Original paper

# Synthesis and application of natural sorbents with silver nanoparticles in reducing the emission of odours

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**Abstract.** The paper concerns the applicability of silver nanoparticles for reducing the emission of odours. Silver nanoparticles were successfully adsorbed on three different dolomitic limestone samples. In the next stage, wastewater from the meat industry was mixed with sorbents modified with a variable content of silver nanoparticles. After 4 days and 7 days, the concentration of ammonia was examined spectrophotometrically. Also, the degree of reduction of total odour concentration by olfactometric method was carried out. Depending on the concentration of the silver nanoparticles on the sorbents, the concentration of ammonia was 15-42 $\mu g/cm^3$ for nAg content equal to 0.08%, while for sorbents containing approximately 36% of nAg the concentration of ammonia was 0.09-16 $\mu g/cm^3$. A leaching test of nanosilver from sorbents confirmed that the particles were bound with the sorbents by strong bonds. The percentage of eluted silver increased from 0.67% to 11%, with increased initial concentrations of nAg on the sorbents from 0.18% to 0.37%.

*Key-words:* nanosilver, dolomitic limestone, odours, ammonia, deodorisation

## 1. Introduction

The continuous increase in urbanisation causes a progressive increase in the production of pollutants, including odours. Odours getting into the air affect the quality of life and human health. As a result, odours are among the major air pollutants and are regulated by law in many countries (Gębicki, Szulczyński 2019).

The addition of nanoparticles improves the physicochemical properties of commercial products. Materials with nanoparticle additives have been widely used in industries such as

chemical, food, construction, medicine, pharmacy, cosmetics and many others (Yang et al. 2019). Nanosilver represents the group of most commonly used nanomaterials. Nanostructured silver is characterised by a high efficiency in destroying a broad spectrum of both Gram-positive bacteria (*Bacillus*, *Clostridium*, *Enterococcus*, *Listeria*, *Staphylococcus* and *Streptococcus*) and Gram-negative bacteria (*Acinetobacter*, *Escherichia*, *Pseudomonas*, *Salmonella* and *Vibrio*), without excluding strains resistant to antibiotics (strains resistant to methicillin and vancomycin, as well as *Staphylococcus aureus* and *Enterococcus faecium*) (Manosalva et al. 2019; Ruttkay-Nedecky et al. 2019). This is due to the interaction of nanosilver with the thiol group located in L-cysteine, which leads to a reduction in the enzymatic activity of proteins. Some coenzymes and enzymes such as NADH or succinate dehydrogenase can react with silver nanoparticles, which leads to a damaging of the chain of cellular respiration. As a consequence, the flow of electrons is blocked (Kailasa et al. 2019; Singh et al. 2019). Although the mechanism of the interaction of nanosilver with fungi is not known in detail, it is clear that it exhibits great activity against fungi strains such as *Candida albicans*, *Candida tropicalis, Saccharomyces cerevisiae* and *Aspergillus fumigates* (Aldossari et al. 2015; Zangeneh et al. 2019).

Odour abatement techniques in the meat industry using nanosilver exist and have been investigated (Kowalski et al. 2009; Kowalski et al. 2010). One of the methods to reduce odour production is the disinfection of food industry facilities and production lines. Antimicrobial agents prevent the development of various bacteria and fungi, especially moulds that are the main cause of odours in the meat industry. Disinfection prevents meat and food product decomposition, which causes the release of odours (Wysocka et al. 2019). The effectiveness of the alcoholic suspension of nanosilver applied with mineral sorbents in the reduction of ammonia emissions from the sheep dung was confirmed (Dobrzański et al. 2010).

The increasing use of nanomaterials results in their increasing release into the environment (Wimmer et al. 2018), which may have a substantial impact on the functioning of living organisms. It is necessary to monitor possible pathways for the release of nanoparticles into the environment. The challenge in determining nanoparticles is their small size and the low concentrations used in products. One should be aware that once introduced into the environment, nanoparticles are pollutants of soil, surface water and groundwater, as well as flora and fauna (Ye et al. 2017). The objective of limiting the uncontrolled release of nanoparticles into the environment may be achieved through the deposition of particles on the surfaces of other materials such as sorbents (McGillicuddy et al. 2017). After use, the materials can be cleaned and activated, thus enabling their reuse (Ebbs et al. 2016).

