

Rock Filters for Enhanced **Phosphorus Removal**

Duncan Mara¹

Michelle Johnson²

M. Alonso Camargo-Valero³

November, 2007





 ¹ Professor, School of Civil Engineering, University of Leeds, UK
 ² Research Assistant, School of Civil Engineering, University of Leeds, UK
 ³ Assistant Professor, Environmental Engineering Section, National University of Colombia, Colombia

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Introduction

Phosphorus pollution in rivers can be attributed to three major sources: industry, agriculture and wastewater treatment works. The use of phosphates in food processing, detergents and agriculture has resulted in the depletion of this essential plant nutrient, yet its presence in industrial wastes and its persistence in wastewater treatment have led to increased concentrations in rivers and therefore widespread concern over its environmental impact. The Environment Agency (2007) reports that in 2006, 50 percent of rivers in England & Wales had high concentrations of phosphate (>0.1 mg/l). The presence of phosphates in river waters causes a rapid growth of aquatic plants such as algae. This process is known as 'eutrophication' and has resulted in fish kills and changes in ecosystem function and diversity. Eutrophication has been a serious environmental concern in the developed world over the last 30 years or so and is now of global concern.

The UK water industry is a major consumer of electrical energy. Energy consumption for wastewater treatment has risen from 437 kWh per megalitre (MI) of wastewater treated in 1998/99 to 634 kWh per MI in 2005/06, with a high of 814 kWh per MI in 2002/03 (Water This increase has been predominantly as a result of UK, 2003, 2004 & 2006). increasingly stringent discharge consents, especially for nutrients (nitrogen and phosphorus), set by the Environment Agency. The EU Urban Waste Water Treatment Directive (91/271/EEC) and the Water Framework Directive (2000/60/EC) require the removal of 75-80% of all phosphorus in municipal wastewaters from large treatment plants discharged into 'sensitive' waters which are eutrophic or which in the near future may become eutrophic if protective action is not taken. Stricter effluent standards for nutrients require more advanced wastewater treatment processes. This will be of particular concern at small domestic wastewater treatment plants which are expected to be required over the next few years to remove both nitrogen and phosphorus. At large treatment plants modified activated sludge processes (generically termed "biological nutrient removal") can be used but they are expensive (especially in terms of energy consumption) and they also require skilled operators because the process is complicated. Similarly, chemical dosing (e.g., with lime, aluminium sulphate, ferric chloride) entails complex removal mechanisms which result in increased sludge production. Simple and inexpensive P removal systems for small wastewater treatment plants have yet to be developed (Keplinger et al., 2004).

Constructed wetlands (CW) have been used in the UK since the mid-1980's for the removal of water pollutants, including nutrients. Plant uptake and adsorption and precipitation reactions have been investigated. Plants take up nitrates and phosphates for growth, yet some researchers have found that plant uptake is negligible considering that the nutrients required for growth are often released back into the water body following biomass death and mineralization of the plant organic matter (Tanner, 2001). In the UK the plant growing season is often limited to just 3 or 4 months (May–August), which implies that plant uptake is ineffective for most of the year.

The main mechanisms of P removal in CW appear to be precipitation (for example, as apatite [Ca5(PO4)3(F, Cl, OH)]) and adsorption on to the bed medium and subsequent crystallization (Brix *et al.*, 2001; Molle *et al.*, 2003). Considerable efforts have been made to identify and evaluate suitable P-adsorbing media. Calcite, crushed marble, crushed waste concrete, sea-shell sand and blast furnace slag (BFS) have all been investigated (Arias *et al.*, 2003; Brix *et al.*, 2001; Arias and Brix, 2005; Korkusuz *et al.*, 2005; Kostura *et al.*, 2005; Molle *et al.*, 2003; Søvik and Kløve, 2005). BFS appears to be a particularly good P-adsorbent (Korkusuz *et al.*, 2005; Kostura *et al.*, 2005). Many of these researchers have attempted sorption equilibrium isotherm experiments, followed by

column experiments fed with phosphorus-spiked water or wastewater. A balance between inlet and outlet P concentrations is made and used to determine the capacity of the medium to retain phosphorus. However, it has often been found that, when experiments are carried out at pilot-scale in the field, the results are disappointing due to hydraulic and/or operational problems, with the latter including biofilm development which reduces the availability of P-binding sites.

