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# A Multiparameter and Multiscale Dataset to Study Sediments and Particle-Bound Pesticide Dynamics of Hydric Transfers in a Wine-Dominated Catchment

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## ABSTRACT

Sediment transport plays a crucial role in water quality at the catchment scale. Yet, access to comprehensive datasets for research on sediment quantity and quality at different spatial scales remains limited. This paper introduces a comprehensive hydro-sedimentological dataset on the Ardières-Morcille catchment and scientific observatory (Beaujolais vineyard, France) available for the period 2020–2023. The observatory was monitored at three nested scales: the Saint-Joseph plot (0.28 ha), the Morcille sub-catchment (3.9 km<sup>2</sup>), and the Ardières catchment (143 km<sup>2</sup>). This dataset includes continuous monitoring of rainfall, water level, and turbidity at the three sites, from which discharge and suspended solids concentrations are derived. In addition, discontinuous yet integrated over time samples were collected for SPM grain size, major elements, and particle-bound pesticides analysis. This dataset has made it possible to assess orders of magnitude for sediment and particle-bound pesticides transfers and to interpret scale effects in time and space responsible for these transfers. We expect this dataset to be a valuable resource for the research community, supporting investigations into sediment transport processes, contaminant fluxes, and hydrological dynamics across multiple scales.

## 1 | Introduction

Soil erosion is often considered to be one of the main threats to agricultural land around the world (Dotterweich 2013; Panagos et al. 2015; García-Ruiz et al. 2015). This gives rise to very diverse associated environmental issues that can be observed at different spatial scales: from erosive runoff on agricultural plots to transfers to large waterbodies downstream of catchment areas. Understanding the temporal dynamics and spatial forms of hydro-sedimentary transfers in catchments must take into account the multi-scalar nature of these processes. Scale effects can be seen in the differences observed between soil losses on plots and the sediment actually transferred at the outlet of

a catchment (Walling 1983), as well as in the non-linearity of processes between different scales in space and time (Mcguinness et al. 1971; Cammeraat 2002; Cossart et al. 2018). Consequently, the analysis of hydro-sedimentary flows should take into account the interactions between the different sub-systems (e.g., from the agricultural plot to the regional scale) and should consider the scale effects that exist between these different compartments (De Vente 2009; Bracken et al. 2015; Keesstra et al. 2018).

In agricultural catchments, these sediment transfers associated with surface runoff must be linked to the transfers of pesticides, which pose environmental and health problems. Indeed, due to regular inputs onto agricultural lands over

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many years, their persistence in soils (Geissen et al. 2021), and the occurrence of storm events, pesticides are ubiquitous in waterways, both in dissolved and particulate phases (Taghavi et al. 2011; Oliver et al. 2012). Most studies have focused on pesticide transfers in the dissolved phase (Cai et al. 2025), but some research has highlighted the importance of the link between pesticide transfers in the particulate phase and surface runoff (Taghavi et al. 2010; Boithias et al. 2011). The contribution of the particulate phase to total pesticide transfer and the potential risk to off-site locations are not yet well understood (Commelin et al. 2022), as few studies have specifically quantified pesticides in the particulate fraction (Chaumet et al. 2022). It is therefore necessary to characterise the respective dissolved and particle fractions of pesticide transfers in different spatial and hydrological contexts.

This short review highlights the need to document hydro-sedimentary transfers and associated particulate pesticides transfers at different scales by means of field measurements. Several works emphasised the need for open-access hydrological datasets to improve the understanding of the processes governing the hydrological behaviour of catchments, to compare different contexts, to calibrate models and to better support transparency and reproducibility of scientific research (Francke et al. 2018; Gaillardet et al. 2018; Boithias et al. 2021; Avila and Rodà 2023; Matthews et al. 2023). The state-of-the-art points out that studies combining hydro-sedimentary transfers and pesticides in particulate phases at nested scales are rare, and that open-access related datasets are even rarer.

This research has been conducted as part of the PULSE project (Fressard et al. 2023) that aimed to develop a better understanding of the landscape influence on sediment and pesticide transfers at different scales, with a view to modelling and scenarios. To this end, it was necessary to obtain data on hydro-sedimentary transport and to establish a link with the capacity of particles to transport pesticides. In this context, field monitoring has been set up to provide initial estimates of specific erosion rates and the proportion of particle-bound pesticides transfers. This paper describes the dataset that has been collected over a period of 3 years, which includes a variety of parameters measured at different spatial scales.

The dataset was collected on the Ardières-Morcille scientific observatory, which belongs to the site-ateliers de la “zone-atelier basin du Rhône” (ZABR, <https://www.zabr.assograie.org/site-ardieres-morcille/>). It has been used by the French National Research Institute for Agriculture, Food and the Environment (INRAE) to study pesticide transfers for over 30 years (Gouy et al. 2021). This observatory aims to identify the factors responsible for the transfers of pesticides and the subsequent concentrations measured in the rivers, as well as to assess the impacts of these pesticides on aquatic ecosystems and possible ways of restoring water quality. Previously to the PULSE project, the measurements have focused on hydrological parameters (water level and flow) and on pesticides in the dissolved phase. The novelty of the dataset presented in this paper is to introduce sediment monitoring on a plot and in the river, as well as pesticides in the particulate phase. Monitoring is organised on three interlocking spatial scales in order to investigate the cascading system: from a vine plot to the integrated monitoring of the catchment area.

The aim of this article is to present and disseminate to the scientific community a complete set of hydro-sedimentological data that includes sediment quantity and quality (pesticides) monitoring over a period of 3 years. We expect this dataset will be used by scientists to improve the knowledge on hydrological processes through the study of the interactions between dissolved and particle-bound pesticides transfers by interpretative or model-based studies.

Following a detailed description of the study area and the characteristics of the monitoring sites, we outline the field data collection protocol. We then present the sediment samples and the laboratory analyses conducted to quantify sediment fluxes and pesticide concentrations. Finally, we characterise the sampling period, given the hydro-climatic context of the studied area, and discuss the representativeness of the collected data.

