Chin. Geogra. Sci. 2010 20(3) 209–217
DOI: 10.1007/s11769-010-0209-7

Freeze-thaw Effects on Sorption/Desorption of Dissolved Organic Carbon in Wetland Soils

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Abstract: The effects of freeze-thaw cycles on sorption/desorption of dissolved organic carbon (DOC) in two wetland soils and one reclaimed wetland soil were investigated. DOC concentrations added were 0–600 mg/L. Laboratory incubations of sorption/desorption of DOC had been carried out at –15℃ for 10 h, and then at +5℃ for 13 h. Soil samples were refrozen and thawed subsequently for 5 cycles. Initial Mass model was used to describe sorption behavior of DOC. The results indicate that freeze-thaw cycles can significantly increase the sorption capacity of DOC and reduce the desorption capacity of DOC in the three soils. The freeze-thaw effects on desorption of DOC in soils increase with the increasing freeze-thaw cycles. The conversion of natural wetlands to soybean farmland can decrease the sorption capacity and increase the desorption capacity of DOC in soils. Global warming and reclamation may increase DOC release, and subsequently increase the loss of carbon and the emission of greenhouse gas.
Keywords: DOC; sorption; desorption; freeze-thaw; wetland soils

1 Introduction

Dissolved organic carbon (DOC) is a major controlling factor in soil formation (Dawson *et al.*, 1978), mineral weathering (Raulund-Rasmussen *et al.*, 1998), nutrient cycling, microbial activity, and organic matter decomposition and transformation in soils (Magill and Aber, 2000; Williams *et al.*, 2000). The sorption of DOC in soil is a dominant factor influencing DOC concentration in soil solutions, transport and transformation, and the microbial availability of DOC. It controls not only the transport of organic matter (OM) and OM-assisted elements (Kaiser and Zech, 1999), but also the stabilization and accumulation of organic matter in soils (Guggenberger and Kaiser, 2003).

Six mechanisms have been suggested to be involved in the sorption of DOC to soil mineral surfaces: ligand exchange, cation bridges, anion exchange, cation exchange, van der Waals interactions and hydrophobic effects (Jardine *et al.*, 1989). The ligand exchange between carboxyl/hydroxyl functional groups of dissolved organic matter (DOM) and iron oxide surfaces was the dominant interaction mechanism, especially under acidic or slightly acidic conditions (Gu *et al.*, 1994). Some researchers investigated the important factors influencing the sorption of DOC in soils. The sorption capacity of DOC in clay soils was greater than that in sandy soils (Moore and Matos, 1999), which appeared to be positively correlated to the soil clay content (Shen, 1999). Moore *et al.* (1992) observed that the sorption of DOC was positively related to soil organic carbon content and negatively related to oxalate-extractable Al and dithionite-extractable Fe. However, the results of another research indicated that soils with higher organic matter content adsorbed less DOC (Jardine *et al.*, 1989). And the sorption of DOC in acid soils was greater than that in alkaline soils (Moore *et al.*, 1992). Maximum sorption of DOC in soils occurred at pH 4, and decreased with either increase or decrease in pH (Ussiri and Johnson, 2004). Lilienfein *et al.* (2004) elucidated that the sorption of DOC in soils increased significantly with increasing soil development, whereas the soil depth did not have a consistent and significant effect. In addition, Vandenbruwane *et al.* (2007) did the comparison of dif-

Received date: 2009-09-11; accepted date: 2010-01-28
Foundation item: Under the auspices of Knowledge Innovation Programs of Chinese Academy of Sciences (No. KZCX2-YW-309), National Natural Science Foundation of China (No. 40871089, 40830535)
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ferent isotherm models for sorption of DOC in soils. However, the studies on DOC sorption capacity of soils have been conducted in a mild temperature range from 5℃ to 25℃, and little is available on the sorption of DOC in freezing and thawing soils.

