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ARTICLE

Evaluation of a combination of phosphorylated fibers and zeolite as a potential substitute to synthetic wetting agents in peat moss products

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Abstract: Water availability and pH are important factors to consider to determine the suitability of a material for use as a growing medium. Unfortunately, most horticultural substrates are characterized by their water repellency. This is the case with peat moss which is hydrophobic and acidic. Synthetic surfactants are required to improve its wettability. In this study, a combination of phosphorylated wood pulp fibers (FLP) and zeolite is proposed as a substitute to surfactants to increase the wettability of peat moss in the presence of lime, an additive generally used as fertilizer or pH regulator. Results show that lime reduces the water retention capacity of FLP. However, the addition of 15% zeolite to the peat moss/FLP system increases the pH and water retention of the substrate. The negative effect of the presence of 1 wt. % lime on the water retention of the peat moss/FLP mixture was corrected by zeolite addition. Optimal conditions were obtained at 10% zeolite for the two types of lime tested with favorable pH and water retention capacity values. Zeolite was shown to have a higher affinity than FLP for calcium ions preventing the detrimental interaction between FLP and calcium ions.

Key words: peat moss, wood pulp fibers, phosphorylation, zeolite, water retention, wetting agent.

Résumé : La quantité d'eau disponible et le pH sont d'importants facteurs à prendre en considération quand on désire établir si un matériau peut servir ou pas de milieu de croissance. Malheureusement, la majorité des substrats horticoles se caractérisent par leur hydrophobicité. C'est notamment le cas de la mousse de sphaigne, qui est à la fois hydrofuge et acide. On doit donc recourir à des agents tensio-actifs synthétiques pour en rehausser la mouillabilité. Dans cette étude, les auteurs ont remplacé ces agents par un mélange de fibres de pâte de bois phosphorylées (FBP) et de zéolite pour accroître la mouillabilité de la mousse de sphaigne en présence de chaux, additif souvent employé pour fertiliser le sol ou corriger le pH. Leurs résultats indiquent que la chaux réduit la rétention d'eau par les FBP. Cependant, ajouter 15 % de zéolite au mélange mousse de sphaigne/FBP relève le pH du substrat et augmente le volume d'eau retenu. La zéolite rectifie les effets négatifs de la présence d'un pour cent de chaux en poids sur la rétention de l'eau dans le mélange mousse de sphaigne/FBP. Pour les deux types de chaux testés, les auteurs ont obtenu les conditions optimales (pH et quantité d'eau retenue) avec 10 % de zéolite. La zéolite se caractérise par une meilleure affinité aux ions calcium que les FBP, donc prévient les interactions indésirables entre ces dernières et les ions calcium. [Traduit par la Rédaction]

Mots-clés : mousse de sphaigne, fibres de pâte de bois, phosphorylation, zéolite, rétention d'eau, agent mouillant.

Introduction

Peat moss is one the most used horticultural substrate because of its availability, low cost, and interesting physical and chemical properties. Peat stimulates plant growth and root development by improving the structure of the growing media, buffering the soil, and preventing the leaching of nutriments. However, the acidic nature of peat requires the addition of lime to neutralize the substrate before its use. Lime is a low-cost natural material that has been used for over 2000 yr to adjust the pH of agricultural land to values between 6 and 7 allowing earthworms to thrive and break down residues into nutriments required for plant growth (Lemaga et al. 2001). Acidity has a negative impact on soil fertility by

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causing nutrient deficiencies and promoting the presence of phytotoxic compounds (Yamoah et al. 1996). Water repellency is another drawback to the use of peat as a growth substrate. In consequence, the addition of a wetting agent is usually required to improve the wettability of the medium. The most common wetting agents are surfactants. In addition to the improvement of wettability and water availability, surfactants can also increase nutrient use efficiency and plant growth (Cid et al. 1993; Kostka 2000). Several studies indicate that surfactants have other direct benefits for the substrate–plant system like increased fertilizer availability, water infiltration, physical characteristics, and reduced water evaporation (Valoras et al. 1969; Urrestarazu et al. 2008).