## 2 | Study Site

### 2.1 | General Context of the Beaujolais Vineyard (France)

Located in the lower part of the Saône catchment, the Beaujolais wine region is approximately 50 000 kilometres long from the northern Lyon conurbation to the southern Mâcon. The vineyards are more precisely located along the eastern hillslopes of the Massif Central (below 500 masl) and are bounded further east by the alluvial plain of the Saône River (Cossart et al. 2020; Pic 2023). The annual precipitation is approximately 850 mm and is regularly distributed throughout the year, although more erosive in summer due to more storms during this period (cf. erosivity factor, Mcgregor et al. 1995). The area receives approximately 2050 h of sunshine per year, and hot temperatures exceeding 30°C are fairly typical during summer (Météo-France 2022).

The dataset was produced on the Ardières-Morcille scientific observatory. The Morcille river is a tributary of the Ardières river, which flows into the Saône, a tributary of the river Rhône. The catchments are characterised by an altered granitic or volcanic basement. The Ardières catchment is mainly characterised by alocrisols and, to a lesser extent, brunisols, both with silty sands or coarse sands (Party 1996). The Morcille sub-catchment is characterised by an altered hercynian crystalline basement of mainly porphyric biotite granite (Pomerol 1984; Party 1996; Frésard 2010; Wahl 2017) which results in three types of soils: (1) sandy soils developed on altered granite on the upper slopes, (2) shallow clay-based soil on the right bank of the Saône river, and (3) sandy soils on lower slopes and thalwegs.

The upstream part of the catchment is representative of the northern part of the Beaujolais vineyard due to its landscape structure (figure 1; Pic 2023). The hillslopes are characterised by vine monoculture on steep slopes. The vineyard is divided into small plots with a high density of vine stocks, along the slope and with frequent use of pesticides. Forested areas and grasslands are mainly located in the head of the catchments or near the rivers. The downstream part of the catchment belongs to the alluvial plain of the Saône river. It is mainly urbanised or oriented to other types of agriculture (cereal crops, vegetable farming, grazing meadows) (Figure 1). The Ardières catchment mean

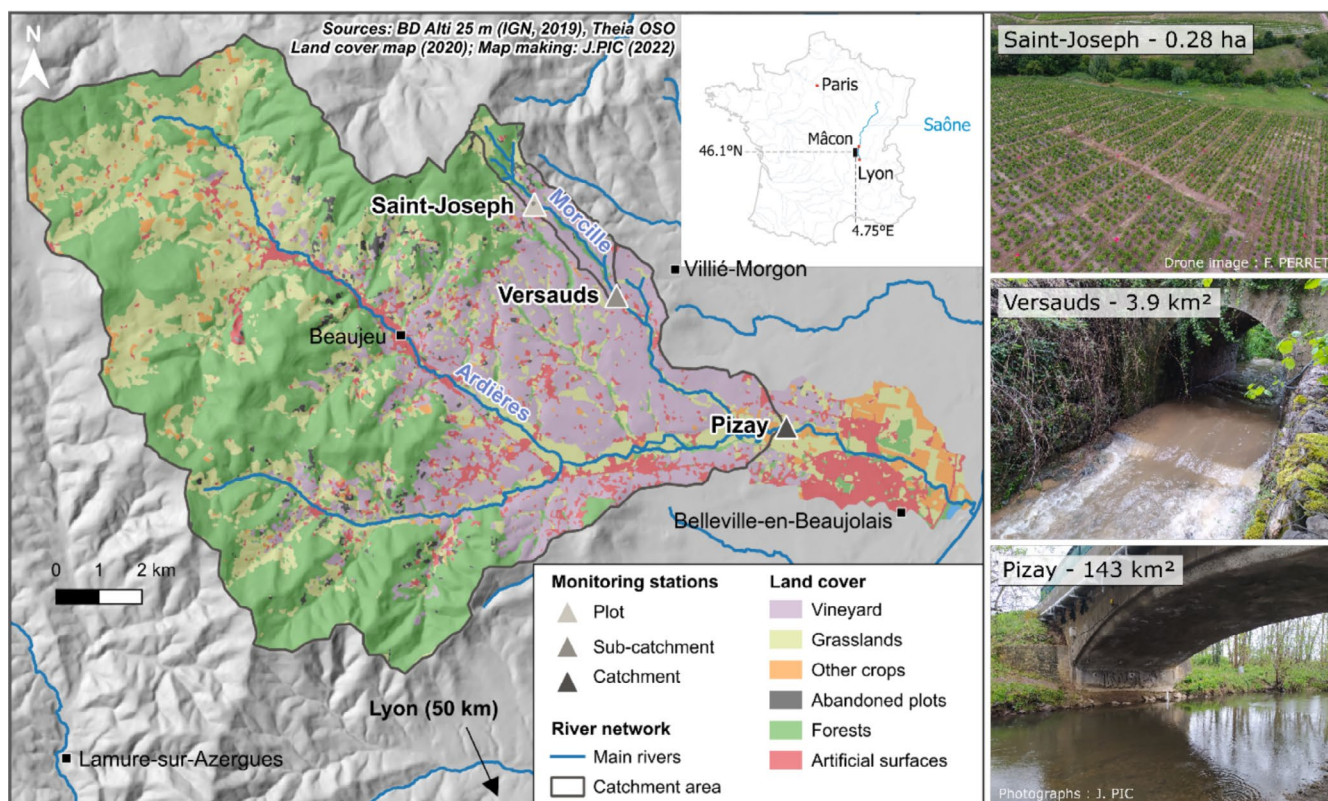


FIGURE 1 | Location of monitoring stations.

and maximum slopes are, respectively, 12° and 42°. The altitude range lies between 170 and 1008 masl. The Morcille catchment includes slopes averaging around 7° but reaching almost 35° in the steepest parts. The mean elevation is approximately 390 m, ranging from 207 to 806 m asl.

## 2.2 | Location of Monitoring Stations and Sites Description

Three measuring stations make up the system: Saint-Joseph, Versauds and Pizay (Figure 1). The rainfall is recorded in parallel according to the location and technology described in (Gouy et al. 2021). This dataset is not included in this paper, but can be downloaded from the same platform (<https://bdoh.inrae.fr/SITE-ATELIER-ARDIERES-MORCILLE/>).

