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Dissolved organic carbon concentrations in runoff from shallow heathland catchments: effects of frequent excessive leaching in summer and autumn

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Abstract Transport and turnover of dissolved organic carbon (DOC) is important in the C cycle of organic soils. The concentration of DOC in soil water is buffered by adsorption to the soil matrix, and has been hypothesized to depend on the pool size of adsorbed DOC. We have studied the effect of frequent artificial excessive leaching events on concentration and flux of DOC in shallow, organic rich mountain soils. Assuming a constant *Kd* value for DOC adsorption to the soil matrix, we used these data to assess the change in the pool of adsorbed (or potential) DOC in the soil. The study involved manipulation of precipitation amount and frequency in summer and autumn in small, heathland catchments at Storgama, southern Norway. The shallow soils (16–34 cm deep on average) limit the possibility for changes in water flow paths during events. The mini-catchments range in size from 75 to 98 m^2. Our data show that after leaching of about 1.2 g DOC m^{-2} the DOC concentration in runoff declines by approximately 50%. From this we conclude that the pool size of adsorbed potential DOC in the shallow soils at any time is of the order 2–3 g m^{-2}. Frequent episodes suggest that the replenishment rate, which depends on the decomposition rate of soil organic matter, is fast and the potential DOC pool could be fully restored probably within days during summer, but with some more time required in autumn, due to lower temperatures. Both pool size of potential DOC and replenishment rate are seasonally dependent. The pool of potential DOC, and thus the DOC concentration in discharge, is at their maximum in the growing season. However, under non-leaching conditions, the concentration of DOC in soil water and thus the pool size of potential DOC seems to level off, possibly due to conversion of DOC to less reversibly bound forms, or to further decomposition to CO_2.

Keywords Dissolved organic carbon · Field manipulation · Catchment · Pool adsorbed potential DOC · Leaching · Replenishment rate

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Introduction

Dissolved organic carbon (DOC), derived from litter and soil organic carbon (SOC) (Fröberg 2004; Michalzik and Matzner 1999; Müller et al. 2009; Qualls et al. 1991), is an important constituent for biological, chemical and physical processes in soils and waters (Steinberg et al. 2006; Wetzel 2001). There are several processes might control the transfer between SOC and DOC, such as the production rate of DOC by microbial and root exudation or microbial turnover rates of soil organic matter, sorption/desorption processes, accessibility of mobile water and hydrology (Qualls 2000; Worrall et al. 2008). Metabolic transformation of organic matter to DOC is also temperature dependent (Kalbitz et al. 2000; Michalzik et al. 2003). After its production, DOC may be leached from the soil, but it also may be stored temporarily, due to adsorption to the soil matrix (i.e. Tipping et al. 2005; Fig. 1). This adsorbed pool buffers the concentration of DOC in soil water and runoff (Tipping 1998), so that the concentrations of DOM often are reported to be relatively unaffected by water fluxes (i.e. Solinger et al. 2001). Recently, this was confirmed in field experiments, where modest amounts of artificial precipitation (10 mm/week), evenly distributed in the growing season in mini-catchments with thin organic soils on bedrock, did not significantly affect the concentration of DOC in runoff (Haaland et al. 2008). Hence, increased precipitation in the growing season does increase the flux of DOC at a rate nearly proportional to the increase in water flux. This suggests that a significant reservoir of adsorbed potential DOC is accumulated in the soil and that leaching is mainly regulated by water throughput.

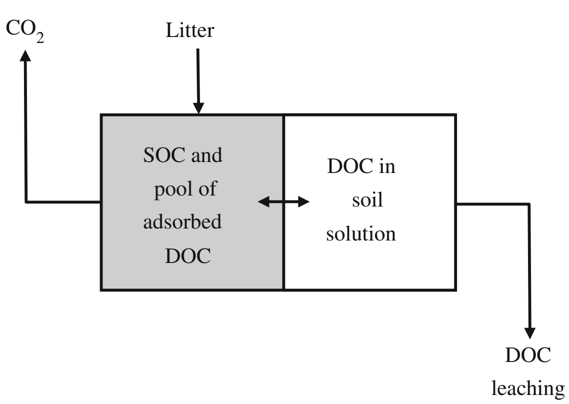

Fig. 1 Adsorbed potential DOC leaves the soil mainly as CO_2 to the atmosphere or as DOC through leaching. DOC is produced from litter and decomposition of SOC and may occur in solution, but it may also adsorb to the soil matrix as potential DOC (Tipping 1998). Principles adapted from the DyDOC model (Michalzik et al. 2003; Tipping et al. 2005)

