

Magnesium Uptake and Palygorskite Transformation Abilities of Wheat and Oat^{*1}

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ABSTRACT

Weathering of clay minerals is a source of nutrients to plants. Palygorskite is a Mg-rich fibrous clay mineral that commonly occurs in the soils and sediments of arid regions. Although many studies have examined the environmental conditions required for the formation and stability of palygorskite, information on the transformation of this mineral in the root zone (or rhizosphere) of agricultural crops is limited. This study explored the possibility of palygorskite transformation in the rhizosphere of wheat and oat and compared the ability of these crops to extract structural Mg from palygorskite. The crops were cultivated in pots consisting of a mixture of sand from Hamadan region, Iran and Florida palygorskite, irrigated with distilled water, and treated with either complete or Mg-free nutrient solutions. After 100 d, Mg uptake by the crops was measured. Clay-sized particles in each pot were also separated from the sand and were analyzed through X-ray diffraction (XRD). The X-ray pattern of the particles was compared with that of the pure palygorskite particles (before cultivation). The results showed that palygorskite could provide sufficient Mg for the growth of wheat in the pots supplied with Mg-free nutrient solution. In spite of the magnesium uptake by both plants from the palygorskite structure, no detectable peaks indicating the transformation of palygorskite were recognized by XRD. A decrease of 1.05 nm in peak intensity was more obvious for the pots containing palygorskite and irrigated with Mg-free nutrient solution than for the pots irrigated with complete nutrient solution. Although the decrease in peak intensity was greater for oat than wheat, indicating greater palygorskite weathering by oat, Mg uptake by oat was low, as evidenced by the deficiency symptoms observed.

Key Words: clay minerals, nutrient solution, rhizosphere, weathering, X-ray diffraction

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INTRODUCTION

Magnesium is an integral part of chlorophyll and, hence, essential for photosynthesis. Magnesium exists in the soil solution (a readily available source of Mg for plants) and in both exchangeable (readily available) and non-exchangeable or mineral forms (McLaren and Cameron, 1996). When the exchangeable form is depleted, usually through root uptake or leaching, the conversion of the non-exchangeable to the exchangeable Mg is slow. Roots absorb Mg from the soil solution, which is in direct contact with the roots. Thus, the quantity of Mg in the soil solution must be greater than the actual Mg requirement of the crop to encourage a sufficiently high flux rate towards the roots to maintain Mg uptake (Grimme and Huttl, 1991).

Weathering of soil minerals is certainly an important source of nutrients for plants (Spyridakis *et al.*, 1967; Hinsinger *et al.*, 1992; Gobran *et al.*, 2005).

Weathering plays a significant role in the biogeochemistry of terrestrial ecosystems because it has a strong and sustained effect on the production of the reactive secondary minerals (clays and oxides) in soils, the supply of nutrients to the biota, the release of potentially toxic elements, the support capacity of mineral substrates, soil genesis, the evolution of landscapes (Drever, 1994), and many other phenomena. Although weathering of clay minerals can replenish Mg levels in the soil that are depleted by cultivation of crops, the speed of replenishment may lead to a temporary Mg deficiency (Gobran *et al.*, 2005).

Palygorskite, also referred to as attapulgite, is a Mg-rich fibrous clay mineral that commonly occurs in the soils and sediments of arid and semiarid regions (Singer, 1989). Palygorskite in arid soils originates from two main sources (Khormali and Abtahi, 2003): 1) inheritance from parent materials and 2) pedogenic formation through neoformation or transformation

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of 2:1 minerals. Khademi and Mermut (1998) found palygorskite, sepiolite, and appreciable amounts of mica and smectite in the clay fraction of the Oligo-Miocene limestone but only traces of palygorskite in the Cretaceous limestone samples obtained from central Iran. The *in-situ* formation of palygorskite in a soil solution is facilitated by the presence of gypsum (Eswaran and Barzanji, 1974) and calcrete or caliche (Rodas *et al.*, 1994; Singer *et al.*, 1995; Lopez-Galindo *et al.*, 1996). Paquet and Millot (1972) showed that palygorskite is not stable and transforms to smectite when the annual precipitation is more than 300 mm. Palygorskite also becomes unstable under acidic conditions and low Mg and Si activities (Bigham *et al.*, 1980; Golden *et al.*, 1985; Singer *et al.*, 1995; Neaman and Singer, 2004). Singer and Norrish (1974) showed that in soil solutions with normal silica concentrations, palygorskite is not stable at pH < 7.7 with a high Mg concentration or at pH < 9 with a low Mg concentration.

