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# Novel use of ochre from mine water treatment plants to reduce point and diffuse phosphorus pollution

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**Abstract.** Treatment of polluting discharges from abandoned mines is producing large quantities of ochre (mainly iron (III) oxides) for which no major end-use has yet been identified. Newcastle and Edinburgh Universities are currently conducting research to develop and test novel field-scale methods for use of ochre for phosphorus removal from sewage effluent and land drainage. Phosphorus pollution is a serious threat to the water environment in industrialised countries, causing eutrophication, algal blooms, fish kills and loss of water resources. Our prior experiments have demonstrated that ochre is an excellent adsorbent of phosphorus from solution. The ongoing research will build upon this preliminary work to develop valuable uses for a low-value by-product of mine water treatment, with benefits for the mining and water industries and the water environment as a whole. The project will assess the performance of ochre for phosphorus removal in three settings: constructed wetlands for sewage effluent treatment; addition of powdered ochre to polluted standing waters (e.g., sewage treatment tanks, septic tanks) so that phosphorus is stripped out as the ochre settles; and treatment of agricultural drainage with ochre-filled filter and dosing units. We will also examine the fate of spent ochre from these applications, when the material is saturated with phosphorus, to assess its performance and environmental acceptability as a slow-release fertiliser and thereby develop a total use cycle for ochre. The paper presents results that demonstrate the potential of ochre for phosphorus removal and discusses current research into this method of ochre use.

## Introduction

The flooding of abandoned mines frequently results in the formation of acidic, ferruginous water due to the oxidation of pyrite in the mine workings. To prevent the pollution of surface watercourses, mine water treatment plants (MWTPs) are employed to treat the most serious discharges of this polluted water. In MWTPs the oxidation and precipitation of reduced Fe (II) is enhanced either by the addition of chemicals (e.g., oxidising reagents, alkali to raise pH and increase the rate of Fe (II) oxidation, and flocculants to assist floc formation and sedimentation) and/or by atmospheric oxidation in storage ponds or constructed wetlands with long retention times. MWTPs therefore accumulate large quantities of Fe(OH)<sub>3</sub> and FeO.OH precipitate, collectively known as “ochre” (of the order of tens of tonnes per annum at a single site). Typically, this is stockpiled and, although a number of possible end-uses have been considered (e.g., colouring bricks/cement and in synthesising coagulants for drinking water), no single end-use has yet been identified which could consume the projected future production. In this paper a further possible large-scale use for ochre is described: the removal of unwanted phosphorus from sewage effluent and agricultural runoff.

Phosphorus pollution from point and diffuse sources is a serious threat to the water environment in the UK and other industrialised countries (D’Arcy et al. 2000). The transfer of phosphorus to rivers and lakes from sewage treatment works, septic tanks and agricultural runoff causes eutrophication, frequently resulting in algal blooms, fish kills and loss of water resources. The costs to the UK water industry are estimated at > £15M per annum (D’Arcy et al. 2000), but, at the same time, farmers are purchasing fertiliser containing phosphorus to

maintain crop yields from agricultural land. To improve the sustainability of phosphorus within the environment, technologies have been developed since the 1950s to remove and recover phosphorus from wastewaters (Morse et al. 1998).

Ochre has the potential to be used in novel technologies for the removal of phosphorus from wastewater due to its high sorption capacity for phosphorus. The predominant mechanism of phosphorus removal from water by ochre is sorption onto iron and aluminium oxides and hydroxyoxides and calcium carbonate. Phosphorus sorption to goethite (a significant component of ochre) initially involves rapid ligand exchange with surface OH groups at very reactive sites and the formation of a binuclear bridging complex between a phosphate group and two surface Fe atoms (Parfitt 1989). Weaker ligand exchange follows with less reactive sites, and finally over time there is a slower penetration of phosphate into the solid matrix by defect sites and pores (Barrow 1983; Parfitt 1989). The sorption of phosphate by iron oxides depends on the number of reactive surface FeOH groups and the crystallinity of the oxides (Parfitt 1989). The long-term storage of phosphorus by ochre is governed by two factors: (1) the phosphorus retention capacity - the maximum number of sites available for phosphorus sorption, and (2) the phosphorus buffer intensity or strength of adsorption (Reddy et al. 1999). These factors are in turn controlled by the physicochemical properties of the ochre and the prevailing redox conditions. Phosphorus removal from wastewater by ochre may also occur by precipitation, although this is believed to be less significant than removal by sorption. Heal et al. (in press) contains a more detailed discussion of the mechanisms.