Over the last 50 yr, a wide range of surfactants has been commercialized as wetting agents for peat moss. They are molecules of middle-range molecular weights containing hydrophilic and hydrophobic groups. According to the nature of the hydrophilic group, they are known as anionic, cationic, amphoteric, or non-ionic surfactants. The global consumption of surfactants in the agricultural industry is estimated to 2.5 $\mathrm{Mt \cdot yr^{-1}}$ (Richard and Thacker 2003). According to their chemical structure, surfactants can have different modes of action. However, they have the common property to attach to the surface of hydrophobic substrates making them wettable through their available hydrophilic groups (Cisar et al. 2000).

Most surfactants used in peat moss products are synthetic petroleum-based compounds such as alkylphenol ethoxylates (APEs) and block copolymers. The environmental persistence of these compounds is not expected to exceed 7 d under frequent irrigation (Ying 2006). Even though these wetting agents are still commonly used, they present some negative aspects such as a relatively rapid loss of efficiency during storage that reduce the shelf life of the finished product. Besides, when high dosages are applied, these compounds can be phytotoxic and cause environmental problems. After use, surfactant degradation products can be directly discharged into the soil, water, or sediments and may constitute an environmental hazard.

In the last few years, the development of less phytotoxic, but less active, wetting agents was reported. Claims were made about their greater longevity and lower water volume required for optimum plant growth (Edser 2007). Recently, a new process to modify wood pulp fibers by phosphorylation using a phosphate ester was developed (Shi et al. 2015). The modification makes wood pulp fibers more hydrophilic by grafting high amounts of phosphate groups while preserving the fiber morphology (Shi et al. 2015). The use of modified wood-pulp fibers as a wetting agent for peat would lead to the replacement of petroleum-based products with renewable bioproducts thus promoting sustainable agriculture and best management practices. Unlike surfactants that are usually applied to horticultural substrates, phosphorylated fibers have a much longer lifetime. At the current time, the only drawback preventing the use of phosphorylated fibers as a wetting agent for peat moss is their high affinity for multivalent cations such as calcium which significantly reduce their hydrophilic character. In horticulture, calcium is derived mainly from the lime used to regulate the pH to growing condition values. Like lime, zeolite has been used to regulate growth medium pH and improve nutrient uptake (Amrhein et al. 1996; Ming and Allen 2001). Zeolites are hydrated aluminosilicates of metals such as sodium, magnesium, or potassium. With a particular structure containing $\mathrm{AlO_4{}^{5-}}$ and $\mathrm{SiO_4{}^{4-}}$ tetrahedral units, zeolite can easily exchange cations without changing its natural structure (Hershey et al. 1980; Boros-Lajszner et al. 2018). Zeolites are already used in certain horticultural applications with no undesired effects. The hypothesis that the water retention capacity of phosphorylated fibers can be preserved by their combination with zeolite will be verified. The main objective of this study is to evaluate the influence of the combination of phosphorylated wood pulp fibers and zeolite on the pH and wettability of peat moss products. On the longer term, the replacement of synthetic wetting agents with phosphorylated natural fibers is considered.

Table 1. Typical characteristics of Na-A zeolite.

Color	White
Chemical composition	17% $\mathrm{Na_2O}$, 28% $\mathrm{Al_2O_3}$ 33% $\mathrm{SiO_2}$, 22% $\mathrm{H_2O}$
pH of 1% dispersion	11
Medial particle (μm)	3–5
Calcium-exchange capacity ($\mathrm{mg \cdot g^{-1}}$ of fiber)	280

Materials and Methods

Peat moss and lime used in this study were provided by Berger (St-Modeste, QC, Canada). Bleached softwood kraft pulp fibers were obtained from a northeastern Canada pulp and paper mill. Other reagents were purchased from different suppliers: decanol, polyphosphoric acid and phosphorus pentoxide (Sigma–Aldrich), urea (Alfa Aeser), and denatured alcohol (Fisher Scientific). The Na-A zeolite was received from PQ Corporation. It was used without any chemical pretreatment. Zeolite properties are listed in Table 1.

Phosphorylation of wood pulp fibers

Phosphorylated fibers were obtained by reacting kraft pulp fibers with a phosphate ester in molten urea according to the procedure described in a previous article (Shi et al. 2014). The phosphate ester used for the phosphorylation of fibers was synthesized by a hybrid approach based on classical procedures using decanol, polyphosphoric acid, and phosphorus pentoxide (Kurosaki and Manba 1982).

