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ORIGINAL ARTICLE

Dissolved organic carbon loss fluxes through runoff and sediment on sloping upland of purple soil in the Sichuan Basin

Keke Hua · Bo Zhu · Xiaoguo Wang

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Abstract Runoff is a major driver for dissolved organic carbon (DOC) diffusing into aquatic ecosystems. Transport of DOC in runoff is important in the C cycle of soils in an agricultural ecosystem. This study provides a combined dataset on DOC loss pathways and fluxes from sloping upland in the purple soil area of southwestern China. A free-drain lysimeter experiment was conducted to quantify DOC loss through overland flow (2010–2012), interflow (2010–2012) and sediment (2011–2012). Average annual cumulative discharges of overland and interflow were 58.3 ± 3.1 mm and 289.4 ± 5.4 mm, accounting for 6.8 % and 33.8 % of the totals during the entire rainy season, respectively. Average annual cumulative sediment loss flux was 183.5 ± 14.6 g m^{-2}. Average DOC concentrations in overland flow and interflow were 3.44 ± 0.36 and 3.04 ± 0.24 mg L^{-1}, respectively. Average DOC content in sediment was 73.76 ± 4.09 mg kg^{-1}. The relationship between DOC concentration and discharge in overland flow events could be described by a significant exponential decaying function ($R = 0.53$, $P = 0.027$). Average annual DOC loss fluxes through overland flow, interflow and sediment were 163.6 ± 28.5, 865.5 ± 82.5 and 9.4 ± 1.5 mg m^{-2}, respectively, and total DOC loss was $1{,}038.5 \pm 112.5$ mg m^{-2}. The results suggest that interflow is the major driver of DOC leaching loss on sloping upland. It is shown that interflow is fundamentally important for reducing DOC loss on sloping croplands in the Sichuan Basin and possibly beyond.

Keywords Sloping upland · DOC loss · Overland flow · Interflow · Sediment

K. Hua · B. Zhu (✉) · X. Wang
Key Laboratory of Mountain Surface Processes and Ecological Regulation, Institute of Mountain Hazards and Environment, Chinese Academy of Sciences, 610041 Chengdu, China
e-mail: bzhu@imde.ac.cn

K. Hua
Soil and Fertilizer Research Institute, Anhui Academy of Agricultural Sciences, 230031 Hefei, China

Introduction

Dissolved organic carbon (DOC) plays a great role in terrestrial and aquatic ecosystems (Jones et al. 2004; Monaghan et al. 2007; Haaland and Mulder 2010). DOC may enhance the sorption and mobility of pesticides and heavy metal in surface waters and leads to drinking water quality problems (David et al. 1991; Li and Shuman 1997; Li et al. 2005). Loss of DOC from agricultural soil occurs at the expense of both soil organic carbon and water quality. Runoff is a major driver for DOC loss from soil to aquatic ecosystems. Recent research focused on DOC concentrations and fluxes in overland flow determined by hydrological processes interacting with the biogeochemistry of terrestrial and aquatic ecosystems (Asano et al. 2009). Furthermore, transport of DOC from soil to runoff is a major mechanism of soil organic carbon loss (Lohse et al. 2009; Froberg et al. 2005).

Transport of DOC in runoff is the complicated biochemical soil processes causing by producing, adsorption and desorption of DOC in soil (Kalbitz et al. 2000; Fujii et al. 2009). Several studies show that two basic conditions both accumulating of soil DOC and movement of soil water should be need simultaneously forming DOC transport in runoff (Michalzik et al. 2003). Rainfall is the main reason causing soil water movement and is also the major driving force for the transport of soil nutrients in runoff. Several results show significant positive correlations between DOC loss discharges and rain events in many catchments (Grieve 1994; Dawson et al. 2002). A great deal of attention has been paid to DOC transport via overland flow in agricultural ecosystems. It was reported that there is a significant relationship between transport of DOC and discharge of overland flow in response to rain events (Dawson et al. 2002). Very few studies, however, pay attention to DOC loss through interflow and sediment from sloping upland. Thus, it is necessary to integrate DOC loss through overland flow, interflow and sediment in order to accurately estimate DOC loss in hydrological process. More specially, there were few papers reporting which was the main hydrological pathway of DOC loss on sloping cropland.

In China, more than 69 % of agricultural lands are located on hills or mountains (Zhong 2000). The hilly area of purple soil in the Sichuan Basin is one of the most important agriculture areas in southwestern China, with an area of 160,000 km^2 (Li et al. 1991).The sloping upland distributed widely in the area has been degraded by severely water erosion. There is tremendous soil organic carbon loss during summer storms due to abundant rainfall (Guo et al. 2008). A great deal of attention has been paid to nutrients transport through overland flow and sediment from sloping cropland of purple soil in the Sichuan Basin, southwestern China (Zhu et al. 2009). In this area, purple soil is typically characterized by thin soil layers and nutrients loss through interflow is a prevailing phenomenon during the rainy season (Wang and Zhu 2011; Zhou et al. 2012). The soil profile is relative thin and easily saturated by rainwater during the rainfall events. There is an obvious soil–bedrock interface for purple soil in which the vertical infiltrating water could quickly reach the interface and turn into interflow moving along the slope (Zhu et al. 2009). However, magnitude of DOC loss through interflow remains uncertain in this area.