Previous work to examine the use of ferruginous materials for phosphorus removal from wastewater has been piecemeal. In the USA, Webster and Wieder (1997) found that the addition of ochre from acid mine drainage to fertilised soils reduced phosphorus concentrations in runoff. In Northern Ireland, Wood and McAtamney (1996) showed that the use of laterite as a substrate in mini constructed wetlands removed 95 % of phosphorus from landfill leachate. The treatment of dairy wastewater has also been investigated in bucket-scale subsurface flow constructed wetlands with an iron ore substrate (Grüneberg and Kern 2000). Other ferruginous media, such as scrap iron, peat treated with bauxite red mud (Roberge et al. 1999) and sand and olivine coated with iron aluminium hydroxyoxides (Ayoub et al. 2001), have been investigated for phosphorus removal from wastewater but few trials have been conducted at the field scale, and attempts to design novel treatment systems are limited. This paper demonstrates the potential of ochre for phosphorus removal and discusses current research into this method for ochre use within three main settings:

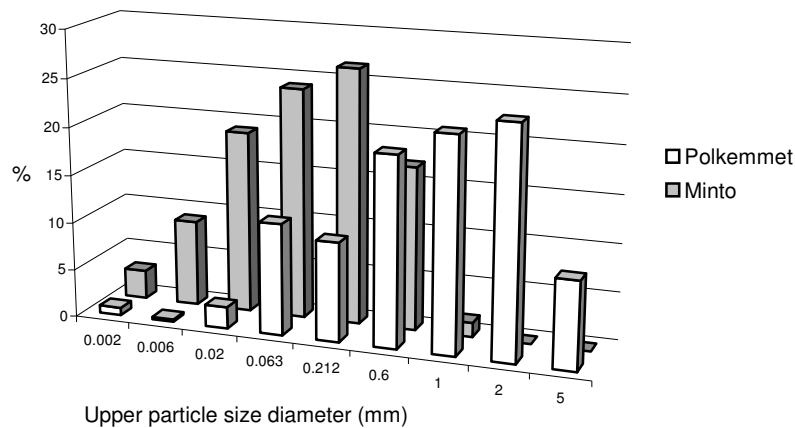
- (1) Removal of phosphorus from sewage effluent with ochre, used either as a substrate in constructed wetlands or in dosing systems for tanks of sewage effluent;
- (2) Removal of phosphorus from agricultural runoff with ochre, either in instream filter units in rivers with elevated phosphorus concentrations, or in constructed wetlands or in dosing systems in drainage ditches;
- (3) Use of ochre saturated with phosphorus from applications (2) and (3) as a slow-release agricultural fertiliser.

## Demonstration of the potential of ochre for phosphorus removal

### Preparation of ochre

Ochre extracted from MWTPs has a very high water content (80-95 %) unless it has been stored in drying beds in good weather conditions. Ochre which has not been air-dried is difficult to handle and transport and consequently most investigations of its phosphorus removal properties have used the air-dried form. Ochres from different MWTPs have similar chemical properties but may have different physical properties. Ochres from two MWTPs in Scotland, Polkemmet and Minto, have a similar chemical composition and mineralogy (identified as a mixture of ferrihydrite and goethite,  $\alpha$ -FeO.OH, by x-ray diffraction) but very different particle size distributions (Fig.1). Polkemmet ochre dries into clods that are readily crushed to a coarse, granular texture which has a high saturated hydraulic conductivity (26-32 m day<sup>-1</sup>, equivalent to coarse sand). In contrast, Minto ochre dries to a fine powder with a considerably lower saturated hydraulic conductivity (0.7-1.7 m day<sup>-1</sup>). The cause of the different physical properties of the two ochres is unclear but is thought to be related to differences in the operation of the MWTPs. At Polkemmet hydrogen peroxide and a polymer are added to the mine water to encourage oxidation and flocculation of iron, whilst at Minto the mine water is unamended.

