

**Dissolved and
particulate nutrient
transport dynamics**

S. T. Harrington and
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Dissolved and particulate nutrient transport dynamics of a small Irish catchment: the River Owenabue

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being the most commonly measured nutrient parameters when monitoring water quality (Schindler, 2006). Agricultural nutrient loss has been estimated to account for almost 50 % of river pollution in Ireland (McGarrigle and Clenaghan, 2004) and is of significant concern.

5 The South Western River Basin District (SWRBD) is classified as being the most pristine river basin district in Ireland, with 92 % of surveyed rivers classed as unpolluted (McGarrigle et al., 2010). Geographically, fertilizer application is higher in southern parts of Ireland than in the rest of the country where the national average usage of fertiliser applied to grazed grassland has been estimated at 65, 3 and 9 kg ha⁻¹ for N, P and potassium respectively. The higher use of fertilisers in the south reflects the more intensive farming and stocking rates (Lalor et al., 2010). High application rates of fertilizers in southern parts of Ireland gives rise to greater risk of non-point pollution to the surface and one of the main pressures to water quality in the River Owenabue, for example, has been identified as being from diffuse agricultural sources, forestry and from domestic wastewater treatment plants leaching to groundwater (Anon, 2010).

15 Scanlon et al. (2004) showed that P loading in the agricultural Dripsey catchment in County Cork were entirely due to increases in particulate P (PP), which were associated to the sediment. Withers et al. (2001) showed that high P levels found in sediment in run-off from agricultural land in the UK was caused by large quantities of artificial fertilisers being applied to land over recent decades.

20 Neill (1989) showed that nitrogen and in particular nitrate in the River Burren in County Carlow, Ireland follows a sine like curve whereby concentrations peak in the winter months of January and February and are lowest in the summer months of July and August. This pattern is consistent with the results of a study in the UK on an agricultural catchment in Devon (Webb and Walling, 1985) and can be explained by water moving through the soil and less nutrient take up during the winter by plant life in addition to plant die-back adding nitrogen to the watercourse which all contribute to higher N levels during the winter.

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The focus today in many nutrient load studies has been to study the filtered (dissolved) part of the water sample. Usually, samples are passed through 0.45 μm filter papers (Drewry et al., 2008; Scanlon et al., 2004) as particles below this size are assumed to be dissolved although larger filter sizes such as 1.2 μm are also used (Quilbé et al., 2006; Salvia-Castellví et al., 2005). Filter papers with a 1.2 μm retention size are practical for use where a relationship or comparison with suspended sediment concentration is investigated because a 1.2 μm pore size is used in most suspended sediment filtration methods. Method 2540 D of the APHA, a commonly used suspended sediment concentration (SSC) testing procedure, defines material passing 2 μm as dissolved. There appears to have been little work presented on maintaining analytical consistency by using the same sized filters or by ensuring only soluble forms are included in the analysis. The use of 1.2 μm filters has the effect of increasing the percentage of N or P included in the dissolved fraction of the water sample, and correspondingly lowers the particulate fractions compared to studies where finer 0.45 μm filter papers are used.

Traditionally, the method of difference has been used to determine the sediment-associated chemical levels of water samples. Firstly, the chemical parameter of the unfiltered whole raw water sample is tested and this is subsequently compared to the filtered sample, with the difference attributed to sediment association. This approach often produces an incorrect result where the filtered sample contains higher levels of the measured chemical than the original unfiltered sample. Apart from sample contamination, analytical error is the only other source of error. The errors are introduced when reporting levels that are on or near the report limit of the equipment or using techniques involving digestion that do not completely release all the available chemicals for analysis (Horowitz, 2008). In fact, the digestion procedures of most chemical and trace element analysis methods are not intended nor designed to completely solubilise sediment associated constituents (Fishman and Friedman, 1989; Eaton et al., 2005). Furthermore, the extent to which the sediment in a sample can be dissolved depends on the matrix of the sample. For example, carbon based sediments may be

determined from filtered samples by Cadmium Reduction Method 8171/Photometric. PN was calculated as the difference between TN and TDN. As an accuracy check, standard samples were included in the analysis of all chemical constituents.