Although several studies have discussed the origin of the environmental conditions for the stability of palygorskite (*e.g.*, Abtahi, 1977, 1980; Bigham *et al.*, 1980; Torres-Ruiz *et al.*, 1994; Khademi and Mermut, 1998; Akbulut and Kadir, 2003; Khormali and Abtahi, 2003; Daoudi, 2004; Bouza *et al.*, 2005), studies on the role of plant roots on the transformation and dissolution of palygorskite are few. The field study of Courchesne and Gobran (1997) showed that the rhizosphere soil was more intensively weathered and had accumulated more acid and base cations as compared to the bulk soil. Changes in the pH of the rhizosphere, *i.e.*, the volume of soil that is influenced by the activity of living plant roots (Darrah, 1993), are by far the best documented chemical interactions occurring at the soil-root interface. The physical and chemical properties of the rhizosphere soil are different from those of the bulk soil, as the chemical interactions in the rhizosphere soil may create gradients in the concentrations of different elements around the roots (Marschner and Römheld, 1996).

Soil scientists have used pure reference minerals or soil clay assemblages in experiments to study the effect of plant roots on weathering (*e.g.*, Spyridakis *et al.*, 1967; Leyval and Berthelin, 1991; Hinsinger *et al.*, 1992; Hinsinger and Jaillard, 1993; Barré *et al.*, 2007a,

b, 2008; Norouzi and Khademi, 2010). The research by Barré *et al.* (2008) confirms the view that 2:1 clay minerals act as a K reservoir; additionally, the research specifies the role of each of the 2:1 clay mineral populations in K absorption and release. This research also showed that the clay mineral populations commonly found in the temperate grassland soil clay assemblages are modified through K absorption or removal and that these modifications appeared to be reversible.

Most of the abovementioned studies were carried out on micaceous minerals and focused on the release or absorption of K. The transformation of palygorskite and sepiolite in the rhizosphere of alfalfa, barley, and canola was investigated by Khademi and Arocena (2008). To our knowledge, their study is the first to report the neoformation of kaolinite from palygorskite and sepiolite in rhizosphere soils. They hypothesized that the lack of fibrous clays as well as the formation of kaolinite can be partly attributed to a plant-induced kaolinization process in the soils of semi-arid to humid regions. The objectives of our study were 1) to investigate the possible transformation of palygorskite in the rhizosphere soils of wheat and oat crops, 2) to compare the ability of these crops to extract the structural Mg from palygorskite, and 3) to determine the effect of the presence or absence of Mg in the soil solution on palygorskite transformation.

MATERIALS AND METHODS

Sample preparation

Florida palygorskite (PF1-1) obtained from the Source Clays Repository of the Clay Minerals Society was used in this study. To remove exchangeable cations like Mg²⁺, Ca²⁺, and K⁺, the clay surfaces were saturated with NH₄⁺ before the experiment using 1 mol L⁻¹ NH₄Cl solution. The saturated palygorskite was washed several times with distilled water to remove the excess NH₄Cl. The palygorskite was then dried and used for the pot experiment. Sand from Hamadan Province, Iran (Hamadan sand) with a particle size of > 0.10 mm was used as a prominent plant growth medium. It consisted of almost pure SiO₂ and was free of clay minerals and other substances (Table I). To prepare the Hamadan quartz sand for the experiment, the sand was first washed with 0.5 mol L⁻¹

TABLE I

Elemental analysis of Hamadan quartz sands and Florida palygorskite used

Mineral	MnO	TiO ₂	Na ₂ O	CaO	K ₂ O	Fe ₂ O ₃	MgO	Al ₂ O ₃	SiO ₂
	g kg ⁻¹								
Palygorskite	0.6	4.9	0.6	19.8	8.0	29.8	102.0	104.0	609.0
Sand	–	–	< 1.0	6.1	< 1.0	5.7	1.1	3.6	975.3

HCl and then with distilled water, oven dried at 105 °C, and then used for the pot experiment.

