TECHNOPARK KRALUPY

Laboratory report: TPK — 570/13/2016

Determination of photocatalytic activity of FN® paints using ISO standard method

a) removal NOx: ISO 22197-1

b) removal of acetaldehyde: ISO 22197-2

c) removal of formaldehyde: ISO 22197-4

d) removal of toluene: ISO 22197-3

Customer: Advanced Materials-JTJ s.r.o

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5th September 2016

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1 Objective

The objective is to determine photocatalytic activity of supplied samples in gaseous phase using ISO standard methods. Photocatalytic paint FN2 was applied on concrete blocks (10x5x2 cm) using paint roller. The coating was done in two layers, the second layer was applied after 24 hours of application of first layer.

Coated samples were tested for its ability to removed pollutants from gaseous phase using following ISO standards methods:

a) removal of NOx: ISO 22197-1

b) removal of acetaldehyde: ISO 22197-2

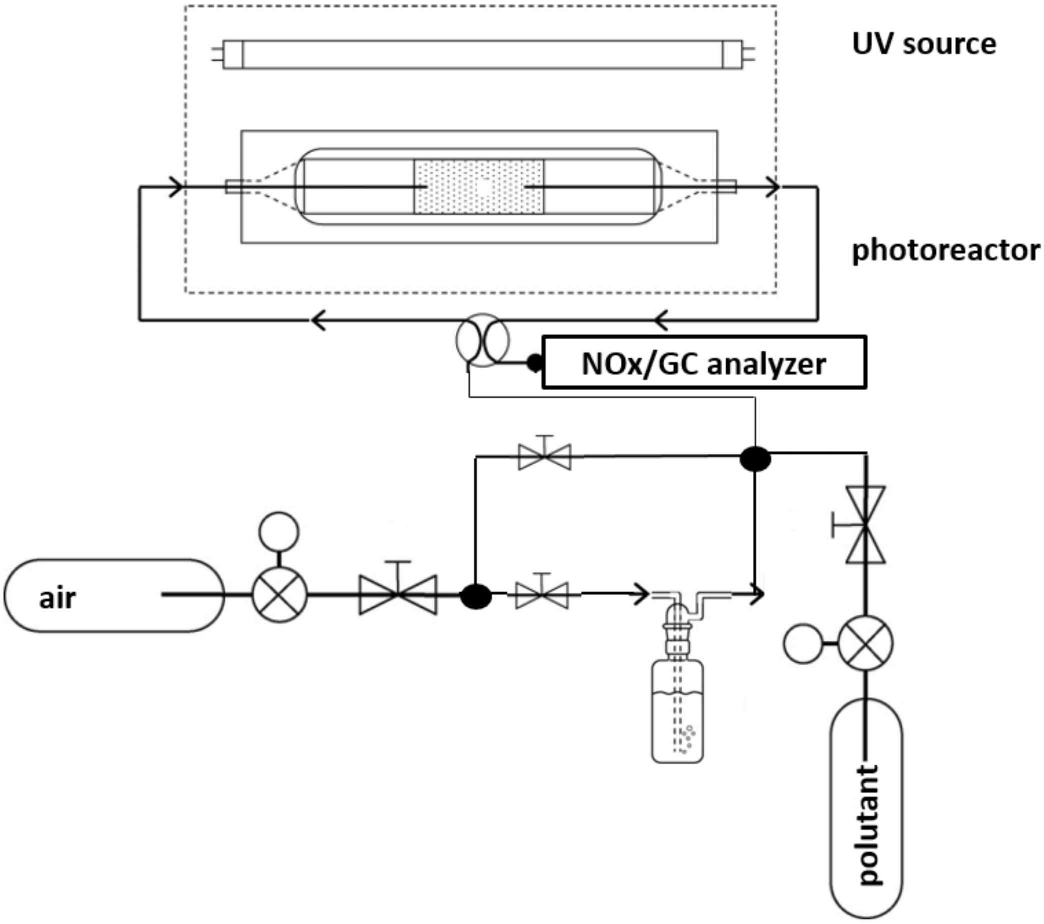
c) removal of formaldehyde: ISO 22197-4

d) removal of toluene: ISO 22197-3

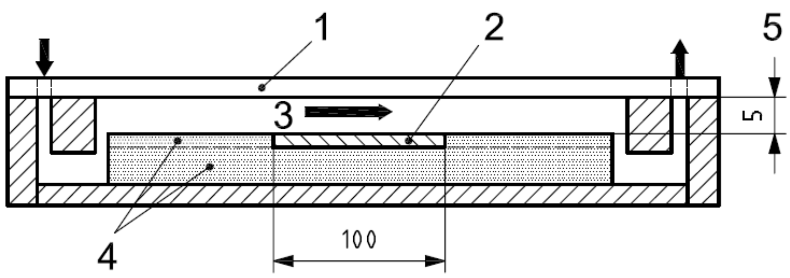
2 Methodology

2.1 Used equipment

The test equipment enables a photocatalytic material to be examined for its pollutant-removal capability by supplying the test gas continuously, while providing photoirradiation to activate the photocatalyst. It consists of a test gas supply, a photoreactor, a light source and pollutant measurement equipment. Schematic diagram of the test equipment is shown on Fig. 2.1. In case of photocatalytic degradation of organic pollutants the analysis is proved by CG-FID, in case of degradation of NOx the concentration of NO, NO_2 and the sum of NOx is realized by chemiluminescence analyzer. A cross sectional view of the photoreactor is illustrated in Fig. 2.2.



2.1: A schematic of the test equipment



2.2: Cross-sectional view of the photoreactor (1-optical window, .2-test piece,3-test gas flow, 4-height-adjusting plate, 5-air layer thickness, 6-test piece

2.2 Operating procedure

Mass controllers were adjusted to achieve the initial concentration of test gas. The ratio of wet/ dry air is set using needle valves to obtain the humidity 50%. Before photocatalytic experiments sample are pretreated by UV irradiation (24 hours, 2x fluorescent tube Eiko, emission maximum 351 nm, intensity of UV light 3 mW/cm^2).

