

## Spectroscopic study of the effect of biological treatment on the humification process of sewage sludge

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### Abstract

In 2005 the treatment plant in Sosnowiec Zagórze was modernized and the processes of nitrification and denitrification were introduced.

The study of the biological treatment influence on the course of the humification process was conducted for the sewage sludge received from each stage of sewage purification. The extracted humic-like substances (HA) were investigated by the use of the spectroscopic and analytical methods. The concentration of free radicals and the *g*-factor was determined with EPR, the presence of the characteristic functional groups was confirmed with IR spectroscopy, whereas the aromatisation of HA was estimated by <sup>13</sup>C NMR method. The results obtained were compared with those for HA extracted from sewage sludge before modernization [1].

It was found that the processes of biological treatment have a significant influence on the changes of the chemical elements in the extracted HA. The HA obtained after modernization are nitrogen-rich (about 9%), in particular after the nitrification and denitrification processes. However, nitrification and denitrification processes only slightly affect the free radical concentration and the *g*-factor values. © 2007 Published by Elsevier B.V.

**Keywords:** EPR; NMR; IR; Free radicals; Sludge; Biological treatment; Humification process; Humic-like substances (HA)

### 1. Introduction

In the sewage treatment process large volumes of sludge are produced. Hence their utilization is of essential importance. These sediments are often used as substitute for manure. The agricultural usefulness of sludge is evaluated by its content of nitrogen, phosphorus, potassium and the ability to form humus [2]. Therefore the monitoring of the humification processes during sewage treatment is of great interest. Recently studies of humification processes of sewage sludge have been conducted during each stage of treatment. This has made the observation of organic matter transformation possible.

The complex study of humification processes during treatment has been performed on samples from Sosnowiec

Zagórze wastewater treatment plant. The spectroscopic and chemical methods were applied. On the basis of the already completed projects it was stated that humification processes are observed during wastewater treatment but the resulting sludge is not mature enough to be applied for agricultural purposes [1,3,4]. The treatment plant in Sosnowiec Zagórze for many years operated without the nitrification and denitrification processes. In 2005 the plant was modernized and processes of biological treatment were introduced. This fact made it possible to study the influence of nitrification and denitrification on the course of the humification process during sewage treatment. It was possible to achieve this aim by comparing the physicochemical properties of humic-like substances extracted from sewage sludge collected from treatment plant in Sosnowiec Zagórze before modernization (without nitrification and denitrification processes) with the analogous properties of humic-like substances extracted from sewage sludge after modernization (with nitrification and denitrification

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processes). Therefore the influence of nitrification and denitrification processes on the sewage sludge quality could be determined.

These biological processes are important since the transformation of nitrogen takes place during nitrification and denitrification takes place and in consequence enrichment of humic substances in nitrogen can be observed. The percentage content of nitrogen bound by humic substances in sewage sludge and in compost is one of the main criteria of usefulness for agricultural purposes [5].

The estimation of the effect of biological treatment on the humification process of sewage sludge can only be obtained by analysis of the humic acid extracted from sewage sludge from the subsequent treatment stages. The properties of humic-like substances (HA) are described by the following factors: parameter  $g$  and the free radical concentration from EPR spectra [5–8], degree of aromaticity from the analysis of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra [6,9,10], the tendency to bind metals from IR and EPR spectra and elementary analysis [5–8,11–19]. The results were compared with the analogous ones obtained for HA extracted from sewage sludge before the treatment plant modernization [1,3,4].

## 2. Materials and methods

Sludge samples for studies were collected three times in various months by workers of the sewage treatment plant in Sosnowiec Zagórze according to Polish standards from:

the recirculation chamber (1), the denitrification chamber (2), the nitrification chamber (3) the digestion chamber (4) and after leaving the press (5) (Fig. 1).

Nitrification and denitrification are the new processes introduced to the sewage purification process after the modernization of the treatment plant. These processes take place in two reactors; the denitrification process precedes the nitrification process (Fig. 1).