Fig. 1. Preparation and treatment of phosphorylated fibers. FLP-Hyb, raw phosphorylated fiber; FLP-H, HCl-treated phosphorylated fiber; FLP-Na, NaOH-treated phosphorylated fiber.

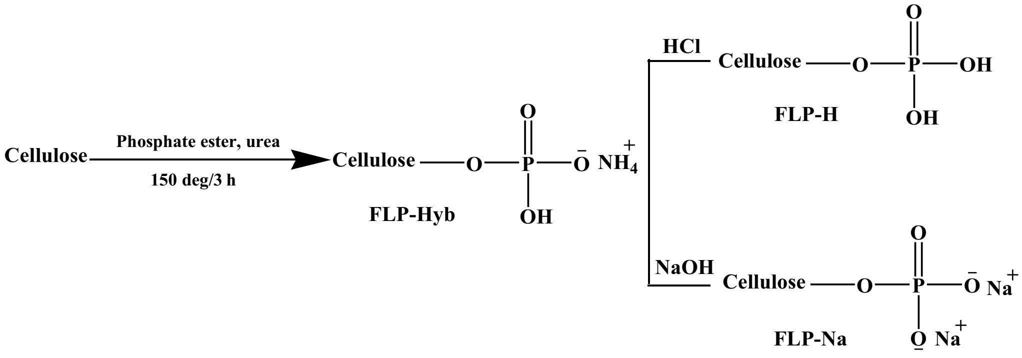
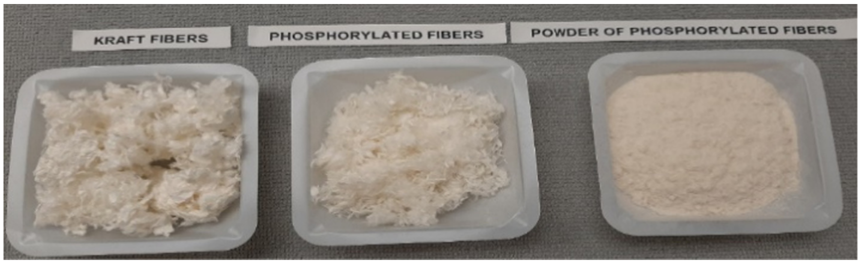


Fig. 2. Visual aspect of kraft pulp and phosphorylated fibers.



Raw phosphorylated fibers (FLP-Hyb) were treated with 0.1 mol·L^{-1} HCl or NaOH solutions to obtain FLP-H and FLP-Na as shown in Fig. 1. Although the ammonium ion carried by FPL-Hyb can be a nitrogen source (plant nutrient), HCl- or NaOH-treated FLP are also interesting materials with specific properties such as higher water retention capacity for FPL-Na (Shi et al. 2015). Treated fibers were washed thoroughly with deionized water to remove residual acid or base. The modified fibers contained close to 1 wt. % of phosphorus.

All types of modified fibers were mechanically treated to decrease their size and improve their dispersion in the substrate. The treatment consists in drying modified fibers at 80–100 °C for 24 h and grinding to obtain a powder as shown in Fig. 2.

Water retention value measurement

The water retention value (WRV) was measured on phosphorylated fibers alone before dispersing them in peat to evaluate their intrinsic ability to hold water after centrifuge and the effect of the presence of calcium ions on this water holding. Water retention value is the most common experimental method used to evaluate the interaction between wood pulp fibers (usually chemical pulp fibers) and water (Scandinavian Pulp, Paper and Board Testing Committee 2000). The method is based on the measurement of the weight of water retained by a wet sample of fibers after centrifugation under specific conditions. This amount of water includes the water in pores, fibril external surfaces, and inter-fiber spaces. For this reason, the WRV of the four wood pulp fiber samples was measured in their powder forms to ensure a maximum availability of surface. The VWR test was carried out to determine which type of phosphorylated fiber can hold the most water. The procedure was realized with 1.5 g of fibers in powder form saturated with 24 mL of deionized water during 24 h at room temperature. The pulp fiber suspension was carefully introduced in a centrifuge tube with a mesh screen at its base and accelerated at a relative centrifugal force of 3000g during 30 min.