The intensity of UV light during photocatalytic experiment was set at 1 mW/cm^2 by changing the optical length (UV source 2x fluorescent tubes Eiko, emission maximum at 351 nm). At first the test gas was loaded outside the reactor (by pass) to achieve stable concentration of pollutant. After that the gas was introduced into photoreactor which was covered by aluminum foil to observe the absorption process of the test piece. When the concentration matches the initial concentration of testing gas the light irradiation started by removal of aluminium foil.

After starting light phase, concentration of test gas was continuously measured for 3 hours in case of formaldehyde, acetaldehyde and toluene and 5 hours in case of NOx respectively. In case of acetaldehyde, formaldehyde and toluene the photocatalytic activity is expressed by the degraded amount of the pollutant in the last hour of the test. In the case of NOx test photocatalytic activity is expressed as an amount of removed NOx during 5 hour of UV irradiation. Condition of ISO standard methods are summarized in Tab. 2.3

Standard	ISO 22197-1	ISO 22197-2	ISO 22197-3	ISO 22197-4
pollutant	NO	acetaldehyde	toluene	formaldehyde
concentration (ppm)	1	5	5	1
total gass flow (dm^3/min)	3	1	0,5	3
time of the experiment (hod.)	5	3	3	3
intensity of UV light (mW/cm^2)	1	1	1	1
expression of photocatalytic activity	amount of degraded NOx during test	amount of degraded acetaldehyde in last hour of the test	amount of degraded toluene in last hour of the test	amount of degraded formaldehyde in last hour of the test

2.3: Conditions of the ISO standards

2.3 Calculations

2.3.1 Removal of NOx

The net amount of NOx removed by the test piece is calculated using formula (1),

n_{NOx}	the amount of NOx removed by the test piece (micromole)
n_{ads}	the amount of NOx adsorbed by the test piece (micromole)
n_{NO}	the amount of NO removed by the test piece (micromole)
n_{NO2}	the amount of NO2 formed by the test piece (micromole)
n_{des}	the amount of NOx removed by the test piece (micromole)

$$n_{NOx} = n_{ads} + n_{NO} - n_{NO2} - n_{des}$$

(1)

amount of NOx adsorbed by the test piece (n_{ads}) is calculated using formula (2),

ϕ_{NOi}	the supply volume fraction of NO (ppm)
ϕ_{NO}	the supply volume fraction of NO at the reactor exit (ppm)
ϕ_{NO2}	the nitrogen dioxide volume fraction at the reactor exit (ppm)
f	the air-flow converted into that at the standard state (0°C, 101,3kPa, and dry gas basis) (dm^3/min)

$$n_{ads} = \frac{f}{22{,}4}\left(\int (\phi_{NOi} - \phi_{NO)} dt - \int \phi_{NO2} dt\right)$$

