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# Changes in the microbial activity and thermal properties of soil treated with sodium fluoride



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#### ABSTRACT

The presence of sodium fluoride in soil can determine its physicochemical and thermal properties as well as microbial activity. In this study, sodium fluoride was introduced to soil in six doses: 1105, 2210, 4420, 6631, 8841 and 11051 mg kg<sup>-1</sup> DM soil. Changes in pH, electrolytic conductivity, soil microbial biomass (SIR-SMB) and glucose biodegradation parameters were determined in soil samples containing different doses of sodium fluoride. Soil microbial activity was investigated by the modified SIR3 (AA) method and isothermal microcalorimetry. Soil samples containing higher doses of sodium fluoride were characterized by elevated pH and increased electrolytic conductivity. Our results revealed that soil microbial activity and the kinetics of glucose decomposition were determined by sodium fluoride concentrations in soil. Sodium fluoride doses of 1105–6631 mg kg<sup>-1</sup> DM soil increased the content of microbial biomass and stimulated glucose degradation in soil samples relative to the control (without sodium fluoride). The two highest doses of sodium fluoride (8841 and 11051 mg kg<sup>-1</sup> DM soil) reduced SIR-SMB and inhibited degradation of organic matter.

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#### 1. Introduction

The fluorine content of rocks can range from 100 to 1300 mg kg<sup>-1</sup>, whereas in most soils, fluorine concentrations are determined between 20 and  $500 \text{ mg kg}^{-1}$ , or even above  $1 \text{ g kg}^{-1}$ (Ozsvath, 2009). According to Davison (1983), soils contain  $20-1000 \,\mu g \, g^{-1}$  of fluoride, excluding natural resources. The total fluoride content of industrially polluted soils can reach 2700 µg  $g^{-1}$ , but fluoride concentrations as high as 3650  $\mu$ g  $g^{-1}$  soil have been reported in cases of severe pollution (Cronin et al., 2000). Fluoride may be present naturally in the soil environment (Weinstein, 1977) as the mineral villiaumite which contains 55% fluorine (Rao, 2003). Fluoride is supplied to the soil environment by industrial processes (EPA, 1978), phosphate fertilizers (Loganathan et al., 2001: Reddy and Kaur, 2008) and volcanic ash (Cronin et al., 2003). Fluorides can also reach soil from atmospheric air, but their content is too low to exert a negative impact on agriculture (McClenahen, 1976). As a halogen salt, sodium fluoride may induce changes in soil microbial activity and the composition of soil-

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dwelling microbial communities (Bagy and Abdel-Hafez, 1984; Asghar et al., 2012). In a study by Ochoa-Herrera et al. (2009), microbial biomass and soil enzymatic activity decreased due to the presence of fluoride. Sodium fluoride can inhibit the activity of soil enzymes, including dehydrogenase, arylsulfatase, alkaline phosphatase (Wilke, 1987), acid phosphatase, peroxidase and ATPase (Reddy and Kaur, 2008). The addition of salt to soil could increase osmotic pressure and contribute to soil erosion. These mechanisms can significantly decrease agricultural output (Omar et al., 1994). The organic matter content of salt-affected soils can undergo visible changes (Walpola and Arunakumara, 2010). Soil alkalinity can also contribute to transformation of organic matter, degradation of soil structure and soil aggregates (Mavi et al., 2012). Wilke (1987) reported that iron, phosphorus and aluminum leaching and increased organic matter content can be attributed to the presence of sodium fluoride in soil. The negative impact of fluorides on the humus content of soil was reported by Zorina et al. (2010). Fluorides may exert toxic effects on plants, cause visible damage to the aboveground parts of plants, or decrease yield (Moeri, 1980; Kumar and Rao, 2008). Sodium fluoride is also an inhibitor of photosynthesis (McLaughlin and Barnes, 1975; Parida and Das, 2005).

According to Arora and Bhateja (2014), fluoride occurs naturally and ranks 13th in terrestrial abundance. Soil contamination with sodium fluoride poses a major environmental problem, which is why new research methods are needed to increase the accuracy of analyses of the impact of sodium fluoride content on soil. Fluoride can be transferred from the soil environment to plants and animals, and it has an indirect effect of humans who consume foods containing this element. Fluoride can also be accumulated in crops (Arora and Bhateja, 2014). According to Cronin et al. (2000), fluoride is characterized by a tendency to persist in the soil, and it can be accumulated in significant amounts over the years. The presence of fluoride at concentrations significantly higher than naturally occurring levels may lead to fluorosis in humans and grazing animals. The estimation of critical fluoride concentrations in soils that are toxic to organisms is a difficult task because the ability to retain fluoride differs between soils. Slightly acidic, fine-textured and amorphous Al-hydroxide-rich soils can retain particularly high fluoride concentrations. The above reduces the fluoride uptake of plants. Fluoride retained in soil can, however, exert a negative impact on soil microorganisms. Elevated fluoride concentrations (380-1803  $\mu$ g g<sup>-1</sup> soil) were found to inhibit microbial growth and activity and decomposition of organic matter, fluoride doses below 200 µg  $g^{-1}$  soil inhibited soil respiration and dehydrogenase activity, whereas doses of 200–2000  $\mu$ gg<sup>-1</sup> soil inhibited denitrification (Cronin et al., 2000). As it has been outlined above, information about critical fluoride concentrations in soils, affecting the activity of soil microorganism, is inconclusive and insufficient. McLaughlin et al. (2001) found that fluoride can accumulate in soil and determined its distribution in the soil profile at more than eight years and at more than fifty years in areas subjected to high levels of phosphatic fertilization. The presence of fluoride, especially in amounts significantly exceeding its natural concentrations in the soil, inhibits microbial growth, slows down the decomposition of organic matter and leads to its accumulation (Ochoa-Herrera et al., 2009). For this reason, the effects of fluoride accumulation in soil have to be carefully assessed and predicted.