Consequently, the purpose of the study was to evaluate the DOC loss flux through overland flow, interflow and sediment on sloping upland of purple soil. Specific objectives were addressed in the study: (1) to quantify DOC loss flux integrating overland flow, interflow and sediment (2) to find out the major pathway of DOC loss on sloping upland of purple soil.

Materials and methods

Site description

The experiment site is located at Yanting Agro−Ecological Station of Purple Soil (N31°16′, E105°28′) at an altitude of 400−600 m in the middle of Sichuan Basin, Southwest China. The station is a member of the Chinese Ecosystem Research Network of Chinese Academy of Science (CERN). The experimental sites have a moderate subtropical monsoon climate with an annual mean temperature of 17.3 °C and mean precipitation of 826 mm over the past 20 years. There are 5.9, 65.5, 19.7 and 8.9 % rainfall occurred in spring, summer, autumn and winter respectively, during the total annual precipitation. Annual precipitations during the entire experiment were 892 mm in 2010, 1,061 mm in 2011 and 1,080 mm in 2012, respectively (Fig. 1).

Soil

The soil is called purple soil and classified as a Pup−Orthic Entisol in the Chinese Soil Taxonomy and an Entisol in the US. Soil Taxonomy due to its color (Gong 1999). Rainfed farming has been maintained on the soil with slope gradients of 3−15 % and shallow soil layer of about 30–80 cm. The soil profile is derived from purplish shale and with a typical "binary structure of soil–bedrock" (Xiong and Li 1986). Interface flow could be generated at the soil–bedrock interface due to different soil water conductivity between soil and bedrock (Zhu et al. 2009). The specific soil used in this study is a loam soil with pH 8.3, a bulk density of 1.33 g cm^{-3}, organic matter content of 8.75 g kg^{-1}, total N content of 0.62 g kg^{-1}, alkali−hydrolyzed N content of 42.29 mg kg^{-1}, and saturated hydraulic conductivity of 16.8 mm h^{-1}.

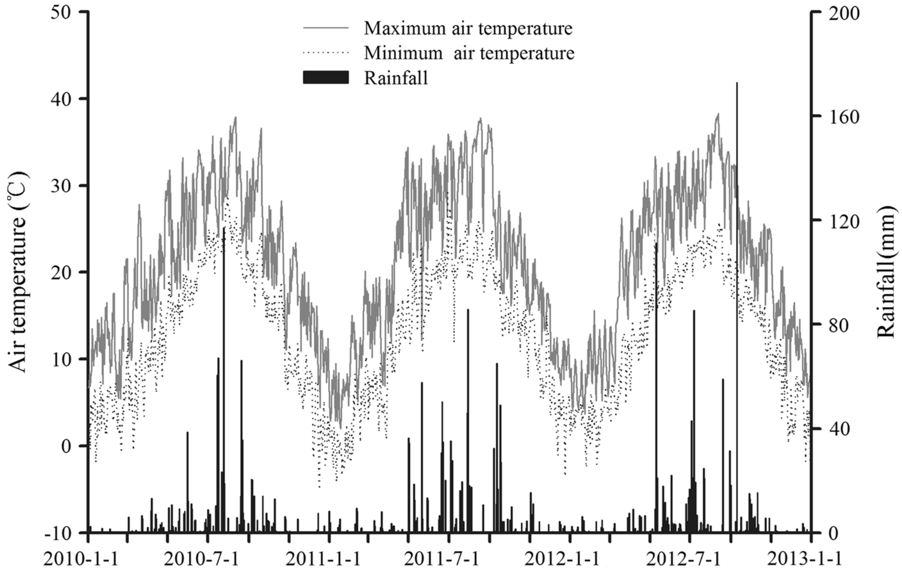


Fig. 1 Daily precipitation, daily maximum and minimum air temperature from 2010 to 2012

Experimental setup

Hillslope hydrologic pathway and runoff plot setup

The purple soil profile of the experimental cropland is shallow and beneath the soil is parent bedrock with poor water conductivity (Li et al. 1991). There is more than 75 % of the annual precipitation in the summer season (from May to October) and the soil profile is easy to be saturated due to poor water conductivity. Hence, the soil water potentially drives overland flow and interflow moving downward along the slope. Based on the sloping upland hydrologic characteristics, field runoff plots were designed and constructed in 2001. The free-drained-lysimeters were previously excavated 11 years ago. The bedrock along the slopes was in the same gradients with upland slope in 6.5° at that time. Then, soil was backfilled with its natural order. After 11 years' deposition, the soils become natural and there are no water logged areas along the bedrock. Meanwhile, to avoid lateral seepage from adjacent plots, each plot was hydrologically isolated with partition walls filled with cement down to the bedrock to a depth of at least 60 cm. The partition walls were built in top and lateral edges of the plots. (Patent: ZL2007100640686) (Zhu et al. 2009). A conflux trough was built at the soil surface and at the soil–bedrock interface to collect both overland flow and interflow (Fig. 2). The collection trough edges for overland flow are lower than the soil surface with 15 cm' difference to ensure overland flow collected completely (Fig. 2b, c). The interflow conflux trough was excavated 10 cm below the soil–bedrock interface and filled with clean arenaceous quartz and pebble till to the level of the soil–bedrock interface. Buckets were installed under each conflux through to collect water samples from both overland flow and interflow. The plots have an area of 8 m (length) by 4 m (width), with a slope gradient of 6.5° and soil depth of 60 cm. The experimental plots were laid out in a randomized block design with three replications. The experimental plots were cropped conventionally with wheat (*Triticum aestivum* L.) from late October to May of the next year, and then rotated with summer maize (*Zea mays* L.) from May through September. Mineral fertilizer was applied once in the beginning of each crop growing season at rates of 130 kg N ha^{-1}, 39 kg P ha^{-1}, and 30 kg K ha^{-1} in the wheat growing season. The maize growing season was applied at the rates of 150 kg N ha^{-1}, 39 kg P ha^{-1}, and 30 kg K ha^{-1}, respectively. The fertilization and crop rotation scheme represent common practice in the region.