(2)

Amount of NOx desorbed by the test piece (n_{des}) is calculated using formula (3),

ϕ_{NO}	the supply volume fraction of NO at the reactor exit (ppm)
ϕ_{NO2}	the supply volume fraction of NO at the reactor exit (ppm)
f	the air-flow converted into that at the standard state (0°C, 101,3kPa, and dry gas basis) (dm^3/min)

$$n_{des} = \frac{f}{22{,}4}\left(\int \phi_{NO} dt + \int \phi_{NO2} dt\right)$$

(3)

the amount of NO removed by the test piece NO (n_{NO}) is calculated using formula (4),

ϕ_{NOi} the supply volume fraction of NO (ppm)

ϕ_{NO} the supply volume fraction of NO at the reactor exit (ppm)

f the air-flow converted into that at the standard state (0°C, 101,3kPa, and dry gas basis) (dm^3/min)

$$n_{NO} = \frac{f}{22,4}\left(\int (\phi_{NOi} - \phi_{NO)} dt\right)$$

(4)

amount of NO_2 (n_{NO2}) formed by the test piece is calculated using the formula (5):

$$n_{NO2} = \frac{f}{22,4}\left(\int \phi_{NO2} dt\right)$$

(5)

ϕ_{NO2} the supply volume fraction of NO_2 at the reactor exit (ppm)

f the air-flow converted into that at the standard state (0°C, 101,3kPa, and dry gas basis) (dm^3/min)

2.3.2 Removal of acetaldehyde

removed quantity of acetaldehyde (n_A) is calculated using equation (6),

R_A is the removal percentage, by the test piece, of acetaldehyde (7) (%)

ϕ_{A0} is the supply volume fraction of acetaldehyde (ppm)

ϕ_A is the quantity of acetaldehyde at the reactor exit (ppm)

f the air-flow converted into that at the standard state (0°C, 101,3kPa, and dry gas basis) (dm^3/min

$$n_A = R_A \frac{\phi_{A0}\, f \cdot 1,016 \cdot 60}{100 \cdot 22,4}$$

(6)

$$R_A = 100 \cdot \frac{\phi_{A0} - \phi}{\phi_{A0}}$$

(7)

When R_A is either below 5% or more than 95%, the removal percentage shall be expressed as below 5% or more than 95%, respectively for uncertainly reasons.

2.3.3 Removal of formaldehyde

The amount of formaldehyde remove by the test piece (n_F) is calculated using (8):

R_F	removal percentage , by the piece, of formaldehyde(9)) (%)
ϕ_{F0}	the supply volume fraction of formaldehyde (ppm)
ϕ_F	volume fraction of formaldehyde at the reactor exit (ppm)
f	the flow rate of the test gas converted into that standard state (l/min, 0°C, 101,3 kPa, dry gas basis)

$$n_F = R_F \frac{\phi_{F0}\, f \cdot 1{,}016 \cdot 60}{100 \cdot 22{,}4}$$

(8)

$$R_F = 100 \cdot \frac{\phi_{F0} - \phi}{\phi_{F0}}$$

(9)

When R_F is either below 5% or more than 95% R_F is expressed as below 5% or more than 95% respectively. Than the quantity of formaldehyde is calculated by equation (10) and (11) respectively.

$$n_F = 0{,}136\phi_{F0} \quad f$$

(10)

$$n_F = 2{,}585\phi_{F0} \quad f$$

(11)

2.3.4 Removal of toluene

Amount of removed toluene by the test piece (n_T) is calculated using formula (12),

R_T	the removal percentage, by the test piece, of toluene (13)) (%)
ϕ_{T0}	the supply fraction of toluene (ppm)
ϕ_T	the volume fraction of toluene at the reactor exit
f	the flow rate of the test gas converted into that standard state (l/min, 0°C, 101.3 kPa, dry gas basis)

$$n_T = R_T \frac{\phi_{T0}\, f \cdot 1{,}016 \cdot 60}{100 \cdot 22{,}4}$$

(12)

$$R_T = 100 \cdot \frac{\phi_{T0} - \phi}{\phi_{T0}}$$

(13)