In this report, phosphorus removal from wastewater was evaluated by pilot-scale tertiary horizontal-flow filters which were tested under laboratory and field conditions in order to determine its performance at small domestic wastewater treatment plants. In the laboratory bench-scale filters were used to estimate the P-sorption capacity of tested media BFS and in the field the performance of pilot-scale filters was monitored for P removal from the effluent of an existing Waste Stabilisation Pond (WSP) System located at Esholt, Bradford.

1. Media Selection

A range of phosphorus adsorbent media were identified as potential media for the filters based on published results in the literature (Wollastonite, filtralite, limestone and BFS). Filtralite proved to be very difficult to acquire, while Wollastonite is only available in the UK in powder form. BFS and limestone were readily available, and their mechanical characteristics are suitable for being used as a filter media; therefore, these two materials were selected for carrying out phosphorus removal experiments. Limestone was acquired from B&Q and BFS was kindly provided by Tarmac Quarry Products Ltd (Scunthorpe, UK).

In order to estimate any disadvantage related to metal leaching from limestone and BFS, each of these two sorbent materials was exposed to a volume of aerated rock filter effluent for a period of six weeks before analysing the liquid for metal concentrations using Inductively coupled plasma atomic emission spectroscopy (ICP-AES). The analyses were carried out in duplicate and the results are shown in Table 1.

	Calcium**	Aluminium	Manganese	Magnesium	Silicon	Titanium	Iron	Potassium
Aer Eff	29.7	0.017	0.042	5.0	3.0	0.015	0.037	7.4
Aer Eff	29.3	0.016	0.041	4.9	3.0	0.016	0.037	7.5
BFS	167.0	0.010	1.430	8.6	34.7	0.215	0.096	39.7
BFS	166.0	0.011	1.490	8.6	33.3	0.216	0.094	40.2
LS	56.4	0.011	0.097	5.0	2.2	0.091	0.036	5.4
LS	56.3	0.010	0.095	4.8	2.2	0.086	0.036	5.5

Table 1 ICP-AES results for limestone and BFS leaching test after 6 weeks*

* Based on a 400-ml aerated rock filter effluent sample containing 276 g of media. **Concentrations in mg/l.

Further to the results reported above, a sample of each of the two sorbent materials was also exposed to a volume of aerated filter effluent for a period of 9 months (equivalent to the period of exposure proposed in the field) before analysing the solution using ICP-AES; the results are shown in Table 2. The results showed that of the constituents which did leach into the liquid, none were regulated by the Environmental Agency. Furthermore, given that BFS is used in construction and passes the Toxicity Characteristic Leachate Procedure (TCLP) tests and also that it has been researched extensively for wastewater treatment, it was assumed that its use would not be of any significant environmental concern.

	Calcium**	Aluminium	Manganese	Magnesium	Silicon	Titanium	Iron	Potassium
BFS	386.0	0.000	0.063	9.3	30.9	0.058	0.054	93.2
LS	84.7	0.006	0.005	9.8	7.6	0.004	0.011	10.4

Table 2 ICP-AES results for limestone and BFS leaching test after 9 months*

* Based on a 400-ml aerated rock filter effluent sample containing 276 g of media. **Concentrations in mg/l.

2. Analytical Determination of Phosphorus in Wastewater Samples

Orthophosphate (reactive phosphorus) and total phosphate (organic and inorganic) concentrations were initially determined using HACH colorimetric methods 8114 and 8190, respectively. These methods were appropriate for the anticipated phosphorus concentrations in the effluents of the filters. HACH chemicals were used for the analyses and a HACH DR-890 colorimeter. However, the reactive phosphorus concentration exceeded the total phosphorus concentration on a number of occasions, possibly due to the low phosphorus concentrations measured. To check accuracy, the ascorbic acid method for the determination of orthophosphate (method 4500-P E; APHA, 1998) was used for duplicate tests, run in parallel with the HACH method. The ascorbic acid method proved to be more accurate and more reliable and therefore, the procedure was developed and adopted for the determination of total and reactive phosphorus from wastewater samples with low phosphorus concentrations (< 3 mg P/L).