3.2 Load calculation

- 5 The nutrient load from a catchment over a period of time can be estimated by integrating, over time, the product of river discharge and nutrient concentration as per Eq. (1).

$$L_c = \int_{t_1}^{t_2} Q_t C_t dt \quad (1)$$

10 where L_c is the load over a time period ($t_2 - t_1$), Q_t is the discharge at time t , C_t is the nutrient concentration at time t , and dt is the time interval ($t_2 - t_1$). C is measured in mgL^{-1} and Q is measured in $\text{m}^3 \text{s}^{-1}$ yielding L_c in g for the selected time period.

15 Where there are omissions or gaps in the record of nutrient concentrations, a means of estimating the unknown concentration is required. Where such situations arise in suspended sediment load analysis, the SSC is often estimated from the discharge using a suspended sediment rating curve between discharge and suspended sediment.

A similar approach can often be adopted for some P parameters, but N is not always well explained by either discharge or suspended sediment concentration (Jarvie et al., 1998) so alternative methodologies must be adopted. Linear interpolation for nitrate load calculation has been found to be the best performing method by Kronvang and Bruhn (1996), Moatar and Maybeck (2005) and Zamyadi et al. (2007). Beale's ratio estimator has been shown to perform well for medium to large sized watersheds (Cohn, 1995), however, it appears to be unverified at smaller scales (Zamyadi et al., 2007). Smart et al. (1999) found that a model which estimates nitrate concentrations from a sine curve gave the best results for the River Don in Aberdeen, Scotland, where
25 concentrations of nitrate ranged between 1 to 6 mgL^{-1} over 11 yr of data and displayed a strong seasonal pattern.

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flooding was reported throughout the SWRBD during this period. The lowest discharge was observed in October 2010 at $0.28 \text{ m}^3 \text{ s}^{-1}$. The River Owenabue exhibits a flashy hydrograph with suspended sediment concentrations and sediment associated nutrient concentrations responding quickly to high flow events.

4.2 Concentrations

The largest TN concentrations occur during the winter months of November, December and January (Fig. 3a). Concentrations were lowest during the July to September period. The annual mean TN concentrations for 2010 and 2011 are 4.7 and 4.6 mgL^{-1} respectively.

The range in TDN was large with concentrations varying between 0.5 and 8.3 mgL^{-1} . The variation in PN was larger with concentrations ranging from 0 to 14 mgL^{-1} . TDN dominates the TN measurement, with the mean TDN content of the samples being 76% . The variation of TDN content was high with a range between 20 and 100% of the TN measurement. Correspondingly, PN constituted 24% of the TN concentrations of the samples, with a range from 0 to 84% .

TP concentrations are generally higher in winter although the winter of 2011 had notably lower concentrations (Fig. 3b). Annual mean TP concentrations were 0.1 mgL^{-1} in both 2010 and 2011. TP is dominated by TDP, with TDP concentrations ranging from 0.01 to 0.19 mgL^{-1} and having an average concentration of 0.06 mgL^{-1} . PP was found to vary from 0.0 to 1.28 mgL^{-1} indicating PP to be much more variable than dissolved phosphorus. The range of TDP within TP samples varied between 11 and 100% with 61% of all samples being dominated by TDP. The range of PP in the TP sample varied between 0 and 89% with an average of 39% .

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4.3 Nutrient relationships and yields

Table 1 shows the result of a correlation analysis between the measured nutrient parameters. Both linear and power based regressions were investigated with the largest r^2 values reported.

All regressions were found to be statistically significant (p values < 0.001). SSC, TP, TRP, and PP were all found to have r^2 values (coefficient of determination) greater than 0.5 when regressed linearly against in-situ turbidity ($Turb_i$). In particular, TP and PP were very well correlated with turbidity. Based on the correlation between P parameters and turbidity, all P parameters were extrapolated from their respective regression curves with turbidity. This approach was taken as using a mixed approach for TDP, which was the only P parameter with an r^2 value lower than 0.5 would have introduced a conflict with the TP/PP/TDP ratios. Nitrogen parameters were calculated using the method in Eq. (2).

