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The Influence of Microfibres in Municipal Sludge on Biogas Production

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Abstract

Wastewater treatment plants (WWTPs) contribute to the release of microplastics into the environment. While the removal efficiency of MPs in WWTPs can reach up to 99.9 %, the highest amount of microplastics is retained in the sludge. Anaerobic digestion, one of the most promising and common processes, can help reduce sludge volume and odour, and due to the formation of biogas, mainly consisting of methane (CH_4) and carbon dioxide (CO_2), can help decrease the operating costs of WWTPs. A test measuring the inhibition of biogas production using the OxiTop® measuring system was employed to determine the effect of added microfibres (MFs) on biogas production. Particles less than 1 mm in size of polyester, polyamide, and polyacrylic, were added to anaerobic sludge at concentrations ranging from 0.05 to 0.10 g l^{-1} to simulate their effect on biogas production. The yields of CH_4 and CO_2 produced during anaerobic digestion of the MFs-contaminated sludge were determined.
The addition of MFs to sewage sludge affects methane production. The results indicated that the lowest added concentrations of 0.05 g l^{-1} of MFs promoted methane production, while the presence of 0.1 g l^{-1} decreased methane production for all types of MFs used. Polyacrylic at 0.1 g l^{-1} had the most negative effect on methane production (up to 27 %), while polyamide at 0.05 g l^{-1} reached the highest methane production (up to 25 %).

Keywords

Anaerobic digestion, methane production, polyester, polyamide, polyacrylic

1 Introduction

Microplastics (MPs) are a ubiquitous anthropogenic pollutant detected throughout the environment, with their concentration steadily increasing.[1] MPs are typically categorised into primary or secondary. Primary MPs are already produced in small, microscopic sizes, and enter the environment as such (through microbeads in personal care products, cosmetics, *etc.*), while secondary MPs result from the degradation of larger plastics into smaller particles due to chemical, physical or biological processes. In the literature, MPs are defined as particles smaller than 5 mm[2] and those smaller than 0.1 µm are termed nanoplastics.[3] Although researchers have proposed new definitions,[4] there remains no universal categorisation for MP sizes. Information regarding the presence of MPs in the environment is limited due to the lack of standardised methods, hindering comparison between studies. Nevertheless, it is widely acknowledged that MPs pose a potential threat to the environment and organisms, inhibiting their growth and reproduction.[5–7] MPs can also enter the human food chain through ingestion or inhalation.[5]

Numerous studies have identified wastewater treatment plants (WWTPs) as significant sources of MPs entering the environment.[6] Wastewater treatment typically involves pretreatment, followed by primary and secondary treatment, with some WWTPs also employing tertiary treatment. Larger particles are removed during pretreatment using screens and meshes, while some MPs settle during primary and secondary sedimentation. Tertiary treatment further reduces MPs, with removal rates reaching even up to 99.9 %[7,8] in the outflow compared to the inflow. During wastewater treatment, up to 90 % of MPs accumulate in the sewage sludge.[6] Various types of MPs may be present in sewage sludge, and determining their origin is challenging. Among the most commonly found plastics in sewage sludge are microfibres (MFs) originating from textiles,[9,10] where polyester (PES) and polyamide (PA) are among the most prevalent textile materials.[10] These materials are also among the most frequently detected types of MFs in WWTPs.[6] The presence of MPs varies, and studies have shown that their concentration depends on the type of treated wastewater, the type of WWTP, seasonal factors, and human habits. In different studies, the quantity of MPs in sewage sludge ranges from 50 to 170,900 particles per kg.[11,12]

Sewage sludge, which is formed during wastewater treatment, comprises solid, semi-solid, and liquid residues. Due to its properties and the presence of useful substances, sewage sludge can be applied for different purposes. One viable option is anaerobic digestion, wherein sludge stabilisation reduces volume and odour while producing methane (CH_4). During anaerobic digestion, microorganisms digest organic matter, forming biogas mainly composed of CH_4 and carbon dioxide (CO_2), which can be utilised for renewable energy production.[13] However, in this process, the microorganisms can be inhibited by adsorbed compounds, such as MPs or MFs, heavy metals,

