



A microcosm experiment to determine the consequences of magnetic microparticles application on water quality and sediment phosphorus pools



A. Funes^{a,b}, A. del Arco^{a,b}, I. Álvarez-Manzaneda^{a,b}, J. de Vicente^c, I. de Vicente^{a,b,*}

^a Departamento de Ecología, Facultad de Ciencias, Universidad de Granada, 18071, Spain

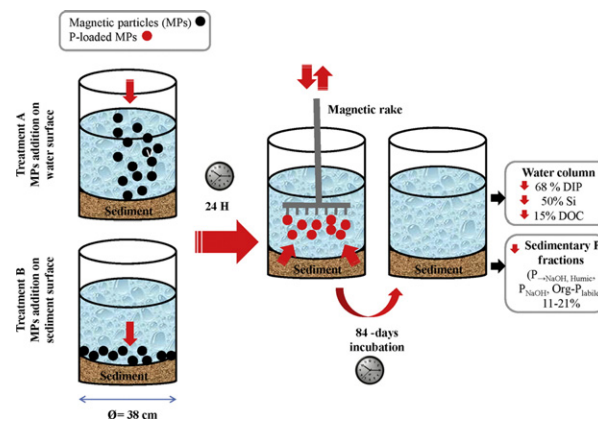
^b Instituto del Agua, Universidad de Granada, 18071, Spain

^c Departamento de Física Aplicada, Facultad de Ciencias, Universidad de Granada, 18071, Spain

HIGHLIGHTS

- Magnetic particles were able to reduce significantly DIP concentration.
- Magnetic particles did not release Fe in anoxic conditions.
- Si and DOC interfered in P adsorption on magnetic particles.
- Nitrogen pools, pH and major cations and anions were not affected.

GRAPHICAL ABSTRACT



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ABSTRACT

This study used microcosms to evaluate the effects of adding iron (Fe) magnetic microparticles (MPs) on water quality, focusing on P concentrations in the water column and sediment. Two treatments were considered for a constant 85:1 MP:P_{Mobile} molar ratio: T-W, applying MPs on the surface water layer; and T-S, applying MPs on the sediment. MP addition reduced P concentrations in lake water and sediment, with both treatments producing a mean reduction of 68 ± 6% in dissolved inorganic P concentration (DIP) over a 70-day oxic period and reductions of 80 ± 8% (T-W) and 80 ± 4% (T-S) over a 5-day anoxic period. MPs also decreased reactive silicate (Si) concentrations by around 50% in both periods, but dissolved organic carbon (DOC) was reduced by only 15% at 24 h after MP addition. Despite the marked decrease in DIP concentration due to MP addition, there was no reduction in chlorophyll *a* (Chla), because post-treatment total P concentrations (>200 µg L⁻¹ vs. >700 µg L⁻¹ before treatments) remained higher than required for changes in the biological community (0.05–0.1 mg L⁻¹). With T-S treatment, there was a reduction of 15% in P bound to Al oxides, clay minerals, and humic substances (P_{-NaOH}) and of 12% in labile organic P (Org-P_{Labile}) versus controls. P bound to humic substances (P_{-NaOH, Humic}) was

* Corresponding author at: Departamento de Ecología, Facultad de Ciencias, Universidad de Granada, 18071, Spain.
E-mail address: ivicente@ugr.es (I. de Vicente).

reduced by 11–22% in both treatments. Finally, T-W rather than T-S treatments are recommended for future whole-lake applications to achieve more effective P removal from water and sediment and a higher percentage MP recovery.

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1. Introduction

The worldwide problem of eutrophication has prompted major investments to improve wastewater treatments and the implementation of measures to reduce the input of phosphorus (P) into aquatic ecosystems (Jeppesen et al. 1999; Gulati and Van Donk 2002; Søndergaard et al. 2005). Internal P loading can have a considerable impact on lake water P concentrations and lake water quality (Phillips et al. 1994), and P inactivation techniques are widely applied to increase sedimentary P binding capacity, including the addition of iron (Fe), aluminum (Al), or Phoslock® (Boers et al. 1992; Egemose et al. 2011; Spears et al. 2013, 2015). However, the effectiveness to remove P of most P-sorbing materials is dependent on the pH, potential redox, and/or presence of other dissolved ions (Westholm 2006; de Vicente et al. 2008; Vohla et al. 2011; Lürling et al. 2014). Thus, the effectiveness of Al and Phoslock® is reduced at the high pH values typically encountered in hypereutrophic aquatic ecosystems (Ross et al. 2008; Reitzel et al. 2013). Moreover, adsorbents that remain within the sediment after P inactivation may eventually be released into the water column under changing physicochemical and biological conditions or through resuspension (Jensen and Andersen 1992; Søndergaard et al. 1992; Rydin and Welch 1998; Egemose et al. 2009).