Many studies about sorption of DOC in soils have been documented, however, almost all of them focused on farmland soils and forest soils, and little attention was paid to the sorption/desorption of DOC in wetland soils. For wetlands in the cold region, freeze-thaw is an important environmental characteristic. Maehlum *et al.* (1995) reported that cold winter climate can affect hydraulic processes and biogeochemical processes of wetlands. Freeze-thaw cycles have effects on soil physical properties, microbial activity and microbial community composition (Schadt *et al.*, 2003; Lipson and Schmidt, 2004; Six *et al.*, 2004; Sjursen *et al.*, 2005). For example, freeze-thaw cycles have disruptive effects on soils structure, which decrease bulk density and penetration resistance (Unger, 1991). Likewise, in controlled laboratory incubations, freeze-thaw cycles resulted in decrease of soil aggregate stability, particularly at high soil moisture (Oztas and Fayetorbay, 2003). Wang *et al.* (2007) have studied the freeze-thaw effects on phosphorus sorption in wetland soils, and the results show that freeze-thaw can promote phosphorus sorption in wetland soils and these effects accumulate over successive freeze-thaw cycles. However, whether and how freeze-thaw affects sorption/desorption of DOC in wetland soils is still unknown. There has been growing interest in how changes in soil freezing initiated by climate change might alter soil nutrient dynamics (Henry, 2007). Thus whether global warming can affect sorption/desorption of DOC in soil through affecting freeze-thaw cycles needs to be concerned. In present, many wetlands have been reclaimed to farmlands in China, especially in the Sanjiang Plain of Northeast China. The effect of wetland reclamation on the DOC sorption/desorption in soils is not examined yet, wetland reclamation might alter the loss of carbon in cultivated wetlands.

The objective of this study was to investigate freeze-thaw effects on sorption/desorption of DOC in wetland soils and reclaimed wetland soils. Particular attention was given to possible effects of reclamation and global warming on the retention and release of DOC in wetland soils. Compared with the related research conducted in other cold region all over the world, the understanding of DOC sorption/desorption in soils in the cold region, therefore, can be verified with higher confidence level.

2 Materials and Methods

2.1 Study area

The study area was located in the Sanjiang Plain, Northeast China (45°01′–48°28′N; 130°13′–135°05′E). It is a cold region and regularly exposed to subzero temperature with mean annual temperature of 3℃ ranging between –21℃ and 22℃. Water and soils in the study area are long-term frozen from late October to next April and begin to melt in the late April (Wang *et al.*, 2006). It is deemed desirable to study the effect of multiple freezing and thawing on the sorption properties of DOC in soils, since the soils in the Sanjiang Plain are subject to lots of freeze-thaw cycles every year.

2.2 Sampling method

Three sampling sites, *Carex lasiocarpa* natural wetland (NW1), *Calamagrostis angustifolia* natural wetland (NW2) and reclaimed wetland (RW) were selected within study area. Specific descriptions of the three sites are shown in Table 1 and soil physicochemical properties are shown in Table 2.

Soils were collected in the surface layer (0–10 cm). At each of the three sampling sites, three soil samples were collected separately. After plant roots were removed, soil samples were homogenized, air-dried, and sieved over a 2-mm mesh and then stored in airtight and light-free plastic bags at 20℃ until chemical analysis and sorption/desorption experiment.

2.3 DOC stock solution preparation

To simulate the field condition, the peat water extract was used as DOC stock solution for the sorption experiments. The surface peat (0–10 cm) was collected from a site in 200 m north of site NW1. The peat sample was filled in a glass bottle, and deionized water was added with water peat ratio of 10∶1. Then the capped glass bottle was placed on a shaker (130 rpm) for 4 h, and the peat water extract was filtered through 0.45 μm polyethersulfone membrane disc filters. Then the filtrate was concentrated in order to obtain the DOC stock solution with high concentration, which was diluted to five DOC concentrations between 0 and 600 mg/L.