Water sampling

Overland flow and interflow water samples were taken separately from the different buckets in each rain event when the water flow stopped completely. Polyethylene bottles of 500 mL were adopted to collect water

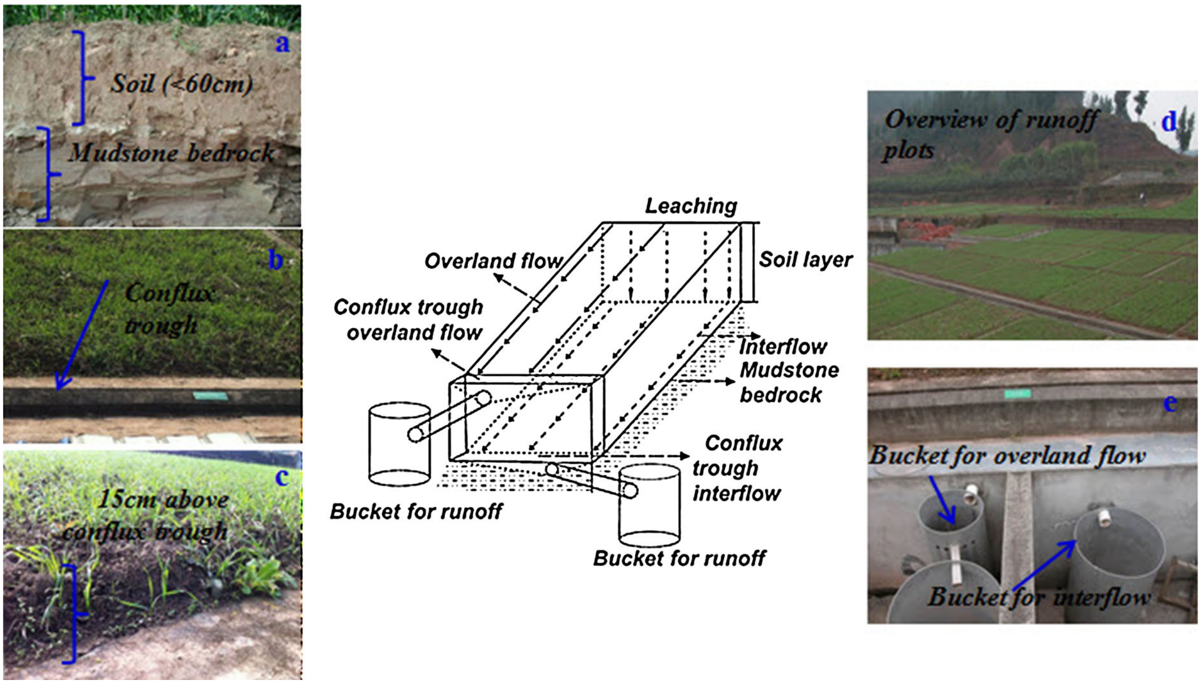


Fig. 2 Schematic illustration of runoff plot structure on the sloping upland of purple soil. Soil profile (**a**); Conflux trough for overland flow (**b**); Height away from conflux trough edges (**c**); Overview of runoff plots (**d**); Collection buckets for overland flow and interflow (**e**)

samples to analyze DOC concentration after the water levels were measured. In addition, the methods for collecting overland flow and interflow samples were obviously different. In overland flow, water and sediment in the buckets were fully roiled and a 500 ml volume polyethylene bottle for runoff sample was taken. After settling for 48 h in collectors, the runoff water was discarded and the sediment was dried in the oven at 105 °C for 24 h. The amount of dry sediment was weighed to determine the sediment loss. In contrast, interflow water in the buckets was collected directly from each bucket for interflow.

Rainfall and intensity measured

Rainfall quantities were measured with an automatic tipping bucket gauge (R13, Vaisala, Finland). This instrument uses a tipping-bucket mechanism to produce a contact closure every time it receives a predetermined small quantity of rainfall (0.2 mm). Rainfall intensity was measured by a siphon rainfall recorder, and maximum rainfall intensity with error 0.1 mm was obtained by intensity recording paper.