Yields of nitrogen and phosphorus passing the gauging station are presented in Table 2 for the years 2010, 2011 and also for the entire monitoring period. The mean TN yield is $4004 \text{ kg km}^{-2} \text{ yr}^{-1}$ for the full monitoring period with a mean PN yield of $982 \text{ kg km}^{-2} \text{ yr}^{-1}$, 25 % of the TN yield. Mean TP yield is $92.6 \text{ kg km}^{-2} \text{ yr}^{-1}$ with a mean PP yield of $48.7 \text{ kg km}^{-2} \text{ yr}^{-1}$, 53 % of the TP yield. These yield values are highly influenced by the major storm event of 2009 as illustrated in Table 2 where the mean yields for the full years of 2010 and 2011 are much lower. The mean contribution of PN and PP is estimated to be 5.66 and 0.28 % respectively of the annualised suspended sediment load which is $17\,331 \text{ kg km}^{-2} \text{ yr}^{-1}$.

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by sandstones and mudstones with background TRP levels of 0.02 mgL^{-1} . The mean TRP value on the Owenabue River for the study period is 0.05 mgL^{-1} suggesting it to be on the threshold of being polluted. In addition, the mean concentrations of SRP on the River Owenabue (0.03 mgL^{-1}) were much higher than those found in the unpolluted River Dee, Scotland (Stutter et al., 2007) which is of particular concern because SRP is the most immediately bioavailable form of phosphorus to aquatic organisms.

N:P stoichiometry is widely used to define P or N limitation for plankton growth, which leads to eutrophication. A molecular N:P ratio greater than 16 (Redfield Ratio) indicates P to be the limiting nutrient, while N:P ratios lower than 16 indicate N to be the limiting nutrient. The mean N:P ratio of the Owenabue River for the entire study period was 43, indicating that plankton growth and thus eutrophication in the Owenabue River is controlled by P inputs, rather than N inputs. This is important in the context of the WFD where rivers must achieve good status by 2015 and the Owenabue River has been identified as being at risk of not meeting this target based on both point source pollution from wastewater treatment plants and diffuse sources such as septic tanks which have a high P content.

5.2 Storm event nutrient dynamics

The dynamics between PN and TDN vary considerably at the intra event scale. Increases in TP and TN during a typical storm event of January 2010 were found to be primarily driven by increases in PP and PN. The profile of both N and P is shown in Fig. 4 for the event. Maximum peak flow recorded during the event was approximately $13.7 \text{ m}^3 \text{ s}^{-1}$, compared to a discharge of $3.1 \text{ m}^3 \text{ s}^{-1}$ at the beginning of the event. Initially there is a sharp decrease in TDN (Fig. 4a) which is partially offset by an increase in PN concentrations which are sufficiently large initially to reverse the overall downward trend in TN concentration. When the initial flush of PN passes, the TN concentration continues to decline. The decline in TN indicates that the nitrogen in the river is being

diluted by increased flows, which has also been reported by others (Salvia-Castellví et al., 2005).

A similar, but stronger trend is found for TP during the same storm event (Fig. 4b). A significant increase in PP results in a significant increase in TP concentrations. The increase in PP is due to the mobilisation of suspended sediment during the initial period of the storm, whereas the sustained slightly elevated TDP concentrations most likely arise from the leaching of P from soils through sub-surface flow. The TP loads, and in particular the PP load is therefore mainly determined by infrequent storm based events, and particularly on the rising limb of the hydrograph of such events. The high correlation of PP with turbidity as presented in Table 1 is further evidence of the important relationship between PP and suspended sediment transport.

5.3 Nutrient loads and yields

Seasonal loads are presented Fig. 5. Winter loads of TN vary from 96 to 311 t for the three winters monitored. TP loads vary from 3.2 to 8.4 t. During the winter, PN and PP contribute 27 and 55 % respectively to the total N and total P load while during the summer PN and PP contribute 19 and 45 % respectively to the total N and total P loads.