The Saint-Joseph station is an experimental plot located upstream of the Morcille catchment and has been studied by INRAE since 2004 (Lacas et al. 2012). The plot covers an area of 0.28 ha and has an average slope of 8.8°. The vines are goblet-pruned over the entire plot and are planted in rows in the direction of the slope. One row out of five has no vines and is grassed. The plot includes four soil bunds to back up the slope connected to a concrete gutter in the centre of the plot. These features are emblematic of the spatial structure of the Beaujolais region for the steepest plots.

The Versauds station is used to investigate the hydro-sedimentary dynamics at the head of the Morcille catchment. The monitoring site is located in the middle of the catchment, under a bridge. The station drains an area of 3.9 km<sup>2</sup> and has

been equipped with a V-shaped weir since 2008. The Pizay station is designed to study river dynamics at a wider scale. It is located on the Ardières river, under a bridge after the confluence with the Morcille river. It drains an area of 143 km<sup>2</sup>. Water levels have been measured at the Versauds station since 2002 and at the Pizay station since 2011. Discharge varies between 0.02 m<sup>3</sup>.s<sup>-1</sup> and 2 m<sup>3</sup>.s<sup>-1</sup> at the Versauds station and between 0.1 m<sup>3</sup>.s<sup>-1</sup> and 30 m<sup>3</sup>.s<sup>-1</sup> at the Pizay station (Gouy et al. 2021).

## 3 | Collected Data and Stations Characteristics

### 3.1 | Summary of Collected Data

All the datasets cited in Table 1 are stored in the 'Hydrological Observatory Database' (BDOH in French; Branger et al. 2014) and accessible via the Ardières-Morcille observatory webpage (Gouy et al. 2015, <https://doi.org/10.17180/obs.saam>). The records can be browsed and visualised by anyone, but registration is required to download them. Use of these records is subject to compliance with the rules of use stated on the website.

### 3.2 | Saint-Joseph Plot

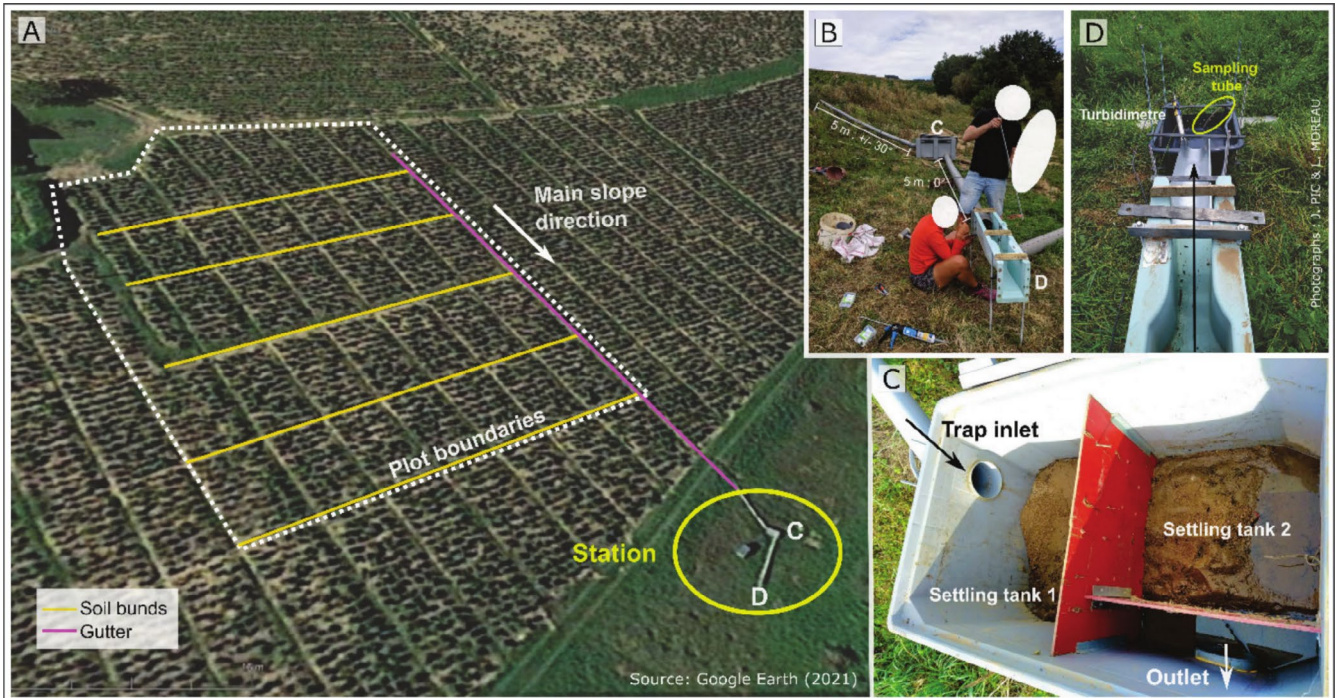
The Saint-Joseph plot was equipped to measure sediment transfers and pesticides in the particulate phase (Figure 2). The monitoring system is made of 2 main components that successively measure: (1) coarse and medium size sediments that settle in the first compartment of the system and (2) residual turbidity at the outlet of the monitoring system.

**TABLE 1** | Summary of collected data as part of the PULSE project.

Site	Parameter	Time-step	Technology
Saint-Joseph	Water level	30s/triggered by the flow rate	Pressure sensor (Keller 36 XiW series) with a 1.2 cm to 3 m measurement range and 0.15 cm uncertainty
	Water flow		Venturi channel Hydrologic E1253AZ with water flow rate between 0.47 and 9.28 L.s <sup>-1</sup> . Relative uncertainty: 15% over the range of flow rates and 20% between 0.47 and 1 L.s <sup>-1</sup> .
	Turbidity		Multiparameter probe (AquaTroll 600) with a 4000 NTU measurement range turbidity sensor and 0.1 mg/L <sup>-1</sup> uncertainty
	Suspended solids concentration		Teledyne Isco automatic sampler
	Coarse sediments: volume, granulometry	Variable—around 2 weeks	Homemade sediment trap and sieving column
Versauds (Morcille river)	Water level	10 min	Pressure sensor (CS420-L) with a 3.5 m measurement range and a 2.8 mm uncertainty. Campbell CR800 datalogger
	Water flow		Calculated from water level and rating curves/gauging methods and devices: Electromagnetic current meter, water surface velocity with radar SVR
	Turbidity		Multiparameter probe (AquaTroll 600) with a 4000 NTU measurement range turbidity sensor and 0.1 mg/L <sup>-1</sup> uncertainty
	Suspended solids concentration		Teledyne Isco automatic sampler
	Granulometry and major elements of suspended solids	Variable—around 2 weeks	Particle traps, CILAS 1190 laser particle sizer, ICP-OES (720-ES, Agilent Technologies)
Pizay (Ardières river)	Water level	10 min	Radar sensor (CS475) with 19.8 m measurement range and 5.1 mm uncertainty. Campbell CR1000 datalogger
	Water flow		Calculated from water level and rating curves/gauging methods and devices: Electromagnetic currentmeter, water surface velocity with radar SVR
	Turbidity		Multiparameter probe (AquaTroll 600) with a 4000 NTU measurement range turbidity sensor and 0.1 mg/L <sup>-1</sup> uncertainty
	Suspended solids concentration		Teledyne Isco automatic sampler
	Granulometry and major elements of suspended solids	Variable—around 2 weeks	Particle traps, CILAS 1190 laser particle sizer, ICP-OES (720-ES, Agilent Technologies)

