Untreated woodchip		
	Pine	Poplar	Oak	Pine	Poplar	Oak
N (%)	22.7	25.7	16.5	0.1	0.1	0.3
Ca (mg/kg)	171.3	240.2	89.7	75.1	80.3	40.4
Fe (mg/kg)	112.0	157.0	58.6	36.8	29.5	ND
Cu (mg/kg)	59.4	83.2	31.1	15.4	41.5	10.2
Zn (mg/kg)	ND	ND	ND	ND	ND	ND
Cd (mg/kg)	ND	ND	ND	ND	ND	ND
Cr (mg/kg)	ND	ND	ND	ND	ND	ND
Ni (mg/kg)	ND	ND	ND	ND	ND	ND

ND, not detected

The permeability of wood varies depending on several factors including wood anatomy and the characteristics of the liquid. Liquid uptake is affected by the poor wettability of the surface of the cell lumen.[13] Banks[15] stated that the permeability of liquid in several commercial timbers is influenced by the properties of the impregnated molecules and the affinity between the solution and the wood. The content of nitrogen and other trace elements in woodchip therefore varies from species to species (Table 1). Owing to the greater porosity of poplar (74%) than of pine (70%) and oak (55%) at 12% moisture content,[23] poplar retained the highest amount of N. In the hardwood species, the vessel arrangement of poplar is diffusely porous, whereas that of oak is ring-porous. It is in agreement with Hassler et al.[24] that diffuse-porous wood has a greater permeability than ring-porous wood.

The presence of air in the cell lumen is the main obstacle preventing rapid penetration of liquid.[25] When liquid starts to enter the woodchip, the existing air inside the void spaces begins to compress. Penetration of liquid is slowed and eventually stopped because of the increasing back pressure. Once pressure equilibrium is achieved, further penetration can be made possible either by increasing or by decreasing the pressure. When penetration is allowed from both sides of the woodchip, the back pressure of trapped air is increased by capillary forces that soon stop further penetration.[26] Although removal of air from dry woodchips is effective,[27] it can be limited by the specific characteristics of wood capillaries because some air can be trapped within the capillaries. Complete penetration can be achieved only if most of the trapped air is removed prior to penetration. Evacuation of dry woodchips has been found to be very effective for removal of air. In practice, complete removal of air is difficult to achieve. Even though the complete removal of air is not possible, it was found that woodchips retained a considerable amount of N when pressure was applied in combination with vacuum (Table 1).

Prime factors responsible for governing the flow are the pressure, fluid viscosity, solvent contact angle, wood pore radius, and wood capillary length.[24] An increase in pressure will enhance the penetration of liquor into the woodchips.[26,28] High pressure is required to overcome the negative effect of surface tension in the liquid–air menisci formed by the capillary condensation of vapor.[29] In addition, high pressure could cause the stretching and bulging of the pit membranes due to plasticity of wood, thus making the pit membrane openings larger.[30] When higher pressure is applied, air is more soluble in the liquid. Thus, more air dissolves, and a higher degree of penetration is achieved. In both sapwood and heartwood, liquid permeability is increased at high pressure. In combination with vacuum and pressure, a considerable amount of nitrogen retention was obtained for different woodchip fertilizers in this study (Table 1).

FE-SEM observation and EDX analysis

EDX spectra of treated samples were taken at different magnifications in order to obtain information on NH_4NO_3 deposits in the cell lumen (Fig. 3C) and in the wood cell wall. Figure 3D shows the EDX spectrum of NH_4NO_3-treated samples. The characteristic signal for N was detected clearly at 0.393 keV and provided qualitative evidence for nitrogen deposition in the samples. A suitable higher magnification allowed observation of the deposition of nitrogen within the cell wall. The same characteristic signal for N was detected at 0.393 keV when measurements were made inside the cell wall of the pine sample. The EDX mapping function furnished information on the relative nitrogen distribution. Figure 4 shows a FE-SEM micrograph of a part of the cross section at 500× magnification. In addition, the figure also shows the nitrogen distribution map. The areas with high nitrogen concentration are shown as bright dots. These results confirmed that NH_4NO_3 solution penetrated into the cell wall. Furthermore, the EDX mapping did not indicate any systematic concentration gradients across the cell wall.