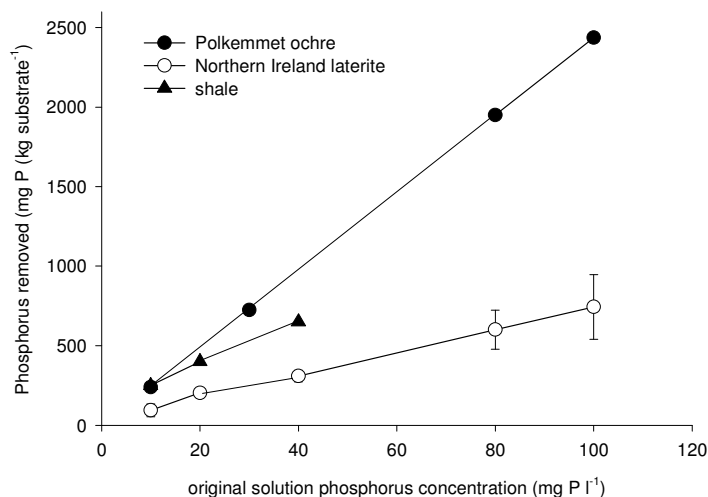
Different physical properties of ochres influence the rate and quantity of phosphorus removal and their suitability for different phosphorus removal applications. Coarse-grained ochres, such as Polkemmet, are more suitable for phosphorus removal in filter units or in the substrate of constructed wetlands. Fine-grained ochres, such as Minto, are difficult to contain and rapidly clog filter units, but rapidly remove phosphorus from wastewater because of the larger surface area of the ochre particles. Such ochres are more suitable for dosing applications to waters with undesirable phosphorus concentrations as long as adequate sedimentation is provided.



**Fig.1.** Particle size distribution of air-dried ochres from two MWTPs, Scotland (after Bozika 2001).

### Capacity of ochre for phosphorus adsorption

Laboratory batch experiments, in which 10 g of air-dried ochre were shaken for 24 hours with 200 ml of artificial phosphorus-enriched water of different concentrations, were used to measure the phosphorus retention characteristics of ochre. The results showed that ochre has a high removal capacity for phosphorus, increasing linearly with solution phosphorus concentration, compared with other wetland substrates (Fig.2). Independent tests by Lamont-Black et al. (2001) also demonstrated that ochre is capable of decreasing very high concentrations of phosphorus (in comparison to those commonly found in polluted waters) to very low residual concentrations. Neither the method of ochre preparation (air-dried versus oven-dried at 105°C), nor the temperature of the experiment (6°C versus 28°C), nor the solution pH affected phosphorus removal (McHaffie et al. 2000; Bozika 2001).



**Fig.2.** Phosphorus removal by three substrates in batch experiments (after McHaffie 2000). Points are the mean of triplicate samples  $\pm$  standard error. Where error bars are not visible they are smaller than the symbols.

The maximum phosphorus adsorption capacities for Polkemmet and Minto ochres have been determined to be 26 and 30.5 mg g<sup>-1</sup>, respectively, in laboratory saturation experiments involving the repeated addition of artificial phosphorus solutions (Bozika 2001). These values are orders of magnitude higher than those measured in other

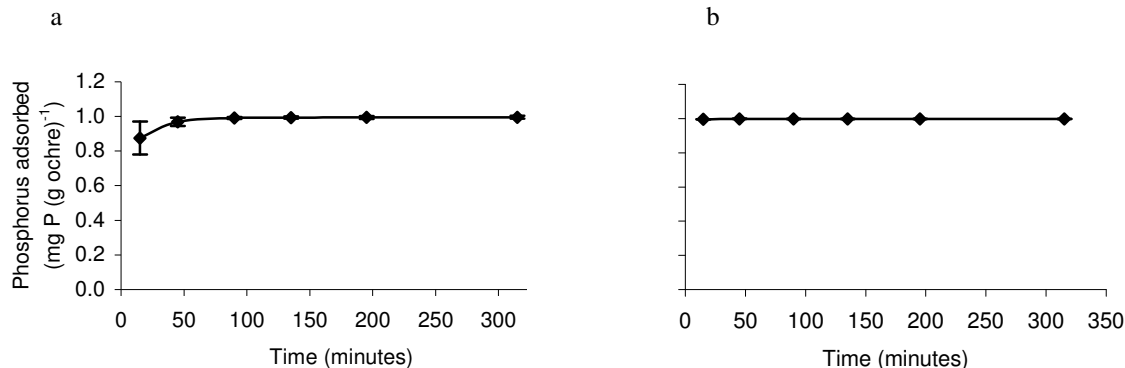
wetland substrates (Table 1), suggesting that ochre has the potential to vastly improve the performance of constructed wetlands for phosphorus removal.