The first device for measuring sediment transfers consists of a settling tank. It was specifically developed and calibrated to match the flow coming from the upstream plot, channelled by a pre-existing concrete gutter used in previous works (Lacas 2005). The

collector was sized by analogy with those installed by the wine growers based on the measurement of volumes/drainage area (m<sup>3</sup>/ha) of a batch of four collectors located nearby. The results of these measurements are very heterogeneous but a 0.5 m<sup>3</sup> settling tank



**FIGURE 2** | Saint-Joseph plot instrumentation. (A)—Plot configuration. (B)—Venturi canal set up. (C)—Inside of the sediment trap. (D)—Venturi canal outlet.

seemed sufficient to collect the flows from the Saint-Joseph plot. The settling tank consists of a 470-l polyethylene tank divided into three compartments, allowing coarse sediments to settle while allowing hydrological flows and suspended solids to flow through (Figure 2). The settling tank is cleaned regularly (approximately every fortnight), enabling the sediments to be weighed on site. A homogenised sample, based on a 250 cm<sup>3</sup> sampling cylinder, is collected to determine its dry mass in the laboratory. The mass of the wet sample is assessed using a balance accurate to one tenth (g). The sample was then placed in an oven at 105°C for at least 24 h. The quantity of sediment trapped in the collector is inferred from the mass difference between the wet and the dry sample.

The outlet of the settling tank is connected to a 160 mm in diameter PVC pipe, which connects the collector to a Venturi gauge channel located downstream (Figure 2). The channel model is a Hydrologic E 1253 AZ with a water flow rate between 0.47 and 9.28 L.s<sup>-1</sup>, and the uncertainty of the system was determined by Lacas (2005). The connection is 5 m long, enabling laminar flow and the accurate measure of water flow rate in the Venturi channel with a pressure sensor (Keller 36 XiW series). A turbidimeter is installed at the Venturi channel outlet to measure residual turbidity and complement the measurements taken in the settling tank. Finally, a Teledyne Isco automatic sampler is installed downstream of the system to collect water samples.

Water level and turbidity data are recorded at 30-s intervals during run-off events. The system is triggered by the flow rate: as soon as a non-zero flow rate is detected, the control unit records the flows. This recording method is suited to the hydrological behaviour of the plot, characterised by short, abrupt phases of activation (Hortonian runoff) followed by long periods without any activity. Turbidity-sensitive sampling was carried

out in as many measurement ranges as possible in order to optimise the correlation between turbidity and suspended solids concentration.

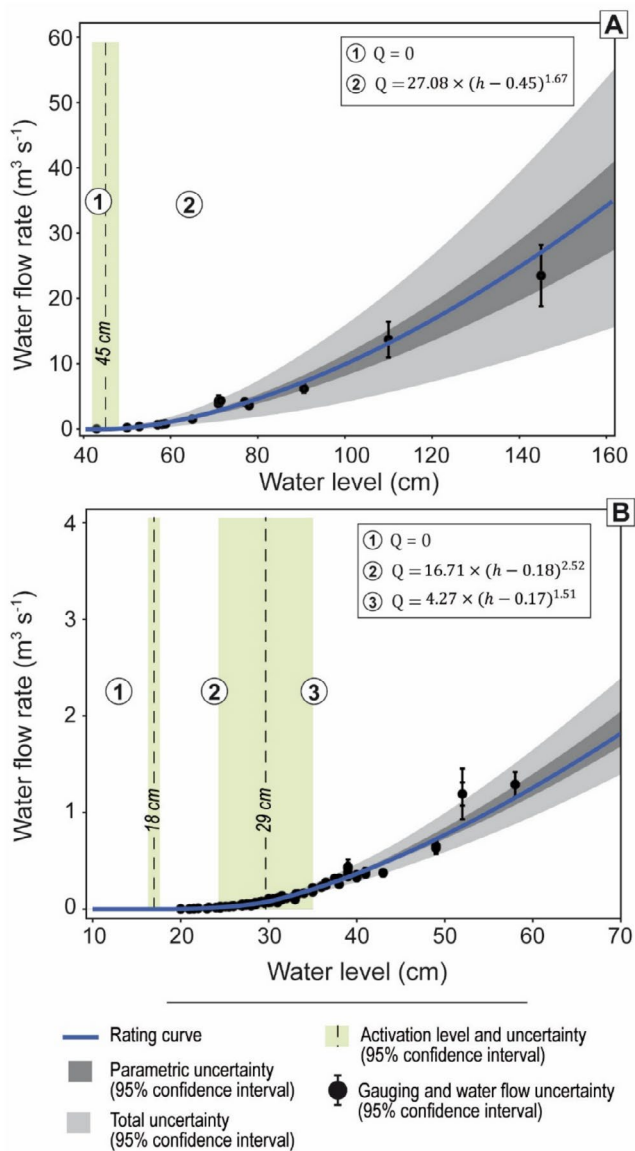
### 3.3 | River Stations

#### 3.3.1 | Continuous Monitoring of Water Level and Turbidity

Water level and turbidity were monitored continuously from May 2020. Water depth has been measured since 2008 at Versauds station with a CS420-L pressure sensor and at Pizay station since 2011 with a CS475 radar sensor (Table 1). A vertical gauge is placed at both sites to check water level measurement. Turbidity is measured at both stations, and samplers have been installed alongside turbidity sensors in order to establish the correlation between turbidity and suspended solids concentration. The entire system (data recording and sampling control) is managed by a datalogger from Campbell Sci. Data are recorded at a time step of 10 min, representing the average of 10 measurements. A field visit for data collection, sampling, battery replacement, and maintenance was carried out every fortnight. Sample collection for calibrating the turbidity/suspended solids concentration relationship has been controlled by turbidity according to variable thresholds in order to have samples representative of the entire range of observed turbidity (Camenen and Melun 2021).