The weight of moist samples was subsequently determined, and samples were dried at 105 °C for 24 h. All measurements were made in triplicates for each sample. The experimental incertitude is given by the standard deviation of the results. The WRV, in gram of water per gram of fibers, was calculated using the following equation:

$$\mathrm{WRV} = \frac{m_\mathrm{w} - m_\mathrm{d}}{m_\mathrm{d}}$$

where m_w and m_d are, respectively, the wet and dry substrate masses in grams.

The effect of lime addition on FLP-Na pH and WRV was determined by applying calcitic lime. The mixture was saturated with deionized water and stored at room temperature for 24 h before measurements were taken.

Water retention measurement

The total water retention (WR) measurements were carried out according to a procedure adapted from Fonteno

Fig. 3. Water retention measurement apparatus.

Fig. 4. Experimental setup used to estimate zeolite and NaOH-treated phosphorylated wood pulp fibers affinity for calcium ions.

et al. (2013). The WR was measured for fibers/peat mixtures to evaluate the water-holding capacity of the system at atmospheric pressure and room temperature. The measuring device consists in a transparent graduated cylinder with a mesh screen at its base and a container to collect water (Fig. 3). Test substrates are prepared by dry mixing. The WR is measured in typical horticultural conditions. The amount of phosphorylated fibers in the mixture was set at 30% to ensure the cohesion of the material and reduce loss during handling. A sample of mixture was placed into a container and saturated with deionized water during 24 h at room temperature. The suspension was carefully introduced in the vertically positioned cylinder. Water percolated through the sample by gravity and was collected in the beaker below.

The weight of the wet sample was subsequently determined when the duration between two drops exceeded 5 min. The sample was dried at 105 °C during 24 h. All measurements were carried out in triplicates for each sample, and the WR in gram of water per gram of mixture was calculated by the ratio of the wet mass to the dry mass with the same equation as WRV. The experimental incertitude is given by the standard deviation of the results.

Relative affinity of zeolite and FLP-Na for calcium ions

The relative affinity of zeolite and FLP-Na for calcium ions was established with a simple setup shown in Fig. 4. FLP-Na (1 g) was inserted in a plastic cup fitted with a mesh screen. The plastic cup was then placed in a 150 mL beaker containing 30 mL of a 3333 ppm $CaCl_2$ solution and zeolite (0.1 g). Two different conditions were tested: P1 where FLP-Na were introduced in the beaker immediately after zeolite, and P2 where FLP-Na were introduced in the beaker containing the zeolite after 10 min. The adsorption of calcium by Na-A zeolite is very fast. It often reaches more than 75% after less than 10 min for the Ca/zeolite ratio that was used (Song et al. 2015). The P2 condition will determine if FLP-Na can desorb calcium already bound to zeolite. All experiments were conducted at room temperature. After 24 h of contact between materials and the calcium solution, fibers were removed from the plastic cups, and zeolite was recovered by filtration. Both solids were washed carefully with deionized water and dried at 100 °C during 5 h.

Solid materials from trials P1 and P2 were digested in an oxidizing system consisting of concentrated sulfuric acid and hydrogen peroxide. The determination of the amount of calcium retained on the phosphorylated fibers and by the zeolite was determined by inductively coupled plasma mass spectrometry (ICP-MS).

Table 2. The pH and water retention value (WRV) of wood pulp fibers and peat moss.

Sample	pH (±0.1)	WRV (g water·g^{-1} fibers) (±0.03)
Peat moss	2.5	Not applicable
Unmodified FL	5.8	0.84
FLP-Hyb	5.7	0.97
FLP-H	3.3	1.24
FLP-Na	6.3	1.34

Note: FLP-Hyb, raw phosphorylated fiber; FLP-H, HCl-treated phosphorylated fiber; FLP-Na, NaOH-treated phosphorylated fiber.