The purpose of this work was to obtain sorption materials based on dolomite modified with silver nanoparticles, which can effectively reduce odour emissions, mainly ammonia. The scope of the work included the preparation of the materials modified with silver nanoparticles with a variable content of nanosilver, and the determination of the degree of nanoparticles' leaching from the surface of the sorbents. At the next stage, a series of studies on the reduction of the odour emissions from prepared wastewater from the meat industry was carried out. The concentration of ammonia was assessed spectrophotometrically. The degree of reduction of total odour concentration was determined by the olfactometry method.

## 2. Experimental

### 2.1. Materials

Silver nanoparticles obtained via the chemical reduction process were used in studies on the reduction of odour emissions. Silver nitrate $AgNO_3$ (POCH), sodium hydroxide (Sigma Aldrich) and tannic acid (Sigma Aldrich) were used in the synthesis of the silver nanoparticles. The initial solution of the silver nanoparticles was prepared by mixing 500 ml of 1.50 g/l silver nitrate(V) solution with 500 ml of tannic acid solution. The molar ratio of tannic acid to silver ions was equal to 0.2. The pH of the solution was adjusted to 8 and was achieved by adding droplets of 5 mol/l NaOH solution. The initial nanosilver suspension concentration was equal to 460 mg/l. The stock solution was diluted with deionised water to obtain the desired concentration of silver nanoparticles. Nanosilver produced by this method has an average particle size of 62 nm and an electrokinetic potential of -29.9 mV.

Three different sorbent materials of natural origin (dolomitic limestone [DL] from three different sources) were used in the research. The sorption characteristics of all materials were determined by the BET method. The results are shown in Table 1.

TABLE 1

Parameters of dolomitic limestones (DL) determined by a low temperature sorption study

| Parameter | DL 1 | DL 2 | DL 3 |
|---|---|---|---|
| Specific surface area $[m^2/g]$ | 2.91 | 7.61 | 5.49 |
| Pores volume $[cm^3/g]$ | 0.007 | 0.020 | 0.021 |
| Pores size [nm] | 1.6 | 1.8 | 1.6 |

The phase composition of DL was defined by the XRD method. In the DL1, the presence of the following compounds was confirmed: calcite ($CaCO_3$), silicon dioxide ($SiO_2$), dolomite ($CaMg(CO_3)_2$), sillimanite ($CaMgSi_2O_6$), diopside ($Mg_3Ca(SiO_3)_4$), magnesium sulphate ($MgSO_4$) and calcium sulphate ($CaSO_4$). The DL2 consisted of anhydrite ($CaSO_4$), dolomite ($CaMg(CO_3)_2$), quartz ($SiO_2$), gypsum ($CaSO_4 \cdot 2H_2O$), magnesium sulphate ($MgSO_4$) and calcium sulphate ($CaSO_4$). In the DL3, the presence of the following compounds was confirmed: anhydrite ($CaSO_4$), calcite ($CaCO_3$), periclase (MgO), quartz ($SiO_2$) and gypsum ($CaSO_4 \cdot 2H_2O$).

Specially crafted sewage composed of water and ground pork in the mass ratio 3:1 served as the source of odours, including ammonia. The ratio of components was selected so that the concentration of gases from the wastewater was adequate to the real conditions occurring in the industry.