**Table 1.** Maximum phosphorus adsorption capacities of different wetland substrates (after Drizo 1998 and Mann 1997).

Substrate	Adsorption capacity ( $\text{mg P (g substrate)}^{-1}$ )
Gravel	0.03-0.05
Bottom ash	0.06
Steel slag	0.38
Blast furnace slag	0.40-0.45
Fly ash	0.62
Shale	0.75
Laterite	0.75
Zeolite	1
Polkemmet ochre	26
Minto ochre	30.5

### Kinetics of phosphorus removal by ochre

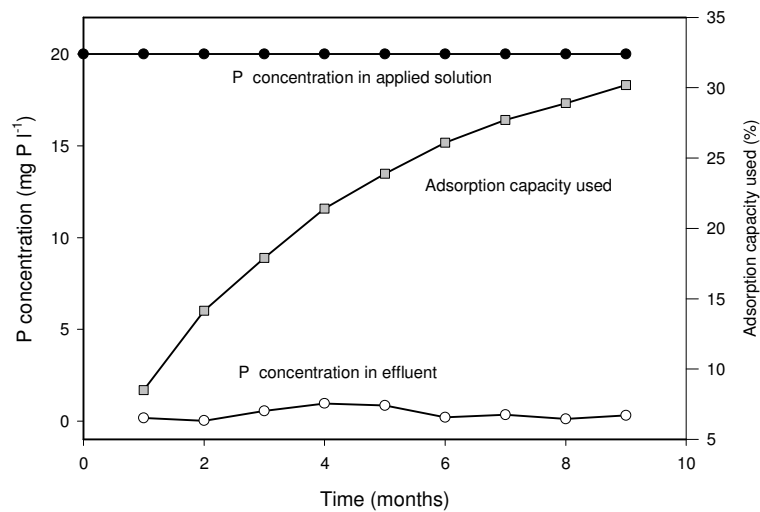
Batch experiments in which samples were removed at different time intervals during a 24-hour period show that the removal of phosphorus by Polkemmet and Minto ochres is rapid (Fig.3). For the Polkemmet ochre almost all phosphorus is adsorbed rapidly within the first hour, after which the solution reaches an equilibrium state. The rate of phosphorus adsorption by Minto ochre is higher (within minutes) due to the larger available surface area for adsorption, resulting from its fine-grained texture. Experiments in which artificial phosphorus solutions were added to ochre in beakers demonstrated that phosphorus removal by Polkemmet ochre is still rapid (phosphorus concentrations decreased from 5 to  $< 0.01 \text{ mg P l}^{-1}$  within eight minutes), even in non-agitated mixtures (McHaffie et al. 2000). These results are supported by independent kinetic tests by Lamont-Black et al. (2001) which also showed that the reaction rate is very rapid, with  $> 98 \%$  phosphorus removal in five minutes of contact time with ochre.



**Fig.3.** Phosphorus uptake from artificial solutions containing  $50 \text{ mg P l}^{-1}$  in batch experiments by (a) Polkemmet and (b) Minto ochres (Bozika 2001). Points are the mean of triplicate samples  $\pm$  standard error. Where error bars are not visible they are smaller than the symbols.

### Long-term phosphorus removal by ochre

Long-term phosphorus removal by ochre was examined by pumping artificial phosphorus solution continuously for nine months onto a gently-angled trough packed with Polkemmet ochre to simulate a horizontal flow filtration system (McHaffie et al. 2000, 2001). Long-term phosphorus removal was consistently high (Fig.4), with the concentration of applied phosphorus dropping from  $20 \text{ mg P l}^{-1}$  to  $< 1 \text{ mg P l}^{-1}$ . Less than one-third of the ochre in the trough was saturated with phosphorus after nine months indicating that it is a suitable medium for long-term phosphorus removal in filter systems. Most of the phosphorus removed by the ochre is unlikely to be readily remobilised since only  $0.4 \%$  of the phosphorus removed was found to be in a plant-available form.