Table 1 Description of three sampling sites in Sanjiang Plain

Site	Wetland type	Vegetation type	Hydrological feature	Soil type
NW1	Marsh	*Carex lasiocarpa*	Perennial flooding	Humus marsh soil
NW2	Marshy meadow	*Calamgrostis augustifolia*	Seasonal flooding	Meadow marsh soil
RW	Farmland	*Glycine max*	Perennial drought	Meadow planosol

Note: NW1 and NW2 represent *Carex lasiocarpa* natural wetland and *Calamagrostis angustifolia* natural wetland soils, RW represents reclaimed soil

Table 2 Soil physical and chemical properties of sampling sites in Sanjiang Plain

Site	Clay content (%)	pH	Organic matter content (%)	DOC content (mg/kg)
NW1	40.20 ± 11.20	5.60 ± 0.50	12.90 ± 3.13	2762.00 ± 182.00
NW2	64.50 ± 8.09	5.40 ± 0.60	11.70 ± 3.27	546.00 ± 76.20
RW	46.80 ± 4.12	6.00 ± 0.50	13.30 ± 3.09	197.00 ± 31.50

Note: Values are given as mean ± standard error, n = 3

2.4 Sorption/desorption experiment with freeze-thaw treatment

For the sorption/desorption experiment, each of the three soils collected from NW1, NW2 and RW was treated by freeze-thaw (FTT) with 3 replicates as described below. Each soil sample had a control that was untreated by freeze-thaw (UT). The flowchart of the sorption/desorption experiment procedures of DOC was shown in Fig. 1.

Sorption procedure: 5 g of soil sample was placed in the 250-ml pre-weighed centrifuge tubes. To each sample, the 50 ml of solution containing varied concentrations of DOC (0 mg/L, 50 mg/L, 100 mg/L, 200 mg/L, 400 mg/L and 600 mg/L) was added, which correspond to 0 mg/kg, 500 mg/kg, 1000 mg/kg, 2000 mg/kg, 4000 mg/kg and 6000 mg/kg DOC added, respectively. Capped tubes were placed on a shaker (130 rpm) at +5℃ for 1 h, then were frozen at −15℃ for 10 h, and finally thawed at +5℃ for 13 h. This is one freeze-thaw cycle (FTC). The control tubes were stored at 5℃ for one 23 h cycle corresponding to one freeze-thaw cycle. The tubes which have been subjected to one to five cycles, respectively, were centrifuged at 5000 rpm for 20 min. Then the supernatant was taken and DOC in the extracts was determined. Adsorbed DOC with one to five cycles was calculated as the difference between the DOC added and DOC remained in the equilibrating solution.

Desorption procedure: Two samples (4000 mg/kg and 6000 mg/kg) after 5 cycles were selected, the supernatant was removed after centrifugation and the tubes were re-weighed to determine the volume of solution entrapped in the residue. Then deionized water was added to bring the total volume to 50 ml. After the first cycle as described in sorption procedure, DOC in the supernatant was determined. The difference between the DOC in the supernatant and the DOC in the residue solution was the desorption amount of DOC. Following the first step again, the amount of DOC desorbed by soils after the second to the fifth cycle was calculated.