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sulphide, and others, thus affecting biogas production.[14–17] Recent studies have investigated the inhibitory effect of MPs or MFs on anaerobic digestion. *Wei et al.*[17] found that polyvinyl chloride (PVC) inhibited methane production by up to 90.6 ± 0.3 % compared to control sample, while *Chen et al.*[18] reported that CH_4 yield depended on the concentration of added polycarbonate MFs. *Li et al.*[19] found up to 10 % inhibition in CH_4 production by adding PES at various concentrations. In a separate study, *Wei et al.*[15] confirmed that polyethylene terephthalate (PET) inhibited sludge hydrolysis, acidogenesis, acetogenesis processes, and hydrogen production in alkaline anaerobic fermentation. The aim of this paper was to examine the inhibitory effect of the most common types of MFs on anaerobic digestion. The impact of different types of MFs (PES, PA, polyacrylic) at various concentrations on biogas production and CH_4 yield was evaluated.

2 Experimental

2.1 Samples

The microfibers (MFs) used in the experiment were manually cut using scissors into particles smaller than 1 mm (Fig. 1), and utilised in two concentrations (0.05 and 0.10 g l^{-1}). Prior to analysis, the MFs were sieved through a stainless steel sieve with mesh size of 1 mm. The MFs were purchased from a fabric store, and their composition was confirmed with Fourier Transform Infrared analysis (FTIR) before and after the experiment. The FTIR spectra after anaerobic digestion indicated no chemical changes.

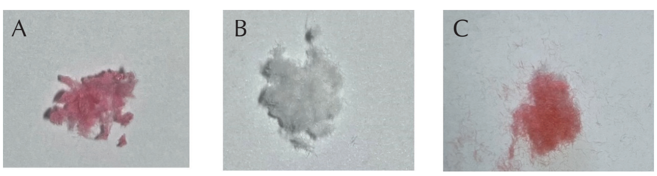

Fig. 1 – Microfibres used in the experiment (A – PES, B – PA and C – polyacrylic)

For the biogas production measurement experiment, anaerobic sludge (after anaerobic digestion) was collected from the municipal WWTP (Ljubljana, Slovenia), with a capacity of 360.000 population equivalents (PE) The daily treated sewage volume is 82.200 m^3 during the dry season and 103.500 m^3 during the rainy season.[20] This WWTP, which mainly treats domestic wastewater, employs a combined sewage system and secondary treatment. The anaerobic sludge was retrieved from the digester and sampled according to ISO 11734:1995[21] into a wide-mouthed expandable flask.

The anaerobic sludge was conditioned at 37 ± 1 °C for two days. Subsequently, the sludge samples were filtered through black ribbon with a pore size of 4–12 μm, and dried to constant mass at 105 ± 2 °C to determine the concentration of total suspended solids (TSS). The concentration of added anaerobic sludge during biogas production measurement was 1.5 ± 0.1 g_{TSS} l^{-1}. Each experiment was conducted in duplicate and repeated twice.

2.2 Inhibition of biogas production

To assess the inhibition of anaerobic microorganisms in CH_4 and biogas production, microfibers (MFs) at various concentrations (0.05 and 0.1 g l^{-1}) were added to monitor biogas production. A modified standard procedure, (SIST EN ISO 11734:1995),[21] using the OxiTop® measuring system, was applied, wherein biogas production, in terms of increased pressure (hPa) in a closed system (Fig. 2), was measured. According to the standard procedure, the anaerobic sludge (at a suspension concentration of 1.5 g_{TSS} l^{-1}) and tap water were dispensed into 250-ml glass bottles (purged with nitrogen to remove any remaining oxygen), followed by the addition of 0.5 ml of glucose (with c = 0.833 mol l^{-1}) as substrate, and 2 ml of buffer solution to maintain constant pH (7.21 ± 0.21). The final volume of the mixture was 100 ml. The PES, PA, and polyacrylic fibres with sizes smaller than 1 mm were added at concentrations of 0.05 and 0.10 g l^{-1}. Control samples without added MFs were simultaneously prepared.