Both P and N loads are primarily transported in the winter months with loads decreasing through spring, summer and autumn (Fig. 5). In the winter, TN loads increase primarily due to an increase in TDN, while TP increases due to elevated TDP and PP loads. The increase in the particulate fractions can be explained by soil erosion and catchment run-off which enters the river during winter floods. This finding is consistent with other studies (Lu et al., 2011) and is supported by the high proportion of the suspended sediment load delivered during high flow events on the River Owenabue (Harrington and Harrington, 2012).

The mean annual yield of TN ($4004 \text{ kg km}^{-2} \text{ yr}^{-1}$) is larger than a reported value of $2545 \text{ kg km}^{-2} \text{ yr}^{-1}$ for the 155 km^2 Burren catchment in Ireland (Neill, 1989) but the 2010 and 2011 TN yields, which do not include the major storm event of 2009 compare

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well to the Burren Catchment. The TN yield is approximately double that of the 73 km² Cadière River in France where TN yields were 2217 kg km⁻² yr⁻¹ (Gouze et al., 2008).

The mean annual TP yield (92.6 kg km⁻² yr⁻¹) is slightly higher than that for the similarly sized River Cherwell catchment in England where the TP yield was 76.4 kg km⁻² yr⁻¹ (May et al., 2001) and also corresponds with a reported range of 54–82 kg km⁻² yr⁻¹ found for similar sized catchments in Italy by Marchetti and Verna (1992). A similar range of values was found in Sweden (10–90 kg km⁻² yr⁻¹) where the higher values were attributed to heavier soils types and a higher proportion of annual crops and associated ploughing (Kyllmar et al., 2006).

6 Conclusions

The results show that discharge and SSC influence the concentration of nitrogen and phosphorus. Nutrient concentrations were found to be highly variable. Significant variations were observed at the within event and seasonal scale. At the seasonal scale most of the SSC, nitrogen and phosphorus are transported in the winter months when run-off rates are generally higher. At the within event scale, the proportions of particulate and dissolved nutrients vary due to changes in the source of supply. Typically, particulate fractions initially dominate before becoming exhausted, and dissolved forms become more dominant.

Turbidity was found to be a suitable surrogate for phosphorus parameters and in particular TP and PP but nitrogen parameters were not well correlated with either turbidity or discharge, as is generally found in the literature. Estimated TN and TP yields were found to be at the higher end of scale in terms of UK and European catchments, but this was significantly influenced by the large storm event of 2009.

The particulate portion of N and P was found, on average, to contribute 24 and 39 % of TN and TP respectively for the samples collected. Overall, the yield of PN and PP was estimated to be 25 and 53 % respectively of the TN and TP yields. Phosphorus

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inputs, in particular from diffuse agricultural sources are a significant potential risk given that they are highly dependent on run-off processes, unlike point sources.

TN concentrations were diluted during storm events, despite an initial increase in particulate concentration signifying that its transport mechanism does not respond quickly to river discharge or overland flow. Conversely, TP was found to respond quickly to river discharge and overland flow where both the dissolved and particulate fractions of phosphorus increase during storm events.

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Table 1. Correlation matrix of r^2 values from regression for the measured variables on the River Owenabue.

	Q	Turb _i	SSC	TN	TDN	TIN	DIN	TP	TDP	TRP	SRP	PP	PN
Q													
Turb _i	<u>0.568</u>												
SSC	<i>0.355</i>	<u>0.860</u>											
TN	0.089	<i>0.021</i>	<i>0.038</i>										
TDN	0.057	0.008	<i>0.009</i>	<u>0.529</u>									
TIN	<i>0.044</i>	0.087	0.080	<i>0.461</i>	0.325								
DIN	<i>0.056</i>	<i>0.058</i>	<i>0.057</i>	<u>0.748</u>	<i>0.441</i>	<u>0.748</u>							
TP	<i>0.379</i>	<u>0.804</u>	<u>0.878</u>	<i>0.036</i>	<i>0.003</i>	<i>0.071</i>	<i>0.057</i>						
TDP	<i>0.368</i>	<i>0.363</i>	<i>0.306</i>	<i>0.020</i>	0.001	<i>0.042</i>	0.190	0.464					
TRP	<i>0.487</i>	<u>0.557</u>	<u>0.520</u>	<i>0.011</i>	0.017	0.159	<i>0.124</i>	<u>0.593</u>	<u>0.541</u>				
SRP	<i>0.603</i>	<u>0.527</u>	<i>0.414</i>	<i>0.006</i>	<i>0.001</i>	<i>0.108</i>	<i>0.090</i>	<u>0.502</u>	<u>0.637</u>	<u>0.749</u>			
PP	<i>0.389</i>	<i>0.778</i>	<u>0.886</u>	<i>0.037</i>	<i>0.007</i>	<i>0.043</i>	<i>0.041</i>	<u>0.967</u>	<u>0.277</u>	<u>0.495</u>	<i>0.381</i>		
PN	<i>0.160</i>	<i>0.018</i>	<i>0.028</i>	<u>0.607</u>	<i>0.003</i>	<i>0.210</i>	<i>0.008</i>	<i>0.032</i>	<i>0.037</i>	<i>0.022</i>	<i>0.011</i>	<i>0.026</i>	