Various studies recently proposed the application of magnetic particles (MPs) to eliminate P from aqueous solutions, utilizing them to adsorb P and then removing them by applying a magnetic separation gradient, minimizing water quality alterations and allowing reutilization of the MPs in subsequent adsorption processes (de Vicente et al. 2010). The advantages of using MPs for lake restoration include: (i) their effective adsorption of P under both batch and flow conditions (de Vicente et al. 2010; Merino-Martos et al. 2011); (ii) their specific capacity to adsorb considerable amounts of P at pH > 7 despite being negatively charged (Daou et al. 2007; de Vicente et al. 2010); (iii) the non-dependence on redox conditions of their P adsorption (Funes et al. 2016); (iv) the reduction in sedimentary P_{Mobile} concentrations caused by their addition (under both oxic and anoxic conditions), potentially contributing to a long-term reduction in P efflux; (v) their lesser cost in comparison to $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ or Phoslock® if reutilized more than twice, considering the maximum P adsorption capacity and price of each adsorbent (Funes et al. 2016); and (vi) the apparent absence of toxic effects on planktonic and benthonic organisms (Álvarez-Manzaneda et al. 2017). With regard to the use of MPs in whole-lake restoration projects, it should be borne in mind that the effectiveness of P adsorption can be reduced by the presence in natural waters of humic acid (HA), reactive silicate (Si), and other major ions, as demonstrated in complex natural matrices (de Vicente et al. 2011) and single-laboratory experiments (Merino-Martos et al. 2015). Therefore, the addition of MPs can be expected to influence the cycling of other nutrients besides P.

The goal of this study was to assess the effectiveness of MP addition to improve the water quality of a hypertrophic Mediterranean lake, using large microcosms containing lake water and lake sediment. The specific study objectives were to evaluate: (i) the long-term effect of MP addition on lake water P concentrations; (ii) the effect of MP addition on the sedimentary P pool; (iii) long-term effects of MP addition on nutrient cycling (carbon C, nitrogen N, Si), and (iv) the optimal working conditions for maximum MP removal and water quality improvement.

2. Materials and methods

2.1. Sampling site

Honda lake is a shallow (surface area = 9 ha, $Z_{\text{mean}} = 1.3$ m; $Z_{\text{max}} = 3.2$ m), hypertrophic, and brackish water ($6000 \mu\text{S cm}^{-1}$) wetland on the coast of Almeria in Southeast Spain (de Vicente et al. 2003; Funes et al. 2016). The catchment area is largely dominated by intensive agricultural activity, despite designation of the lake as a Natural Reserve (1989) and Ramsar Site (1994). Besides high external P inputs, there is an important internal P loading due to frequent resuspension events and anoxic periods during the summer, when abundant sedimentary P is continuously released into the overlying water (de Vicente et al. 2006).

2.2. Materials

MPs used as P adsorbents (HQ grade, BASF, Germany) were micron-sized soft magnetic spherical particles with negligible coercive field and remanent magnetization (*i.e.*, zero magnetization upon removal of the magnetic field). Further details on these particles have been reported by de Vicente et al. (2010) and Merino-Martos et al. (2011).

A device was designed to remove MPs from the sediment in microcosms (Fig. 1). It comprises a rake (maximum diameter of 20 cm) fitted with a platform (plus handle) containing an array of 89 cylindrical neodymium magnets individually inserted into its base (Fig. 1a). A one-piece hard-plastic shell (1 mm thickness) protected the magnet from direct contact with MPs to facilitate their recovery and cleaning. Fig. 1b depicts a detail of the base and hard-plastic shell, while Fig. 1c displays a surface plot of the magnetic field density on the shell, and Fig. 1d exhibits the magnetic field density profiles in x- and diagonal (45°) directions. Fig. 1c and d show that there was minimal interference between magnets. Comsol Multiphysics software (COMSOL Inc., Burlington, MA) was used to model the size (diameter 4 mm, height 25 mm) and arrangement (cubic array) of the magnets and the separation between them (14 mm), achieving an optimum magnetic flux density gradient of approximately 20 T/m to remove the maximum number of MPs.

2.3. Microcosm experiment

On July 2015, 100 dm³ of surface sediment were collected with an Ekman dredge at the deepest site of the lake. Surface water was also collected near the shore with a peristaltic pump and kept at 14 °C until use. Sediment was thoroughly homogenized in a large tank before being divided among 15 PVC microcosms ($\varnothing = 38$ cm; $h = 58$ cm). Once the microcosms were in the laboratory, 6600 cm³ of surface sediment and 40 L of surface lake water were added to each. The microcosms were randomly distributed in an outdoor roofed area to minimize bias.

After a one-week stabilization period, baseline physicochemical data were gathered from the microcosms (day 0), which were then allocated in a randomized manner to one of three treatments (5 replicates per treatment): (1) MP addition on the surface of the water (T-W); (2) MP addition on the surface of the sediment (T-S); or (3) no MP addition (control). MPs were added to the microcosms in aqueous dispersion (120 g L^{-1} distilled water), using a peristaltic pump with a grid to achieve a homogeneous distribution of the MPs. The MP: P_{Mobile} molar ratio was 85:1, three-fold higher than the ratio selected by de Vicente

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