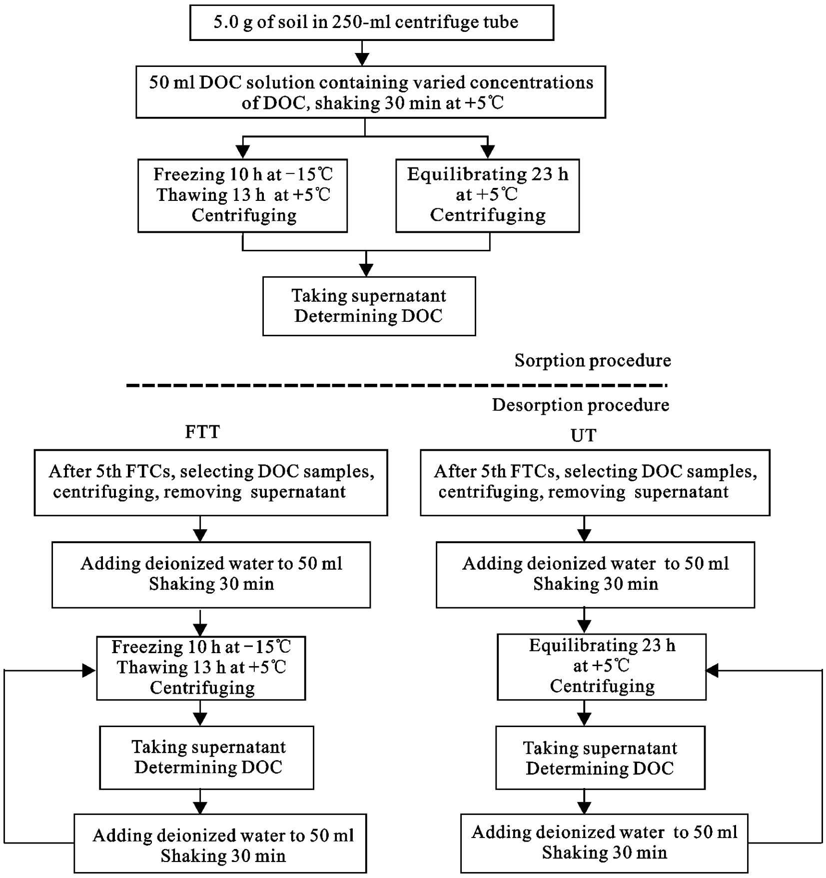
2.5 Analytical method

Soil samples were analyzed for clay content, organic matter content, DOC content and pH. DOC stock solution was analyzed for pH, DOC, NO_3^-, NH_4^+ and PO_4^{3-} contents.

Clay content of soils was determined using a Mastersizer 2000 Laser Grainsize (Manufactured by Malvern Instruments Ltd. UK; measuring range: 0.02–2000 μm) as described by Wang *et al.* (2006). Organic matter content of soils was measured by potassium dichromate volumetric method—external heating (Bao, 2000). Soil DOC in the extract was determined with a TOC-V_{CPH} (Shimazu, Japan). The pH was determined in situ with composite electrodes. The NO_3^-, NH_4^+, and PO_4^{3-} in stock solution were determined using SAN^{++} Continuous Flow Analyzer (SKALAR, Netherland). The supernatant samples were analyzed for DOC concentration. The DOC was determined by SAN^{++} Continuous Flow Analyzer (SKALAR, Netherland).

2.6 Statistical analysis

Nonparametric test and one-way analysis of variance (ANOVA) were performed using SPSS for Windows, Release 11.0, Standard Version (SPSS Inc., US), and graphics using OriginPro 7.5, (OriginLab Corp., US). Nonparametric test was performed to compare the dif-



FTT denotes freeze-thaw treatment; UT denotes control treatment

Fig. 1 Flowchart of experiment procedures of dissolved organic carbon (DOC) sorption/desorption in wetland soils

ferences of sorption/desorption of DOC in FTT and UT soils. One-way ANOVA was performed to compare the differences in sorption/desorption of DOC after varied FTCs.

3 Results and Discussion

3.1 Sorption behavior of DOC

The sorption amount of DOC increases with DOC concentration added. This trend can be described by Initial Mass (IM) isotherm model, which is the most common model based on simple partitioning. This model is based on the linear adsorption isotherm (Travis and Etnier, 1981), especially fit for the condition when the substances initially exist in the soils:

$$S = m \times C_1 - b \qquad (1)$$

where S is the total amount of DOC adsorbed (mg/kg); C_1 is the concentration of DOC added (mg/kg); m is the partition coefficient of DOC between soil and solution, used to estimate sorption capacity; b is the intercept (mg/kg).

Parameters of IM isotherms model are presented in Table 3, and IM isotherms for sorption of DOC in FTT soils and UT soils are given in Fig. 2. IM isotherm has been used to describe sorption of DOC in soils in many studies (Nodvin *et al.*, 1986; Guggenberger and Zech, 1992; Riffaldi *et al.*, 1998), and the highest concentration of DOC added in experiment is 81 mg/L (Nodvin *et al.*, 1986). However, in this study, for NW2 and RW soils, when DOC concentration is added from 0 mg/L to 400 mg/L, IM isotherm can describe sorption behavior of DOC in soils very well. When DOC added is higher than 400 mg/L, IM isotherm is not suitable. Moreover, for NW1 soils, the highest concentration of 600 mg/L is still suitable to IM isotherm.