2.1. Total phosphorus determination

Wastewater samples contain both organic and inorganic phosphorus species; therefore samples should be previously prepared before using the ascorbic acid method, in order to transform all phosphorus species into orthophosphate. Total phosphorus determination included a digestion under strong oxidant conditions with either ammonium persulfate or potassium persulfate at 150 °C for 2 hours by using a closed reflux system. This method was validated with phosphorus standard solutions (2.5, 3.5, 5.0, 7.5, and 10.0 mg PO₄³⁻/L) and the results from seven replicates were statistically processed to generate a calibration chart (Figure 1). For this method, uncertainty and detection levels were \pm 0.04 mg PO₄³⁻/L and 0.34 mg PO₄³⁻/L, respectively.

2.2. Reactive phosphorus determination

Reactive phosphorus was determined from original wastewater samples after filtration by the ascorbic acid method which was validated under our laboratory conductions. Phosphorus standard solutions (2.5, 3.5, 5.0, 7.5, and 10.0 mg PO₄³⁻/L) were analysed by seven replicates and the results were statistically processed to generate a calibration chart (Figure 2). Corresponding figures for uncertainty and detection levels were \pm 0.04 mg PO₄⁻³/L and 0.40 mg PO₄³⁻/L, respectively.



Figure 1 Total Phosphorus calibration chart for ascorbic acid method prior sample digestion



Figure 2 Reactive phosphorus calibration chart for ascorbic acid method

3. Phosphorus Sorption Capacity of Limestone and Blast Furnace Slag

Phosphorus adsorption capacity was estimated in 14-litre bench-scale horizontal-flow filters containing limestone and BFS (25 mm average size each) under laboratory conditions at 20°C (see, Figure 3). The filters were fed continuously for eight days, until equilibrium conditions were attained, with P-spiked water by pumping from a 25-litre feeding tank with a peristaltic pump (model 101UR, Watson Marlow Bredel Inc., Wilmington, USA) at a hydraulic loading of 1.8 m³/m³ d which corresponded to a retention time (θ) of 0.25 d. The filter effluent was returned to the feeding tank and recycled during

the experiment. P-spiked water solutions were made in 20 litres of tap water supplemented with KH_2PO_4 to give initial P concentrations (C_o) of 5, 10 and 100 g P/L for BFS filters and 2.5, 5.0 and 50 g P/L for limestone (LS) filters, to reach the maximum P-adsorption capacity as demonstrated by Sakadevan and Bavor (1998). The phosphorus concentration was determined daily in samples collected from the filter effluents using the previously validated ascorbic acid analytical method. The average equilibrium concentrations (C_e) from duplicate experiments are given in Table 2.



Figure 3 Experiment set-up for P-adsorption capacity in the laboratory

The results from P-adsorption capacity experiments are shown in Table 3. BFS showed a higher phosphorous adsorption capacity and lower C_e values than LS. BFS showed a good affinity for inorganic phosphorus species (PO₄³⁻) as adsorption capacity values reached up to 30.2 g P/kg BFS. However, typical P concentrations in domestic wastewaters are lower than 20 mg P/L (Tchobanoglous and Burton, 1991) and it is expected that the adsorption capacity would also be lower under typical operational conditions. In order to reach a low final effluent P-concentration (e.g., <2 mg P/L), additional experiments were carried out with BFS as it showed a better performance in these adsorption experiments.

Medium	Initial P concentration C _o (g P/L)	Final P concentration C _e (mg P/L)	Mass of Medium (kg)	P-adsorption capacity (mg P/kg)				
BFS	100	177	23.1	30,200				
	10	104	22.0	3,100				
	5	94	23.8	1,400				
LS	50	325	26.1	13,300				
	5	163	26.5	1,300				
	2.5	124	26.5	600				

Table 3 Results from P-sorption capacity experiments with BFS and LS

Results at low C_o values with BFS (Table 4) reveal a much more realistic sorption capacity for P removal by adsorption on this material which was considerably lower than previous results. This suggests that the sorption process of P species from the bulk of the liquid phase to active points on BFS surface depends on P availability in the liquid. Hence, the surface provided for micropores located in the internal structure of the BFS media could not be reached by P species when tested under low driving force ($C_o - C_e$) conditions.

