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Waste paper mill sludges originating from different effluent treatment and de-inking installations are complex mixtures of inorganic and organic particles. Due to their favourable physico-chemical, and microbiological characteristics, they may be conveniently reused for different purposes as such or after appropriate pretreatment. Sludges from the Slovenian paper industry were extensively tested for their chemical, stability and sealing properties. During the biodegradability tests, evolutions of greenhouse gases CO_2, CH_4 and H_2S as well as the concentrations of released volatile organic acids, such as acetic, propionic, butyric, lactic and glycollic acids as the typical degradation products of organic materials, were measured. Some other important parameters of water leachates such as pH, redox potential, content of starch and leachable ions were also evaluated. The results indicate that most of them can be efficiently applied as alternative hydraulic barrier layers for landfill construction and covering instead of the more expensive clay due to their good geomechanical properties, chemical inertness and microbiological stability. Such replacement brings about considerable economical and ecological benefits as the waste is reprocessed as secondary raw material.

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Introduction

Relatively large volumes of solid waste are generated during paper production. They consist mainly of paper rejects and sludge which originates from different mechanical, chemical and biological effluent treatments and from de-inking processes. The quantity of released waste sludge corresponds to 3–9% of paper production. Its chemical composition and physical properties depend on the manufactured paper grade, specific freshwater consumption and the wastewater cleaning technique applied. Paper mill sludge is a complex material, typically composed of water, organics such as wood and cellulose fibres, synthetic polymers, coatings, starch, sizing agents and resins as well as inorganics, including calcium carbonate, kaolin and talc. With regard to their chemical structure, sludges may be either primarily organic or primarily inorganic in character. In printing paper production, inorganic sludges usually prevail, whereas in the packaging paper industry, sludges of more organic character are produced (Hynninen 1998).

Up to now, approximately 70–75% of paper mill solid waste has been disposed of in landfill, which is inconvenient from both economical and ecological standpoints. Another 15–20% is currently incinerated for energy production and volume reduction. In both cases much of the material is lost, whereas this biosolid could be conveniently treated as a secondary raw material instead. Depending on its physicochemical and microbiological properties, the accumulated waste may be further processed and used for composting, production of soil-covering paper, insulating and fire-resistant materials, barrier layers, conversion to fuel components and carriers of artificial fertilizers. Waste solids of more inorganic character may be efficiently utilized in the production of building materials such as brick and cement (Charlie 1977, Hudson & Lowe 1996, Jokela *et al.* 1997, Krogman 1998, Liaw *et al.* 1998).

However, the most promising application of sludge seems to be its use as hydraulic barrier layer in landfills since its geotechnical properties, such as shear angle, cohesion, unit weight, permeability and modulus of elasticity resemble those of compacted clays which constitute natural barrier zones (Andersland & Laza 1972, Moo-Young & Zimmie 1997). Landfills represent a potential threat to the surroundings if they contain poisonous substances, which is frequently the case. Old landfill sites have to be closed for this reason in order to prevent the emission of toxic gases, contaminated water and dust into the area. In the process of closing, a landfill is covered or capped by different layers of natural and synthetic materials. Clay and bentonite serve as hydraulic barrier layers and may be conveniently replaced by paper mill sludge, provided that its water permeability is lower than 10^{-8} m s^{-1} and that it is chemically as well as microbiologically stable. Such replacement is economically wholly justified as the costs of 'classical' landfill closing are increasing from 40 € m^{-3} to about 60 € m^{-3} whereas the costs are reduced to about 12 € m^{-3} when paper mill sludge is applied instead of the usual covering or capping materials. By implementing appropriate technology for controlled waste biodegradation, high value biogas may be produced in abundant quantities at closed landfill sites as well. Thus, they can conveniently function as power stations after closing (Quiroz & Zimmie 1998, 1999, 2001, Palko *et al.* 2001, Simpson & Zimmie 2004).

Countries such as Finland and the United States have already started to use paper mill sludge for landfill capping and some others are preparing similar projects.