Analytical methods

Soil physical and chemical characteristics were analyzed using standard methods (Lu 1999). Water samples of overland flow and interflow for DOC analysis were filtered through 0.45 μm membrane, DOC concentration in filtrate was automatically analyzed by flow injection technology through a special DOC module with AA_3 Auto-analyzer (Bran + Lubbe, Norderstedt, Germany). 2.5 g of moist sediment was shaken with 25 mL distilled water (1:10 w/v) for 120 min in 50 cm^3 polypropylene bottles on a reciprocating shaker at a speed of 200 rev min^{-1}. The sediment extracts were then centrifuged at 5,000 rev min^{-1} for 30 min (Jones and Willett 2006) and were filtered through 0.45 μm membrane, DOC concentration in the filtrate was automatically analyzed by flow injection technology through AA_3 Auto-analyzer.

Data analysis

Water levels in the buckets were measured using a ruler after each runoff event during 2010–2012 in order to calculate the runoff discharges. To decrease errors caused by measuring manually, water levels in the buckets were measured 4 times and means of the measured water levels were determined.

The DOC loss flux in an individual flow (Q_i) produced for each rainfall event was calculated as:

$$Q_{\mathrm{i}} = C_{\mathrm{i}} \times q_{\mathrm{i}} \tag{1}$$

Where Q_{i} is DOC loss flux for overland flow or interflow (mg m^{-2}), C_{i} is DOC concentration in overland flow or interflow water (mg L^{-1}); q_{i} is the runoff depth per unit (mm).

The annual cumulative DOC loss flux was calculated as:

$$Q = \sum_{i=1}^{n} Q_i \tag{2}$$

Where Q indicates the annual cumulative DOC loss flux (mg m^{-2}), $\mathrm{i} = 1 \sim n$ (n is the number of runoff event in the entire year).

Runoff coefficient (RC) for overland flow and interflow was calculated as:

$$RC = q/R \tag{3}$$

Where q is the cumulative runoff depth in overland flow or interflow per unit from May to October (mm), R is cumulative rainfall from May to October (mm). All the statistical analysis was performed with SPSS 13.0 software package (SPSS, Inc., USA). Significant differences were analyzed using ANOVA, followed by the least significant difference test (LSD, $P < 0.05$ or 0.01 levels). Sigma plot 10.0 was used for graph preparation (Systat Software, Inc., Chicago, IL, USA).

Results

Water discharge through overland flow and interflow

During the whole experiment period, there were 26 rainfall events observed in the three rainy seasons from 2010 to 2012. Rainfall ranged from 16.2 to 177.1 mm per event and max rainfall intensity ranged from 1.1 to 60 mm h^{-1} (Fig. 3a). There were 17 overland flow events and the observed overland flow discharges ranged from 1.5 to 53.9 mm (Fig. 3b). Annual cumulative overland flow discharges through overland flow were 43.7 ± 2.2 mm, 59.4 mm ± 3.6 and 71.7 ± 3.4 mm for 2010, 2011 and 2012, respectively. In addition, there were 23 interflow events observed during the whole experiment. Interflow discharges ranged from 1.4 to 121.6 mm during the entire period (Fig. 3c). Annual cumulative interflow discharges were 269.6 ± 5.2 mm, 299.1 ± 3.9 mm and 299.6 ± 7.1 mm for 2010, 2011 and 2012, respectively. Average annual cumulative discharge for interflow was 289.4 ± 5.4 mm. Average runoff coefficient for overland flow and interflow in the three years were 6.8 % and 33.8 %. The discharge of interflow was 5 times higher than that of overland flow. The results showed that the discharge through interflow was the main pathway in runoff discharge on sloping upland of purple soil.

Sediment concentration and loss flux

Figure 4 presents average sediment concentrations and loss fluxes for all overland flow events monitored from 2011 to 2012. Sediment concentration and loss flux could not be analyzed because of data loss in 2010. Based on the measured data, sediment concentration through each overland flow event ranged from 0.2 to 6.8 g L^{-1}, with an average 1.5 ± 0.08 g L^{-1} for the entire experimental period. Average sediment concentrations in 2011 and 2012 were 1.76 ± 0.07 and 1.19 ± 0.10 g L^{-1}, respectively. From 2011 to 2012, the efflux of sediment ranged from 0.8 to 194.2 g m^{-2} per rainfall event. Average sediment loss fluxes were 20.43 ± 1.46 and 33.9 ± 2.95 g m^{-2} for 2011 and 2012, respectively. Annual estimated cumulative sediment loss fluxes in 2011 and 2012 were 163.45 ± 11.68 and 203.49 ± 17.59 g m^{-2}, respectively, and average annual cumulative sediment loss flux was 183.47 ± 14.64 g m^{-2} per year.

