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Flood survey of nitrate behaviour using nitrogen isotope tracing in the critical zone of a French agricultural catchment

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ABSTRACT

Measurements of $\delta^{15}\text{N}-\text{NO}_3^-$ were taken in a highly flood-responsive agricultural catchment in the southwest of France to trace the sources and transfer pathways of nitrates during flood events. From January to March 2013, surface water samples were collected every week at the outlet, and four floods were sampled with a high resolution. Sampling was also performed in surface waters and sand lenses from the rest of the basin to trace nitrate sources and processes spatially. Nitrate extractions were performed using a method based on the solubility difference between inorganic salts and organic solutions. The $\delta^{15}\text{N}$ values were in the range of surface water contaminated by N-fertilisers. Depending on the hydroclimatic event, nitrates resulted from a combination of sources and processes. At the start of the floods, the values of $\delta^{15}\text{N}-\text{NO}_3^-$ and nitrate concentrations were low, demonstrating the dilution of water with rainwater. During a second phase, the nitrate concentration and the $\delta^{15}\text{N}$ were higher. Deeper waters and soil solutions were the second source of nitrates. When the water level was low, both nitrate concentration and isotopic composition were high. These values reflected the denitrification processes that occurred in the soil under anaerobic conditions. An analysis of $\delta^{15}\text{N}-\text{NO}_3^-$ in stream water in a small agricultural catchment was efficient at determining the origin of nitrates during flood events using a simple method.

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1. Introduction

Nitrogen concentrations in the atmosphere and hydrosphere have been increased by human activities in recent decades (Galloway et al., 2004; Harker et al., 2015; Vitousek et al., 1997). One major source of nitrogen is the burning of fossil fuels, which releases nitrogen

compounds such as nitric oxides (NO_x), nitrous oxides (N_2O) and ammoniac (NH_3) into the atmosphere. Waste from sewage also contributes to the increase of nitrogen in water. Furthermore, one of the main sources of nitrates is the input of nitrogen fertilisers and animal manure in soil in order to enhance crop yields. Cultivated plants only absorb half the fertilisers being spread (FAO, 2001), therefore nitrates are transported in large quantities to surface water and groundwater and, finally, to the rivers through surface runoff, soil leaching and soil erosion (Kattan et al., 1986; Mariotti and Létolle, 1977; Probst, 1985). The change in the nitrogen cycle leads to the

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eutrophication of aquatic ecosystems and the acidification of soil (Brunet et al., 2011; Hastings et al., 2009; Perrin et al., 2008; Semhi et al., 2000; Vitousek et al., 1997).

Controlling nitrate concentration in stream water is essential in agricultural areas in order to limit the surface water and ground water pollution. Acquiring knowledge of the origin and pathway of nitrates is the first step in taking proper action.

The aim of this paper was to monitor nitrate concentration and its nitrogen isotopic composition during flood events to highlight their origin and transfer pathways and to enhance understanding of the controlling factors in a highly flood-responsive catchment.

This small agricultural catchment, Montoussé, located in the Southwest of France, is dominated by mineral fertilisers. It has been monitored since 1980 to control nitrogen flux and nitrate concentration, reduce nitrogen losses and improve water quality through the use of better agricultural practices. Research is currently being developed with farmers (“Groupement des agriculteurs de la Gascogne toulousaine”) in order to improve crop yields and reduce the effects of nitrate pollution. One of the main actions was to create grassed buffer strips along the river in 1995. Several studies (Mayer et al., 2007; Sebilo et al., 2006; Vidon and Hill, 2004) have highlighted that nitrate leaching can be reduced in the stream by creating grassed buffer strips between the cultivated fields and the creek.

Since 1985, nitrate concentrations have been measured weekly and since 2006 measured continuously (Ferrant et al., 2013) at the outlet of the watershed using an YSI multiparameter probe equipped with a nitrate sensor. Nitrate dynamics depend mainly on floods and droughts, leaching and retention processes and fertiliser application at the small catchment scale. The hydrochemical conditions at the Montoussé catchment are mainly controlled by floods. The concentration as well as the flux of pollutants such as nitrate and pesticides is higher during floods events and they are mainly leached by subsurface run-off in this catchment (Idir et al., 1999; Taghavi et al., 2010).