Experience from abroad confirms the fact that landfills collecting municipal and industrial waste may be conveniently sealed by means of sludge only when its generation site is less than 100 km away. This could be easily achieved in Slovenia where 33 landfills are to be closed and the annual generation of primary paper mill sludge is quite large and amounts to about 45 000 t. However, sludges that are to be used as impermeable sealing layers at landfill sites have to be thoroughly tested for their properties, which depend on the current production programmes in individual paper mills (Gillespie 1970, Bower 1973, Zimmie *et al.* 1997, Tähtinen 1999, Ishimoto *et al.* 2000, Fourie & Brown 2001, LaPlante & Zimmie 2003).

Most of the research on the properties of paper mill sludge that has been conducted so far has been focused on the determination of dry matter and ash contents as well as specification of leachable heavy metals, chloride and sulphate concentrations (Mroueh *et al.* 2001, Sorvari & Tenhunen 2001a, Tenorio *et al.* 2001, Simpson *et al.* 2004). Biodegradation tests have also been the subject of investigation (Tähtinen 1999, Palko *et al.* 2001), however less has been reported on emission of odour-causing substances (Černec *et al.* 2005).

The aim of our extensive work was to establish whether the chemical and biostability properties of primary waste biosolids originating from five Slovenian paper mills producing different paper grades from fresh and recycled fibers present suitable substitutes for clay in the capping process of municipal landfills.

Materials and methods

Sampling

Freshly generated sludge samples from three different chemimechanical water treatments (S1, S2 and S3) and two deinking facilities (S4 and S5) of five paper mills were collected at various points from large piles to obtain about 3 kg of representative material for subsequent chemical and stability analyses. Samplings of individual sludge types were performed several times in all seasons during a period of 1 year. After having been collected, sludge samples were first homogenized by means of a laboratory mixer and afterwards analysed for dry matter and ash contents. Water extracts were prepared to determine leachable organic and inorganic compounds.

Chemical and geomechanical analyses

Dry matter content was determined according to the DIN EN 12880 standard and ash content at 550°C according to the DIN EN 12879 standard. Organic matter content was calculated on the basis of these results.

Water extracts were prepared according to the DIN 38414-S4 standard. They were subsequently analysed for pH, redox potential (measured against standard hydrogen electrode), starch, anions such as chloride, sulphite, sulphate, thiosulphate, sulphide, nitrate, phosphate and carbonate, as well as for the content of volatile organic acids such as formic, acetic, propionic, butyric, glycollic and lactic acid.

Starch content in water extracts was qualitatively evaluated as either non-present or low or high by the J_2/KJ reagent (7.5 g KJ and 5 g J_2 per litre of deionized water). Fifty millilitres of diluted HCl (1 + 9), 25 mL of water extract and 5 mL

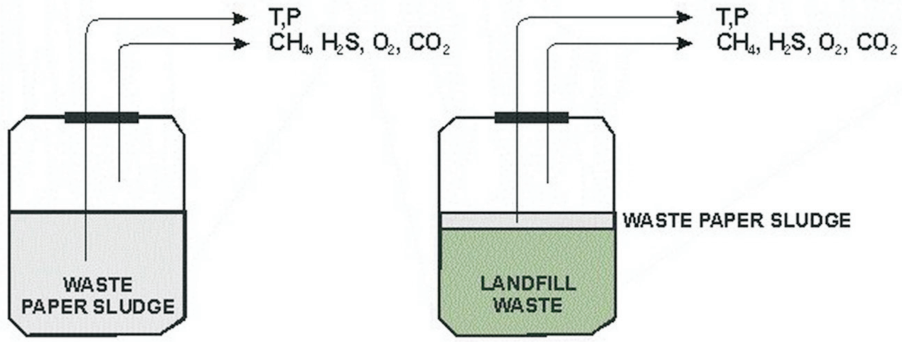

Fig. 1: Schematic presentation of biodegradation and sealing tests of waste paper mill sludges.

of J_2/KJ were added to a 100 mL measuring flask which was than filled with deionized water up to the marker. Blue colour developed if starch was present. The colour intensity indicated the level of starch content.