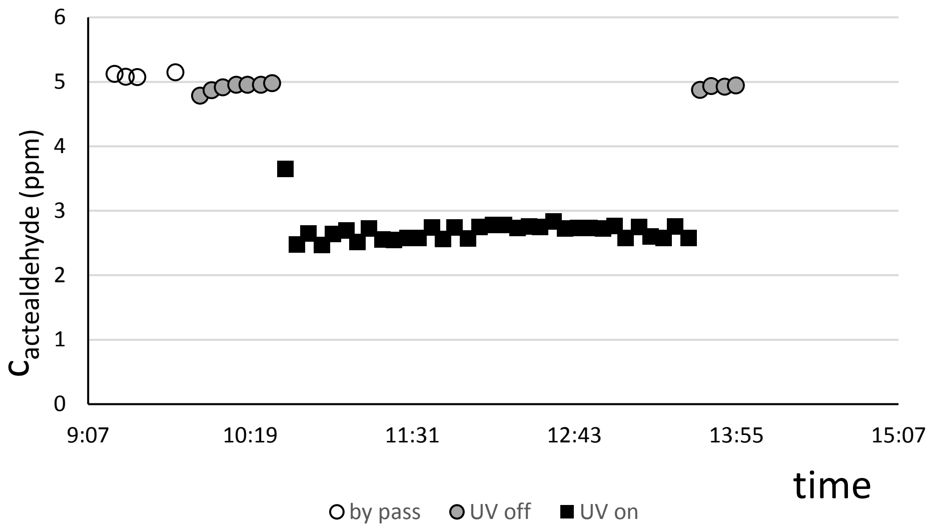
The quantity of toluene removed (n_T) is calculated by Equation 2. Where R is either below 5% or more than 95%, it is expressed as below 5% or more than 95% respectively.

3 Results and discussion

3.1 Determination of photocatalytic activity using acetaldehyde (ISO 22197-2)

date of the test:	1.9. 2016
temperature in laboratory (°C)	24°C
relative humidity in the lab.(%)	37
beginning of the experiment	9:19
dark phase	9:57
UV light on	10:35
UV light off.	13:34

3.1: Conditions of the acetaldehyde test



3.2: Dependence of acetaldehyde concentration on time of photocatalytic experiment

On the Fig. 3.2 there is shown dependence of acetaldehyde concertation on time of exposition. During photoiradiation it comes to decrease of acetaldehyde with conversion about 46% which is proportional to 6,2 micromole of removed acetaldehyde in the last hour of the test.

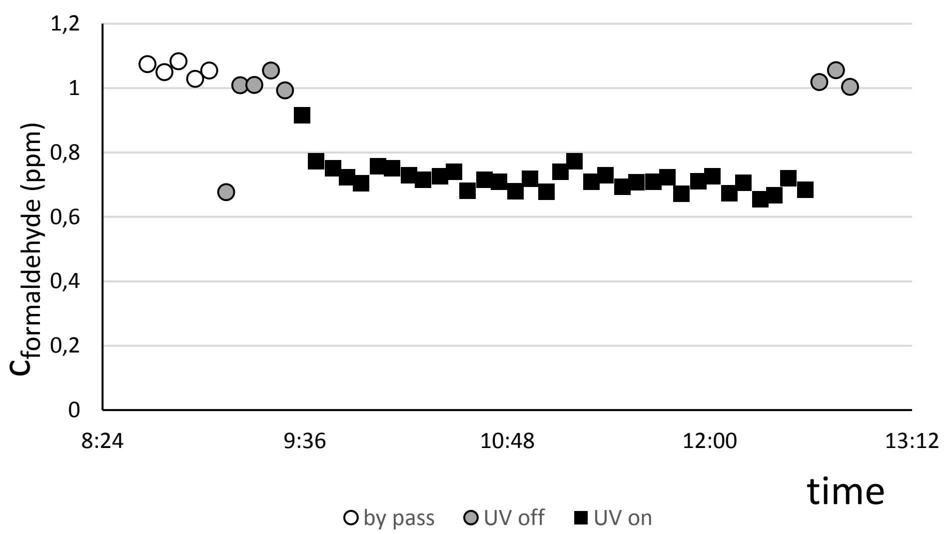
R_A	46%
n_A	6,2 micromole

3.3: The amount of removed acetaldehyde

3.1 Determination of photocatalytic activity using formaldehyde (ISO 22197-4)

date of the test:	31. 8. 2016
temperature in laboratory (°C)	22,5°C
relative humidity in the lab.(%)	31
beginning of the experiment	8:40
dark phase	9:08
UV light on	9:35
UV light off.	12:39