C _o , mg P/L	C _e , mg P/L	<i>q</i> , mg P/kg BFS	P removal, %
19.5	4.0	18	79.5
11.2	2.8	10	75.0
5.1	1.2	5	76.4

 Table 4 P-sorption capacity of BFS under low P-concentrations

The Freundlich model is one of the most widely used expressions for sorption process under equilibrium conditions; it represents the relationship between q (mg P/kg BFS) and C_e (mg P/L) as an exponential equation.

$$\log_{10} q = (1/n)\log_{10} C_e + \log_{10} K_f$$
(1)

In Equation 1, the Freundlich model is transformed into a linear equation where Kf and (1/n) are empirical constants which may be used to compare the P-sorption performances of either different sorption media being tested under the same conditions or one sorption medium tested under different conditions.

Sorption isotherms based on the results from the experiments with high and low C_o values followed Freundlich model (Figure 3); corresponding K_f and (1/n) values were 0.25 and 4.66 for high P concentrations and 3.89 and 1.04 for experiment with low P concentrations, respectively. The isotherms suggest that BFS would perform better if the P concentration in the influent to a BFS filter was grams of P per litre but this condition is far from the real-life scenario for domestic wastewater treatment. However, these results show that BFS has the potential to produce a final effluent which meets P discharge consents as the aim of P removal from domestic wastewaters is obtain a treated effluent with a low P concentration (e.g., <2 mg P/L).



Figure 3 P-sorption isotherms at 20°C based on the Freundlich model for BFS

4. Pilot-scale Waste Stabilisation Pond System

4.1 Existing WSP system

The University of Leeds pilot-scale Waste Stabilisation Pond (WSP) experimental facility is located at Yorkshire Water's Esholt Wastewater Treatment Works in Bradford, West Yorkshire, UK. It comprises primary treatment by three primary facultative ponds (PFP) in parallel, each receiving screened wastewater (50% industrial wastewater, 50% domestic wastewater) and secondary treatment in either two maturation ponds (M1 and M2) and a reed-bed channel (RBC) in series, or three horizontal-flow rock filters in parallel including one working as a control filter (CRF), one as a subsurface horizontal-flow constructed wetland (CW), and one as aerated rock filter (ARF), Figure 4.



Figure 4 Pilot-scale reactors: the effluents from the primary facultative ponds (top) are 'polished' either in maturations ponds and a reedbed channel (left) or in aerated and unaerated rock filters and in a subsurface horizontal-flow constructed wetland (right).

4.2 Pilot-scale facilities for phosphorus removal

A simple, cost-effective P removal filter was designed to remove P from our WSP system at Esholt. The wastewater treatment train for this particular project included a primary facultative pond (PFP1), an ARF and two new horizontal-flow filters in parallel. The PFP1 was loaded at 80 kg BOD/ha d with an average nominal retention time (θ) of 30 days within the experimental timeframe reported herein. The PFP1 effluent was pumped into the base of the ARF using a peristaltic pump (Watson Marlow, model 504S) at a hydraulic loading rate of 0.6 m³/m³ d (θ = 0.8 d). The ARF filter (4.0 × 0.5 × 0.5 m) was constructed aboveground, lined with a low-density polyethylene liner and filled with 40–100 mm limestone aggregate; it was aerated one-third of the way along its length, using an oil-free compressor (Jun-Air, Nørresundby, Denmark) at an air flow rate of 20 L/min.

Two 50-litre tanks filled with BFS and limestone aggregate for phosphorus removal were placed on site and fed with ARF effluent. A known volume of aerated filter effluent was pumped to each P removal filters in parallel using a Watson Marlow model 504S

peristaltic pumps fitted with a Watson Marlow model 501RL pumpheads and 8.0-mm internal diameter Marprene tubing. Table 5 shows the flows applied to each filter.

Media	Flow (ml/min)	Flow (m³/day)	Effective* volume (I)	Retention time (hours)
BFS	60	0.09	21.8	6
LS	43	0.06	15.7	6

Table 5 Flows, volume and retention time in each filter

*Effective volume is the available volume for wastewater treatment (i.e., the total volume minus that occupied by the medium).