3.2 Freeze-thaw effects on sorption of DOC

In Fig. 2, the sorption isotherms of FTT soils are always higher than that of UT soils. With a nonparametric test, the difference of the adsorbed DOC amount between the FTT soils and UT soils is significant (Z = –7.784; p < 0.001). These results indicate that freeze-thaw cycles increase the adsorbed amount of DOC in soils. Moreover, values of *m*, used to estimate the sorption capacity of FTT soils, are always higher than that of UT soils (Table 3). With a nonparametric test, the difference of *m* values between FTT soils and UT soils is also significant ($Z = -3.408$; $p = 0.001$). It indicates that freeze-thaw increases the sorption capacity of DOC in soils, which may be resulted from the freeze-thaw effects on the soil structures. The main clay mineral of these soils is hydrous mica. Freezing can break down aggregates in soils, and soil mineral surfaces may be increased, which can provide more sorption positions for DOC (Hinman, 1970; Bullock, 1988; Oztas and Fayetorbay, 2003; Six *et al.*, 2004). In addition, DOC sorption is affected by iron oxides in soils. About 50%–70% DOC is adsorbed by oxides in soils (Jardine *et al.*, 1989). We speculate that the amorphous iron oxides in soils can be increased by alternate freeze-thaw cycles. The increased iron oxides amount can affect DOC sorption in soils, and the increased DOC sorption capacity by freeze-thaw can confirm this speculation. Moreover, the increased sorption capacity of DOC caused by freeze-thaw cycles can increase DOC retention in wetland soils. Under conditions of low plant and microbial activity in winter, the sorption of DOC may represent an intermediate DOC sink in soil. Thus, the increased DOC retention by freeze-thaw cycles likely increase the DOC sink in wetland soils. For DOC sorption can decrease the bioavailability of DOC, freeze-thaw can have effects on the microbial contacting to the DOC in the following growing season.

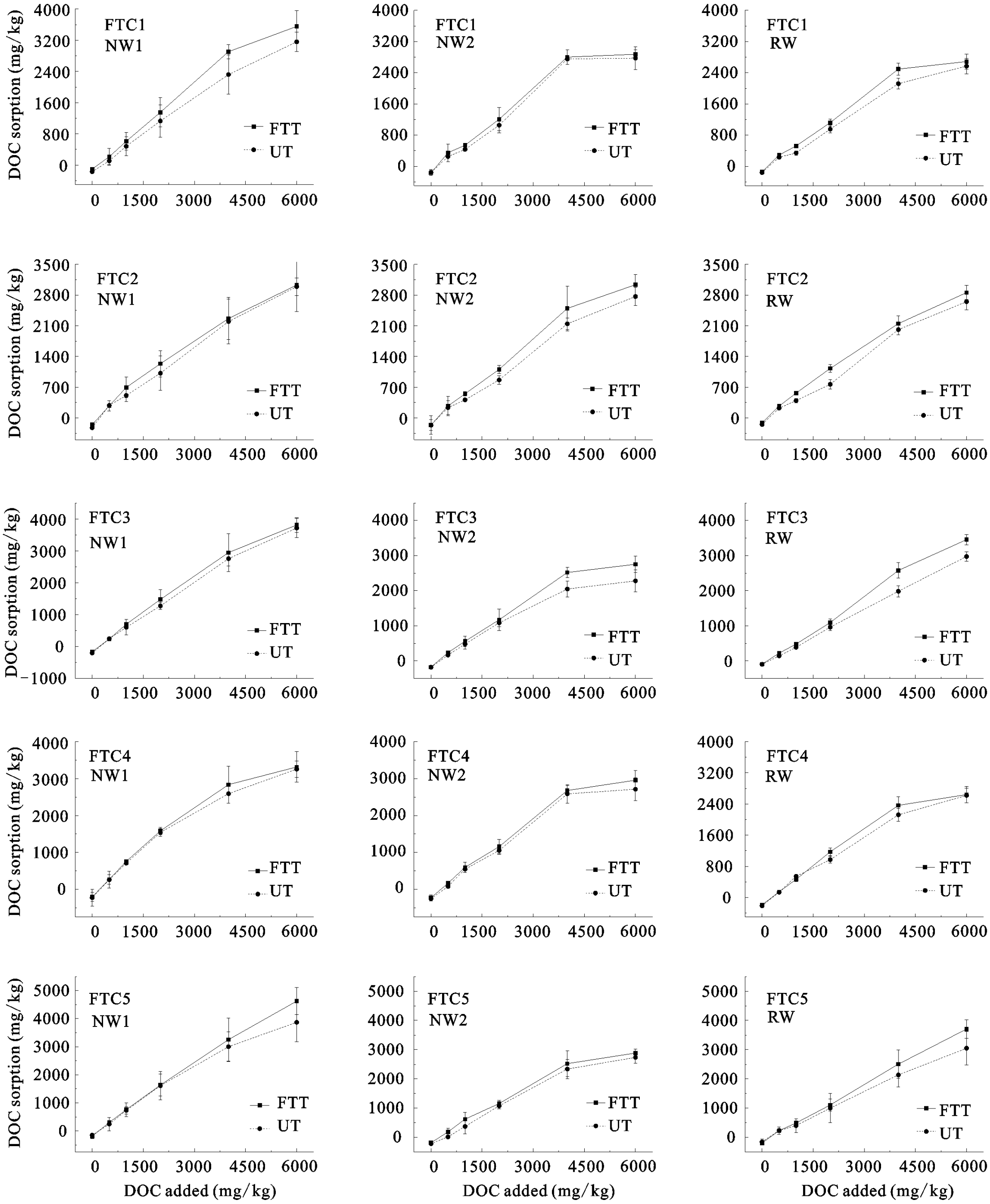
Furthermore, the difference of the adsorbed DOC amount at varied FTCs (from FTC1 to FTC5) is not significant (NW1 soils: $F = 0.43$, $p = 0.996$; NW2 soils: $F = 0.05$, $p = 1.000$; RW soils: $F = 0.31$, $p = 0.998$). The results indicate that the FTCs can not significantly affect the adsorbed DOC amount in soils. The difference of *m* values is not significant at varied FTCs (NW1 soils: $R = 0.588$, $p = 0.298$; NW2 soils: $R = 0.243$, $p = 0.294$; RW soils: $R = 0.319$, $p = 0.600$), which indicates that FTCs do not have significant effects on the sorption capacity of DOC in soils. Therefore, the first FTC increases the DOC sorption in soils, while the following FTCs do not increase or reduce these effects.