There is substantial uncertainty about processes that control DOC dynamics in soils and runoff water, even in shallow soils where event-dependent flow paths are of minor concern. For example, do we know little about the in situ pool size of adsorbed potential DOC, and its depletion and replenishment rates (i.e. McDowell 2003). Estimates of potential DOC have been done before in laboratory studies (i.e. Qualls 2000), still a better understanding of this store of potential DOC in soils and how it varies in time is crucial for improving existing models for DOC dynamics in soils and runoff water (i.e. Futter et al. 2007; Michalzik et al. 2003; Neff and Asner 2001; Tipping et al. 2005; Worrall et al. 2008). Therefore, the main objectives of the present study were to estimate the pool size of adsorbed potential DOC, and the rate of leaching and replenishment in field conditions.

Using artificial precipitation events, we estimated the leaching of DOC and the pool size of potential DOC in the soil. To assess replenishment rates of potential DOC as well as seasonal differences, artificial events were repeated at short term intervals both in summer and autumn.

The study was conducted in relatively simple systems, viz. non-forested heathland mini-catchments in southern Norway, with shallow organic soils (Christophersen et al. 1982; Haaland et al. 2008; Strand et al. 2008). In the shallow, organic-rich soils (average depth 16–34 cm) water flowpaths are not likely to vary much in response to hydrological conditions. Thus, they are unlikely to play a major role in explaining changes in runoff DOC concentrations during the artificial events. The selected mini-catchments provide unique opportunities to conduct full scale in situ manipulation experiments involving DOC leaching in response to increased precipitation in summer and autumn.

Methods

Site description

The Storgama catchment (8°32′E, 59°01′N, altitude 450–600 m), is located in Telemark County, Southern Norway (Fig. 2). The bedrock consists of acid, crystalline granite. At about 30% of the small catchments bedrock occurs at the surface, whereas

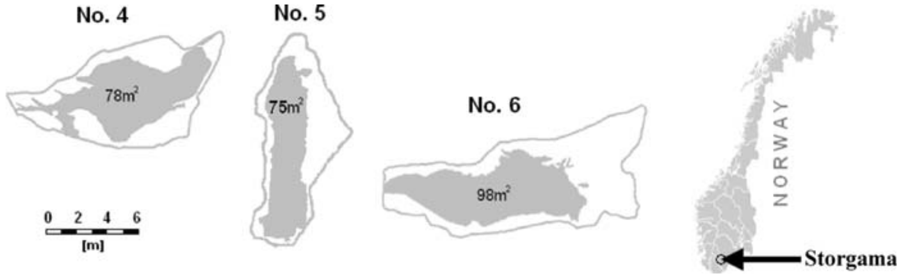

Fig. 2 Catchments number 4, 5 and 6 at Storgama. Soils and vegetation are in *grey*, whereas exposed (lichen covered) bedrock is in *white*. Values represent total surface area in each catchment. Average soil depth is about 0.1 m to 0.3 m and the total relief within the catchments is 1–2 m. The catchments are situated close to each other and within a radius of about 50 m

the remainder is covered by shallow soils rich in organic matter. Soils are classified (FAO-Unesco) as Folic Histosols and Dystric or Lithic Leptosols (Strand et al. 2005). The soils have low pH and base saturation (BS), relatively high effective cation exchange capacity (ECEC), and high content of soil organic matter (SOM) (Strand et al. 2008). The vegetation is mainly Calluna heathland with unproductive and spread Scots pine (*Pinus sylvestris* L.), birch (*Betula pubescens* L.) and junipers (*Juniperus communis* L.). Moorgrass (*Molina coerulea* L.) and peat moss (*Sphagnum* sp.) are predominant in depressions, while heather (*Calluna vulgaris* L.) dominates elsewhere. Concentrations of DOC in runoff are in general smallest after snowmelt (about 2–5 mg l^{-1}) and greatest during the growing season (about 10–15 mg l^{-1}; Austnes et al. 2008; Strand et al. 2008). Mean annual (1961–1990) temperature and precipitation amounts at the nearest official meteorological station (Tveitsund) is 5.7°C and 993 mm. The greater Storgama catchment, situated at slightly higher elevation than the mini-catchments selected for the present study, has been used as a permanent monitoring site for runoff chemistry since the early 1970s (Johannessen and Joranger 1976).