The concentrations of inorganic ions and low molecular weight organic acids were measured on the Metrohm 761 Compact IC ion chromatograph under the following experimental conditions.

Anion analyses: column: Metrosep Anion Dual 2; eluent: 2.0 mmol $NaHCO_3$/1.8 mmol Na_2CO_3, 15% (v/v) acetone; flow: 0.8 mL min^{-1}; suppressor solution: 50 mmol H_2SO_4; injected volume: 20 µL; detector: conductivity detector.

Organic acid and carbonate analyses: Column: Metrosep Organic acids; eluent: 0.5 mmol $HClO_4$; flow: 0.5 mL min^{-1}; suppressor solution: 10 mmol LiCl; injected volume: 20 µL; detector: conductivity detector.

Concentrations of individual ionic species were calculated from corresponding calibration curves of standard solutions. The results are expressed as milligrams of measured species per gram of dry sludge.

Fourier transform infrared (FTIR) spectra of ash samples were recorded on a BioRad Excalibur spectrophotometer as transmission spectra on KBr pellets in the range between 4000 and 400 cm^{-1}.

The geomechanical properties of sludges, such as uniaxial compressive strength, elastic modulus, Poisson's ratio and hydraulic conductivity coefficient were determined according to standard methods.

Biodegradability and sealing tests

Stability properties of sludge samples were determined by experiments on a laboratory level.

A 50 L plastic barrel served as reaction vessel for the simulation of characteristic landfill circumstances. It was filled with fresh sludge to two-thirds of its volume, after which it was tightly covered. The barrel was equipped with temperature and pressure sensors, as well as a gas measuring detector (IR biogas analyser, Geacomm GA 94) that measured emissions of CH_4, CO_2, H_2S and O_2 at regular time intervals, namely every 12 h for 30 days. After each measuring, air was blown through the surface of the tested material to ensure aerobic conditions on the upper side of the sludge. All sensors were connected to a computer that was recording the results onto a graph.

During sealing tests, which were essentially conducted in the same way, paper mill sludge was replaced by common landfill waste that was covered or sealed by a thin layer of sludge (20 : 1, v/v).

All experiments were performed at room temperature (18 ± 2°C, Figure 1).

The same chemical analyses as in the case of fresh sludges were also performed with materials after they had been subjected to biodegradation and sealing tests, namely after 30 days. However, no samples were taken from the barrel during that period.

Results and discussion

Chemical analyses

The results of chemical analyses clearly indicate that there were specific differences among sludges originating from different water treatments (S1, S2 and S3) and de-inking devices (S4 and S5) of individual paper mills, as well as among those collected at the same paper manufacturing process on different occasions. Considering the fact that their exact composition depended on the current production programme, the evaluation of their characteristics was significant only if we took into consideration their changing properties over longer periods of time. Therefore, the results of chemical analyses of all examined sludge types were averaged. In total, 12 measurements were performed for each sludge sample. The average values as well as standard deviations are presented in Tables 1–4.

Industrial sludges and biosolids are usually dewatered in paper mills before further handling to reduce total weight and increase their dry matter content. Centrifugation, filtra-

Table 1: Dry matter and ash content of sludges.