3.4: Conditions of the formaldehyde test



3.5: Dependence of concentration of formaldehyde on time of photocatalytic experiment

In the Fig. 3.5 there is illustrated dependence of acetaldehyde concertation on time of photoiradiation. During exposition it comes to decrease of concertation at the reactor exit with the conversion of 31% which is proportional to 2,6 micromole in last hour of the test.

R_F	31%
n_F	2,6 micromole

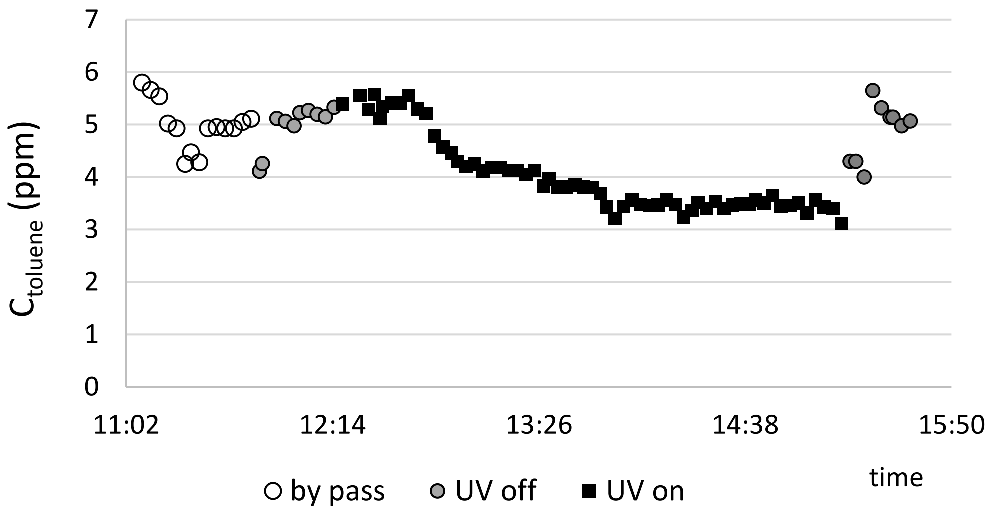
3.6:The amount of removed formaldehyde

3.2 Determination of photocatalytic activity using toluene (ISO 22197-3)

Determination of photocatalytic activity using formaldehyde was realized with the Department of Inorganic Technology UCT Prague. Used equipment was practical the same as was shown in Fig. 2.2.

date of the test:	2.9. 2016
temperature in laboratory (°C)	24,8
relative humidity in the lab.(%)	40
beginning of the experiment	11:08
dark phase	11:49
UV light on	12:18
UV light off	15:12

3.7: Conditions of the test – unexposed sample (removal of toluene))



3.8: Dependence of concentration of toluene on time of photocatalytic experiment

In the Fig. 3.8 there is decrease of toluene concentration on time of photocatalytic experiment. During exposition of UV light it comes to decrease of toluene with the conversion about 34% which is equal to 2,4 micromole of removed toluene in the last hour of the test.