## 2.2. Methods

### 2.2.1. Sorption process

Sorption of silver nanoparticles on the DLs was performed by dynamic methods using nanosilver solutions at concentrations of 40.92, 89.62, 130.43 and 187.78 mg/l, obtained by diluting the stock solution. 100 ml of nanosilver suspension and 4.9-5.1 g of the sorbents were stirred on a magnetic stirrer (350 rpm). After 60 min, each suspension was filtered and the obtained filtrate was subjected to ICP-OES analysis to determine the silver concentration. The analysis was performed on a Plasma 40 apparatus from Perkin Elmer. After the sorption process, the sorbent material was subjected to SEM-EDS (Vegall-Tescan Company) analysis in order to confirm the adsorption of silver nanoparticles and the FT-IR (Nicolet 380) technique to determine the nature of the interactions between the sorbent and nanoparticles.

### 2.2.2. Nanosilver leaching studies

Elution of nanosilver was performed by placing 1.0 g of the DLs with adsorbed silver nanoparticles into 20 ml of water, which was stirred on magnetic stirrer for 60 min. The sorbents were filtered off and the filtrates were subjected to ICP-OES analysis to determine the concentration of silver.

### 2.2.3. Ammonia determination

In order to investigate the reduction of ammonia emissions by the sorbents modified with silver nanoparticles, wastewater from the meat industry was prepared. For this purpose, samples of 10 g of meat, 30 ml of water and 1 g of the modified DLs were mixed. The samples were sealed and stored at 20°C for 4 days and 7 days. After this, the ammonia concentration was measured in each sample. Figure 1 presents a schematic diagram of the apparatus used.
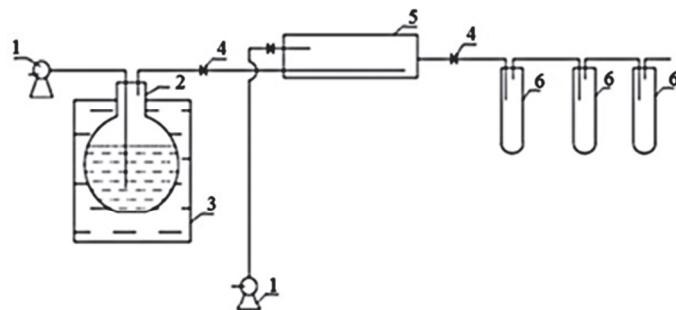


Fig. 1. Diagram of the apparatus in the ammonium absorption process (1: peristaltic pump, 2: round-bottomed flask, 3: water bath, 4: valves, 5: glass container, 6: Poleżajew wash).

The measurement of the concentration of the secreted ammonia consisted of passing a constant volume of the air through the sewage containers. The air flow was at a rate of 555.6 cm³/s. After 10 min, the air mixed with gases from the sample was transferred through tubes into Poleżajew washes with a 5% solution of sulfuric (VI) acid, which absorbs gases, including ammonia. Ammonia concentration analysis was performed by the spectrophotometrically indophenol method according to PN-91 Z-04009/02. The following equation of the calibration curve was obtained ($R^2 = 0.96$):

$$A = 0.0682c_{NH_3} + 0.0510$$

where $A$ signifies absorbance and $c_{NH_3}$ the ammonia concentration [μg/ml].

## 2.2.4. Olfactometric studies

The olfactometric method was used in order to define the odours' intensity as a sum of all odours emitted from the sample. This study was performed in order to test the applicability of nanosilver in terms of limiting a range of odour emissions, not only ammonia (Hayes et al. 3013).

Since the smell of a substance is a subjective characteristic, the best organ for measuring the intensity of odours is the human nose. Four people were the representative group. The people involved in the experiment were healthy and had correct sense of smell. The method consisted of sniffing the diluted gas above the samples, and then each person responded to the question "Do you smell gas?". The answer had to be unambiguous (i.e. yes/no), otherwise the measurement was repeated (Kośmider et al. 2012).

TABLE 2

Example of sensory evaluation card

| R | Sample volume | Individual results of each person | | | | % of „yes" |
|---|---|---|---|---|---|---|
| | | A | B | C | D | |
| | | | | | | |
| | | | | | | |
| $x_i$ | | | | | | |
| $R = \sqrt{x_i}$ | | | | | | |
| $R_{50\%}$ | | | | | | |

where: $x_i$ – result of multiplying of neighbouring dilutions at which the reply was "yes" and "no", $\overline{R} = \sqrt{x_i}$ – individual score calculated as geometric mean of neighboring dilutions at which the reply was "yes" and "no", $R_{50\%}$ – geometric mean of individual results.