Release pattern of nitrogen from woodchip fertilizer

Different types of fertilizer exhibited significant differences in release patterns over time. Soaking time affected the absolute amount of N released during the experimental period. Owing to the micro-structural differences among wood species, the release patterns of nitrogen varied (Fig. 5). For poplar woodchip fertilizer, a marked rapid release of N occurred incrementally during the first 192 h of the experiment. Thereafter, steady rates of release were observed in poplar and oak. However, the rate of nitrogen release from oak was significantly less than that of poplar. In contrast, no significant difference in nitrogen release was observed between oak and pine, particularly after 96 h of incubation time. Even though the oak woodchip fertilizer retained the lowest amount of N, oak showed the slowest nitrogen release of the three wood species studied. After 768 h of incubation, pine, poplar, and oak were found to have released only about 47%, 56%, and 42%, respectively, of their impregnated nitrogen. Thus, woodchip fertilizers can be used as SRFs.

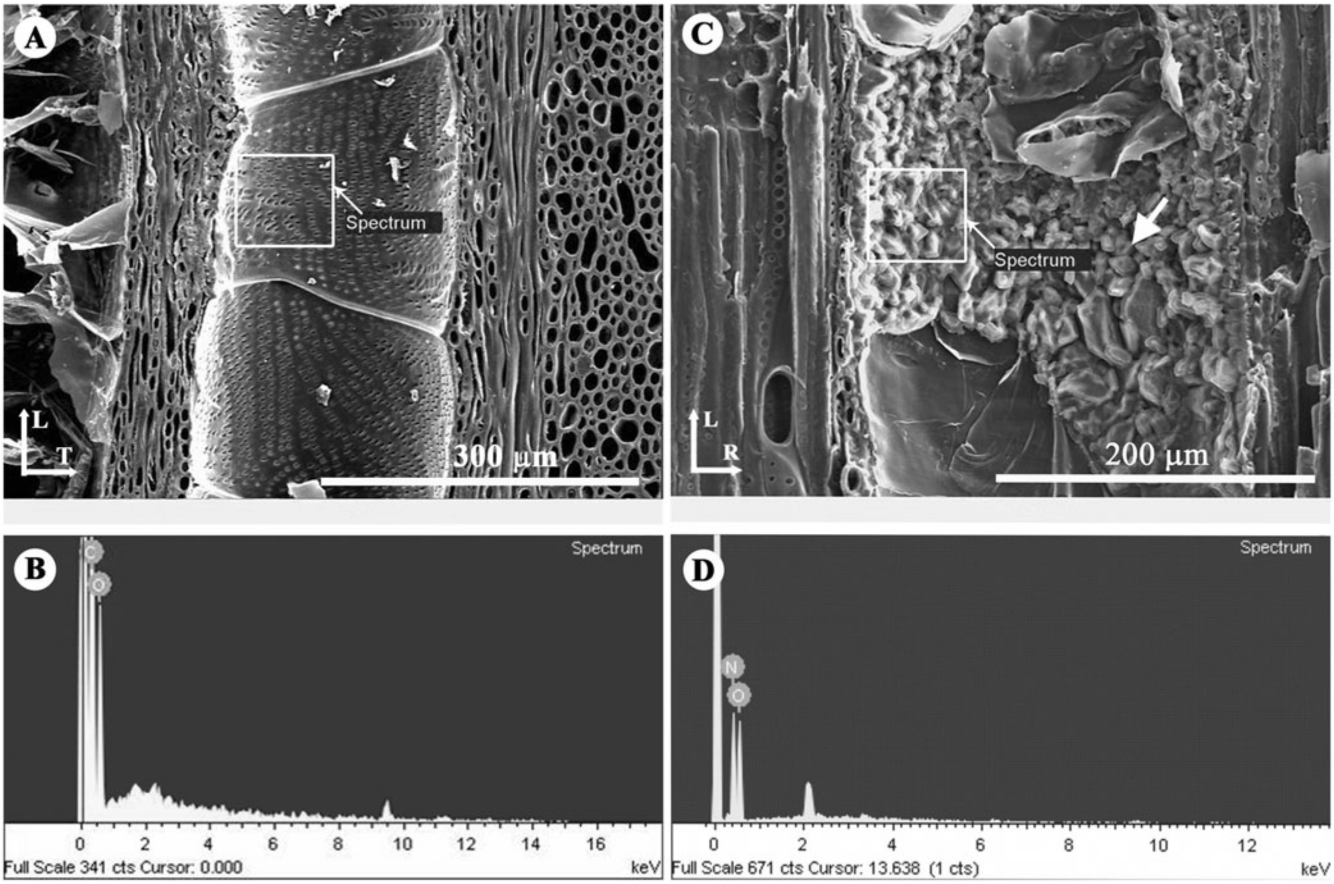


Fig. 3. Field-emission electron microscope (FE-SEM) micrographs and energy-dispersive X-ray (EDX) spectra of oak large vessels in untreated (**A** and **B**) and treated woodchips (**C** and **D**). The *white box* indicates the area for which the spectrum was measured. The *large arrow* shows the nitrogen deposition. *L*, *T*, and *R* are the longitudinal, tangential, and radial planes, respectively

Waldron et al.[31] showed that release occurs by means of a number of concurrent and consecutive phenomena, each affected by variation in wood product, nutrient solutions, and exposure conditions. Dissociation of precipitated or reacted nutrients and their diffusion from the cell walls to free water inside the cell lumens create the