Note: Red/italic font indicates linear regressions, black plain font indicates power based regression and underlined values are those where the regression equation explains greater than 50% of the variation.

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Table 2. Annual nutrient yields ($\text{kg km}^{-2} \text{yr}^{-1}$) for the River Owenabue for the full years of 2010 and 2011 and for the entire monitoring period (16 September 2009 to 9 February 2012).

	TN	TDN	TIN	DIN	PN	TP	TDP	TRP	SRP	PP
2010	3353	2853	3330	2791	500	79.0	31.2	28.1	18.4	43.0
2011	3141	1857	3123	2219	1284	59.8	36.6	24.0	15.4	27.1
Entire monitoring period (Annualised)	4004	3022	3662	2878	982	92.6	51.9	33.7	22.9	48.7

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**Dissolved and
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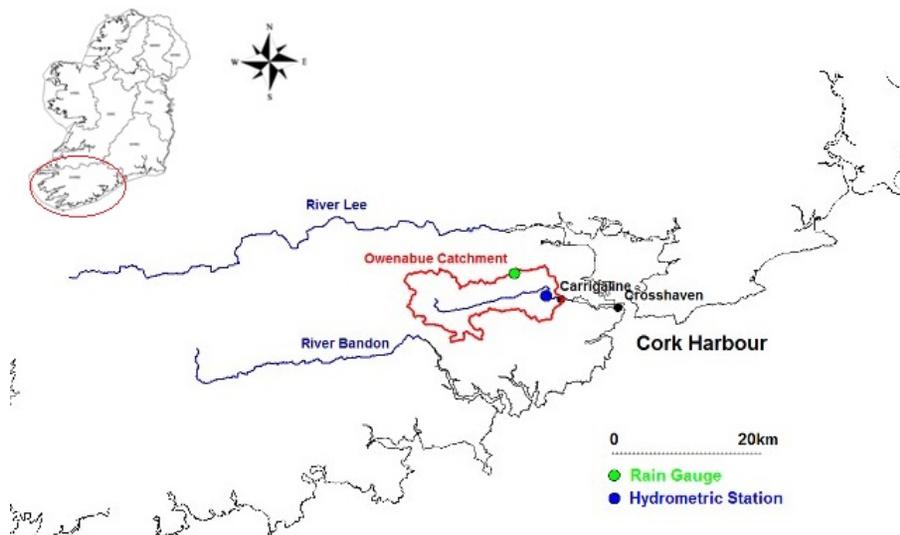


Fig. 1. Location of the River Owenabue catchment and gauging station.