Comparison of adsorbed DOC amount among varied soils at sites NW1, NW2 and RW is presented (Fig. 2). NW1 soils adsorb more DOC than NW2 and RW soils do (NW1 > NW2 > RW). The *m* values of soils at site NW1 are about 11.8% higher than that at site NW2, and are about 18.2% higher than that at site RW. This indicates that the DOC sorption capacity of soils at site

Table 3 Initial Mass isotherms parameters for DOC sorption in freeze-thaw treated and control soils

Site	Cycle	*m*		*b*		*R*	
		FTT	UT	FTT	UT	FTT	UT
NW1	FTC1	0.758	0.628	142	170	0.999	0.999
	FTC2	0.590	0.580	22	114	0.994	0.996
	FTC3	0.776	0.729	126	160	0.999	0.999
	FTC4	0.754	0.697	83	72	0.995	0.990
	FTC5	0.856	0.793	132	101	0.999	0.998
NW2	FTC1	0.725	0.718	142	211	0.997	0.995
	FTC2	0.649	0.560	123	143	0.999	0.996
	FTC3	0.664	0.553	134	111	0.999	0.997
	FTC4	0.715	0.702	199	251	0.999	0.998
	FTC5	0.666	0.650	138	259	0.999	0.999
RW	FTC1	0.646	0.562	114	145	0.999	0.997
	FTC2	0.556	0.522	36	135	0.998	0.994
	FTC3	0.668	0.525	147	112	0.998	1.000
	FTC4	0.640	0.570	169	142	0.999	0.997
	FTC5	0.648	0.534	150	89	0.999	0.999

Note: FTT and UT represent soils with and without freeze-thaw treatment, respectively; FTCs represent freeze-thaw cycles; *m* is partition coefficient of DOC between soils and solution; *b* is the intercept of Initial Mass Isotherm model; *R* is correlation coefficient



FTT and UT represent soils with and without freeze-thaw treatment, respectively; NW1 and NW2 represent *Carex lasiocarpa* natural wetland and *Calamagrostis angustifolia* natural wetland soils, RW represents reclaimed soil