#### 3.3.2 | Integrated Sampling of Suspended Solids

In parallel, particle traps were installed to sample suspended solids. The particle traps used are identical to those used by



**FIGURE 3** | Rating curves and associated uncertainties (A) Pizay and (B) Versauds monitoring stations.

(Schulze et al. 2007; Masson et al. 2018). It has been shown that the size of particle traps has an influence on the granulometric fractions they collect (Berni et al. 2019). At the Versauds station, the trap dimension was reduced to three-quarters to suit the size of the river. At the Pizay station, two particle traps (one full size, and one reduced to three-quarters) were used in parallel in order to compare the differences in granulometric fractions in relation to site characteristics and flow rate. The trap consists of a stainless-steel cage with two settling troughs. Water flow is ensured through three inlet and outlet ports. The particle traps are placed directly on the riverbed to collect the suspended solids. These traps allow for an integrative measurement of the sediments in suspension and are not suitable for studying saltation and bedload. The particle traps were collected every fortnight, and their contents carefully recovered: an aliquot (20 mL) was taken for particle size analysis in the laboratory, and the remainder poured into a 2L brown bottle. After settling in the laboratory and removal of the supernatant, the sample was freeze-dried and ground for subsequent analysis of granulometry (analysed

before freeze-drying and grinding), major element content (particulate organic carbon, total nitrogen, phosphorus, sodium, potassium, magnesium, calcium) and pesticides for some samples.

## 4 | Samples and Data Analysis

### 4.1 | Water Flow Estimation

Instantaneous flow rates at the outlet of the sediment collector on the experimental plot were measured using a Venturi-type channel gauge and a pressure sensor. It is characterised by a known head/flow relationship, the quantification limits of which correspond to the validity of the calibration curve (Lacas 2005). Flow rates at the Saint-Joseph station are based on the following equation:

$$Q = 0.002929 \times (H)^{1.58} \quad (1)$$

Where, Q: water flow (L.s<sup>-1</sup>); H: water level (mm).

At the Pizay and Versauds stations, most of the gauging was carried out using an acoustic currentmeter at different verticals spaced in proportion to the width of the watercourse. Three readings were systematically taken at 0.2, 0.6, and 0.8 times the height. Flows were then calculated using the central section method via the formula (Perzyna 2016):

$$Q = \sum (v \times A) \quad (2)$$

Where, Q: flow rate of the wetted subsection (m<sup>3</sup>.s<sup>-1</sup>); v: flow velocity of the wetted subsection (m.s<sup>-1</sup>); A: area of the wetted subsection (m<sup>2</sup>).

During flood periods, gauging was carried out using a velocimetric radar according to the method proposed by Costa et al. (2006) and Dramais et al. (2012). Flows are calculated by considering an increase in flow velocity between the bottom and the surface following a logarithmic progression. The uncertainty of this method is still higher than that of the currentmeter method, but it allows flow values to be obtained during flood events.

The rating curves for the two stations were then determined using the BaRatin (Bayesian Rating curve) method developed by INRAE since 2010 (LE Coz et al. 2014; Horner et al. 2018). This method is used not only to construct the rating curve, but also to calibrate it and estimate the associated uncertainties. The rating curve is determined by combining information from gauging and the hydraulic context of the site.

The correlation between water level and flow at the Versauds station is based on 70 gauges, 3 of which were taken using a radar sensor. The flow is governed by two rating curves. Between 17.5 cm and 29 cm, the flow is controlled by a triangular weir. Above 29 cm, the flow is controlled by a rectangular weir. Few gauging operations have been carried out at water depths in excess of 40 cm, which means that the section of the rating curve is less reliable (Figure 3B). However, since 2008, the water height has exceeded 40 cm only 0.6% of the time at this station. On this

site, the Regional Directorate of Environment, Planning and Housing (DREAL) records also the water level and calculates the water flow. This data may be used to complete missing data in our dataset (<https://www.hydro.eaufrance.fr/stationhydro/U450601001/series>).

Although there were gauges prior to 2018 at the Pizay station, the rating curve is only based on gauges from 2018 onwards. Changes in the hydromorphological context were observed at this date, making the previous rating curve obsolete. The rating curve is based on 16 gauges, including 4 from a radar sensor. Below a water level of 45 cm, the flow at the station is considered to be zero. Above 45 cm, it is governed by the equation in Figure 3A.

## 4.2 | Suspended Solids Concentration Estimation

The estimation of sediment flows is based on the correlation between turbidity and the concentration of suspended solids. As the granulometry and colorimetry of the suspended solids influence the turbidity measurement, it is necessary to calibrate this relationship specifically for each study site. The samples taken are then filtered and the suspended solids weighed according to the NF EN 872 protocol. The suspended solids concentration is given in  $\text{mg/L}^{-1}$  by the expression:

$$[\text{SSC}] = \frac{M_1 - M_0}{V} \times 1000 \quad (3)$$

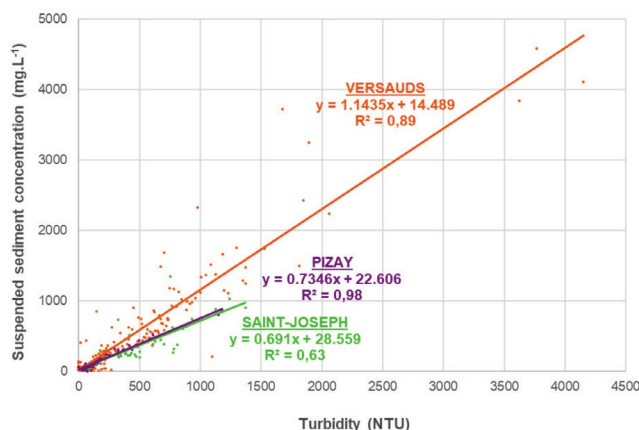
Where,  $M_0$ : initial mass of the filter, weighed dry (mg);  $M_1$ : mass of filter after filtration, weighed dry (mg);  $V$ : filtered volume of sample (mL).