A 30-litre Nalgene bottle was used to 'spike' each filter with a P solution (KH_2PO_4), in order to increase P influent concentration up to 10 mg P/L (Figure 5). That due to the effluent from the aerated rock filter often contained less than 2 mg P/I (presumably due to the excellent P removal in the waste stabilisation pond due to algal assimilation and subsequent removal of the algae in the aerated rock filter). Initially, the filters were drip-fed using a calibrated dripping rate. However, after various problems caused by changing flow rates as the head pressure changed plus problems with freezing during the winter, it was decided to use a capillary pump and tubing to supply a pumped flow of the spike to each filter. This system was put in place in March 2007 and was monitored weekly until mid-June 2007.



(a) P-removal filter

(b) P-spiking system

Figure 5 P-removal filters on site

5. Overall Performance of Pilot-scale Limestone and BFS Filters

A monitoring programme was carried out weekly until mid-June, in order to determine the performance of P-removal filters under on-site conditions. Grab samples of influents and effluents were taken for analysis of suspended solids (SS), biochemical oxygen demand (BOD), total Kjeldhal nitrogen (TKN), ammonium-N, chlorophyll-a and alkalinity. Grab samples were also taken from each of the sampling points and stored in acid-washed glass bottles for monthly phosphate analysis using ion chromatography (IC-ED; DX500, Dionex Cop., Sunnyvale, USA). The influent flow rate to each filter was measured using a measuring cylinder and stopwatch.

Analysis of the data from the P removal filters revealed that removal of SS, TKN, ammonium-N, BOD, chlorophyll-a and alkalinity were negligible since each of these parameters were removed in the aerated rock filter. Many of these concentrations were

below Environment Agency consent requirements and around the minimum detection limits so it was decided that they should only be measured on a monthly basis from March 2007 onwards. Figure 6 shows the results from these analyses.



Figure 6 Filter influent and effluent SS, BOD, ammonia-N, TKN, chlorophyll-a and alkalinity concentrations.

There was no real difference in removal performance in either filter for any of the parameters measured though the concentrations of all of the parameters were very low, often around the limit of detection. Towards the end of the sampling period, the concentration of all of the parameters increased in the effluents of each filter, but also in the influent. This shows how the removal performance was generally dependent on the removal performance in the preceding ARF, and also proves the inability of the BFS and limestone filters to buffer fluctuating influent quality due to a short retention time ($\theta = 6$ hours).

Considering that the performance of a wastewater treatment unit depends mostly on adherence to hydraulic design and a phenomenon such as short-circuiting deeply affects the facility's overall effectiveness and efficiency. A tracer experiment was conducted in the BFS filter for hydraulic characterization using Rhodamine WT. Tracer concentrations in the BFS filter effluent were measured in-situ, every 5 minutes for one day before spiking and for 30 afterwards, with a Rhodamine WT fluorometric sensor (model YSI 6130, YSI Inc., Yellow Springs, USA) coupled to a multiparameter sonde (YSI 6820; YSI Inc.) with continuous data-logging system.

The Rhodamine WT results were corrected for background content based on results from readings recorded before tracer injection (negative values were taken as zero as they included only Rhodamine WT). Tracer responses in the BFS filter effluent were plotted against normalised time (t/ θ), as shown in Figure 7.



Figure 7 Tracer response curve for the Rhodamine WT spikes in BFS filter

Data from tracer experiments were also processed following the method described by Levenspiel (1999) for dispersion number (δ) and actual retention time; the dead-space and short-circuiting indices were calculated by the method given by Kilani and Ogunronbi (1984). The hydraulic characteristics of the BFS filter are summarized in Table 6. Although the dead-space index indicates that 22 percent of the effective volume in BFS filter was not being used (and therefore the actual retention time was lower than the nominal retention time), the dispersion number of 2.9 (and 3 is very close to infinity in dispersion-number terms, infinity being the value of the dispersion number in a completely stirred tank reactor – CSTR) shows that the BFS filter was behaving essentially as a CSTR with some by-pass flow and dead space.

Mean nominal retention time, h	Retention time, h	Dispersion number	Dead-space index	Short-circuiting index	Hydraulic regime
6.0	4.7	2.9	0.78	0.50	Completely mixed

6. Phosphorus Removal by Pilot-scale Limestone and BFS Filters

The effluent total phosphorus concentrations from each of the two filters are showed in Figure 8; results from IC-ED readings (Dionex) are also reported. The Dionex results prove the accuracy of the lab-based analysis method for phosphorus determination since both data sets are comparable. The total P results show that phosphorus was successfully removed in each filter until February 2007, but this removal declined over time. Removal performance in the BFS filter exceeded than in the limestone (LS) filter but after approximately four months of operation, both filters were not removing phosphorus at the same rate that they had done at the start of the experiment.