Pot experiment

A pot experiment was carried out under greenhouse conditions. Pots were filled with a 718-g mixture of palygorskite and sand. The weight of palygorskite of each pot was 25 g kg⁻¹. Four seeds of wheat (*Triticum aestivum*) or oat (*Avena sativa*) were planted and grown for a period of 100 d in each pot. A factorial experiment was performed with three replicates per treatment for a total of 30 pots in a completely randomized design. The experimental treatments are given in Table II.

Pots without plants (Treatments 9 and 10) were also used to determine the possible effects of nutrient solution on palygorskite transformation. All the pots were irrigated with either distilled water as needed or weekly with nutrient solution for a period of 100 d. The nutrient solution had the following composition per liter: 5 mL 1 mol L⁻¹ Ca(NO₃)₂, 2 mL 1 mol L⁻¹ MgSO₄·7H₂O, 5 mL 1 mol L⁻¹ KNO₃, 1 L 1 mol L⁻¹ KH₂PO₄, 2 mL Fe-EDTA, and 1 mL trace compounds containing H₃BO₃, MnCl₂·4H₂O, ZnSO₄·7H₂O, CuSO₄·5H₂O, and H₂MoO₄·H₂O. Analytical grade chemicals were used to prepare the Mg-free nutrient solution according to the process described by Stegner (2002) to avoid the introduction of any impurities containing Mg into the rhizosphere.

Laboratory analyses

At the end of the growing period (100 d), the length and diameter of the plant stems were measured. Then, the plants were harvested and their shoot and root biomass were measured separately after oven drying at 75 °C for 24 h. The roots and shoots were weighed and then ashed at 550 °C for 5 h. The ashes were dissolved in 10 mL 1 mol L⁻¹ HNO₃, and the Mg concentration in the solution was measured by titration with EDTA (0.02 mol L⁻¹). The titration was first conducted under conditions where both Mg²⁺ and Ca²⁺ ions reacted

with EDTA and the end point was detected after both were titrated. Then, Mg²⁺ ions were precipitated as Mg(OH)₂ by increasing the pH of the solution. Hence, subsequent titration of the solution measured only the remaining Ca²⁺ ions, which remained soluble even at the increased pH. After that, the Mg²⁺ content was determined by the difference between the titration levels.

The palygorskite particles in the growth media surrounding the roots in each pot were separated from sand by washing with distilled water in a sieve of pore size < 0.05 mm. The clay-sized particles were then separated from the solution using a centrifuge, and equal-sized clay samples (40 mg of dried clays) were used for X-ray diffraction (XRD) analysis in order to obtain a more reliable comparison between peak height intensities for the palygorskite samples that had undergone different treatments. Oriented slides were prepared for both K- and Mg-saturated samples. The Mg-saturated samples were solvated with ethylene glycol while the K-saturated samples were heated at 550 °C. The mineralogy of the clay-sized particles of the rhizosphere, before and after the pot experiment, was determined using XRD (Bruker D8 model, Germany). The CuK_α radiation was generated at 40 kV and 30 mA. The peak height intensity of 001 reflection indicated the abundance of clay minerals.

Statistical analysis

The data from the XRD analysis were subjected to an analysis of variance using the SAS software 9.1 (SAS Institute Inc., USA). Mean values from different treatments were compared through the least significant difference (LSD) test at 5% level of significance using the MSTATC software.