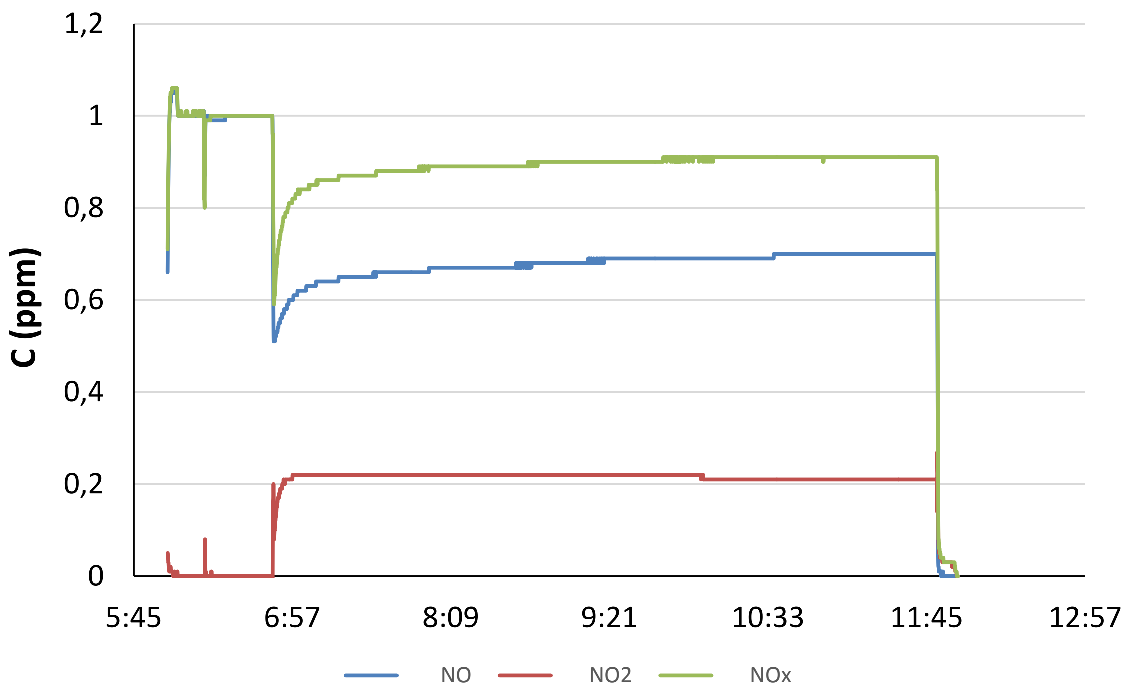
R_T	34%
n_T	2,4 micromole

3.9: The amount of removed toluene

3.3 Determination of photocatalytic activity using removal of NOx (ISO 22197-1)

date of the test:	6.9. 2016
temperature in laboratory (°C)	23
relative humidity in the lab.(%)	39
beginning of the experiment	6:01
dark phase	6:17
UV light on	6:48
UV light off	11:50

3.10: Conditions of the NOx test



3.11: Dependence of concentration of NOx on time of photocatalytic experiment

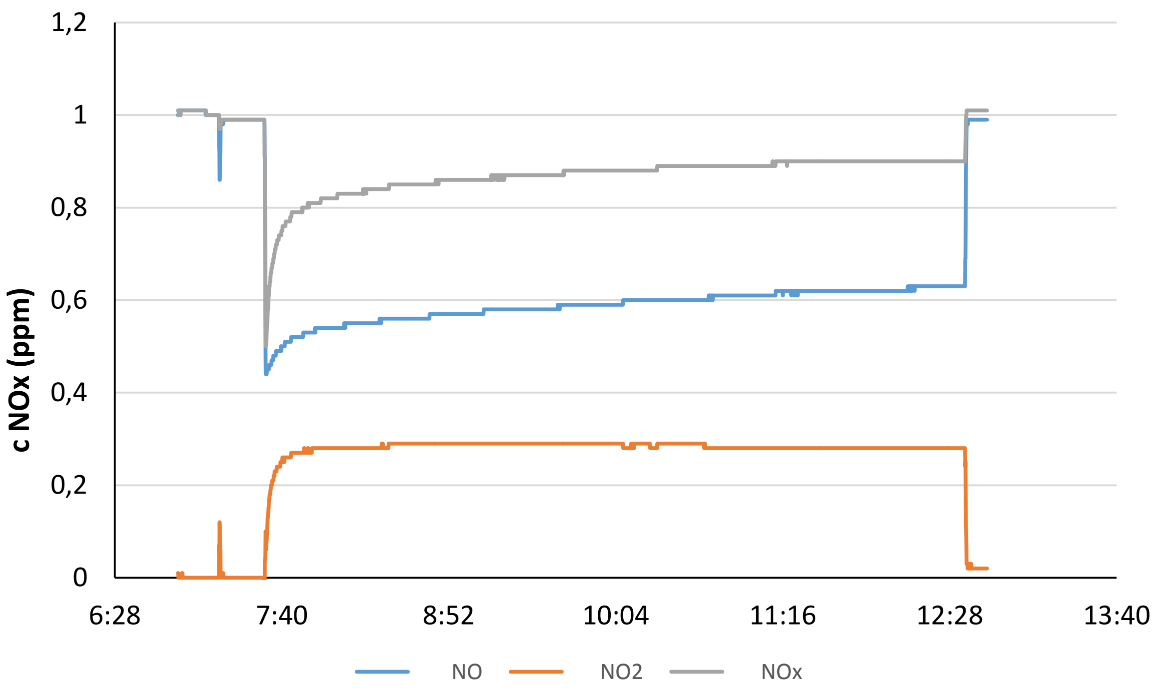
n_{ads}	0,06 micromole
n_{des}	0,09 micromole
n_{NO}	32 micromole
n_{NO2}	21 micromole
$\mathbf{n_{NOx}}$	11 micromole