Figure 8 BFS (top) and limestone (bottom) filter effluent phosphorus concentrations.

Tertiary horizontal-flow BFS filters were also tested under laboratory conditions. The laboratory-scale filter was similar to that described previously (see Section 3) but it was fed continuously with fresh (i.e., un-recycled) P-spiked water (10 mg P/L); samples were collected from the filter effluent and processed for P content as detailed above. Results from the continuous-flow pilot-scale filters at Esholt (Figure 9) show that the BFS filter performed better (<2 mg P/L) for longer than the laboratory-scale filters, even though the

latter received the same hydraulic and P loadings (1.8 m^3/m^3 d and 18 g P/m³ d, respectively). This may mean that the environment provided by the ARF effluent could have improved the P-sorption capacity of BFS by introducing changes linked to pH, organic matter and dissolved oxygen. For instance, results from the leaching test carried showed that Fe is leached from BFS (0.08 mg Fe/kg BFS in 6 weeks) and this could have contributed to improved P removal.



Figure 9 Comparison of effluent phosphorus concentrations from laboratory and on-site BFS filters

Surface coating samples from the original BFS medium (Sample A), saturated medium after laboratory experiments (Sample B), saturated medium after on-site experiments (Sample C), and media fully saturated with a 2M H_3PO_4 solution (Sample D) were processed for X-ray diffraction (XRD) with a X-ray diffractometer equipped with a copper tube (Cu K α radiation and a graphite monocromator (model PW1050-Cu K α , Royal Philips Electronics N.V., Amsterdam, The Netherlands).

Observations with a scanning electron microscope (SEM) (Stereoscan model 360, Cambridge Instruments Ltd, Cambridge, UK) were also conducted on the secondary electron mode at an acceleration voltage of 14 KV to collect the images and these were then analysed by the image analysis technique.

XRD scans (Figure 10) from samples B and C did not showed any major change in the mineralogy of the surface coating on the BFS (sample A) which was mostly akermanite $(Ca_2MgSi_2O_7)$, thus indicating that sorbed P species were amorphous.

The presence of gypsum (CaSO₄•2H₂O) in sample D, confirmed by XRD, suggests that amorphous phosphorus forms are linked to Ca/S/O-rich phases as reported by Pratt *et al.* (2007). SEM images (Figure 11) confirmed the presence of amorphous phosphorus forms on BFS after P removal.



Figure 10 XRD scans $(\theta - 2 \theta)$ from surface coating of BFS samples: original material (A), material from laboratory experiments (B), material from on-site experiments(C) and fully saturated BFS (D).



Figure 11 SEM images from the surface coating of BFS samples: original material (sample A), saturated material from laboratory experiments (sample B), saturated material from on-site experiments (sample C) and fully saturated BFS (sample D).

Conclusions

BFS filters reported a better performance for P removal than limestone filters as BFS material has a higher P-adsorption capacity. Isotherms for P-sorption showed that BFS has the potential to produce a final effluent which meets P discharge consents (e.g., <2 mg P/L). BFS and LS filter make any additional contribution for the removal of SS, TKN, ammonium-N, BOD, chlorophyll-a or alkalinity, which can be attributed to short retention times. The sorption capacity of BFS was higher in the field pilot-scale filter than in the laboratory bench-scale filter, possibly due to changes introduced in the aerated rock filter linked to pH and organic matter and dissolved oxygen concentrations. BFS filters are thus an appropriate low-cost technology for upgrading small wastewater treatment systems to remove phosphorus.

Deliverables

Paper to be presented at the II International Congress 'SmallWat07' Wastewater Treatment in Small Communities, Seville, 11–15 November 2007:

Camargo Valero, M. A., Johnson, M. L., Mather, T. and Mara, D. D., Enhanced phosphorus removal in a waste stabilisation pond system with blast furnace slag (BFS) filters.

Acknowledgements

We are extremely grateful to all our funder, The BOC Foundation, and especially to Yorkshire Water who also generously provided the site and give us almost day-to-day operational support in one way or another at Esholt; and also to Mr. Saranagon Hemavibool for scanning samples for SEM images.

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