Dry matter (%)	Fresh sludge	Biodegraded sludge
S1	37 ± 5	39 ± 4
S2	45 ± 7	51 ± 5
S3	45 ± 5	48 ± 5
S4	53 ± 8	56 ± 6
S5	67 ± 10	70 ± 7
Ash 550°C (%)		
S1	63 ± 2	68 ± 4
S2	69 ± 4	77 ± 2
S3	65 ± 3	75 ± 5
S4	66 ± 6	72 ± 5
S5	70 ± 7	82 ± 2

Table 2: pH and redox potential of water extracts of sludges.

pH value	Fresh sludge	Biodegraded sludge
S1	7.6 ± 0.2	7.9 ± 0.1
S2	7.9 ± 0.3	7.3 ± 0.2
S3	8.1 ± 0.2	8.0 ± 0.3
S4	9.0 ± 0.3	7.8 ± 0.2
S5	7.3 ± 0.2	7.0 ± 0.2
Redox potential (mV)		
S1	162 ± 20	143 ± 22
S2	165 ± 10	136 ± 15
S3	172 ± 27	117 ± 17
S4	182 ± 18	122 ± 14
S5	28 ± 11	6 ± 4

tion and pressing methods are usually applied for this purpose to reach values as high as 60% or more if the materials are to be further recycled.

The results from Table 1 indicate that de-inking sludges (S4, S5) contained less water and for this reason need less dewatering than chemi-mechanical sludges.

It is also evident from Table 1 that the examined samples had predominantly inorganic character, as the inorganic portion was the larger in all cases (ash contents > 60%). The latter consisted mostly of carbonates and silicates (kaolin), which was confirmed by FTIR analyses of the corresponding ash samples. The quantity of inorganic fraction increased from 8 to 20% in 30 days during the stability tests, meaning that the corresponding part of the organic material was decomposed. It is well known that polymeric carbohydrates, such as cellulose, wood polyoses, starch and coatings, which are the predominant constituents of waste biosolids from paper mills, are liable to gradual chemical and microbiological decomposition. They represent an excellent carbon source for micro-organisms. Anaerobic bacteria ferment carbohydrates and release volatile fatty acids as metabolic by-products, which may cause odour problems. The most typical are formic, acetic, propionic, butyric, glycollic, lactic and pyruvic acids. Some lower alcohols, CO_2 and CH_4 may also

Table 3: Contents of volatile organic acids released from sludges.

Acetic acid (mg g^{-1})	Fresh sludges	Biodegraded sludge
S1	< 0.01	0.71 ± 0.12
S2	< 0.01	< 0.01
S3	0.68 ± 0.17	0.82 ± 0.14
S4	< 0.01	0.15 ± 0.04
S5	2.10 ± 0.25	4.80 ± 0.47
Propionic acid (mg g^{-1})		
S1	< 0.,01	0.18 ± 0.02
S2	< 0.01	< 0.01
S3	0.35 ± 0.12	1.04 ± 0.22
S4	< 0.01	< 0.01
S5	0.89 ± 0.23	5.44 ± 0.77
Butyric acid (mg g^{-1})		
S1	< 0.01	< 0.01
S2	< 0.01	< 0.01
S3	< 0.01	< 0.01
S4	< 0.01	< 0.01
S5	0.02 ± 0.01	0.52 ± 0.15
Glycollic acid (mg g^{-1})		
S1	< 0.01	0.04 ± 0.01
S2	< 0.01	0.03 ± 0.01
S3	< 0.01	< 0.01
S4	< 0.01	< 0.01
S5	1.40 ± 0.27	1.70 ± 0.30

Table 4: Content of leachable anions in sludges.

Chloride Cl^- (mg g^{-1})	Fresh sludge	Biodegraded sludge
S1	0.06 ± 0.02	0.11 ± 0.03
S2	0.80 ± 0.12	0.08 ± 0.04
S3	0.18 ± 0.08	0.15 ± 0.07
S4	0.03 ± 0.01	0.02 ± 0.01
S5	0.09 ± 0.03	0.07 ± 0.03
Sulphate SO_4^{2-} (mg g^{-1})		
S1	1.29 ± 0.27	0.12 ± 0.04
S2	0.08 ± 0.01	0.01 ± 0.01
S3	0.32 ± 0.10	0.11 ± 0.07
S4	0.29 ± 0.11	0.12 ± 0.09
S5	0.58 ± 0.14	0.64 ± 0.22
Carbonate CO_3^{2-} (mg g^{-1})		
S1	3.7 ± 1.0	2.4 ± 0.4
S2	2.4 ± 0.8	2.2 ± 0.5
S3	3.7 ± 0.7	2.3 ± 0.7
S4	1.9 ± 0.4	2.9 ± 0.7
S5	2.9 ± 0.6	1.3 ± 0.2