Experimental design

Artificial episode studies with fixed precipitation intensity on small catchments with shallow soils were conducted to quantify the effect of leaching on DOC concentrations in runoff water. The accumulated leached DOC per event, together with the change in DOC concentration in catchment runoff were used to estimate the pool size of potential DOC for each event (Table 2). For the study we selected three small heathland catchments (viz. catchments 4, 5 and 6), which are 75, 78 and 98 m^2 in size, respectively (Fig. 2). The selected catchments were just below the watershed divide of the greater Storgama catchment (Strand et al. 2008). Catchment characteristics and soil properties are presented in Table 1. The catchments are surrounded by exposed bedrock, partly covered by lichens, defining the catchment's natural boundaries (Fig. 2; Christophersen et al. 1982). The episode studies were conducted in 2005 and 2006, both in summer and autumn. In autumn 2005 and summer 2006, short artificial precipitation episodes (35–70 mm of artificial precipitation in 6–10 h) were repeated with 1–3 weeks intervals (Table 2). In addition to, prolonged artificial episodes (182 and 237 mm, respectively, in 2 days) were conducted in autumn 2006.

In the evaluation of the results we also re-analysed data from a previously published experiment (Haaland et al. 2008), with respect to the effect of increased weekly precipitation on the accumulated flux of leached DOC throughout the growing season. In this experiment, precipitation in two catchments (catchments 4 and 6) was increased artificially with 10 mm per week between July 1 and October 15 in 2004, 2005 and 2006, respectively. Precipitation was added at night at about 4.0 mm h^{-1}, using a sprinkler system (see below). Five small heathland catchments at Storgama, receiving natural precipitation only, were used as references (Haaland et al. 2008; Strand et al. 2008).

Episode studies; set-up and sampling

Water from a nearby oligotrophic pond was transported (by gravitation) through polyethylene (PE)

Table 1 Study catchments and soil characteristics

	Catchment 4				Catchment 5				Catchment 6			
	Litter	Top 5 cm	Middle	Bottom 5 cm	Litter	Top 5 cm	Middle	Bottom 5 cm	Litter	Top 5 cm	Middle	Bottom 5 cm
pH	4.3	4.4	4.8	4.9	4.3	4.2	5.1	5.0	4.5	4.2	4.7	4.8
SOC (g/kg)	428	175	53	50	392	174	41	45	421	199	78	81
C:N ratio	25	19	17	15	24	20	21	19	28	23	21	20
Area (m^2)	78				78				98			
Soil volume (m^3)	7				7				13			
Soil depth (cm)	16				16				34			
Bare rock (%)	51				51				88			
Slope (°)	11				11				9/7[a]			
Calluna cover (%)	75				75				56			
Tree crown cover (%)	12				12				20			
pH	4.5 (0.1)				4.5 (0.1)				4.6 (0.1)			
SOC (kg/m^2)	8.0 (4.5)				8.0 (4.5)				11.8 (5.1)			
C:N ratio	20 (2)				20 (2)				23 (5)			

Soil profiles were separated into four depth intervals, if depth allowed, including litter, the top 5 cm, the bottom 5 cm, and a midsection (middle) of varying depth. Content in kg m^{-2} for the soil organic carbon (SOC) has been calculated by multiplying the SOC concentrations in each soil layer with layer bulk density (Strand et al. 2008). Standard deviations are shown in brackets