Prior to sealing the glass bottles, they were purged with nitrogen to remove any remaining oxygen. Measurement of biogas production (hPa) started at a constant temperature of 37 ± 1 °C for 8 days. On day 7 of the experiment, 2 ml of 6.0 M NaOH were added through the side rubber of the glass bottle to absorb the formed CO_2. The remaining change in pressure after one day was attributed to the production of CH_4. To calculate the amount of produced bi-

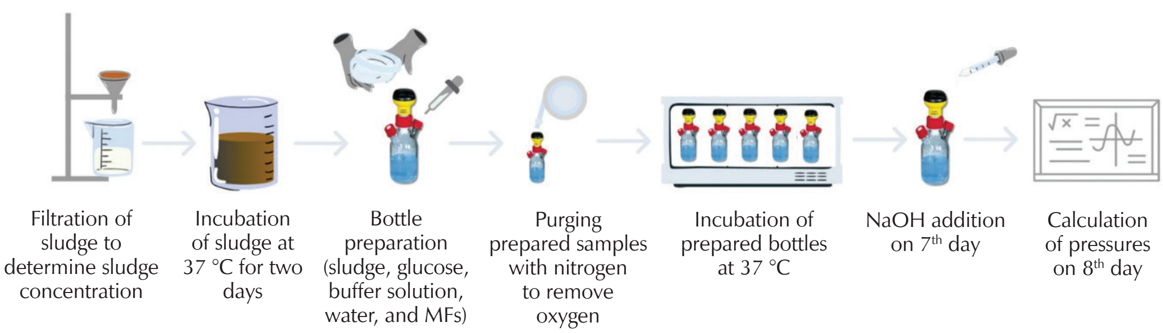

Fig. 2 – Procedure for determining biogas and methane inhibition

ogas (sum of CO_2 and CH_4 moles) at the end of anaerobic digestion, the general gas equation (Eq. (1)) was utilised.

$$n = \frac{\Delta p \cdot V}{R \cdot T} \tag{1}$$

In Eq. (1), n stands for the number of moles (CO_2 and CH_4 moles), Δp for the pressure difference [Pa], V for the volume of the gas [m^3] and R for the gas constant [8.314 J mol^{-1} K^{-1}].

It was assumed that CO_2 and CH_4 were the only gaseous products formed during the process.

The same equation was employed to calculate the amount (n) of CO_2 remaining after the addition of NaOH. The yield of CO_2, D, was calculated according to Eq. (2).

$$D = \frac{n_1}{n} \cdot 100 \tag{2}$$

The quantity n_1 stands for the number of moles of CO_2 produced and n for the number of moles of the total gas mixture.

The remaining proportion represented CH_4 yield (Fig. 3). All experiments were conducted in duplicate (n = 2).

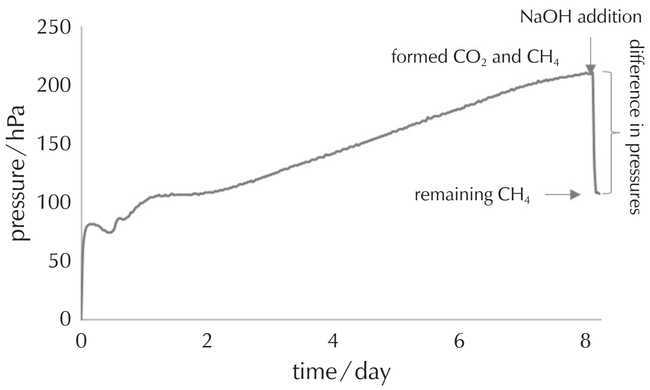

Fig. 3 – Pressure curve and pressure determination

3 Results and discussion

The aim of this study was to assess the inhibitory effect of added MFs at different concentrations on CH_4 and biogas production. Control experiments with samples without added MFs were run simultaneously to determine the impact of the added MFs. The percentage of formed gases was calculated based on pressure changes after the addition of NaOH on day 7 of the experiment using Eqs. (1) and (2).

With the control samples without added MFs, CH_4 yield reached 41 ± 11 %, whereas according to the standard procedure, biogas should contain 50 % CH_4. Upon the addition of PES to the anaerobic sludge, CH_4 yield reached 50 ± 5 % (0.05 g l^{-1}) and 33 ± 2 % (0.10 g l^{-1}). Similarly, after the addition of PA, CH_4 yield reached 50 ± 2 % (0.05 g l^{-1}) and 26 ± 9 % (0.10 g l^{-1}), while in the case of polyacrylic, CH_4 yield reached 41 ± 3 % (0.05 g l^{-1}) and 23 ± 3 % (0.10 g l^{-1}).