**Fig.4.** Phosphorus removal and % adsorption capacity used in Polkemmet ochre after nine months operation of trough experiment (after McHaffie et al. 2001).

## Current research into the use of ochre for phosphorus removal

Laboratory investigations of phosphorus removal by ochre demonstrate that it is highly effective in rapidly removing phosphorus from wastewaters. A number of potential applications exist for using ochre to treat phosphorus-rich waters, including sewage effluent, agricultural runoff and eutrophic watercourses. We are currently undertaking further basic research to provide the scientific basis for using ochre for phosphorus removal from wastewater in addition to conducting field-scale trials of the process.

### Resolution of engineering issues and preparation of ochre

Recent experiences have shown that a major obstacle to the use of ochre for phosphorus-removal purposes is the difficulty of handling the material. Ochres, particularly the Minto material, are often very weak compared with most engineering soils and therefore are vulnerable to erosion by running water and wind disturbance and have poor-loading capacities. If a more granular form can be prepared at reasonable cost, without compromising its phosphorus removal properties, widespread applications become possible. Preliminary laboratory results show that pea-sized spherical ochre granules can be formed by adding ordinary Portland cement, a surfactant and water. The granules produced are quite porous and seem reasonably robust when subjected to wet-dry and freeze-thaw susceptibility tests and end-over-end mixing in water. Phosphorus is removed when the granules are added to artificial phosphorus solutions. The amount of water added to ochre to achieve granulation is critical. Too much water prevents granulation but, if there is not enough present to satisfy the hydration reactions in the cement, then strength and durability are lost after air-drying for about seven days. Further testing is required to establish the durability and phosphorus sorption ability of these granules in the longer term (e.g., permeability, compaction characteristics, erosion potential, batch experiments).

### Influence of phosphorus form on the efficiency of removal by ochre

The form in which phosphorus occurs in wastewaters may affect its removal by ochre. Most investigations of phosphorus removal by ochre have used artificial solutions, containing soluble inorganic phosphorus, but it is necessary to examine whether phosphorus removal is as effective in raw wastewaters (where some of the phosphorus may be in organic form) before full-scale field testing of the treatment process.

## Treatment of sewage effluent with ochre

With stricter controls on sewage discharge into receiving waters (e.g., the EU Urban Wastewater Treatment Directive (Farmer 2001)), the development of new methods for phosphorus removal are required. Constructed wetlands are increasingly used for tertiary treatment of effluent in sewage treatment works and also for sewage treatment in rural communities where it is uneconomic to provide full-scale chemical and biological treatment works. Although constructed wetlands are effective in removing nitrogen from sewage effluent they are less efficient in removing phosphorus (Cooper et al. 1996). The incorporation of ochre, which has been demonstrated to have a high capacity for phosphorus removal, into the substrate of constructed wetlands is therefore anticipated to improve their treatment efficiency for phosphorus.

The Polkemmet ochre is particularly suitable for such an application as it has a relatively high saturated hydraulic conductivity. Realistically, a constructed wetland might absorb only half its maximum phosphorus capacity before flow rates declined significantly, but this would still give a long operational life time compared to other substrates (Table 2). Field trials of ochre as a substrate in constructed wetlands treating sewage effluent are currently being conducted. In these trials water quality (including heavy metal concentrations) and longevity of phosphorus sorption are being assessed, in addition to the phosphorus removal efficiency and hydraulic performance of the ochre.

**Table 2.** Estimated operational lifetimes for constructed wetlands using 3 or 5 m<sup>2</sup> wetland area per person with shale or Polkemmet ochre as a substrate with three concentrations of sewage effluent, 20, 5 or 3 mg P l<sup>-1</sup> (McHaffie et al. 2001).