The mean temperature of the biological treatment processes in the months in which the samples were collected was  $14^\circ\text{C}$ , i.e. near to the annual mean temperature of  $15^\circ\text{C}$ . However, pH for sewage before the biological processes on the inflow into the denitrification chamber in the sampling months was 7.5 which are also near to the annual mean pH value of this process – 7.4. After nitrification and denitrification the pH value decreases on the outlet from the nitrification chamber to the value 7.1 in the sampling months which is also close to the annual mean pH value on the outlet from the nitrification chamber – 7.0. The nitrification and denitrification processes can occur in the biological chamber in this range of temperature and pH.

Humic-like substances (HA) were extracted from sludge samples according to the method described previously [1]. Electron paramagnetic resonance (EPR) spectra were obtained with Bruker EMX EPR spectrometer operating at X-band frequency at room temperature. The EPR was applied for both quantitative (free radicals concentration) and qualitative ( $g$ -factor) analysis at each stage of treat-

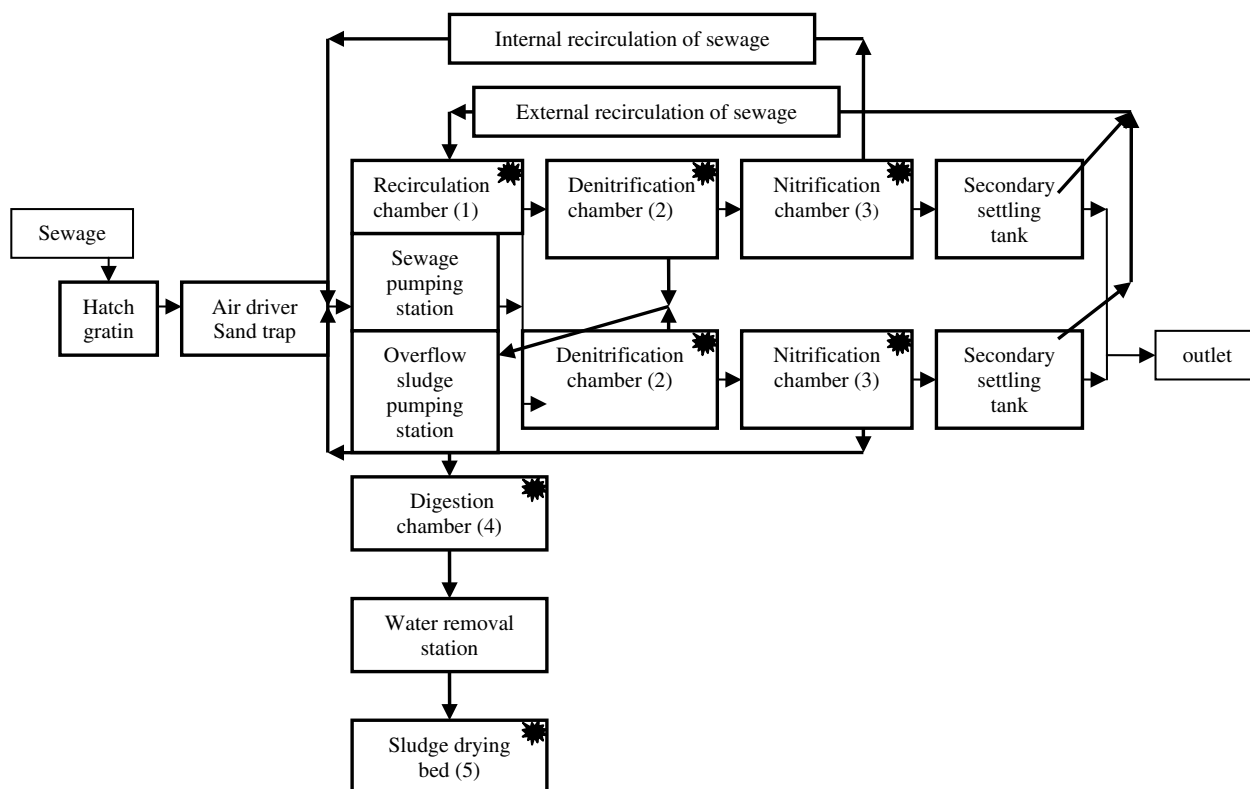


Fig. 1. Block diagram of the treatment plant in Sosnowiec Zagórze (Poland) after modernization with marked points of sampling.

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