RESULTS AND DISCUSSION

Plant growth

The mean values of the shoot and root dry weights

TABLE II
Treatments of the pot experiment

Code	Growth medium	Crop	Nutrient solution
1	Palygorskite + quartz sand	Wheat	Full nutrient solution
2	Palygorskite + quartz sand	Oat	Full nutrient solution
3	Palygorskite + quartz sand	Wheat	Mg-free nutrient solution
4	Palygorskite + quartz sand	Oat	Mg-free nutrient solution
5	Quartz sand	Wheat	Full nutrient solution
6	Quartz sand	Oat	Full nutrient solution
7	Quartz sand	Wheat	Mg-free nutrient solution
8	Quartz sand	Oat	Mg-free nutrient solution
9	Palygorskite + quartz sand	No	Full nutrient solution
10	Palygorskite+ quartz sand	No	Mg-free nutrient solution

and yields (sum of the shoot and root dry weights) are shown in Fig. 1. There were no significant differences in the dry matter and yield of oat that was supplied with Mg-free nutrient solution (Treatments 2 and 8), while a significant difference ($P < 0.05$) was observed in the case of wheat. The least dry weight of the wheat biomass was obtained from the treatment of the pure quartz media supplied with the Mg-free nutrient solution (Treatment 7). No significant difference ($P < 0.05$) of wheat was observed between the treatment of the palygorskite-containing media supplied with the Mg-free nutrient solution (Treatment 3) and those with the complete nutrient solution (Treatments 1 and 5).

Palygorskite was the sole source of Mg in the pots containing palygorskite and irrigated with the Mg-free nutrient solution. The results of the dry matter weight

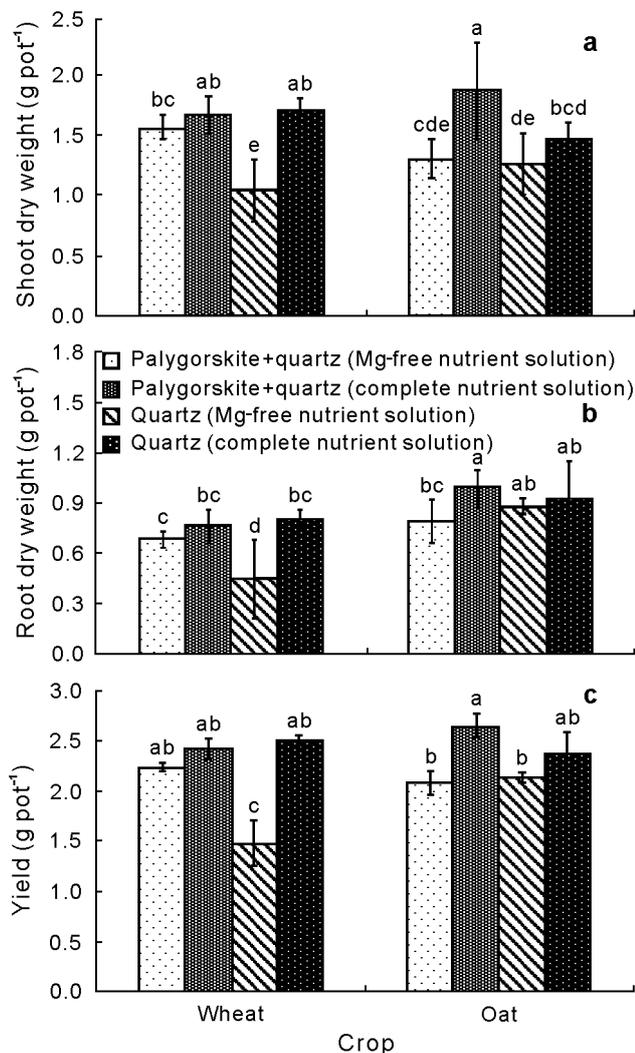


Fig. 1 Mean and standard deviation values of shoot dry weight (a), root dry weight (b), and yield (c) of wheat and oat grown in the media of palygorskite and/or quartz supplied with the Mg-free nutrient or complete nutrient solution. Mean values followed by the same letter(s) are not significantly different ($P < 0.05$).

and yield indicated that the plants supplied with the Mg-free nutrient solution did not receive any Mg from other sources and that the plant roots had an important role in mineral weathering. These findings suggested that palygorskite could provide enough Mg for the growth of wheat. On the other hand, palygorskite as the sole source of Mg was unable to support oat growth.