The results of the experiments revealed that the concentration of 0.05 g l^{-1} of MFs promoted methane production, whereas the presence of 0.10 g l^{-1} of MFs decreased methane production for all added types of MFs (Fig. 4). These findings are consistent with those of *Chen et al.*,[18] who found that low concentrations (10 to 60 particles g^{-1} TS) of polycarbonate MFs promoted CH_4 production, while higher concentrations (200 particles g^{-1} TS) inhibited CH_4 production. The PA significantly increased methane production (up to 25 %), PES up to 10 %, while polyacrylic improved methane production, but had no significant impact. The presence of polyacrylic at a concentration of 0.10 g l^{-1} had the most pronounced inhibitory effect, reducing methane production by up to 27 %, while PA had the lowest but still significant effect, decreasing methane production by up to 9 %. *Chen et al.*[18] suggested that promoted CH_4 production may be due to increased activity of enzymes, such as protease, acetate kinase, and F_{420}, which participate in and accelerate anaerobic digestion.

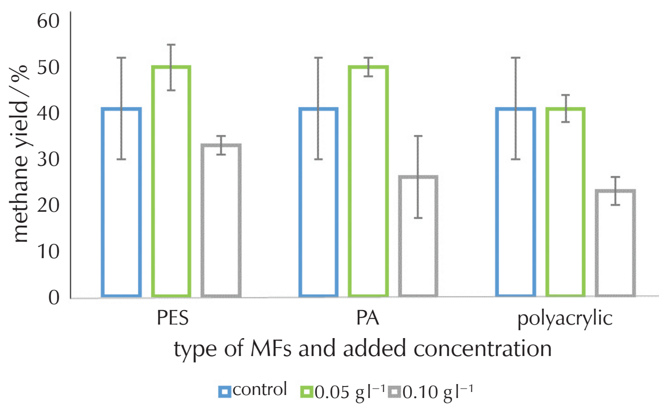

Fig. 4 – Methane yields of contaminated sludge compared to control sample ± SD [%]

Based on the results, it appears that the addition of MFs influences methane production. The type of MFs (PA) that most effectively promoted methane production at lower concentrations exhibited the lowest inhibitory effect on methane production at higher added concentrations, while polyacrylic, with the lowest production impact at lower added concentrations, exhibited the highest inhibitory effect on methane production. The presence of MFs also influenced the production of biogas (Fig. 5), which consisted of CO_2 and CH_4. At concentrations of 0.05 g l^{-1}, biogas production significantly increased by 114 % (with PA), 55 % (with PES), and 39 % (with polyacrylic) compared to the control. However, at concentrations of 0.10 g l^{-1}, biogas production was inhibited by 11 % and 29 % with PES and polyacrylic MFs, respectively, while PA increased total biogas production by up to 7 % compared to the control.

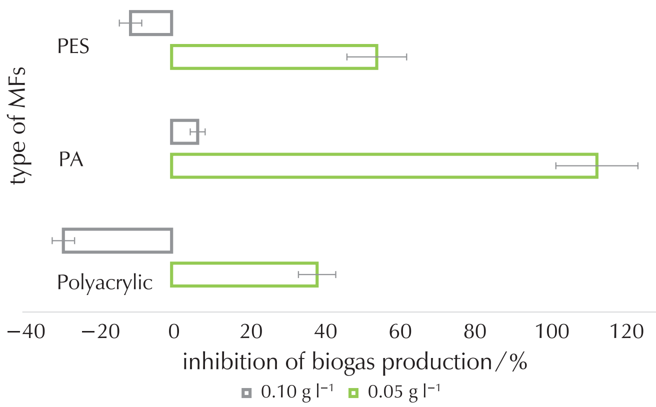

Fig. 5 – Biogas production of contaminated sludge compared to control sample ± SD [%]

Based on the results presented in Figs. 4 and 5, it is evident that both CH_4 and biogas production are similarly influenced by the same type and concentration of MFs added.