[a] Catchment 6 has two outlets, hence two slope measurements

Table 2 Event ID abbreviations. S = summer event, A = autumn event

Event ID	Date	Events			Pre-conditions		
		Added (mm)	Duration (h)	Intensity (mm h^{-1})	24 h (mm)	7 days (mm)	14 days (mm)
S5_1	30.06.2006	70	10	7.0	0	9	38
S5_2	06.07.2006	70	10	7.0	0	0	30 (+70)
S5_3	08.08.2006	70	10	7.0	0	14	64
S5_4	31.08.2006	70	10	7.0	0	91	155
A4_1	03.10.2005	44	6	7.4	0	49	54
A4_2	20.10.2005	52	7	7.4	0	1	8
A4_3	28.10.2006	237	42	5.7	0	61	91
A6_1	03.10.2005	35	6	5.7	0	49	54
A6_2	20.10.2005	40	7	5.7	0	1	8
A6_3	28.10.2006	182	42	4.3	0	61	91

First digit is catchment number. Last digit is the chronologic event number within the catchment. For example, S5_2 is summer episode number 2 in catchment 5. The pre-condition columns show the accumulated amount of precipitation during 24 h, 7 and 14 days prior to the event, respectively. The notation 30 (+70) mm as pre-condition 14 days prior to event S5_2, reflects that 70 mm of the 100 mm in total originates from the previous summer event (S5_1)

tubing and filtered through active coal granulate before applying it to the catchments (Haaland et al. 2008). The chemical composition of the artificial precipitation was close to that of natural precipitation in the area (Haaland et al. 2008) with low DOC concentrations (<1.0 mg l^{-1}). Water flow was regulated by valves connected to digital controllers (Motorola). Commercially available disc irrigation sprinklers (Netafim), one per 4 m^2, were sufficient for an even distribution of the added water. Runoff was sampled and gauged manually (using graduated cylinders) at the natural outlets of the catchments. Irrigation rate was kept constant for each event, assuring a constant discharge rate after the soil had been moistened (often less than 5 mm needed). Since the rate of discharge was close to being constant throughout a single episode (Table 2) we did not take volume proportional discharge samples.

For the previously reported study, where we added artificial precipitation at a rate of 10 mm/week, discharge was measured using tipping buckets and digital loggers. Volume proportional samples were taken from discharge (Haaland et al. 2008).

Water analyses

Water samples were stored dark and refrigerated (4°C), and analysed within a few days. TOC analysis was done using a Shimadzu TOC-V total organic carbon analyser and a Phoenix 8000 TOC-TC analyser. DOC samples were filtered through 0.45 µm Millex-HA syringe-driven filters before analysis.

Computational methods

Fluxes of leached DOC were computed, by multiplying DOC concentration with the appropriate discharge flux for the same time interval. The leaching of DOC during an episode was taken as the sum of all DOC fluxes for the episode (in g) and divided by the surface area of the catchment (in m^2). Weekly average volume proportional samples taken from catchment 4 and catchment 6 have been used to compute differences in concentrations in runoff between years (Table 3).

Table 3 TOC concentrations in runoff (volume proportional samples) from catchment 4, catchment 6 and the average ± SD of five reference catchments, between July 1 and October 15 in 2003, 2004, 2005 and 2006

Year	TOC concentration (mg l^{-1})		
	Catchment 4	Catchment 6	Reference catchments
2003	11.9	11.5	11.6 ± 3.2
2004	10.8[a]	10.7[a]	11.6 ± 2.4
2005	11.2[a]	10.7[a]	11.6 ± 3.7
2006	11.0[a]	11.5[a]	13.5 ± 2.5

From Haaland et al. (2008)

[a] 10 mm of extra precipitation added weekly

As a first approximation, we estimated the pool of adsorbed potential DOC, assuming the DOC concentration to be in equilibrium with the adsorbed content of potential DOC and that this equilibrium may be described using a constant distribution constant, *Kd*:

$$Kd = \text{adsorbed potential DOC (mg/kg)} / \text{DOC concentration (mg/l)}.$$

Based on this assumption, a reduction in DOC concentration by 50%, implies a reduction in the adsorbed potential DOC pool by 50%. Thus the export of DOC associated with a reduction in DOC concentration by 50% represents 50% of the pool of adsorbed potential DOC.