Fig. 2 compares shoot length and shoot diameter for both crops undergoing different treatments. The lack of Mg in the nutrient solution as well as the absence of palygorskite in the growth media did not seem to be the limiting factors for oat shoot length. On the other hand, palygorskite significantly increased ($P < 0.05$) the diameters of oat and wheat shoots in the pots supplied with Mg-free nutrient solution (Treatments 3 and 4). In other words, lack of Mg in the nutrient solution as well as the absence of palygorskite (Treatments 7 and 8) resulted in a significant decrease ($P < 0.05$) in the shoot diameters of both crops. It seemed that different parts of the plants showed a different response to the Mg deficiency.

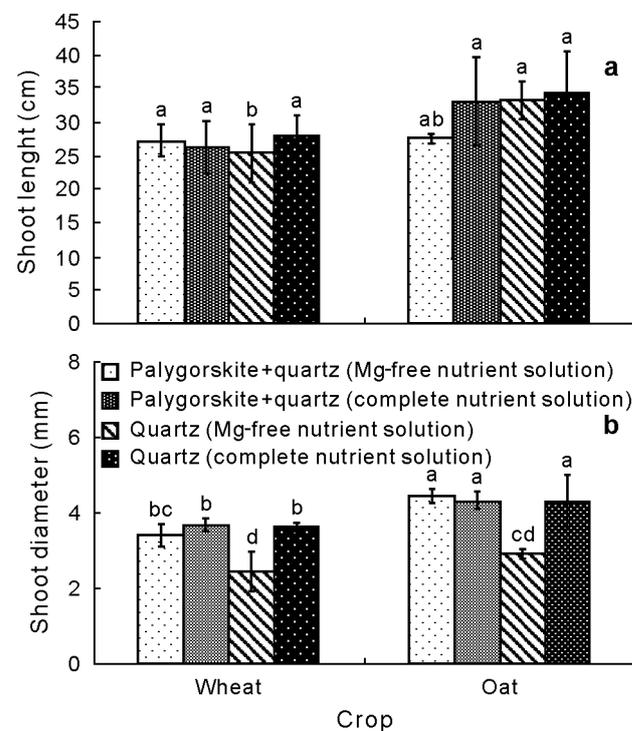


Fig. 2 Mean and standard deviation values of shoot length (a) and shoot diameter (b) of wheat and oat grown in the media of palygorskite and/or quartz supplied with the Mg-free nutrient or complete nutrient solution. Mean values followed by the same letter(s) are not significantly different ($P < 0.05$).

Mg content and Mg uptake

Mean values of Mg content in the shoots and roots

and Mg uptake by the plants are presented in Figs. 3 and 4. These values also showed a trend similar to those of the dry weight and yield. There were no significant differences in the Mg content and the Mg uptake by oat among the growing media supplied with the Mg-free nutrient solution (Treatments 2 and 8) while

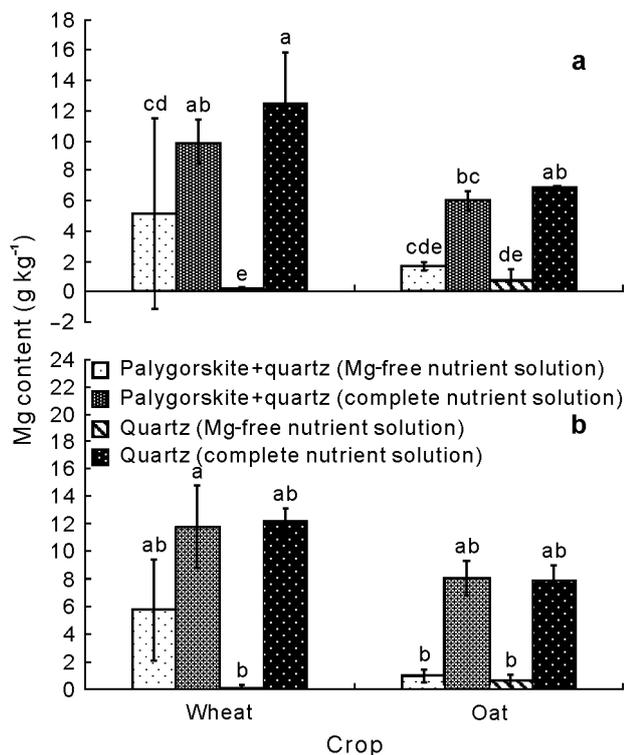


Fig. 3 Mean and standard deviation values of Mg content in shoots (a) and Mg content in roots (b) of wheat and oat grown in the media of palygorskite and/or quartz supplied with the Mg-free nutrient or complete nutrient solution. Mean values followed by the same letter(s) are not significantly different ($P < 0.05$).