Olfactometric measurements were made for seven samples prepared with three different dolomitic materials differing in silver concentration, and one without dolomitic materials. The study began with a minimum gas concentration undetectable by the human nose. Next,

this was increased up to the point at which all the people confirmed the perception of the smell. Gas samples were diluted dynamically with air using a bypass air pump with Teflon valves. The concentration of odours in sensory tests is expressed as the number of odour units (NOUs) (Kośmider et al. 2012). Table 2 presents the sensory evaluation card, which was filled by each person (A–D) taking part in the study. The NOUs is equal to the value of $R_{50\%}$ [NOU/m$^3$]. The NOUs was also graphically determined. For this purpose, a graph of the dependence of the answer 'yes' (in %) on subsequent dilutions (R) was made. The R values on the X axis are on a logarithmic scale. Then, the corresponding x value is equal to the logarithm of $R_{50\%}$ (Brattoli et al. 2011).

## 3. Results and discussion

### 3.1. Sorption and leaching

Table 3 presents the results obtained after the sorption process and after the process of elution of the nanosilver from the modified DLs. Based on the data presented in Table 3, it can be seen that the nanosilver is almost completely deposited on the sorbents. The leaching test confirmed that the particles were bound with the sorbents by strong bonds. The maximum percentage of eluted silver was equal to 11.5%.

TABLE 3

Silver content in the sorbents after the sorption and the elution processes

| Initial concentration of nanosilver solution used in the sorption process [mg/l] | Concentration of solution after sorption process [mg/l] | Content of nanosilver in the sorbent [%] | Concentration of solution after elution process [mg/l] | Percentage of leached nanosilver [%] |
|---|---|---|---|---|
| dolomitic limestone 1 (DL1) | | | | |
| 40.92 | 0.077 | 0.08 | 0.003 | 1.47 |
| 89.62 | 0.116 | 0.18 | 0.003 | 0.67 |
| 130.43 | 0.555 | 0.26 | 0.051 | 7.85 |
| 187.78 | 2.357 | 0.37 | 0.106 | 11.43 |
| dolomitic limestone 2 (DL2) | | | | |
| 40.92 | 1.357 | 0.08 | 0.005 | 2.53 |
| 89.62 | 0.755 | 0.18 | 0.045 | 10.13 |
| 130.43 | 0.584 | 0.26 | 0.056 | 8.63 |
| 187.78 | 12.160 | 0.35 | 0.077 | 8.77 |
| dolomitic limestone 3 (DL3) | | | | |
| 40.92 | 0.139 | 0.08 | 0.011 | 5.39 |
| 89.62 | 2.483 | 0.17 | 0.010 | 2.30 |
| 130.43 | 0.119 | 0.26 | 0.012 | 1.84 |
| 187.78 | 0.085 | 0.37 | 0.061 | 6.50 |