Results

Climatic conditions during the episode studies

A4_2 and A6_2 had the driest pre-conditions with only 8 mm of natural precipitation the 2 weeks before the artificial event, while S5_4 had the wettest pre-event conditions with a total of 155 mm the 2 weeks prior to the event (Table 2). Except for a little rain during S5_3 (<0.5 mm), there was no interference with natural precipitation while conducting the episode studies. Soil temperatures at 5 cm depth during the summer episodes ranged between 13°C and 19°C. The autumn episodes were carried out at relatively low soil temperatures (2–9°C). The catchment soils were not exposed to frost or drought in the weeks prior to or during the experiments (Fig. 3).

Concentrations and fluxes of DOC

Concentrations of DOC in runoff from the small catchments ranged from 3 to 13 mg l^{-1} (Fig. 4). In general, the highest DOC concentrations appeared after 5–15 mm of applied precipitation, but this varied slightly between catchments and events. Events during autumn (studied in catchments 4 and 6) had somewhat higher DOC concentration and fluxes compared to those in summer (studied in catchment 5; Fig. 4), but probably this was related to generally slightly lower DOC concentrations in catchment 5 (Fig. 3). In particular the concentration and flux of DOC in runoff were elevated during the artificial episode in early October 2005 (A4_1 and A6_1) and higher than values