DOC concentration and loss flux through overland flow

Dissolved organic carbon concentrations and loss fluxes for all the overland flow events were measured for the three full rainy seasons from 2010 to 2012 (Fig. 5). During the full experimental period, DOC concentrations in overland flow ranged from 1.68 to 5.34 mg L^{-1}, with average 3.44 ± 0.36 mg L^{-1}. Average DOC concentrations were 3.93 ± 0.50, 3.50 ± 0.32 and 3.10 ± 0.33 mg L^{-1} for 2010, 2011 and 2012, respectively. The efflux of DOC

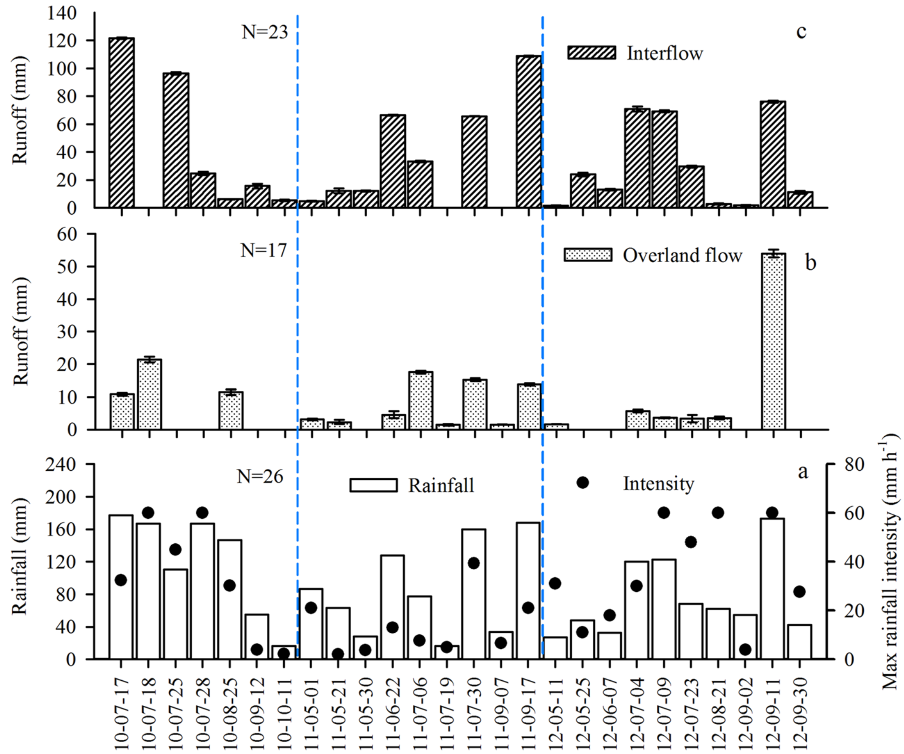


Fig. 3 Runoff discharges in each rain event and its response to rainfall and rainfall intensity from 2010 to 2012. Rainfall and max rainfall intensity (**a**), overland flow (**b**) and interflow (**c**), *Vertical bars* indicate the standard deviation of three different replicates

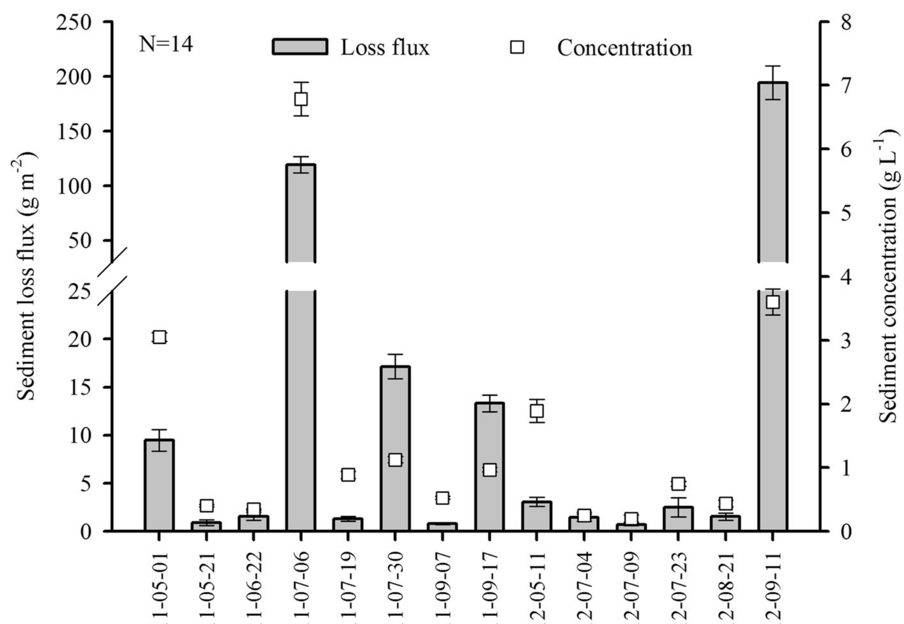


Fig. 4 Sediment concentration and loss flux in each overland flow event from 2011 to 2012. *Vertical bars* indicate the standard deviation of three different replicates

through overland flow events was from 5.3 to 90.4 mg m^{-2} per event. Average DOC loss fluxes were 57.1 ± 9.8, 21.2 ± 3.5 and 11.9 ± 0.6 mg m^{-2} per rainfall event for 2010, 2011 and 2012, respectively. Annual cumulative DOC loss fluxes through overland flow water were 171.2 ± 29.4, 169.8 ± 28.2 and 149.8 ± 27.9 mg m^{-2} for 2010, 2011 and 2012, respectively. Average annual cumulative DOC loss flux through overland flow for the 3 years period was 163.6 ± 28.5 mg m^{-2}.