Figure 2 presents the micrographs taken with an SEM. The microphotographs present the initial surface of sorbents and their surfaces after nanosilver sorption. The initial concentration of nanosilver solution used in the sorption process was equal to 187.78 mg/l. It can clearly be seen that silver particles are deposited on sorbent surface (Fig. 3). EDS analysis confirmed the presence of silver particles on the surface of all three sorbents (Table 4).
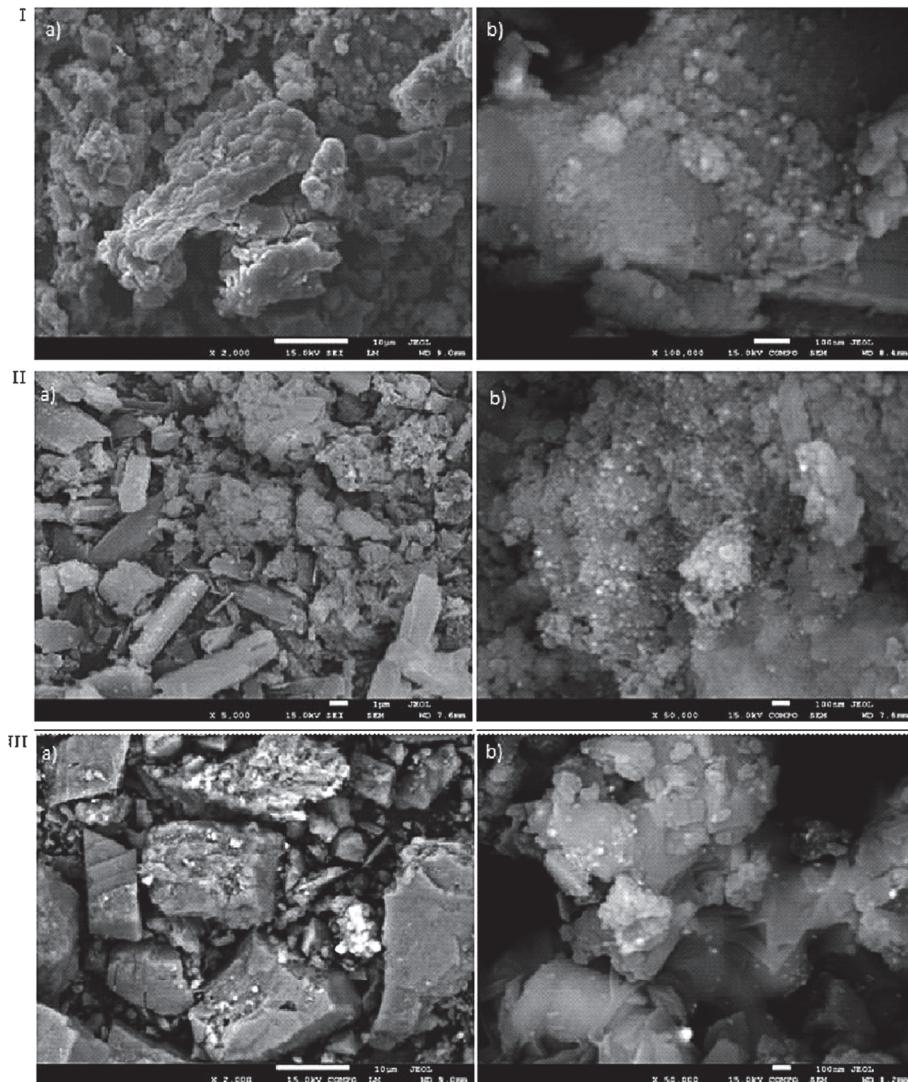


Fig. 2. SEM images of sorbents before (a) and after (b) the sorption of nanosilver ($C_{0,nAg}$ = 187.78 mg/l): I – sorbent DL1, II – sorbent DL2, III – sorbent DL3.
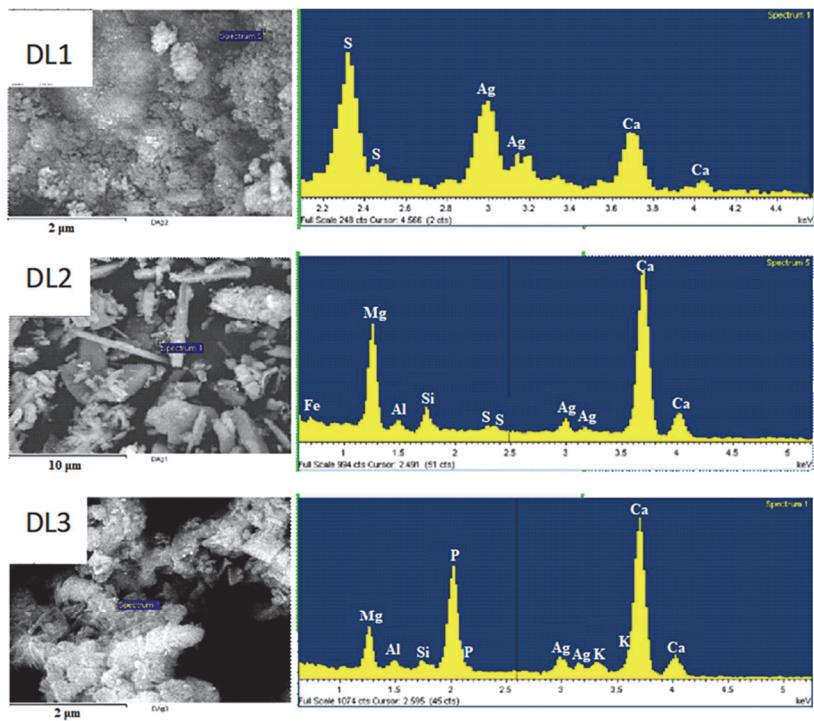
Fig. 3. EDS analysis results of sorbents after the sorption of nanosilver ($C_{0,nAg}$ = 187.78 mg/l).