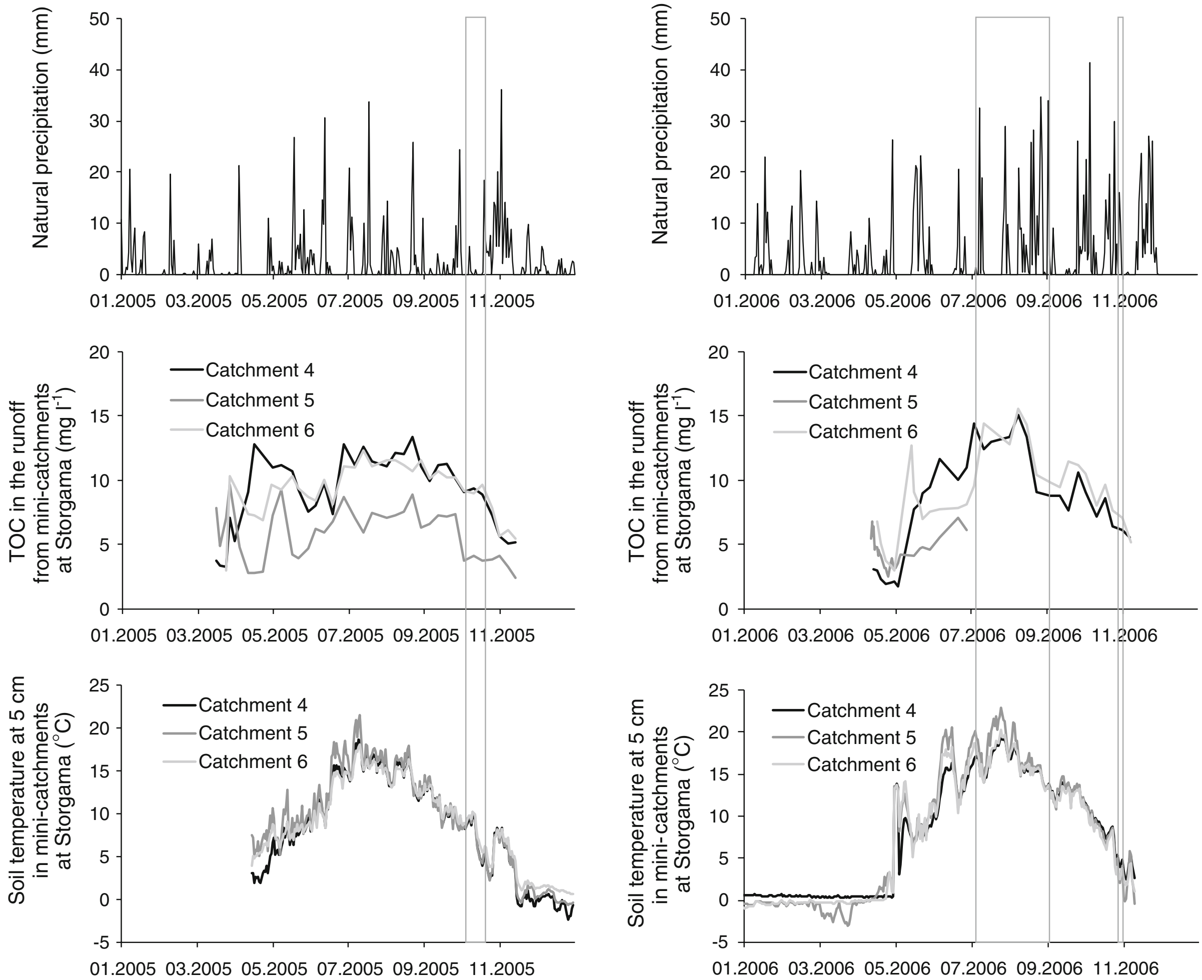

Fig. 3 Daily precipitation at Tveitsund metrological station (about 8 km west of Storgama), total organic carbon TOC concentrations in runoff (volume proportional weekly averages) for the mini-catchments, and soil temperature at 5 cm depth in the mini-catchments (20 min resolution, average of 3 measurements). Catchment 4 and catchment 6 have similar TOC concentrations, while catchment 5 has somewhat lower TOC concentrations. Differences between catchments in runoff DOC concentrations are explained by catchment specific characteristics such as SOC content and soil depth (Strand et al. 2008). *Grey* windows depict the periods with artificial precipitation events. The figure is modified from Haaland et al. (2008)

observed during the episode 17 days later (A4_2 and A6_2). Also autumn 2006 (A4_3 and A6_3) showed relatively high DOC concentrations and fluxes during the episodes. All episodes showed a tendency of decreasing DOC concentrations in runoff, the longer the episode lasted. This effect was most pronounced in the autumn episodes and less so in summer. The artificial, excessive autumn 2006 episodes (A4_3 and A6_3) showed marked declines in DOC concentrations of about 50%, after receiving up to about 25% of the annual normal precipitation in 42 h (Fig. 4).

Concentrations of DOC are highest in runoff during the growing season, when temperatures are highest (Fig. 3; Strand et al. 2008). Earlier temperature was reported to be an important factor in controlling leaching of DOC (Kalbitz et al. 2000; Michalzik et al. 2003; Qualls 2000). In the non-manipulated reference catchments, accumulated DOC fluxes for the growing season were between 1.7 and 4.2 g DOC m^{-2}. The additional 10 mm of weekly precipitation at catchment 4 and 6 (about 150 mm in total for the growing season), was nearly all transferred to runoff (little being used for