concentration gradient that drives the diffusion process. This diffusion rate is associated with different factors such as wood permeability, direction of movement in wood, nutrient concentration, temperature, and moisture content, among others. Once the nutrients on the surface are removed, a concentration gradient is again created and drives the diffusion process. This repeating phenomenon is believed to cause nitrogen components to dissolve out from the woodchip fertilizer. In this study, nitrogen release from the three wood species varied depending on the pore distribution.

Based on the nutrient demands of the plants, woodchip fertilizers can be recommended according to their nutrient-release pattern. For example, short duration plants can be fertilized with poplar woodchip fertilizer, whereas oak woodchip fertilizer can be used for plants that need nutrients over a long period of time. As no coating material was used for manufacturing woodchip fertilizer, application of this kind of fertilizer to cultivated land will ensure environmental safety.

Conclusions

Saturated solution of NH_4NO_3 was successfully impregnated into the microspores of different woodchips to formulate SRFs from pine, poplar and oak. The specific findings of this study were:

1. Among the three wood species tested, poplar retained the highest and oak retained the lowest amount of N after impregnation.
2. Some secondary and microelements such as Ca, Fe, and Cu were also found in woodchip fertilizers.
3. Once woodchips were dried after treatment with NH_4NO_3 solution, nitrogen deposits were found in the cell lumens. Furthermore, EDX analysis confirmed the occurrence of nitrogen deposition inside the cell wall.
4. The soaking experiment using distilled water for a 768 h incubation period showed that oak woodchip fertilizer had the slowest pattern of release, and poplar woodchip fertilizer the highest.