The initial element composition of the dolomitic limestones

| Sorbent I | | | Sorbent II | | | Sorbent III | | |
|---|---|---|---|---|---|---|---|---|
| Element | Weight [%] | Atom ic [%] | Element | Weight [%] | Atom ic [%] | Element | Weight [%] | Atom ic [%] |
| Mg | 22.12 | 31.97 | Mg K | 0.67 | 1.02 | Mg K | 7.31 | 11.38 |
| Al | 0.95 | 1.24 | Al K | 0.55 | 0.76 | Al K | 1.23 | 1.72 |
| Si | 5.85 | 7.32 | Si K | 2.24 | 2.94 | Si K | 1.33 | 1.79 |
| S | 1.04 | 2.13 | S K | 39.06 | 44.90 | P K | 20.74 | 25.36 |
| Ca | 58.79 | 51.55 | Ca K | 49.54 | 45.56 | K K | 3.06 | 2.97 |
| Fe | 7.99 | 5.03 | Fe K | 6.64 | 4.33 | Ca K | 56.03 | 52.95 |
| Ag | 2.36 | 0.77 | Ag L | 1.29 | 0.44 | Fe K | 0.62 | 0.42 |
| | | | | | | Ag L | 9.69 | 3.40 |
| Total | 99.1 | | Total | 99.9 | | Total | 99.1 | |

In the spectrum of sorbent 1 before sorption (Fig. 4Ia), there are peaks with wavenumbers 3643 and 3468 cm$^{-1}$, which represent a hydroxyl group -OH. The peaks at 2983, 2512 and 712 cm$^{-1}$ confirmed the presence of the bonding of $CO_3^{2-}$. The peaks found within the ranges of 1200–800 and 600–400 cm$^{-1}$ confirmed the presence of the stretching vibration of Si-O. The peaks of wavenumbers equal to 1110, 671 and 611 cm$^{-1}$ confirmed