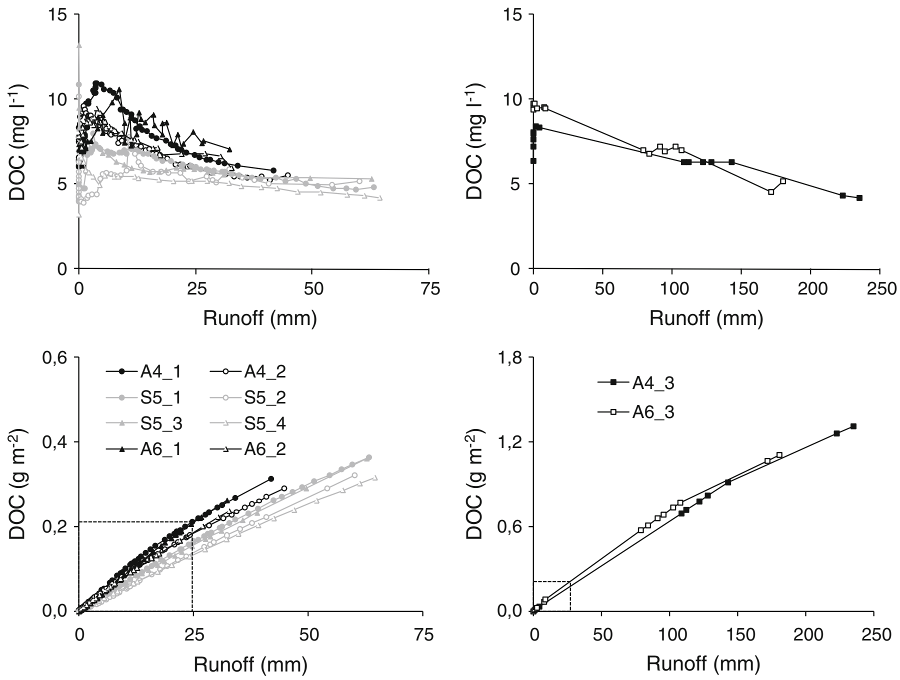

Fig. 4 DOC concentration and DOC flux in runoff during artificial episodes from three small headwater catchments at Storgama. *Left panels* show graphs of two short autumn episodes in catchments 4 and 6, and four short summer episodes in catchment 5. The *right panels* show graphs of two excessive autumn episodes in catchments 4 and 6. Event id abbreviations are explained in Table 2. The DOC fluxes are similar for the autumn events in the *left* and *right lower panels* after an accumulated runoff of 25 mm

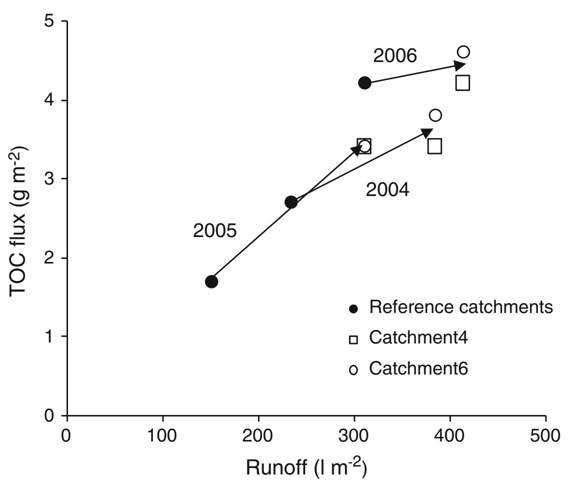

Fig. 5 TOC leaching from mini-catchments at Storgama comparing reference catchments with catchments 4 and 6, which received 10 mm extra precipitation between July 1 and October 15. The increase caused by additional precipitation is not linear. This is particularly so in wetter years

evapotranspiration), which caused a slight decrease in DOC concentration (Table 3), but an increase in DOC flux (Fig. 5). During wetter years (2004 and 2006) additional precipitation caused an increase in DOC fluxes of about 0.9–0.2 g DOC m^{-2}. In comparison did DOC leaching increase with about 1.7 g DOC m^{-2} during the dry growing season of 2005 (Fig. 5).

Discussion

A substantial decline in DOC concentration with about 50% was seen during the excessive autumn precipitation treatments, 2006 (A4_3 and A6_3; Table 2). Leaching of about 1.2 g DOC m^{-2} in 2 days, due to about 200 mm of artificial precipitation in autumn, reduced the concentrations of DOC from about 10 to 5 mg l^{-1} (Fig. 4). If sorption of DOC may be described by a fixed *Kd* (Tipping et al. 2005), this implies that the store of potential DOC in catchment 4 and 6 during late autumn was reduced by 50%. Hence, this suggests a pool size of potential DOC of about 2–3 g m^{-2} A similar estimate is obtained from the DOC flux data of the first artificial autumn event of 2005 (A4_1 and A6_1), which although smaller in water throughput, shows the same DOC leaching pattern as the one for 2006. By contrast, the last artificial autumn event in 2006 has lower DOC concentrations and fluxes, indicating a slightly smaller pool of potential DOC. The potential DOC pools may be compared with the much larger SOC pool sizes of 8,000 and 11,800 g m^{-2} in catchment 4 and 6, respectively, suggesting a store of about 0.2–0.3 g potential DOC per bulk kg SOC. This is lower than reported by Qualls (2000) in a laboratory experiment for a pine forest floor, where 2.4% of the SOC pool in 2 g of grinded, but not dried soil, was found to be soluble (by extracting with water).