Substrate	Operational lifetime / years					
	3 m <sup>2</sup> per person <sup>a</sup>			5 m <sup>2</sup> per person <sup>a</sup>		
	20 mg P l <sup>-1</sup>	5 mg P l <sup>-1</sup>	3 mg P l <sup>-1</sup>	20 mg P l <sup>-1</sup>	5 mg P l <sup>-1</sup>	3 mg P l <sup>-1</sup>
Shale <sup>b</sup>	1.6	6.5	10.7	2.7	10.7	17.8
Ochre <sup>c</sup>	18.8	75	125	33.5	134	224

Constructed wetland substrate assumed to be 0.6 m deep. <sup>a</sup>Each person assumed to produce 0.2 m<sup>3</sup> sewage a day. <sup>b</sup>From data in Drizo (1998). <sup>c</sup>Calculated with half the measured maximum phosphorus adsorption capacity to take account of the anticipated decline in flow rates.

The Minto ochre is less suitable for use as a filter material for removal of phosphorus from sewage effluent because of its lower saturated hydraulic conductivity. However, its fine-grained texture gives it a high capacity for phosphorus adsorption, and it has a potential for sewage effluent treatment in a system in which the effluent is dosed with the ochre and mixed in holding tanks. For example, it is estimated that a septic tank of 7 m<sup>3</sup> capacity serving a household of five people would require the addition of 5.1 kg of Minto ochre per week to remove the phosphorus (McHaffie et al. 2001).

## Treatment of agricultural runoff with ochre

Approximately 40 % of agricultural land in the UK (excluding rough grazing) is underlain by subsurface field drains which have been shown to be a major conduit of soluble and particulate phosphorus export to watercourses in storm events, even when best management practices are implemented (Dils and Heathwaite 1996). Although it is not feasible to install wastewater treatment works or constructed wetlands for every field drain, treatment with ochre may form a cheap, low maintenance means of reducing phosphorus exports from field drains. Treatment at the field scale could take the form of filter cartridges containing coarse-grained ochre. Alternatively, dosing with powdered ochre, followed by settlement, has already been demonstrated to be effective in reducing phosphorus concentrations in simulated agricultural runoff in laboratory scale flume experiments (Sweetman 2001). In a short-term pilot study in a river affected by agricultural runoff, instream filter units, containing the coarse-grained Polkemmet ochre, reduced phosphorus concentrations and altered the algal community composition, from one dominated by algae typical of eutrophic conditions to one dominated by algae typical of mesotrophic conditions (Bush 2001). Longer-term field investigations, which include a complete assessment of the environmental benefits and impacts, especially with regard to aquatic ecology, are necessary to develop further this potential application for ochre use.

## Recycling of phosphorus-saturated ochre as a slow-release fertiliser

When the phosphorus removal capacity of ochre in a constructed wetland or filter unit is finally exhausted, the 'spent' ochre will require removal and disposal. A more sustainable alternative to disposal to landfill is the use of phosphorus-saturated ochre as a slow-release fertiliser. A pilot pot experiment, in which barley was grown in soils amended with ochre containing different amounts of sorbed phosphorus, has already demonstrated that some of this phosphorus is available for uptake to plants (Smith 2001). Pot experiments are currently being conducted at Edinburgh to compare the performance of barley and grass grown in soils amended with ochre with that of plants grown with conventional inorganic fertiliser. The trials will be extended to include field plot experiments in 2003. In all these experiments, heavy metal concentrations in soil, leachate and vegetation and the soil pH and nutrient status will be assessed to ensure that there are no adverse environmental and agricultural consequences of recycling sorbed phosphorus by using the saturated ochre as an agricultural fertiliser.

## Conclusions

Laboratory experiments and small-scale trials have demonstrated that ochre, formed as a by-product from mine water treatment, has a high capacity for phosphorus removal from wastewater, as a result of sorption to the high concentration of iron oxides and hydroxides contained within the ochre. Potential applications of phosphorus removal by ochre are widespread, including sewage effluent treatment in constructed wetlands and by dosing systems, and treatment of agricultural runoff by instream filter units and/or dosing and settlement systems. Furthermore, phosphorus sorbed to ochre has the potential to be recycled as a slow-release fertiliser. Full-scale implementation of phosphorus removal from wastewater by ochre requires field trials to assess the performance and costs of the treatment process. The use of ochre for phosphorus removal from wastewater is particularly attractive not only because treatment costs are potentially lower than more traditional methods, but also due to the sustainability advantage of using a by-product of treating one form of water pollution (i.e., mine water pollution) to address another water problem (eutrophication).

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