the presence of the stretching vibration of anionic group $SO_4^{2-}$. In the spectrum obtained after the sorption process (Fig. 4Ib), there are peaks at the following wavenumbers: 3542 and 3406 cm$^{-1}$. These peaks originate from the stretching vibrations of the phenolic (-OH) groups of tannic acid, which was used during the preparation of nanosilver.
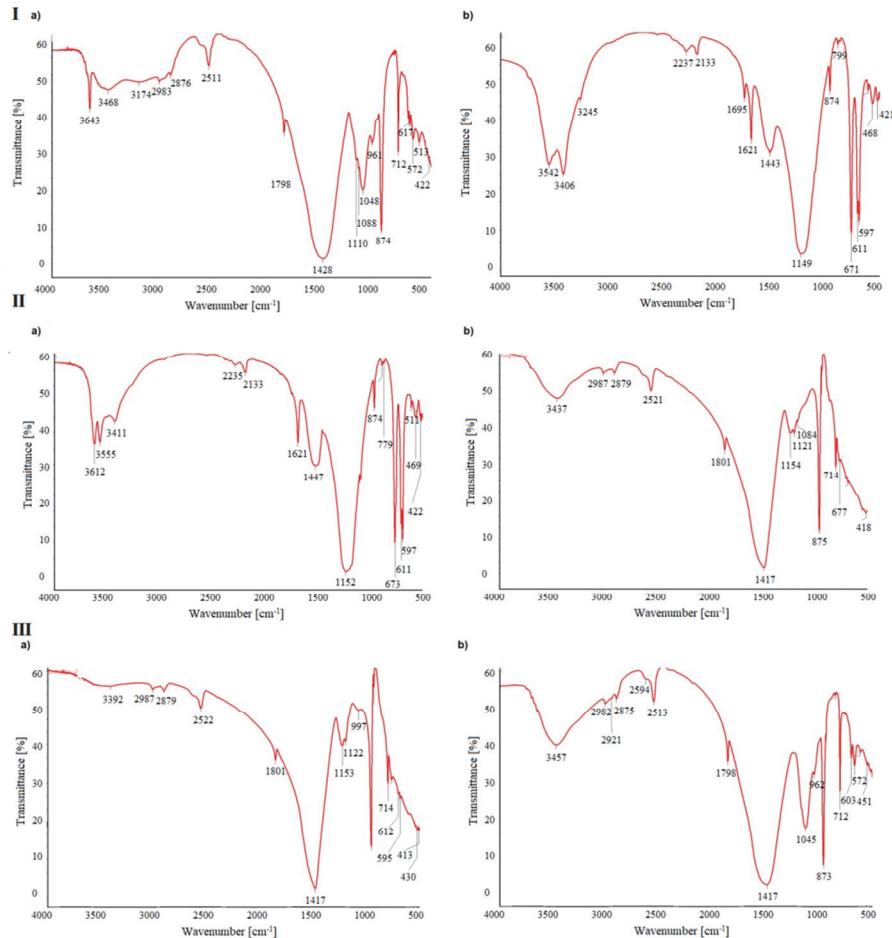


Fig. 4. FTIR spectra before (a) and after (b) the sorption process: I – sorbent DL1, II – sorbent DL2, III – sorbent DL3.

The spectrum of the sorbent DL2 before sorption (Fig. 4IIa) contains peaks with the maximum at 3612, 3555 and 3411 cm$^{-1}$, which represent hydroxyl groups (-OH). The peaks found within the ranges of 1200–800 and 600–400 cm$^{-1}$ confirmed the presence of the stretching vibration of Si-O, while the peaks at wavenumbers 1012, 673 and 611 cm$^{-1}$ confirmed the presence of the stretching vibration of anionic group $SO_4^{2-}$. In the spectrum obtained after sorption (Fig. 4IIb), the presence of the peak at 3437 cm$^{-1}$ was confirmed. This peak, as in the case of the sorbent DL1, is derived from the stretching vibration of the phenolic groups of tannic acid. In the spectrum of the initial sorbent DL3 (Fig. 4IIIa), peaks

at 2987, 2522 and 714 cm⁻¹ correspond to the binding of $CO_3^{2-}$. The peaks found within the ranges of 1200–800 and 600–400 cm⁻¹ originate from the stretching vibrations of Si-O, and those at wavenumbers 1153, 677 and 612 cm⁻¹ correspond to the stretching vibration of anionic group $SO_4^{2-}$. The spectrum obtained after sorption (Fig. 4IIIb) presents peaks at 3542 and 3406 cm⁻¹. These peaks also come from the stretching vibration of the phenolic group of tannic acid.

Based on these results, it can be concluded that the process of sorption of silver nanoparticles on the sorbents is determined by the presence on their surface of tannic acid anion, which stabilises the nanometric structure (electrosteric stabilisation).