be synthesized in the process. The magnitude of organic deterioration may be efficiently established by determining released leachable organic acids as well as various chemical properties of water extracts. The latter become progressively acidic with the ongoing deterioration of tested materials, while their redox potential decreases due to chemical and biological oxidation (Geller 1984, Sand 2000). This phenomenon is clearly illustrated in Table 2.

Slightly lower redox values of water extracts of biodegraded sludges indicated oxygen consumption within slowly decomposing organic materials, while pH was always close to neutral, meaning that no significant release of organic acids took place during the biodegradation tests.

Among the released low molecular weight volatile fatty acids, acetic, propionic and glycollic acids prevailed, while formic, lactic and pyruvic acids were detected in only a few cases and in trace amounts.

Individual acid species are typical of specific oxidation and fermentation mechanisms that make them good indicators of degradation processes within the sludge. Fortunately, the quantities of leached butyric acid were too low to cause emissions of characteristic but extremely unpleasant smell (Dyer 1996, Gudlauski 1996, Göttsching 1998). The concentration ranges of released organic acids in fresh sludges and in sludges submitted to stability or biodegradation tests are presented in Table 3.

The value 0.01 mg g^{-1} represents the experimentally determined limit of quantification (LOQ) for organic acids analysis in sludge, whereas the limit of detection (LOD) is about 0.005 mg g^{-1}.

According to the results, some sludges proved to be significantly more stable and chemically inert than others. The most probable reason for their stability behaviour was the absence or very low content of starch, which was among all the present organic compounds most easily oxidized or attacked by micro-organisms. Typically, starch concentration levels of water extracts were always larger in those cases where the contents of organic acids were higher, so the highest levels were detected in S3 (chemi-mechanical) and S5 (de-inking) sludges.

After fibres and fillers, starch is the third most important raw material in the manufacture of paper. It is used both as a dry-strength additive and as a binder for coating colours. If it is not completely retained in the paper structure, it finishes first in process waters and finally as a component of sludge after water treatment. As such, it causes lower stability.

The results of anion determinations are collected in Table 4. Among the anionic species, only chloride, sulphate and carbonate demonstrated measurable values, whereas the concentration of leachable nitrate in all cases was found only in traces. No other anions such as phosphate, sulphite, thiosulphate or sulphide were detected. The most interesting fact is that the concentrations of leachable ions were always higher in fresh sludges, whereas the other materials displayed lower leachability and chemical reactivity with time, after the decomposition tests had been performed. The absence of sulphide ions indicated poor activity of sulphate-reducing bacteria (SRB). The latter utilize sulphate ions during anaerobic respiration as electron acceptors. During electron transfer, sulphate (SO_4^{2-}) is reduced to sulphide (S^{2-}). H_2S, which is a typical product of anaerobic degradation, is extremely toxic and causes a bad smell even if present only in small concentrations.

The LOQs for individual anions are 0.001 mg g^{-1} for chloride and 0.005 mg g^{-1} for sulphate and carbonate.

The results are in perfect agreement with the suggested Finnish leaching values for the mineral materials used in earth construction (Sorvari & Tenhunen 2001b), where the limiting values are 1.5 mg g^{-1} for sulphate and 0.25 mg g^{-1} for chloride.

Biodegradability

There ares no consistent European standardized methods for evaluation of aerobic and anaerobic biodegradability of non-homogeneous solid materials. Individual EU member states use different techniques for determination of biological stability. In Germany, for example analytical methods, such as respiration activity (AT4), gas formation potential (GB21), accumulated gas production (GS21) and dynamic respiration index (DRI) are used to estimate the biostability of waste samples (Heerenklage *et al.* 2005), whereas in Finland the OECD 310F biodegradation test is commonly applied for testing the total biodegradable organic carbon content in paper sludges (Palko *et al.* 2001)

In our research an in-situ simulation model was used for this purpose.