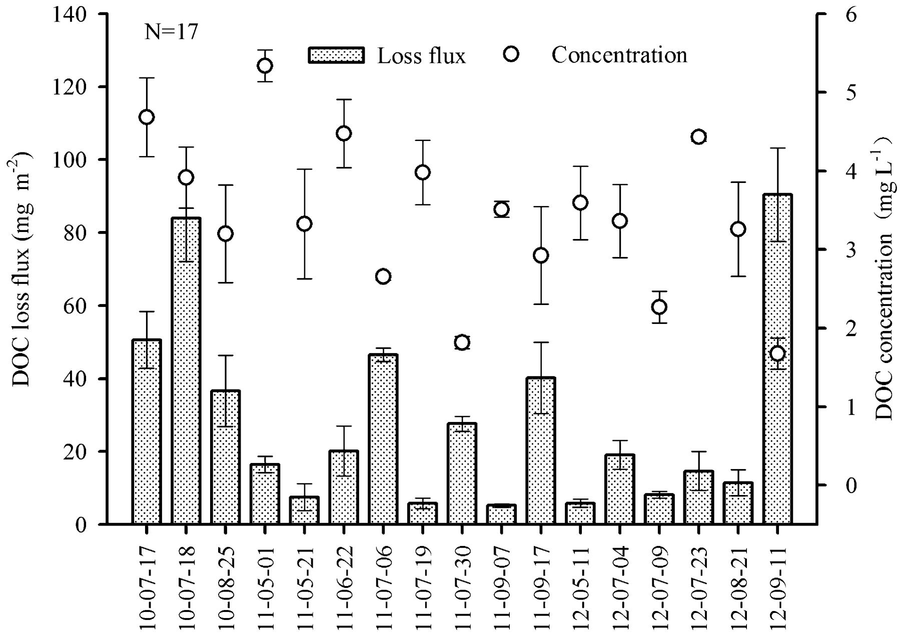


Fig. 5 DOC concentration and loss flux of each overland flow event from 2010 to 2012. *Vertical bars* indicate the standard deviation of three different replicates

DOC concentration and loss flux through interflow

During the entire experiment period, DOC concentrations through interflow were from 1.45 to 4.71 mg L^{-1} with average 3.04 ± 0.24 mg L^{-1} (Fig. 6). Average DOC concentrations were 2.80 ± 0.29, 3.47 ± 0.24 and 2.88 ± 0.21 mg L^{-1} for 2010, 2011 and 2012, respectively. Efflux of DOC through overland flow events ranged from 3.0 to 402.4 mg m^{-2} per event. Average DOC loss fluxes were 130.4 ± 13.7, 150.6 ± 12.1 and 76.1 ± 8.0 mg m^{-2} per rainfall event for 2010, 2011 and 2012, respectively. Corresponding, annual cumulative DOC loss fluxes through interflow were 782.1 ± 82.4, 1,054.3 ± 84.9 and 760.1 ± 80.2 mg m^{-2}. Average annual cumulative DOC loss flux through overland for the 3 years period was 865.5 ± 82.5 mg m^{-2}. Annual cumulative DOC loss flux differed between the 3 years.

DOC content and loss flux through sediment

Based on the observed data, DOC content in sediment was from 35.11 to 106.13 mg kg^{-1}. Average DOC contents for 2011 and 2012 were 82.68 ± 6.17 and 64.83 ± 2.01 mg kg^{-1} per rainfall event (Fig. 7). DOC efflux in sediment per event ranged from 0.03 to 7.0 mg m^{-2}. Average DOC efflux in sediment was 1.4 ± 0.2 and 1.3 ± 0.2 per rainfall event for 2011 and 2012, respectively. Corresponding, annual cumulative DOC loss flux through sediment was 11.2 ± 1.8 and 7.6 ± 1.1 mg m^{-2} with average 9.4 ± 1.5 mg m^{-2}.

Distribution of DOC loss through overland flow, interflow and sediment

Overland flow, interflow and sediment were the three major hydrologic pathways of DOC loss from soil to water. The annual cumulative DOC loss fluxes through the three pathways were analyzed (Table 1). Average annual cumulative total DOC loss flux through runoff and sediment was 1,038.5 ± 112.5 mg m^{-2}. Average annual cumulative DOC loss fluxes through overland flow, interflow and sediment were 163.6 ± 28.5, 865.5 ± 82.5 and 9.4 ± 1.5 mg m^{-2}, presenting for 15.8 %, 83.3 % and 0.9 % of the total flux, respectively. Compared with overland flow and interflow, DOC loss flux in sediment was low. The cumulative DOC loss flux through interflow was 5 times greater than that through overland flow. This indicates that interflow is the main transport pathway of DOC loss in the rainy season on sloping upland of the purple soil.