Results and Discussion

Water retention value measurement

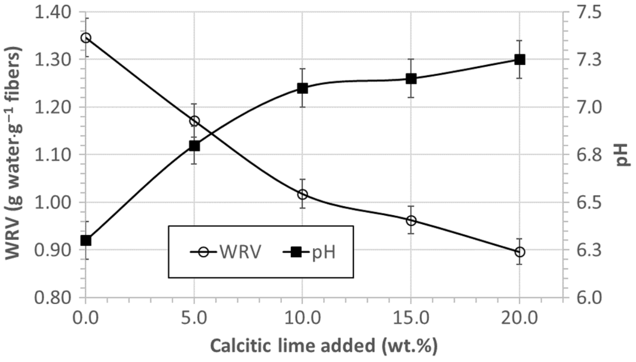
Table 2 shows that pH and WRV of fibers are influenced by the phosphorylation reaction and the post-treatment with sodium hydroxide or hydrochloric acid. Among all fibers used in this study, unmodified FL and FLP-Hyb exhibited the lowest WRV values. The WRV is viewed as the penetration of water through fiber pores and cavities leading to fiber swelling. The retention of water was found to correlate with the fiber pore volume (Joutsimo and Asikainen 2013). The presence of hydrogen on the phosphate group promotes the formation of inter- and intra-fiber hydrogen bonds and the aggregation of cellulose chains which reduce their water retention capacity (Miao et al. 2018). However, the sodium form FLP-Na exhibited the highest WRV among the three phosphorylated fiber types with a mean WRV of 1.34 and a suitable pH for horticulture. Its counter-ion is mobile and able to break hydrogen bonds, preventing treated fibers to aggregate and promoting water penetration. Given the pH and WRV of the different types of phosphorylated fibers, the sodium form FLP-Na was selected for further studies.

Effect of lime addition on the pH and WRV of FLP-Na

The effect of lime on sample pH and WRV was determined by applying calcitic lime to the sodium form of phosphorylated fibers. The mixture was saturated with deionized water and stored at room temperature for 24 h before measurements were taken. pH and WRV results are reported in Fig. 5.

The addition of calcitic lime increases pH but lowers the WRV. The highest increase in pH was observed at 20% added lime. It was associated to a decrease of the WRV of FLP-Na to a similar value to unmodified fibers (less than 1). Lime consists essentially of CaO which occurs as an alkaline $Ca(OH)_2$ solution in a moist environment leading to the neutralization of the medium pH. Phosphorylated fibers exhibits a perfect ions exchanger behavior. The free calcium ion from lime presents a high affinity to phosphate groups and can be exchanged with sodium ions. Given the charge density of the phosphate group, calcium fixation prevents water molecules from approaching the hydrophilic part of phosphorylated fibers. Adding a material with a higher affinity toward calcium ions could restore the water-holding capacity of FLP-Na by acting as a temporary trap for calcium ions released by the lime.

Fig. 5. Effect of calcitic lime on pH and water retention value (WRV) of NaOH-treated phosphorylated wood pulp fibers.



Effect of zeolite addition on the pH and WR of peat moss/FLP-Na mixtures

The substrate used in this study has a pH varying from 3.7 to 4.1 which is typical of natural peat moss. The addition of up to 30 wt. % FLP-Na to this substrate did not significantly affect its pH. However, Table 3 shows that adding only 5% zeolite to the mixture increases the pH from 3.8 to 6.2. At higher levels of zeolite, the pH varies slowly and reaches 6.8 at 15 wt. % zeolite. This near neutral pH can be associated to the exchange of Na^+ ions with H^+ from peat (Ramesh et al. 2015). In fact, ion exchange depends on the structure of zeolite. Only the open structure of the microparticle ensures free ion exchange (Nibou et al. 2010). The hydrophobic character of peat results in a low WR. The addition of FLP-Na in the same mixture increases WR from 6.139 to 8.507. Zeolite addition improves the water-holding capacity of the mixture with the highest value measured for 15 wt. % zeolite which corresponds to a 19.5% increase of WR as shown in Table 3.

The WR improvement with zeolite addition can be associated with the powder form of zeolite used in this work. According to Xiubin and Zhanbin (2001), high available surface area improves the water-holding capacity of the zeolite.