The correlation between turbidity and suspended solids concentration is based on 54 samples taken at Saint-Joseph, 234 at Versauds and 68 at Pizay. The correlations between turbidity and suspended solids concentration are generally good at all three stations. The Pizay station had an  $R^2$  of 0.98, with the sample with the highest concentration having a turbidity of 870 NTU. The turbidity measurement range is wider at Versauds, where the highest turbidity sampled was 4152 NTU. The turbidity/concentration correlation has an  $R^2$  of 0.89. The Saint-Joseph station had a wider dispersion of samples (collected downstream the tank), with an  $R^2$  of 0.63. The highest turbidity value sampled was 1370 NTU (Figure 4).

## 4.3 | Grain Size Analysis of Solid Matter Samples

The granulometry of the samples collected in the Saint-Joseph trap is measured to compare hydrological events and the proportion of fine and coarse sediments. Given the coarse nature of the sediments collected, sieving was the preferred method. Granulometry is carried out using dry samples placed in a column of sieves. The sieve range extends from 0.063 mm to 8 mm.

The particle size distributions of samples taken by particle traps in the river were analysed using a CILAS 1190 laser particle sizer in liquid mode. This instrument measures the



**FIGURE 4** | Correlation between turbidity and suspended solids concentrations determined by the analysis of the samples from the automatic samplers at the 3 monitoring sites.

particle size distribution of a sample in the form of volume distributions made up of 100 particle size classes ranging from 0.04 to 2500  $\mu\text{m}$ . The granulometer is equipped with a 450-mL tank into which solid samples are manually introduced. After adding mineralised water to the tank up to a defined level, a stirring system homogenises the particles in the tank. The quantity of sample introduced into the granulometer is adjusted to achieve between 5% and 25% obscuration (recommended by standard ISO 13320:2020). Two successive analyses are carried out on the sample: the first without the use of ultrasound, then a second with the application of ultrasound (38 kHz, 50 W) 30s before and during the analysis. Ultrasound dissociates the aggregates: the aim was to assess whether the difference between the particle sizes obtained using these two protocols provided a better understanding of particles mobilisation and transfer processes. The optical model used to transform the measured signal into a particle size distribution is the Fraunhofer model, with the particle shape considered to be spherical.

## 4.4 | Major Elements Analysis Protocol

The concentrations of particulate phosphorus, sodium, potassium, magnesium, and calcium were analysed in the suspended solids using an inductively coupled plasma optical emission spectrometer after mineralisation of the particles with *aqua regia*. The *aqua regia* extraction method was carried out in a microwave oven in closed reactors in accordance with ISO standard 15587-1:2002. Approximately 300 mg of dry, ground sample was introduced into a reactor with 6 mL of SUPRAPUR 30% HCl and 2 mL of SUPRAPUR 65% HNO<sub>3</sub> acid. Once the reactors were closed, they were introduced into the microwave for mineralisation (15 min at 180°C with a power of 1500 W). The mineralised samples were taken up with a few millilitres of ultrapure water and transferred to a 50 mL PP tube (SARSTEDT) topped up with ultrapure water. The mineralised solutions were kept refrigerated and protected from light before analysis.

The mineralised samples were then analysed using an ICP-OES (720-ES, Agilent Technologies) in accordance with ISO standard

**TABLE 2** | Suspended solids samples used for pesticides analysis.

Sample name	Sampling method	Site	Period
VER-PAP-200519-200 528-1	Particle trap 1:1	Versauds	2020-05-19–2020-05-28
PIZ-PAP-200519-200 528-1	Particle trap 1:1	Pizay	2020-05-19–2020-05-28
VER-PAP-201117-201 209-2	Particle trap 3/4	Versauds	2020-11-17–2020-12-09
PIZ-PAP-201117-201 209-1	Particle trap 1:1	Pizay	2020-11-17–2020-12-09
PIZ-PAP-201117-201 209-2	Particle trap 3/4	Pizay	2020-11-17–2020-12-09
VER-PAP-210503-210 518-2	Particle trap 3/4	Versauds	2021-05-03–2021-05-18
PIZ-PAP-210503-210 518-1	Particle trap 1:1	Pizay	2021-05-03–2021-05-18
PIZ-PAP-210503-210 518-2	Particle trap 3/4	Pizay	2021-05-03–2021-05-18
VER-PAP-210609-210 622-2	Particle trap 3/4	Versauds	2021-06-09–2021-06-21
PIZ-PAP-210609-210 622-1	Particle trap 1:1	Pizay	2021-06-09–2021-06-21
PIZ-PAP-210609-210 622-2	Particle trap 3/4	Pizay	2021-06-09–2021-06-21
SJO-PAP-210510-210 511	Sediment trap	Saint Joseph	2021-05-10–2021-05-11
SJO-WAT-210510-210 511	Automatic sampler	Saint Joseph	2021-05-10 – 2021-05-11
PIZ-WAT-210510-210 511	Automatic sampler	Pizay	2021-05-10–2021-05-11
SJO-PAP-210620-210 622	Sediment trap	Saint Joseph	2021-06-20–2021-06-22
SJO-WAT-210620	Automatic sampler	Saint Joseph	2021-06-20
PIZ-WAT-210616-210 622	Automatic sampler	Pizay	2021-06-16–2021-06-22

11 885:2007. Calibration solutions were prepared in *aqua regia*. Quality controls were systematically included in the sample analysis. For a 300 mg test sample, the limits of quantification for P, Na, K, Mg, and Ca concentrations in the suspended solids were 5 mg/kg (the concentrations measured in the project samples were more than 10 times higher than this value). Analytical uncertainties are 20%.