## 3.2. Analysis of ammonia emissions

Table 5 lists the concentrations of ammonia gas in the tested samples. The DL sorbents with different content of nanosilver were tested in two replications (sample 1 and 2). The ammonia concentrations in samples after 7 days were in most cases higher than the concentrations of ammonia after 4 days. The reason for that may be the fact that after long periods of time, nanosilver loses its antibacterial properties (due to oxidation and the formation of sulphides). Increasing the concentration of nanosilver reduced the emissions of ammonia, which may be caused by the antimicrobial action of nanosilver against spoilage organisms whose vital functions lead to the formation of ammonia.

TABLE 5

Results of ammonia emission

| Content of nanosilver in the sorbent [%] | Ammonia emission after 96 hours [µg/cm³] | | Ammonia emission after 168 hours [µg/cm³] | |
|---|---|---|---|---|
| Sorbent DL1 | | | | |
| | Sample 1 | Sample 2 | Sample 1 | Sample 2 |
| 0.08 | 15.675 | 42.271 | 57.858 | 39.541 |
| 0.18 | 5.636 | 26.155 | 53.103 | 27.036 |
| 0.26 | 5.548 | 21.047 | 17.877 | 18.406 |
| 0.37 | 0.352 | 16.556 | 8.366 | 16.996 |
| Sorbent 2 | | | | |
| | Sample 1 | Sample 2 | Sample 1 | Sample 2 |
| 0.08 | 23.073 | 33.905 | 87.977 | 34.874 |
| 0.18 | 13.122 | 25.803 | 45.970 | 32.056 |
| 0.26 | 3.082 | 22.456 | 39.629 | 31.263 |
| 0.35 | 0.088 | 9.863 | 26.772 | 27.036 |
| Sorbent 3 | | | | |
| | Sample 1 | Sample 2 | Sample 1 | Sample 2 |
| 0.08 | 21.312 | 32.936 | 86.303 | 40.686 |
| 0.17 | 20.695 | 20.079 | 69.835 | 39.541 |
| 0.26 | 4.844 | 13.914 | 53.191 | 34.874 |
| 0.37 | 4.139 | 6.781 | 50.989 | 31.263 |

## 3.3. Olfactometry

Table 6 summarises the results obtained after the olfactometric tests. Olfactometric studies have shown that increasing the concentration of nanosilver reduces the odour emissions. The influence of the sorbent type is also noticeable. Sorbents No. 2 and No. 3 have been found to be the most effective. They are characterised by a much larger surface area and pore volume in comparison to sorbent No. 1.

TABLE 6

Results of olfactometric studies

| Content of nanosilver in the sorbent [%] | Sorbent | Number of fragrance units [NFU/m$^3$] | Number of fragrance units [NFU/m$^3$] – obtained by graphic method |
|---|---|---|---|
| 0.08 | 1 | 2354.7 | 2354.3 |
| 0.18 | 1 | 1400.4 | 1483.5 |
| 0.26 | 1 | 1400.4 | 1483.5 |
| 0.37 | 1 | 700.1 | 789.2 |
| 0.35 | 2 | 123.7 | 141.5 |
| 0.26 | 3 | 174.9 | 171.9 |
| 0.37 | 3 | 52.0 | 43.3 |

## 4. Conclusion

The study confirmed that nanosilver is deposited on the DL sorbents. The deposition rate on each sorbent was higher than 95%. Tannic acid used as a reducing and stabilising agent in the process of obtaining nanosilver is perhaps the factor that allows the process of sorption. Nanosilver particles were adsorbed successfully on the sorbents by strong bonds, so that particles were not removed by physical force during the leaching process. The percentage of leaching was equal to 10%.

The reduction of ammonia emissions through the use of nanosilver has been proved to be effective. This method can also be efficient in reducing the emissions of other odorants detected by olfactometric analyses.

Sensory analysis is carried out in order to determine the intensity of the collective odour rather than its individual components. Studies have shown a reduction in the intensity of odours with increasing nanosilver concentration. For this reason, it can be concluded that nanosilver reduces odour emissions.

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