The presence of PA (0.05 g l^{-1}) had the most pronounced effect on both CH_4 and biogas production compared to the control sample, while polyacrylic (0.05 g l^{-1}) exhibited the lowest effect on CH_4 production. PES, at a concentration of 0.10 g l^{-1}, showed the lowest inhibitory effect on CH_4 production. PA (0.10 g l^{-1}) resulted in the highest increase in biogas production (7 ± 2 %), while polyacrylic (0.10 g l^{-1}), which had the highest inhibitory effect on CH_4 production (27 ± 4 %), also exhibited the greatest inhibitory effect on biogas production (29 ± 3 %). Other studies investigating the impact of MPs/MFs on anaerobic digestion have indicated that inhibition is primarily caused by the physical disruption of microbial cells or the leaching of adsorbed toxic compounds, which can also affect microbial activity thus affecting biogas and CH_4 production.[17,18,22] Since CH_4 production was promoted at lower concentrations (0.05 g l^{-1}) and inhibited at higher added concentrations (0.10 g l^{-1}), it can be assumed that with the addition of higher concentrations of MFs, a higher amount of adsorbed chemicals started leaching, thus influencing methane production. *Li et al.*[19] found that the addition of PES affected the structure and diversity of the microbial community, although the effect was insignificant. They also noted that the addition of up to 200,000 particles per kg of total solids inhibited the anaerobic digestion process. However, *Wang et al.*[23] focused on the inhibitory effect of aged MPs and leachates on methane production, and found that the inhibitory effect of MPs on methanogenesis was primarily due to leachates from the added MPs. These leachates induce oxidative stress, which could damage microbial cells, reduce microbial activity, and consequently inhibit methane production.

4 Conclusion

Microfibres (MFs) present in sewage sludge at lower added concentrations promoted methane production, whereas they inhibited it at higher concentrations. This could be attributed to the leaching of MF-adsorbed compounds, which correlates with the amount of MF particles in the test system. Further studies should be devised and conducted to explore the quality and quantity of adsorbed compounds, aiming to comprehend their impact on biogas and CH_4 production.

In conclusion, the presence of MFs influences anaerobic digestion in terms of quantity and composition of the biogas produced. By gaining a better understanding of the mechanisms and compounds affecting the consortia of anaerobic species in the sewage sludge, the potential of anaerobic digestion to generate renewable energy could be enhanced.

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List of abbreviations

MPs	– microplastics
WWTPs	– wastewater treatment plants
MFs	– microfibres
TSS	– total suspended solids
TS	– total solids
FTIR	– Fourier Transform Infrared Spectroscopy
PE	– population equivalent
PES	– polyester
PA	– polyamide
PVC	– polyvinyl chloride
PET	– polyethylene terephthalate
n	– number of moles
Δp	– pressure difference
V	– volume of gas
R	– gas constant
D	– proportion of gas
SD	– standard deviation

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SAŽETAK

Utjecaj mikrovlakana u mulju uređaja za pročišćavanje komunalnih otpadnih voda na proizvodnju bioplina

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Postrojenja za pročišćavanje otpadnih voda (UPOV) doprinose ispuštanju mikroplastike u okoliš. Iako UPOV-i mogu ukloniti čak 99,9 % mikroplastike, najveća količina zadržava se u mulju. Anaerobna digestija, jedan od najperspektivnijih i najčešće primjenjivanih procesa, može pomoći u smanjenju volumena i neugodnih mirisa mulja. Također, proizvodnja bioplina, koji se u najvećoj mjeri sastoji od metana (CH_4) i ugljikova dioksida (CO_2), može smanjiti operativne troškove uređaja za pročišćavanje otpadnih voda. Za ispitivanje utjecaja dodanih mikrovlakana (MF) na proizvodnju bioplina upotrijebljen je mjerni sustav OxiTop®. Čestice poliestera, poliamida i poliakrila, veličine manje od 1 mm, dodane su anaerobnom mulju u koncentracijama od 0,05 do 0,10 $g\,l^{-1}$ da bi se simulirao njihov učinak na proizvodnju bioplina. Mjereni su prinosi CH_4 i CO_2 nastali tijekom anaerobne digestije mulja onečišćenog MF-om. Rezultati su pokazali da dodavanje MF-a utječe na proizvodnju metana. Najniže koncentracije od 0,05 $g\,l^{-1}$ MF-a potiču proizvodnju metana, dok koncentracija od 0,10 $g\,l^{-1}$ smanjuje proizvodnju metana za sve vrste upotrijebljenih MF-a. Poliakril s 0,10 $g\,l^{-1}$ imao je najnegativniji učinak na proizvodnju metana (smanjenje do 27 %), dok je poliamid s 0,05 $g\,l^{-1}$ postigao najveći porast proizvodnje metana (do 25 %).

Ključne riječi

Anaerobna digestija, proizvodnja metana, poliester, poliamid, poliakril

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