The determination of particulate organic carbon (POC) and particulate nitrogen (NTP) analyses was conducted using a carbon and nitrogen analyser (Thermo Electron, CN Flash 2000) and a method adapted from the NF ISO 10694 standard method (AFNOR, 1995). Decarbonation was completed using phosphoric acid according to manufacturer recommendation. The LOQ was estimated to be 0.5 g/kg. A reference material from a proficiency interlaboratory testing (Aglae, 15 M9.1; 40 g/kg) was systematically used to control analytical accuracy (93%) and uncertainty (20%;  $k = 2$ ).

#### 4.5 | Pesticide Analysis Protocol

The samples analysed were only collected in 2020 and 2021, and different sampling methods were applied according to each site. The excessively dry climate in spring–summer 2022 meant that it was not possible to sample the same rainfall event at all 3 scales. At the Saint Joseph station, both the deposits in the trap and the suspended solids samples were analysed. It should be noted that during the period in which these samples were collected, the automatic sampler was triggered by turbidity (threshold set at 20 NTU, and samples taken every

minute thereafter), while the turbidimetric probe was fixed in the outlet compartment of the sediment trap where the water level was subject to oscillations: this led to very noisy turbidity records. These do not question the concentrations analysed on the samples taken by automatic samplers but complicate the analysis of flow dynamics. At the Versauds station, samples were only taken using a particle trap, while the water sampler also made it possible to analyse two flood events at the Pizay station. All the suspended solids samples analysed during the project are presented in Table 2 below.

A multi-residue analysis method adapted to suspended solids was developed and validated, and was used to quantify 34 pesticides (30 active substances and four transformation products). A QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) extraction method is carried out on the dry ground samples, followed by purification by dPSE (dispersive Solid Phase Extraction). The pesticides were then analysed and quantified using ultra-high performance liquid chromatography (UHPLC), coupled with a tandem mass spectrometer (MS–MS). The extraction yields of the analytical method were evaluated by adding deuterated compounds to each sample. Assessment of the method performance and determination of the measurement uncertainties in accordance with the standards (NF T90-210, 2018 and NF ISO 11352, 2013 respectively) allows validating the analytical method for 34 pesticides (see (Marie 2021) for a detailed presentation of the analytical protocols and determination of the associated uncertainties). The pesticide concentrations presented here are concentrations in suspended solids expressed in ng/g dry, and systematically corrected for extraction yields to take into account any artefacts or matrix effects observed during the analysis.

**TABLE 3** | Characteristics of the analysed pesticides.

<b>Code</b>	<b>Pesticide</b>	<b>Family</b>	<b>Use</b>	<b>Authorised (yes/ban year)</b>	<b>Koc (mL/g)</b>	<b>Kfoc (mL/g)</b>	<b>Kow (log)</b>	<b>DT50 (days)</b>
ATC	Acetochlor	Herbicide	Pre-emergence	2008	156	NA	4.1	14
ACF	Aclonifen	Herbicide	Pre-emergence	Yes	NA	7126	4.4	117
ATZ	Atrazine	Herbicide	Pre-emergence	2003	100	174	2.7	75
AZS	Azoxystrobin	Fungicide	Cereal	Yes	589	423	2.5	78
BOS	Boscalid	Fungicide	Wide use	Yes	NA	772	3.0	484
CBZ	Carbendazim	Fungicide	Cereal	2009	NA	225	1.5	40
CFV	Chlorfenvinphos	Insecticide	Root pest	2007	680		3.8	40
CTU	Chlortoluron	Herbicide	Cereal	Yes	NA	147	2.5	34
DCPMU	1-(3,4-dichlorophenyl)-3-methylurea	DIU transformation product	NA	NA	NA	928	2.7	127
DCA	Dichloroalanine	DIU transformation product	NA	NA	NA	485	2.7	33
DFZ	Difenoconazole	Fungicide	Seed or spray	Yes	NA	3760	4.4	130
DFE	Diflufenicanil	Herbicide	NA	Yes	5504	2215	4.2	95
DMM	Dimethomorph	Fungicide	Vine	Yes	NA	419	2.7	73
DIU	Diuron	Herbicide	Pre-emergence	2008	680	757	2.9	146
FLZ	Flazasulfuron	Herbicide	Pre- and post-emergence	Yes	46	283	-0.1	41
FZN	Fluazinam	Fungicide	Soil pathogens	Yes	16430	1958	4.9	124
FMX	Flumioxazine	Herbicide	Pre-emergence	Yes	889	NA	2.6	22
FTM	Flurtamone	Herbicide	Pre- and post-emergence	Yes	NA	257	3.2	11
IMD	Imidacloprid	Insecticide	NA	2018	NA	225	0.6	191
IDX	Indoxacarb	Insecticide	Wide use	2021/2024	4483	NA	4.7	113
IPU	Isoproturon	Herbicide	Cereal	Yes	NA	122	2.5	12
IPUD	Isoproturon demethyl	IPU transformation product	NA	NA	NA	147	NA	33
IXB	Isoxaben	Herbicide	Pre-emergence	Yes	909	354	3.9	105
LINU	Linuron	Herbicide	Pre- and post-emergence	2017	842	559	3.0	58
MTM	Metamitron	Herbicide	NA	Yes	77	86	0.9	30
MTMD	Metamitrone deaminated	MTM transformation product	NA	NA	NA	103	NA	31
MTZ	Metazachlor	Herbicide	NA	Yes	54	79	2.5	9
NPM	Napropamide	Herbicide	Pre-emergence	Yes	839	885	3.3	70

(Continues)

TABLE 3 | (Continued)

Code	Pesticide	Family	Use	Authorised (yes/ban year)	Koc (mL/g)	Kfoc (mL/g)	Kow (log)	DT50 (days)
NFZ	Norflurazon	Herbicide	Pre-emergence, industry	2003	700	295	2.5	90
PPZ	Propizamide	Herbicide	Residual post-emergence	Yes	840	1626	3.3	50
SMZ	Simazine	Herbicide	NA	2001	130	750	2.3	60
MTC	S-metolachlor*	Herbicide	Pre-emergence	Yes	120	163	3.4	90
SPX	Spiroxamine	Fungicide	Plants and fruit	Yes	NA	14 567	2.9	25
TBZ	Tebuconazole	Fungicide	Leaf diseases	Yes	NA	769	3.7	63

Note: The physico-chemical characteristics come from the Pesticides Properties Database (<https://sitem.herts.ac.uk/aeru/ppdb/>) consulted in November 2022. Koc is the organic-carbon normalised adsorption coefficient. Kfoc is its equivalent in the case of a non-linear adsorption isotherm (Freundlich). Kow is the octanol–water partition coefficient, given in log. DT50 is the half-life of the molecule in water, expressed in day.