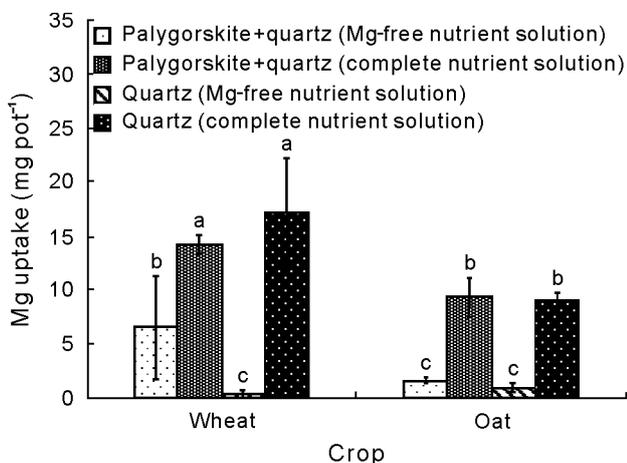


Fig. 4 Mean and standard deviation values of Mg uptake by wheat and oat grown in the media of palygorskite and/or quartz supplied with the Mg-free nutrient or complete nutrient solution. Mean values followed by the same letter are not significantly different ($P < 0.05$).

a significant difference ($P < 0.05$) was observed for wheat.

Mg content and Mg uptake were significantly higher ($P < 0.05$) in both the crops grown in the pots supplied with the complete nutrient solution (Treatments 1, 2, 5, and 6) when compared with those grown in the pots supplied with the Mg-free nutrient solution (Treatments 3, 4, 7, and 8). The lowest Mg content and uptake were observed in the plants grown in pure quartz media supplied with the Mg-free nutrient solution (Treatments 7 and 8). There were no statistically significant differences between the Mg content and Mg uptake by wheat and oat supplied with the complete nutrient solution. The reason was that Mg required by the plants grown in these media was supplied mainly from the nutrient solution.

The mean values of the plant Mg uptake by the end of the experiment showed that the wheat grown in the media supplied with the complete nutrient solution (Treatments 1 and 5) had a significantly greater Mg content ($P < 0.05$) than the oat grown in the same media (Treatments 2 and 6). Besides, wheat had a significantly ($P < 0.05$) higher Mg uptake than oat when both were grown in pots containing palygorskite and supplied with Mg-free nutrient solution (Fig. 4). This suggested that the ability of Mg uptake was higher in wheat than oat. The optimum quantity of Mg required for the vigorous growth of wheat and oat has been calculated by Jones *et al.* (1991) to be 0.16%–1.00% and 0.15%–0.50%, respectively. Thus, Mg uptake by wheat was sufficient for optimum growth, while that by oat was not, with the deficiency symptoms being visible in the old leaves of oat. Khademi and Arocena (2008) reported that Mg uptake by alfalfa from either palygorskite or sepiolite was not sufficient for optimum growth, while that by barley and canola from sepiolite was almost enough to maintain growth. The results obtained by Barré *et al.* (2007b) suggest that 2:1 clay minerals serve as a huge, renewable K reservoir whose theoretical capacity in fertile soils could exceed 3 t ha⁻¹.

XRD diffractograms

X-ray diffractograms obtained for the different treatments are shown in Fig. 5. In all samples, 001 and 002 plane diffraction peaks at 1.05 and 0.64 nm were easily detectable. These two peaks are characteristic of palygorskite. The XRD patterns of the samples from the pots without plants (Fig. 5f, g) were similar to those of the control sample (pure palygorskite before the experiment (Fig. 5a).