Evaluation of pH and WR of a lime-treated peat/FLP-Na mixture in the presence of zeolite

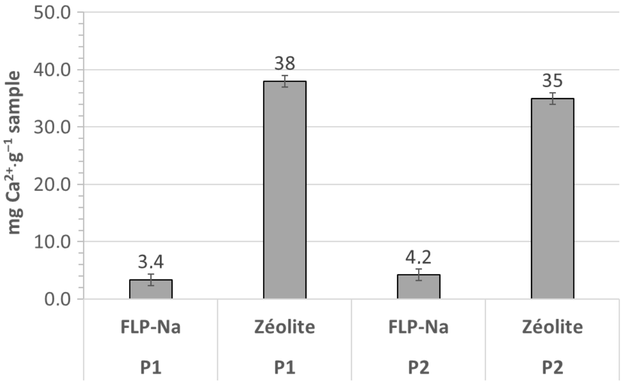
Throughout the study, the composition of the mixture was fixed at 30 wt. % FLP-Na and 1 wt. % lime. This amount of lime, either calcitic or dolomitic did not significantly affect the pH and the water retention capacity of the sample as shown in Table 3.

According to results shown in Table 3, zeolite has not the same effect on the pH of the sample whether lime

Table 3. Effect of zeolite addition on the pH and water retention (WR) of a lime-treated peat/NaOH-treated phosphorylated fiber (FLP-Na) mixture.

pH (±0.1)	Lime (wt. %)	Zeolite (wt. %)	WR (g water·g^{-1} fibers) (±0.007)
3.8	0	0	8.507
6.2	0	5	9.460
6.6	0	10	9.538
6.8	0	15	10.169
3.9	1% calcitic	0	8.384
4.6	1% calcitic	5	9.179
6.4	1% calcitic	10	9.326
3.9	1% dolomitic	0	8.365
5.0	1% dolomitic	5	9.314
6.0	1% dolomitic	10	9.687

Fig. 6. Relative calcium ion affinity of NaOH-treated phosphorylated wood pulp fibers (FLP-Na) and zeolite: simultaneous (P1) and sequential (P2) addition of FLP-Na to the zeolite–Ca^{2+} solution.



is present or not. In the absence of lime, zeolite exchanges its monovalent cations such as Na^+ to increase the pH of medium. The presence of lime in the medium seems to disrupt this mechanism. The zeolite affinity with calcium could cause a competitive exchange between H^+ and Ca^{2+} from lime with Na^+, which would explain the low pH increase.

Although water retention variations are not significant for the two types of lime, neutral conditions appear to be best for most plants, but the optimum pH conditions depend on crop varieties. However, the 6–7 pH range and significant moisture allow a good microorganism activity promoting plant growth. These conditions were obtained at 10 wt. % zeolite.

Determination of the relative affinity of zeolite and FLP-Na towards calcium ions

The relative Ca^{2+} adsorption by FLP-Na and zeolite was estimated in two different conditions: zeolite and FLP-Na are either simultaneously introduced in the calcium ion solution (P1) or FLP-Na are introduced in the calcium ion solution 10 min after zeolite (P2). In both cases, zeolite exhibits a greater ability to sequestrate calcium ions than FLP-Na as shown in Fig. 6. The calcium ions sorption ratio FLP-Na/zeolite remains close to 1/10. With its high ion-exchange capacity, zeolite can release calcium ions and make them available at a later time for plant growth and development. This high ability of zeolite to adsorb free calcium can substantially reduce the detrimental effect of these cations on the water retention of phosphorylated fibers.

Conclusions

The addition of a combination of phosphorylated fibers and zeolite leads to an efficient regulation of pH and water retention capacity of peat moss in the presence of lime. Zeolite, which has more affinity for calcium ions than FLP-Na, acts as a calcium binder preventing the loss of the FLP water-holding capacity, while keeping calcium ions available for plants when needed. Results show that at 1 wt. % lime (calcitic or dolomitic) in a peat/FLP-Na homogenous mixture, zeolite allows obtaining a favorable system for plant development with near neutral pH values and water retention capacity reaching 10 g water per gram of mixture. Therefore, the phosphorylated fibers/zeolite combination could constitute a suitable replacement for synthetic wetting agents in horticulture. However, there is still a need for a follow-up study on the plant toxicity and environmental risks of this system.

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