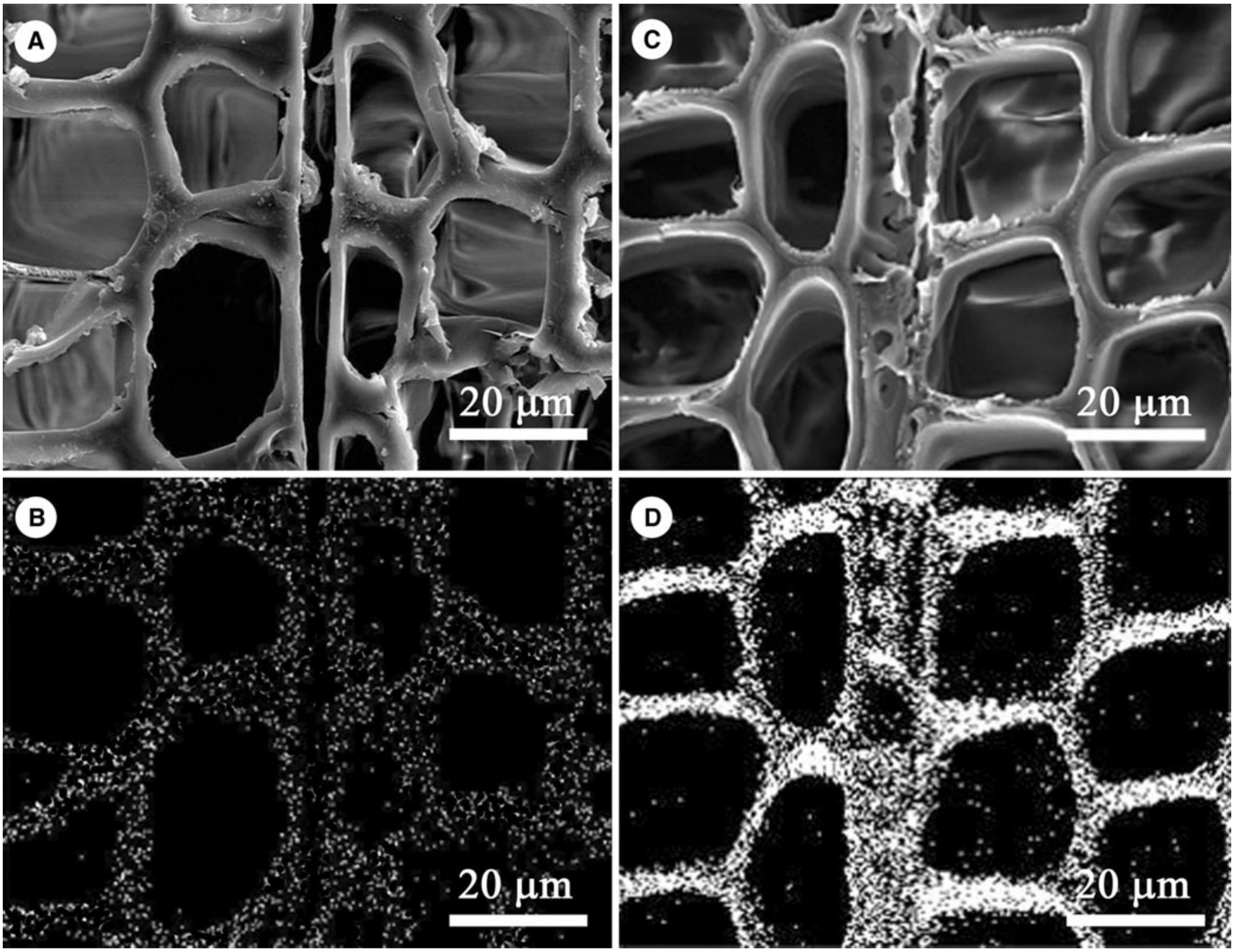
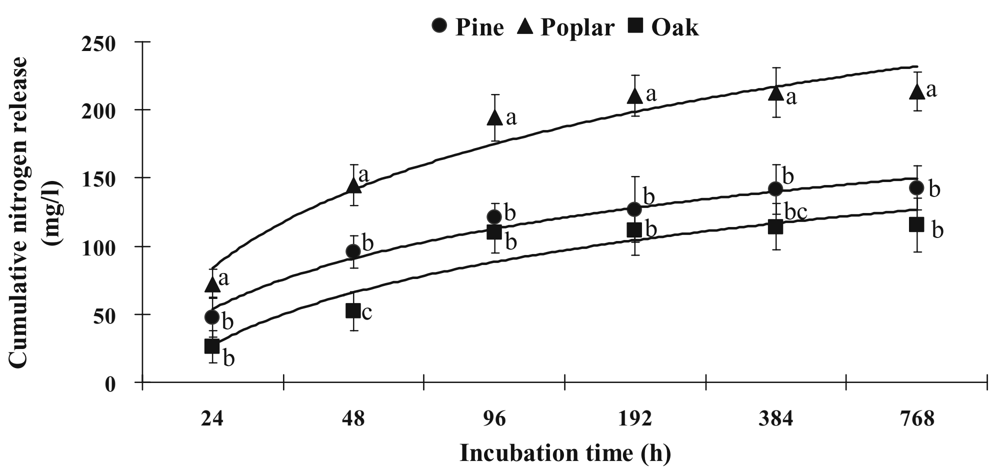


Fig. 4. FE-SEM micrographs and EDX mapping of untreated (**A** and **B**) and treated pine woodchip (**C** and **D**). The areas with high nitrogen concentrations are displayed as bright dots

Fig. 5. Pattern of nitrogen release from different woodchip fertilizers to distilled water



Based on the above findings, it is suggested that woodchips impregnated with NH_4NO_3 could be used as slow-release nitrogen fertilizers for plants.

Acknowledgments This study was supported by the Korea Environmental Industry & Technology Institute and Kangwon National University, Republic of Korea.

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