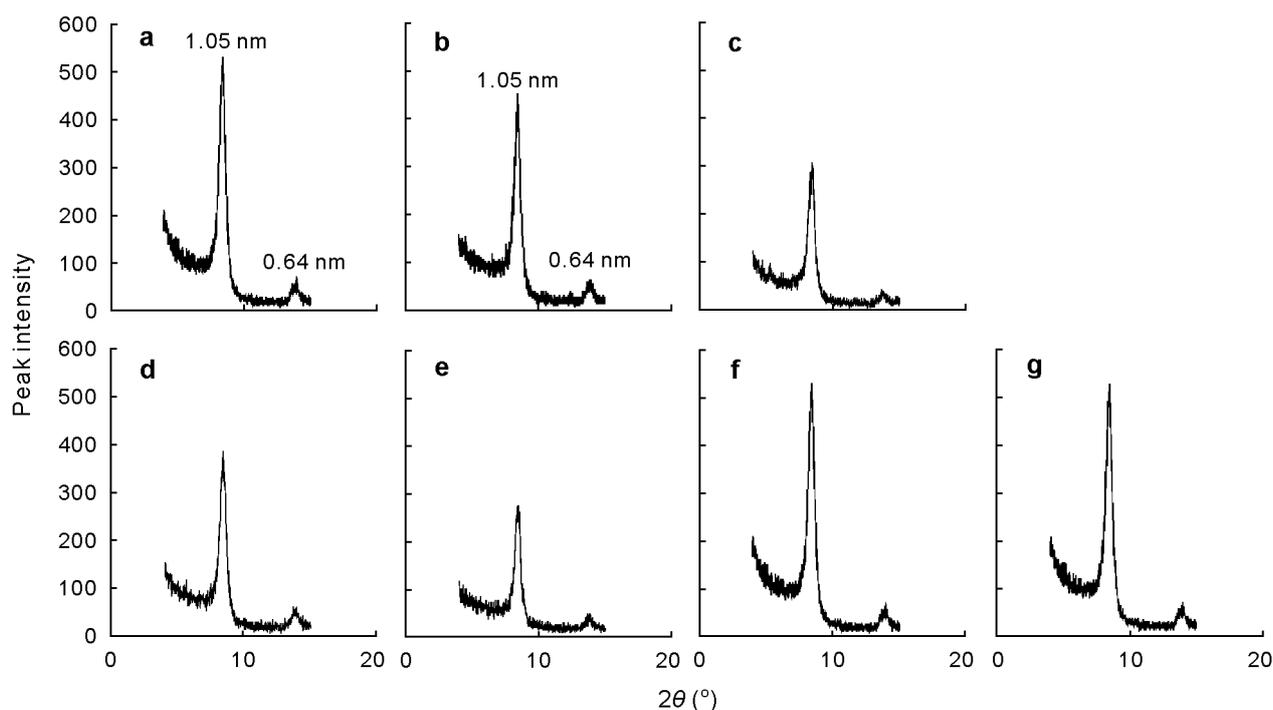


Fig. 5 X-ray diffractograms of clay-sized particles of K-saturated samples of palygorskite before cultivation (a), palygorskite after interaction with wheat rhizosphere supplied with the complete nutrient solution (b), palygorskite after interaction with wheat rhizosphere supplied with the Mg-free nutrient solution (c), palygorskite after interaction with oat rhizosphere supplied with the complete nutrient solution (d), palygorskite after interaction with oat rhizosphere supplied with the Mg-free nutrient solution (e), palygorskite without interaction with plant and supplied with the complete nutrient solution (f), and palygorskite without interaction with plant and supplied with the Mg-free nutrient solution (g) at ambient temperature.

In spite of the Mg uptake by both wheat and oat in the pots supplied with the Mg-free nutrient solution (Treatments 3 and 4) (Fig. 4), no detectable peaks, which could indicate the transformation of palygorskite to other clay minerals, were recognized by XRD. Visual (qualitative) interpretation of diffractograms showed that at the end of the growing period of the plants irrigated with the Mg-free nutrient solution, the 1.05 nm peak (001 reflection of palygorskite) showed a considerable decrease in intensity (Fig. 5c, e) when compared to the peak corresponding to pure palygorskite before cultivation. The decrease in the peak height intensity was greater in the oat rhizosphere soils than in the wheat rhizosphere soils; this is probably due to the greater root weight (Fig. 1b) and root density of oat, as observed in the pots after harvesting (data not shown). The greater root weight and density may have resulted in greater weathering of palygorskite as observed from the physical and chemical changes of the rhizosphere soils. A minor decrease in the peak intensity was observed in the samples from pots containing plants supplied with the complete nutrient solution (Fig. 5b, d).