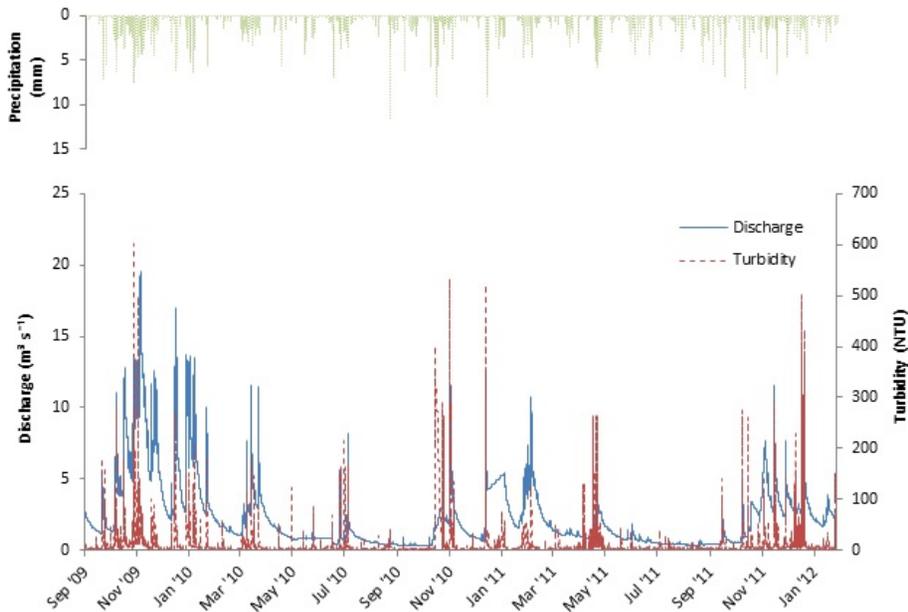


Fig. 2. Precipitation, discharge and turbidity values during the monitoring period.

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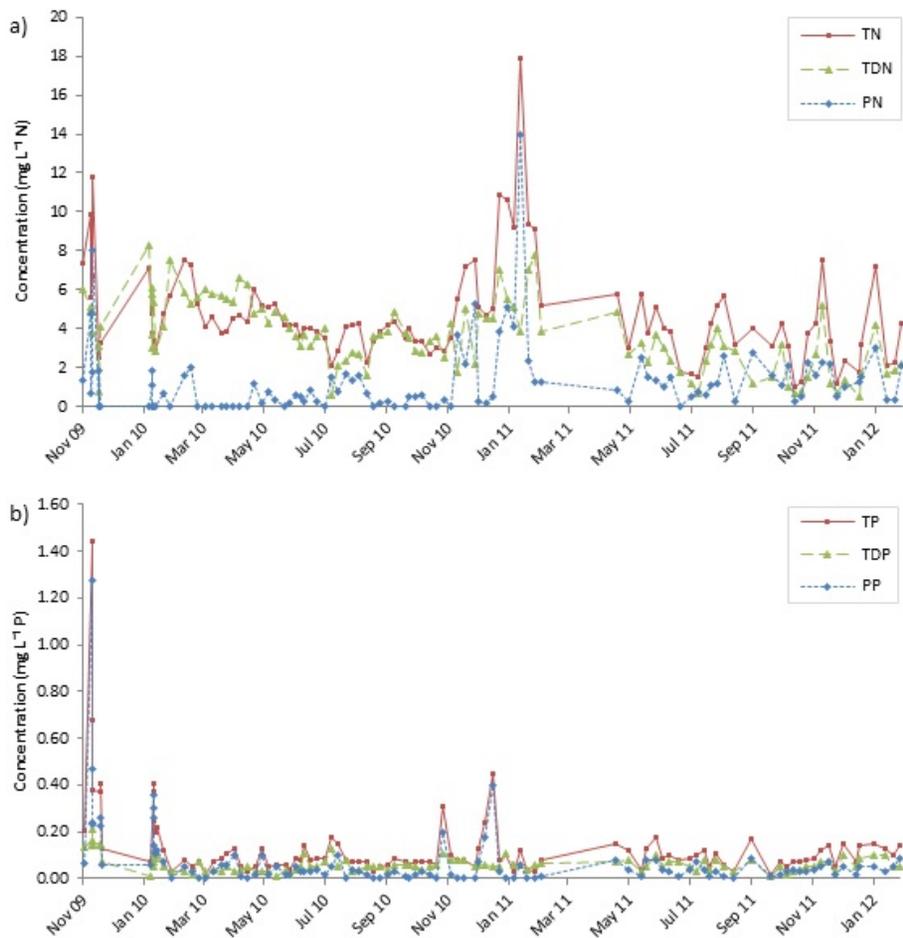


Fig. 3. Mean monthly concentrations of (a) nitrogen and (b) phosphorus.