The aim of this study was to determine the effect of sodium fluoride on microbial activity and the thermokinetics of glucose decomposition in soil. Fluoride concentrations considerably higher than those typically found in soils have also been examined in this experiment.

#### 2. Materials and methods

#### 2.1. Experimental site

The experiment was performed on soil samples collected at the Research Station in Tomaszkowo, Poland (53°43'N, 20°24'E), operated by the University of Warmia and Mazury in Olsztyn. Mean annual air temperature in the experimental site is 7.81 °C, and mean temperature during the growing season is 14.8 °C. Annual precipitation approximates 656 mm, including 439 mm in the growing season. The growing season begins in April and ends in October. The experimental field was composed of class IVb rusty brown soil (Cambisol) (Olszewska and Grzegorczyk, 2008; Kostrzewska et al., 2014). The experimental field was sown with oats (Avena sativa L.) in the year of soil sampling. The following fertilizers were applied before sowing: nitrogen at 50 kg ha<sup>-1</sup> (as 46% urea), phosphorus at  $50 \text{ kg} \text{ ha}^{-1}$  (as 40% granular triple superphosphate) and potassium at  $50 \text{ kg} \text{ ha}^{-1}$  (as 60% potassium salt). 46% urea was applied at 100 kg ha<sup>-1</sup> in the tillering stage, and 34% ammonium sulfate was applied at 50 kg ha<sup>-1</sup> during heading. Soil was also enriched with straw. Soil was characterized by the following parameters:  $C_{org}$ : 33 g kg<sup>-1</sup>, N<sub>tot</sub>: 1.23 g kg<sup>-1</sup>, C:N ratio of 26.83, P<sub>2</sub>O<sub>5</sub>: 152 mg kg<sup>-1</sup>, K<sub>2</sub>O: 287 mg kg<sup>-1</sup> and 5.74% organic matter.

### 2.2. Sample collection and physicochemical and microbial properties of soil samples

Soil was sampled at a depth of 0–20 cm. The 15 soil samples were collected. Samples were taken from 5 locations of 3 randomly chosen blocks, from the center and corners of each block. Then the samples were homogenized into one complex sample. Soil was stored in a laboratory  $(20–22 \,^{\circ}C)$  to produce air-dry samples. Soil was passed through a 2 × 2 mm mesh. The samples were stored in the polyethylene bags at 4  $^{\circ}C$ . Three samples of soil, taken from a homogenized complex sample were used for each analysis.

Soil reaction (pH) was measured potentiometrically with the Hanna pH meter 213 (Hanna Instruments). Electrolytic conductivity (EC) was determined with the use of the Hanna conductivity meter 9033 (Hanna Instruments). The microbial biomass (SIR-SMB) (mg C kg<sup>-1</sup>) content of soil samples was calculated by the modified SIR3 (AA) method (SIR: substrate-induced respiration; AA: alkaline absorption) (Beck et al., 1997; Nakamoto and Wakahara, 2004; Cabral and Sigstad, 2011). Soil reaction, electrolytic conductivity and SIR-SMB measurements were performed in three replicates.

#### 2.3. Microcalorimetric measurements

Calorimetric methods are used to determine the microbial activity (Barros et al., 2007a) and in kinetic studies of soil (Barros et al., 2007b). The calorimetric set used in the measurements was composed of two types of heat-conduction microcalorimeters, a popular thermostat and a precise temperature controller (Fluke 2100). Every calorimeter consisted of five individual calorimetric cells, including four measuring cells and one reference cell. Reference cells contained vessels filled with Al<sub>2</sub>O<sub>3</sub> and water whose weight was similar to that of the measuring vessels. One of the four remaining cells in calorimeters contained a vessel with thermally inactive material. Soil samples were placed in 30 cm<sup>3</sup> HDPE vessels (0.D. -31 mm) in the first calorimeter and in  $20 \text{ cm}^3$ glass ampules (O.D. – 22.5 mm) in the second calorimeter. The results of measurements were recorded with the Data Logger (Pico Technology). The results of calorimetric measurements were used to calculate the rate of heat production (RHP), peak time (PT), thermal effects  $(Q_t)$  (Wadsö and Goldberg, 2001), apparent growth rate constant (k), generation time  $(t_G)$  (Wang et al., 2008) and inhibitory ratio (I) (Yao et al., 2008) of the examined processes.A water solution of ammonium sulfate (1 mg of ammonium sulfate per 1 g of DM soil) and a water solution of sodium fluoride in the appropriate doses were introduced to soil 12 h before measurements. Soil samples were treated with six doses of sodium fluoride: 1105, 2210, 4420, 6631, 8841 and 11051 mg kg<sup>-1</sup> DM soil. The doses were calculated based on the mass of fluoride ions per 1 kg DM soil (Table 1). The control samples did not contain sodium fluoride.

Table 1	
Doses of sodium fluoride and fluoride ions	

Dose of NaF (mg kg <sup>-1</sup> DM soil)	Dose of F <sup>-</sup> (mg kg <sup>-1</sup> DM soil)
0	0
1105	500
2210	1000
4420	2000
6631	3000
8841	4000
11051	5000

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