In the pots supplied with the Mg-free nutrient solution, the plants probably took up most of the Mg

of the soil solution and accelerated Mg release, which caused greater weathering of palygorskite. The plant physiological requirement of Mg causes the level of the Mg, released in the soil solution by high acidity, to decrease continuously, thus accelerating the weathering of palygorskite in the rhizosphere soils. The roots and organic ligands such as humic and fulvic acids serve as chelating agents, which further facilitates the release of structural Mg from palygorskite (Khademi and Arocena, 2008). Therefore, palygorskite weathering in the rhizosphere can be due to: 1) the acidity of the rhizospheric environment arising from root and microorganism activities and 2) the destabilization of fibrous clays as a result of Mg uptake by plants.

As mentioned earlier, in spite of the greater palygorskite weathering by oat, its Mg uptake from the root zone was less than that of wheat (Fig. 4). This can be explained by the lesser Mg requirement of oat than wheat (Jones *et al.*, 1991) and/or the lesser ability of oat to absorb Mg from the soil solution. Deficiency symptoms such as chlorosis in the older leaves of oat (data not shown) confirmed the idea that oat had a lesser ability than wheat in absorbing Mg from solution. It seemed that the different physiology of the studied crops played an important role in the uptake of

Mg from the solution. More investigations are needed to better understand the mechanism of changes induced by plants and microorganisms in palygorskite weathering.

No change could be observed by XRD in the palygorskite growing media without plant cultivation (Treatments 9 and 10) (Fig. 5g). This clearly showed that the peak height intensity decrease in the other treatments (Treatments 1–8) could only be attributed to plant root activity. In other words, the acidity of the nutrient solution (pH = 6) did not affect palygorskite weathering. Khademi and Arocena (2008) reported that the clay fraction of the alfalfa rhizosphere soil with no peat amendment showed no new reflection in the XRD pattern. In their study, pots with peat amendment had much lower pH values than those without peat amendment. This can be attributed to the production of CO₂ and organic acids as a result of decomposition of organic matter by microorganisms (Kelly *et al.*, 1998). Several authors have concluded that the ability of different plants to excrete protons and organic acids through the roots is responsible for the low pH values of rhizosphere soils (*e.g.*, Spyridakis *et al.*, 1967; Leyval and Berthelin, 1991; Courchesne and Gobran, 1997; Rufyikiri *et al.*, 2004). Hinsinger *et al.* (1992) used phlogopite as the sole source of both Mg and K for Italian ryegrass. Their study reported that root-induced weathering caused the release of a significant amount of interlayer K, but not of structural Mg. Hosseinifard (2010) compared the effect of the growing period of plants on the transformation of muscovite in the rhizosphere of wheat and pistachio. His results exhibited that muscovite was transformed to vermiculite in the wheat rhizosphere 150 d after planting the crop, whereas no vermiculitization was observed 100 d after planting. He also showed that pistachio showed no transformation of muscovite 6 months after cultivation of the seedling and that vermiculitization was observed only after one year from cultivation of the seedling. All the above studies demonstrated that the growth period, plant type, and rate of root secretion played crucial roles in the release of Mg from palygorskite structure and palygorskite transformation, or weathering.

CONCLUSIONS

The adequate Mg uptake by wheat grown in palygorskite media supplied with Mg-free nutrient solution suggested that the structural Mg of clay minerals like palygorskite might contribute significantly to the supply of Mg in agricultural crops. The lack of fi-

brous clays, even in the soils of arid regions, could be attributed to a plant-induced process. Different crops had a different ability to take up nutrients from clay mineral weathering. More studies with a focus on the influences of rhizospheric conditions, clay type, and different plant secretions on mineral transformation were suggested to better understand the mechanisms of support provided by the rhizosphere for the growth of crops.

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