3.12: Amount of removed NOx

date of the test:	5.9. 2016
temperature in laboratory (°C)	24
relative humidity in the lab.(%)	40
beginning of the experiment	6:56
dark phase	7:13
UV light on	7:33
UV light off	12:35

3.13: Conditions of the test NOx test

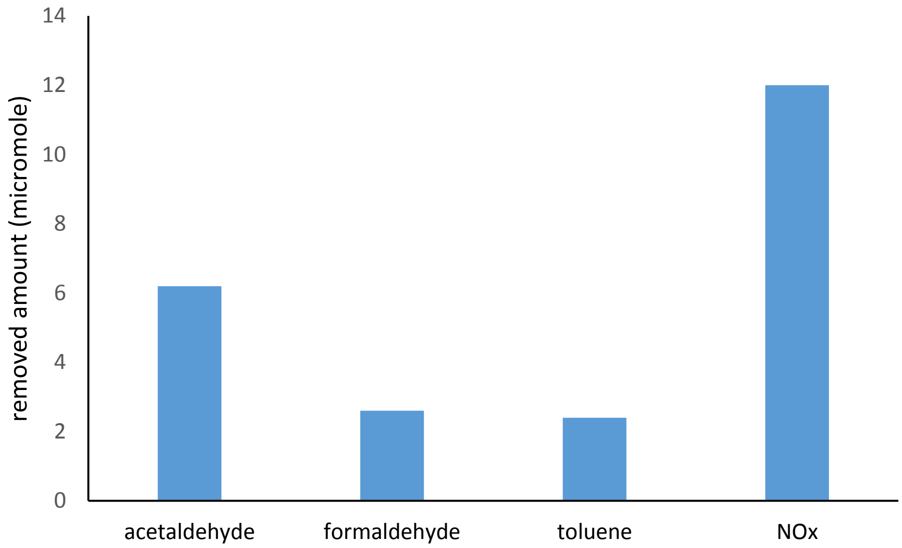
In this case photocatalytic activity was determined in sample which was tested before using formaldehyde and acetaldehyde. Comparing the Fig 3.11 and 3.14 and the results in 3.12 and 3.15 it follows that the sample after tests with organic pollutants exhibit similar photocatalytic activity as untested sample.



3.14: Dependence of concentration of NOx on time of photocatalytic experiment sample which was tested before using acetaldehyde and formaldehyde

n_{ads}	0,1 micromole
n_{des}	-
n_{NO}	39 micromole
n_{NO2}	27 micromole
$\mathbf{n_{NOx}}$	12 micromole

3.15 Amount of removed NOx sample which was before tested using acetaldehyde and formaldehyde



3.16: Comparison of removed amount of the pollutant during photocatalytic test

In the Fig. 3.16 there is comparison of photocatalytic activity expressed as an amount of removed pollutant during test. It can be assumed that sample FN2 is able to decomposed acetaldehyde, formaldehyde, toluene and also NOx. The removed amount differ by used ISO method. In case of organic pollutants the degraded amount is calculated using average conversion in the last hour of the test, in case of NOx the degraded amount is proportional to the difference of integrated values of NO and NO_2 concentrations respectively during 5 hours of the test.

4 Conlucions

Photocatalytic activity of FN2 paint was determined using various standard ISO method. It was found that FN2 paint is able to decomposed formaldehyde, acetaldehyde, toluene and also NOx. Sample which was tested before using formaldehyde and acetaldehyde exhibit similar photocatalytic activity to NOx as sample which wasn't tested to organic pollutants.

According to the archived results it can be concluded that FN2 paint can be applied in air treatment process especially in application on large surfaces as a facade of buildings.