Fig. 6 DOC concentration and loss flux of each interflow event 2010–2012. *Vertical bars* indicate the standard deviation of three different replicates

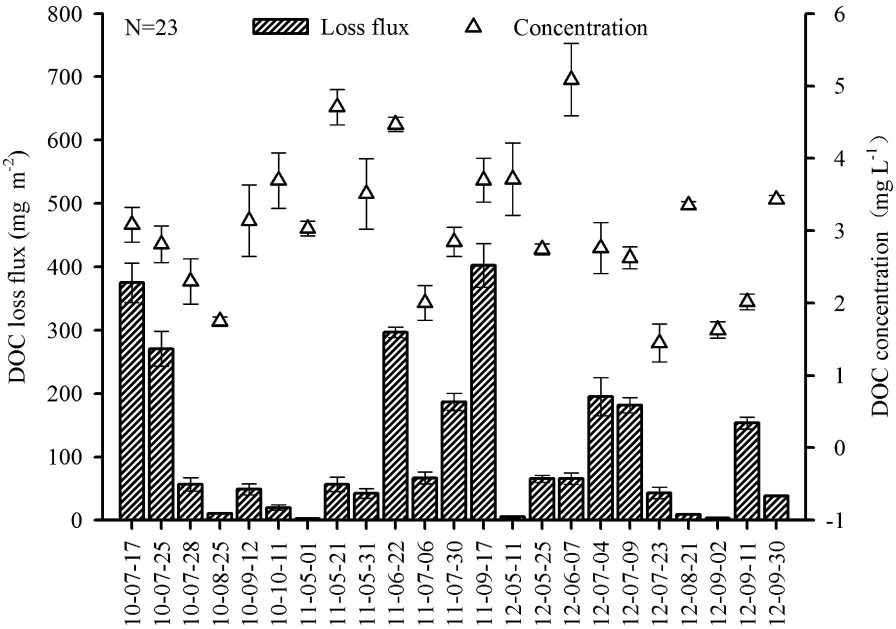
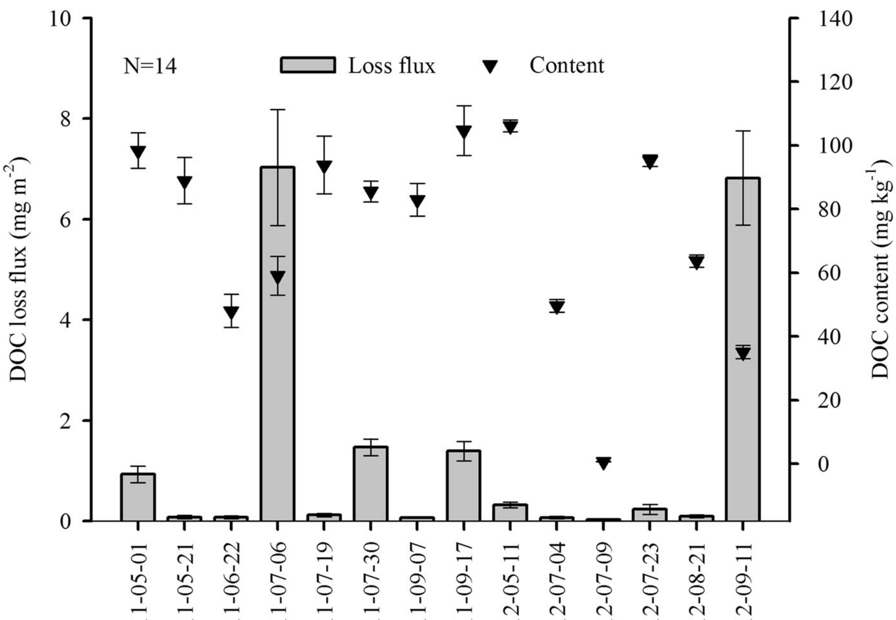


Fig. 7 DOC concentration and loss flux of each overland flow event from 2011 to 2012. *Vertical bars* indicate the standard deviation of three different replicates



Discussion

Relationship between runoff discharge and DOC concentration

The key finding of the present study is that overland discharge is an important regulating factor of DOC concentration in overland flow on the sloping upland. By contrast, there was no significant relationship between runoff discharge and DOC concentration through interflow ($R = -0.05$, $P = 0.819$). Several studies have reported that solute transfer from the soil surface to overland flow is greatly complicated due to the fact that there are many complex processes occurring simultaneously. These processes include the transfer of solutes from soil surface by diffusion,

Table 1 Ratio (%) of DOC loss flux (mg m^{-2}) through overland flow, interflow and sediment accounting for total DOC loss

Year	Overland flow		Interflow		Sediment		Total
	DOC flux	Ratio	DOC flux	Ratio	DOC flux	Ratio	DOC flux
2010	171.2 ± 29.4[a]	–	782.1 ± 82.4[b]	–	–	–	–
2011	169.8 ± 28.2[a]	13.75	1,054.3 ± 84.9[a]	85.35	11.2 ± 1.8	0.90	–
2012	149.8 ± 27.9[b]	16.33	760.1 ± 80.2[b]	82.84	7.6 ± 1.1	0.83	–
Mean	163.6 ± 28.5		865.5 ± 82.5		9.4 ± 1.5		1,038.5 ± 112.5

Mean ± SD; means shown in each column followed by the same letter identifier are not significantly different (LSD test, $P > 0.05$)