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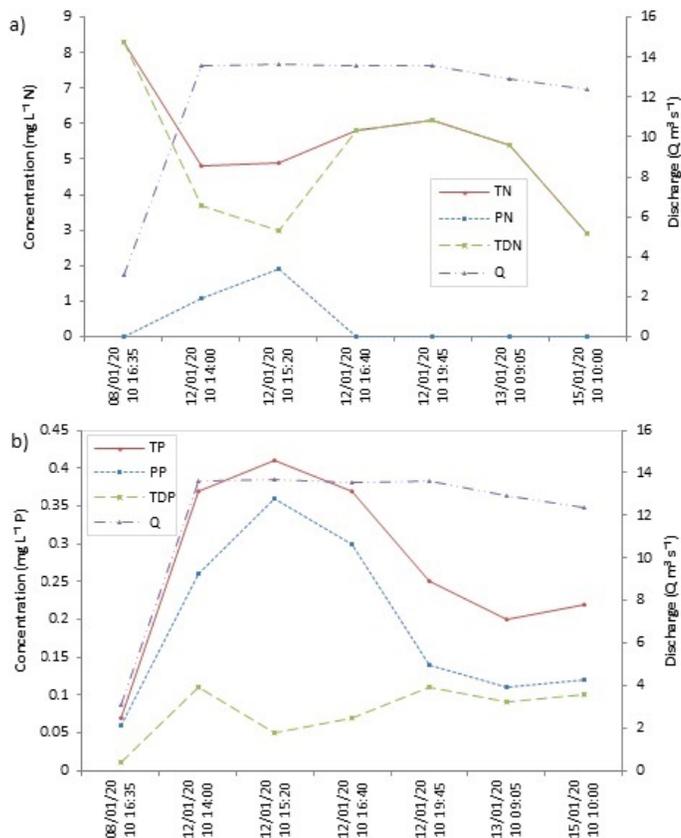


Fig. 4. Discharge and (a) nitrogen and (b) phosphorus concentrations during a storm event from January 2010.

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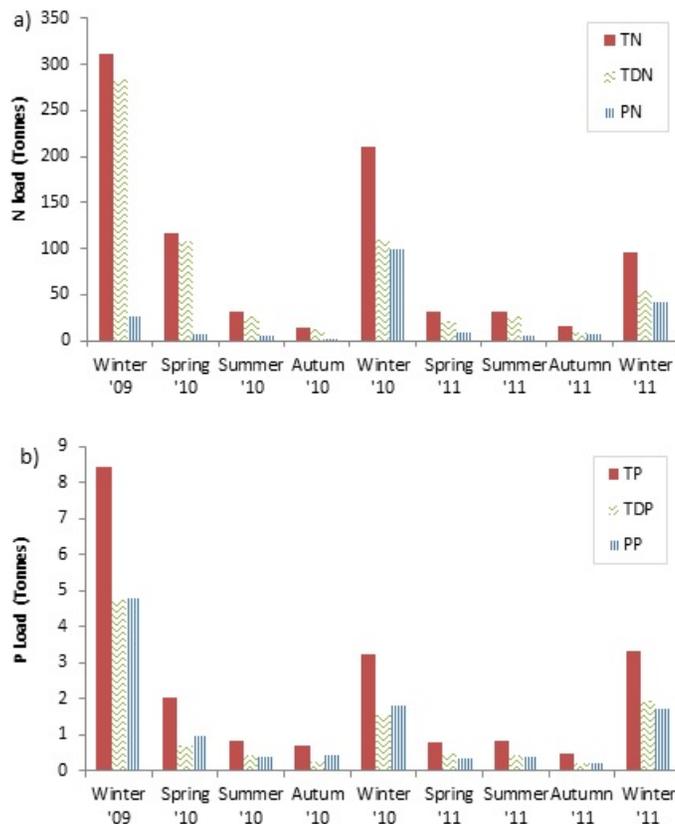


Fig. 5. Seasonal loads of **(a)** nitrogen and **(b)** phosphorus on the River Owenabue.