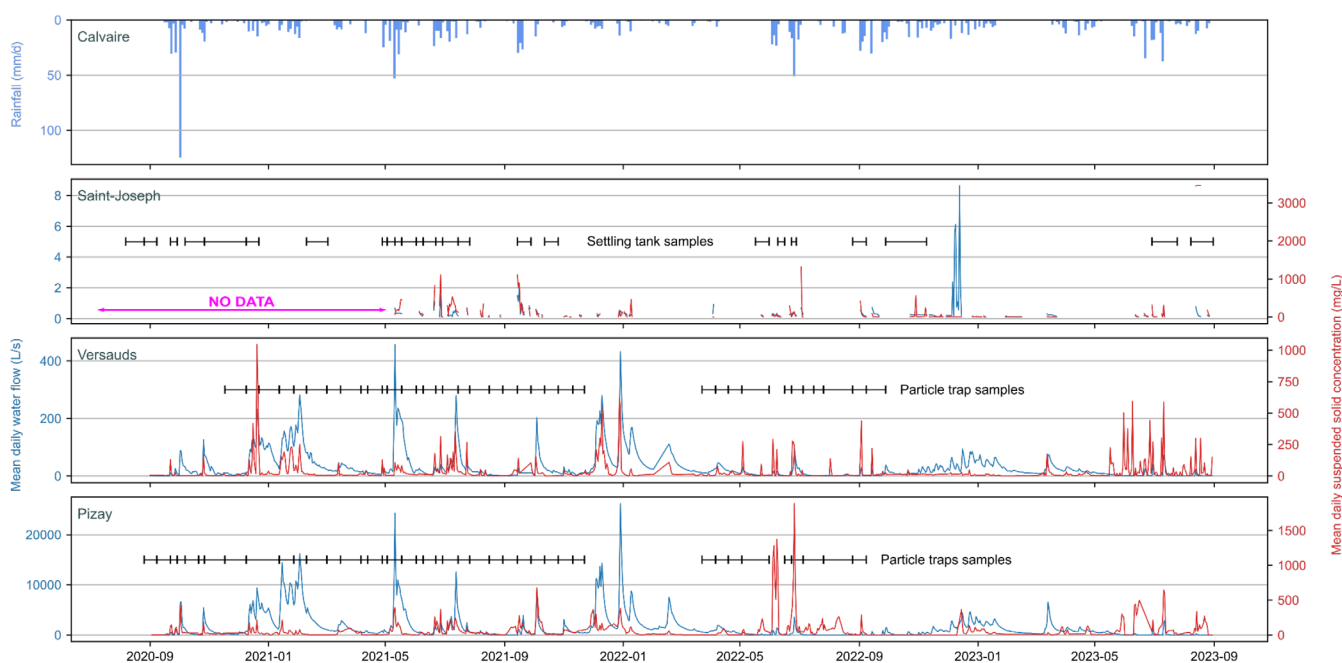


FIGURE 5 | Chronicles of water and solid flows measured at stations on the Ardieres-Morcille sites between September 2020 and August 2023. Precipitation data are taken from the Calvaire station presented in Gouy et al. (2021).

The main characteristics of the 34 selected pesticides are summarised in Table 3: family (herbicide, fungicide, insecticide), main uses, crops and main periods of application, date of ban when the molecule was no longer authorised at the time of the study in 2021, physico-chemical characteristics that may influence environmental fate. In parallel, the quantities applied can be approximated from the “Banque Nationale des Ventes de produits phytopharmaceutiques par les Distributeurs agréés” (BNV-D), which publishes annual quantities purchased by postcode (<https://ventes-produits-phytopharmaceutiques.eauffrance.fr/>).

## 5 | Period and Data Representativeness

In an interpretation perspective, it should be noted that 2020–2022 are ordinary years with a dry trend and the existence of

local particularities (at station scale) on the sediment transfers. Indeed, designing the suitable station on the Saint-Joseph site took some time explaining the flow rate and turbidity recording from, respectively, January and July 2021. Moreover, even if the sampling technique did not result in samples in the upper range, the turbidity values frequently reach the probe limits of quantification due to site characteristics and, in this case, were set at 4000 NTU. At the Versauds station, the granulometry recorded appeared to be high (mostly sandy) which probably leads us to minimise the sediment transfers on this site.

## 6 | Conclusion

This paper introduces a comprehensive hydro-sedimentological dataset on the Ardieres-Morcille scientific observatory (Beaujolais

vineyard, France) for the period 2020–2023. Three nested scales were monitored: the Saint-Joseph plot (0.28 ha), the Morcille sub-catchment (3.9 km<sup>2</sup>) and the Ardières' catchment (143 km<sup>2</sup>). This dataset includes continuous monitoring of rainfall, water level, and turbidity at the three sites, from which discharge and suspended solids concentrations are derived (Figure 5). The dataset is available on BDOH via the Ardières-Morcille observatory webpage (<https://bdoh.irstea.fr/SITE-ATELIER-ARDIERES-MORCILLE/>). The dataset is complete for the period, and further data for water level and turbidity are still being acquired at the river stations and will be extended in time. Besides, discontinuous data on sediments' granulometry, major elements, and particle-bound pesticides were recorded. This dataset is available on <https://doi.org/10.57745/J7GQDW>. By making these data publicly available, we aim to support the scientific community in advancing research on hydrosystem functioning, particularly in relation to the understanding and modelling of sediment transport and the interactions between dissolved and particulate-phase pesticide transfers.

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### Disclosure

The authors have nothing to report.

### Data Availability Statement

All the datasets cited in the Table 1 are stored in the 'Hydrological Observatory Database' (BDOH in French; Branger et al. 2014) and accessible via the Ardières-Morcille observatory webpage (Gouy et al. 2015, <https://doi.org/10.17180/obs.saam>). Discontinuous data on sediments' granulometry, major elements and particle-bound pesticides, as well as the GIS layer of catchment area are available on <https://doi.org/10.57745/J7GQDW>.

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