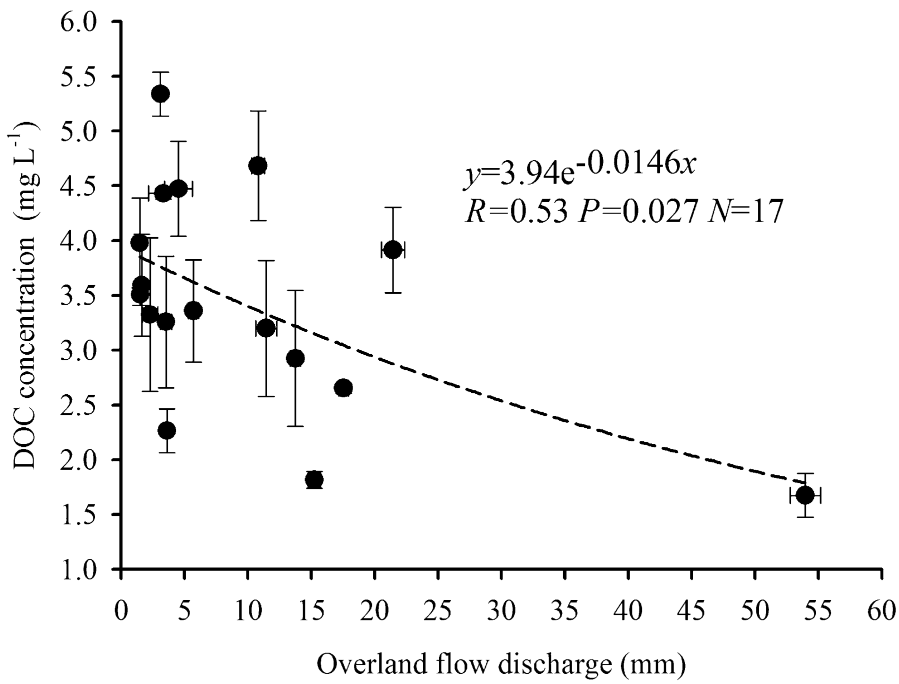


Fig. 8 Correlation relationship between DOC concentration and runoff discharge. *Vertical bars* indicate the standard deviation of three different replicates

ejection of solution from the soil surface by raindrops, erosion by raindrops and surface flow of sediment with adsorbed chemicals and adsorption–desorption of the adsorbing chemicals (Shi et al. 2011). The influence of rainfall on solute transfer at soil surface is reflected in increasing overland flow and vertical mechanical actions to soil surface (Fraser et al. 1999; Kleinman et al. 2006). The runoff discharge can either increase or decrease the solute concentration of overland flow depending on the type of solutes. For instance, Kleinman et al. (2006) reported that dissolved reactive P concentrations are positively correlated while NO_3^- concentrations are negatively correlated to runoff discharge. Similarly, in the present study, a strong relationship between runoff discharge and DOC concentration through overland flow was observed, which could be described best by a significant exponential decaying function (Fig. 8). Thus, our study indicates that overland discharge is a potential regulating factor of DOC concentration in surface runoff, especially in regions as ours where high overland flow occurred. Since the interaction between soil DOC and runoff is extremely complicated, other major factor influencing DOC concentrations through overland flow should be father studied by artificial simulated rainfall.

The dominant hydrological pathway of DOC loss on sloping upland

DOC loss is an interactive process between soil DOC and runoff water movement (Martin 2003).

Despite several documents reported DOC concentration and loss flux via overland flow (Asano et al. 2009; Lohse et al. 2009), few papers simultaneously monitored DOC loss through interflow and sediment at a plot scale. Hence, it is difficult to accurately estimate DOC loss flux in hydrological process. Our original hypothesis was that interflow dominated the total runoff of sloping upland in the area, thereby DOC loss via interflow was probably the major DOC loss pathway in hydrological processes. By comparing the DOC loss flux through interflow and those in overland flow and sediment, we found that interflow was the dominant hydrological pathway of DOC loss, which was further supported by the field plot observations during the 3 years. It is fatherly suggested that interflow water is the major driving force for the transport of DOC. It is mainly attributed to the characteristic of interflow abundant in the area. Purple soil is characterized by coarse texture, weak water retention, and higher saturated hydraulic conductivity and well-developed macropores. Zhu et al. (2009) also demonstrated that interflow was the predominant water flow pattern due to prevalent macropore flow. Overland flow only accounted for a very small part of the total runoff in this area (Wang and Zhu 2011; Zhou et al. 2012). In the area, the soil profile is relatively thin and can be easily saturated by rainwater during the events (Li et al. 1991). When the saturated soil water was formed, the vertical infiltrating water can quickly reach the soil–bedrock interface fatherly, turning into interflow lateral moving along the slope and result in a high average flow rate in the total water runoff (Wang and Zhu 2011). Consequently, the efforts to reduce DOC loss in hydrological process should take into account the DOC loss through interflow. Any soil organic carbon (SOC) loss in hydrologic process mitigation strategy needs to consider the way of DOC loss on sloping upland. In addition, the spatial heterogeneity of hillslope hydrology has been widely recognized in hydrological studies, which is major challenge in accurately estimating DOC loss via hydrological process and other similar catchments. Our DOC loss flux monitoring was limited to a relatively small plot scale (32 m^2), which constrained the interpretation of results to upscaling. A monitoring of larger sloping runoff plots would better reveal the DOC loss via hydrological processes.

Conclusions

Average annual DOC loss flux through interflow was 865.5 ± 82.5 mg m^{-2}, which was 5 and 92 times higher than those via overland flow and sediment. Our results suggest that interflow is the major driver of DOC leaching loss on sloping upland. Interflow is fundamentally important for reducing DOC loss on sloping croplands in the Sichuan Basin.

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