Fig. 2 Initial Mass (IM) isotherms for DOC sorption in freeze-thaw treated wetland soils

NW1 is greater than those at site NW2 and RW, while the DOC sorption capacity of soils at site NW2 is greater than that at site RW. It is reported that soils with higher organic matter content adsorb less DOC (Jardine *et al.*, 1989), and there is positive correlation between soil sorption capacity of DOC and the soil clay content (Shen, 1999). Consistent with these previous studies, the adsorbed amount and sorption capacity of DOC in soils at site NW2 are greater than that at site RW. The soil clay content at site NW2 is about 37.8% higher than that at site RW, while the soil organic matter content at site RW is about 13.7% higher than that at site NW2. On contrast, the adsorbed amount of DOC at site NW1 was greater than those at site NW2 and RW, although the soil organic matter content at site NW1 is greater, which is not consistent with the previous studies. Thus another factor may exist for the greater sorption capacity of NW1 soils. In fact, a certain content of peat exists in the surface soils at site NW1, which is a ubiquitous phenomenon for perennial flooded wetland soils. The peat is porous and has great sorption capacity, which may be the reason for NW1 soils with higher organic matter content and lower clay content adsorbing more DOC than NW2 and RW soils doing.

3.3 Freeze-thaw effects on desorption of DOC

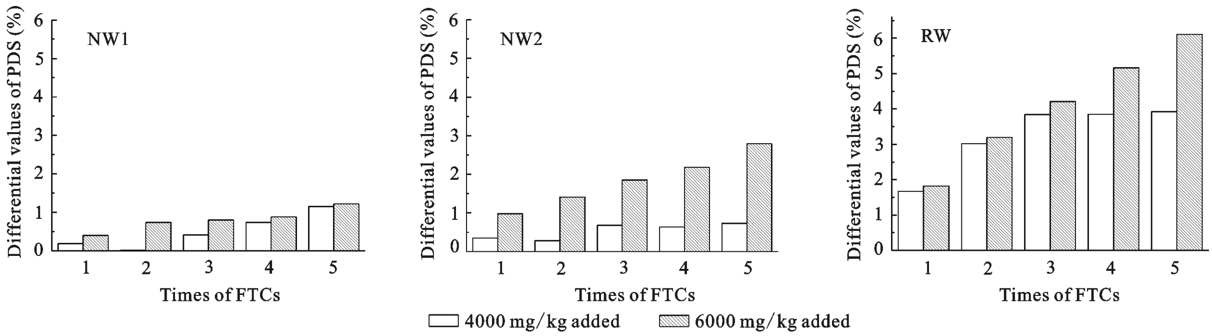
After the sorption experiment, some soil samples were reused for desorption experiment of DOC. DOC is desorbed from soil samples which have adsorbed DOC in the sorption experiment. The desorption capacity of DOC is estimated by the percentage of the desorbed DOC amount to the adsorbed DOC amount (PDS). PDS in FTT and UT soils are presented in Table 4. With a nonparametric test, there is significant difference of PDS in FTT and UT soils ($Z = -4.082$; $p < 0.001$). PDS of FTT soils is lower than that of UT soils, which indicates that freeze-thaw reduces the desorption capacity of DOC. The comparison of PDS at different sampling sites shows that PDS order is RW > NW2 > NW1. For example, after FTC5, PDS of NW1, NW2 and RW soils (with DOC concentration of 4000 mg/kg added in sorption experiment) is 31.7%, 33.3% and 45.0%, respectively.

Moreover, the difference of PDS in UT soils and FTT soils increases with increasing FTCs (Fig. 3). It may indicate that the freeze-thaw effects on DOC desorption can be improved by the increasing FTCs, which meant that the decreased desorption of DOC caused by freeze-thaw treatment are increased with FTCs. Moreover, in the cold region, the average annual FTCs are stable, so the freeze-thaw effects on the DOC desorption capacity may also be stable. If air temperature increases in the cold region and the FTCs reduce, the DOC desorption capacity can increase because of the reduced effects of freeze-thaw. The increased desorption can improve the release of DOC. Therefore, DOC released from soil mineral surfaces to soil solution can be increased by global warming. And DOC transporting from soil solution to overlying water column through the process of molecular diffusion may be affected, which affects DOC concentration in soils and the overlying water column. However, besides reducing FTCs, global warming may also affect freeze-thaw time, freeze-thaw intensity, and so on. Whether these factors response to global warming can affect sorption/desorption of DOC needs further investigation.