The driest pre-event conditions in summer were prior to S5_1 and S5_3, resulting in almost identical fluxes of DOC from catchment 5 in response to artificial episodes of 70 mm. By contrast, events S5_2 and S5_4, having wetter antecedent conditions (Table 2), had smaller fluxes of DOC leaving the catchments, indicating a smaller pool of potential DOC at the start of these episodes. In addition, declining soil temperature from >20°C in July to about 12°C at the end of August (Fig. 3), may explain some of the reduced store of potential DOC prior to S5_4 (Fig. 4), as it would be associated with a smaller DOC production rate caused by decreased microbial activity at lower temperature. Yet, even after the extra addition of three times 70 mm of artificial precipitation during summer, in addition to the naturally wet summer of 2006 (Fig. 3), S5_4 resulted in only slightly smaller DOC concentrations and fluxes in runoff as S5_1. This suggests rapid replenishment rates during summer, and probably a complete replenishment of the potential DOC pool within days. The time between events would hence be important for the replenishment. In this context others have reported that results from best fit models, diffusion of the produced DOC from within aggregates of soil organic matter to adsorption sites at aggregate surfaces, rather than the production of DOC itself, seems to be what is limiting the supply of potential DOC between runoff events (Worrall et al. 2008).

During autumn, soils have lower temperatures and in particular events A4_2 and A6_2 occur after a cold period (Fig. 3). The DOC flux was slightly lower than that observed for A4_1 and A6_1, despite relatively dry antecedent conditions and thus little DOC leaching prior to the artificial event. Probably, temperature-limited replenishment of DOC may explain the slightly smaller DOC leaching fluxes and hence a smaller pool of potential DOC at the start of the artificial episode (i.e. Kalbitz et al. 2000; Michalzik et al. 2003).

The addition of 10 mm of precipitation per week at catchments 4 and 6 throughout the growing seasons of 2004, 2005 and 2006, did not cause a pronounced dilution of DOC in runoff (Haaland et al. 2008; Table 3). Yet, the wetter the summer the smaller the absolute and relative increase in DOC flux in response to the additional precipitation (Fig. 5). Similar observations were made by others in laboratory studies (Christ and David 1996; Gödde et al. 1996). This suggests that the pool of adsorbed potential DOC becomes less depleted during drier years, thus sustaining greater DOC concentration values in runoff. During autumn there was a significant decline in DOC concentrations during both dry and wet years in the catchments receiving extra artificial precipitation (Fig. 3). Previously, similar observations have been reported for organic surface horizons of forest soils in areas with considerable amounts of precipitation in autumn (e.g. Fröberg et al. 2006; Michalzik et al. 2003; Vogt and Muniz 1997). In reference catchments, without the addition of artificial precipitation, a similar decline in DOC concentration was seen only during wetter years (2004 and 2006; see Haaland et al. 2008).

It is interesting to note that reference catchments did not show pronounced increases in concentration of DOC after relatively dry conditions during summer, despite a limited depletion of the potential DOC pool (Haaland et al. 2008; Table 3). This may suggest that even without leaching the increase in the adsorbed potential DOC pool is leveling off and there is no further net production of potential DOC. Thus, there seems to exist a maximum pool size of potential DOC at Storgama, which may be explained by a balance between DOC production rates and rates of potential DOC consumption (e.g. mineralization to CO_2 or conversion to SOC (Tipping et al. 2005)).

Conclusions

Our data suggest that the size of the potential DOC pool at Storgama during autumn is in the range of about 2–3 mg C m^{-2}. This is only 0.2–0.3 g of potential DOC per kg of SOC. The replenishment rate was fast and the DOC pool could be fully restored probably within days. In autumn, replenishment rates may be slower however, due to lower soil temperatures. DOC concentration in runoff, and thus the pool of potential DOC in the soil, is smallest after snowmelt in spring. During the growing season, the pool of potential DOC reaches its maximum. Under non-leaching conditions the DOC pool does not increase continuously in size, but levels off. This might suggest that with an increasing pool of potential DOC there is a gradual increase in the net decomposition of DOC to CO_2 (or an increase in the conversion of DOC to soil humic material), thus keeping the total pool of potential DOC pool in balance.

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