The water contributions during floods are very important because of the different water origin and the addition of various nutrients from different part of the catchment, such as nitrate (Buda and DeWalle, 2009).

The origin of nitrate pollution can be determined by analysing the isotopic composition of nitrogen (Heaton, 1986; Kendall, 1998). Indeed, the source, climatic events

and physical and chemical processes change the isotopic ratio of nitrogen and oxygen. In this study, it was decided to analyse only nitrogen isotopes. $\delta^{15}\text{N}$ is a good discriminator for assessing the principal origin and main processes in a small agricultural catchment. Table 1 provides a summary of the $\delta^{15}\text{N}$ values of different sources of nitrogen in the environment. In surface water, $\delta^{15}\text{N}-\text{NO}_3^-$ is comprised of between +4 and +7‰ for unpolluted rivers and between +6 and +12‰ when stream water is contaminated by fertiliser (Cravotta, 1984; Fogg et al., 1998; Xue et al., 2009). $\delta^{15}\text{N}-\text{NO}_3^-$ of nitrate derived from animal manure and sewage is typically higher (Kendall et al., 2007). The nitrogen isotopic signature varies with the source of the nitrate, but the isotopic signature also changes when the nitrate is involved in chemical reactions. Nitrification and denitrification are the two main processes that fractionate the most. Nitrification reaction generates fractionation from -29 to -12‰. In contrast, the denitrification process leads to high ^{15}N -enrichment in the residual nitrate. Fractionation of the total denitrification ($\text{NO}_3^- > \text{N}_2$) is very significant, ranging from -40 to -5‰ (Kendall, 1998). Therefore, the range of values of $\delta^{15}\text{N}$ is an indicator of the different processes (Kellman and Hillaire-Marcel, 2003; Mariotti et al., 1981; Sebilo et al., 2006).

The denitrification process occurs when anaerobic conditions are present (Kendall, 1998; Sebilo et al., 2003; Sebilo et al., 2006), there are enough denitrifying bacteria in the soil and there is a source of organic carbon to provide energy to the bacteria (Birgand et al., 2007). Grassed buffer strips allow the retention of nitrate and therefore trigger the denitrification process in low-oxygen environments (Galloway et al., 2004; Sebilo et al., 2006; Vidon and Hill, 2004). In the studied watershed, leaching of nitrate into the river should be limited by the grassed buffer strips.

Nitrate leaching and retention processes at catchment scale are not very well understood due to the various sources of nitrate, hydrological pathways and variations in hydroclimatic conditions (Billy et al., 2013). The main objective of this study was to determine the different factors affecting nitrate concentration during flood events in an agricultural watershed.

Thanks to a simple extraction method, $\delta^{15}\text{N}$ of nitrate in the surface water was measured for three months from January to March 2013 at the outlet of the catchment and in other water springs around the watershed. The main

Table 1
Range of values of $\delta^{15}\text{N}$ for different nitrogen sources.

Nitrogen source	$\delta^{15}\text{N}$ (‰)	References
Soil organic nitrogen	+4 to +9	Heaton, 1986; Kendall, 1998
Total soil nitrogen	-10 to -15	Kendall, 1998
Fertilisers	-4 to +4	Heaton, 1986; Fogg et al., 1998; Kendall, 1998
Manure	-10 to +20	Cravotta, 1984; Fogg et al., 1998; Kendall, 1998
Rain	-6 to +2	Heaton, 1986; Freyer, 1991; Russell et al., 1998; Hastings et al., 2009; Xue et al., 2009
Sewage waste	>+10	Heaton, 1986; Cravotta, 1984; Fogg et al., 1998; Kendall, 1998
Vehicles (NO_x)	-13 to -2	Heaton, 1990
Coal fire NO_x	+6 to +13	Heaton, 1990
Atmospheric nitrogen	0	Heaton, 1990; Freyer, 1991
Surface water contaminated by nitrates	+4 to +7	Cravotta, 1984; Fogg et al., 1998; Xue et al., 2009
Surface water not contaminated by nitrates	+6 to +12	Cravotta, 1984; Fogg et al., 1998; Xue et al., 2009

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