Table 4 Percentage of total desorbed amount of DOC to adsorbed amount of DOC (%)

Soil	Cycle	Samples with 4000 mg/kg DOC		Samples with 6000 mg/kg DOC	
		FTT	UT	FTT	UT
NW1	FTC1	8.8	9.2	8.0	8.2
	FTC2	15.6	16.3	14.9	14.9
	FTC3	21.3	22.1	19.4	19.8
	FTC4	26.7	27.6	23.6	24.3
	FTC5	31.7	32.9	27.3	28.5
NW2	FTC1	9.2	9.6	9.7	10.7
	FTC2	17.3	18.0	17.3	18.7
	FTC3	23.0	23.7	23.5	25.4
	FTC4	28.3	28.9	29.0	31.2
	FTC5	33.3	33.6	34.1	36.9
RW	FTC1	12.1	13.8	9.8	11.6
	FTC2	23.0	26.0	17.9	21.1
	FTC3	30.9	34.8	23.9	28.1
	FTC4	38.1	41.9	28.9	34.1
	FTC5	44.9	48.7	33.8	39.9

Note: FTT and UT represent soils with and without freeze-thaw treatment, respectively. FTCs represent freeze-thaw cycles



NW1 and NW2 represent *Carex lasiocarpa* natural wetland and *Calamagrostis angustifolia* natural wetland soils, RW represents reclaimed soil

Fig. 3 Differential values of percentage of desorbed DOC amount to adsorbed DOC amount (PDS) in freeze-thaw treated wetland soils

3.4 Effects of wetland reclamation on soil sorption/desorption behaviors of DOC

The DOC sorption capacity in soils at site RW is lower than that at site NW2, and the DOC desorption capacity in soils at site RW is greater than that at site NW2 (Fig. 2, 3). However, the soil type of site RW is the same as that of site NW2 before the reclamation for soybean production. This indicates that the reclamation appears to reduce the sorption capacity of DOC and to increase the desorption capacity of DOC. The iron oxides complex decreases after the reclamation (Zou *et al.*, 2008). The decreased iron oxides complex may reduce the sorption capacity and increase the desorption capacity of DOC. The altered hydroperiod can affect amorphous iron oxides, which may be another reason. Su *et al.* (2001) observed that water logging increases amorphous iron oxides of soils. The increased amorphous iron oxides can result in the increased sorption of DOC. Due to the conversion of the wetland to the farmland, shortened hydroperiod in farmland may cause decreased amorphous iron oxides of soils and thus reduces sorption capacity of DOC in farmlands. Under the cultivation condition, lots of crop residues and other organic matters are returned to soils, which increases the organic matter content in soils. This results in the decreased sorption of DOC in the reclaimed wetland soils (Jardine *et al.*, 1989). In addition, the decreased retention of DOC may increase the loss of carbon and the emission of greenhouse gas.

5 Conclusions

Freeze-thaw cycles can increase the sorption capacity of DOC and reduce desorption capacity of DOC in wetland soils and reclaimed wetland soils. The freeze-thaw effects on the desorption capacity of DOC can be improved by increasing FTCs. Greater sorption capacity of DOC and weaker desorption capacity of DOC in soils of perennial flooding wetland than that of seasonal flooding wetland are observed. Wetland reclamation reduces the sorption capacity of DOC and increases the desorption capacity of DOC in soil. The DOC released from soil mineral surfaces to soil solution can be increased by global warming.

Acknowledgements

We gratefully acknowledge Prof. Liu Xiaobing for his constructive comments on the manuscript and Zou Yuanchun, Zhao Haiyang, Wang Chaoying and Lou Bokun for sampling and samples analyses. Special thanks go to the Sanjiang Mire Wetland Experimental Station of Chinese Academy of Sciences and Honghe National Nature Reserve.

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