Typical emissions of CH_4, CO_2 and H_2S as well as temperature changes during biodegradability tests for different sludges are presented in Figures 2–5.

The temperature of the tested sludge samples (measured in the middle of the sample) slightly oscillated during the test but did not show any tendency towards increasing (Figure 5). A typical de-inking sludge sample S5 always manifested both CH_4 and CO_2 emissions, indicating that anaerobic as well as aerobic decomposition took place within different layers of the sludge. This particular sludge usually contained much starch. Volatile organic acids were present already in the fresh material and their concentrations increased with time. Sludge S3 from a water treatment plant in most cases emitted CO_2, indicating that it was liable to increased aerobic biodegradation. It usually also contained high levels of starch as S5. Despite this, its production of leachable acids was very low so there was no release of bad

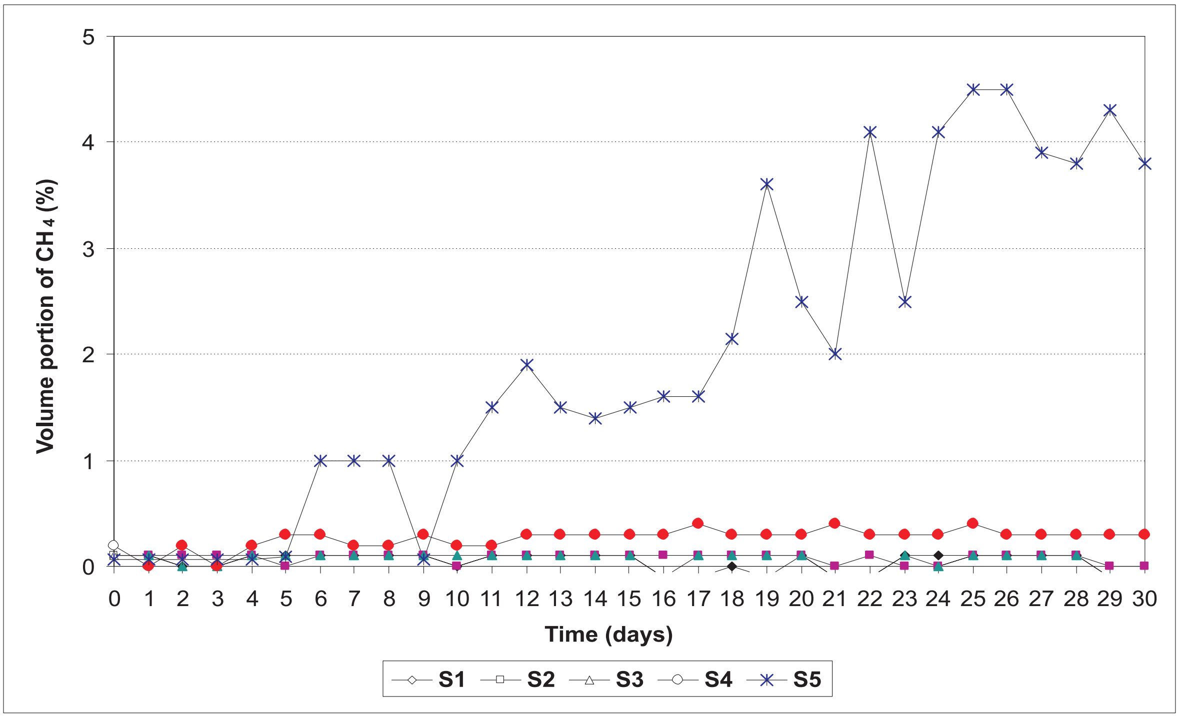

Fig. 2: Comparison of the typical dynamics of CH_4 emission from tested sludges (S1–S5).

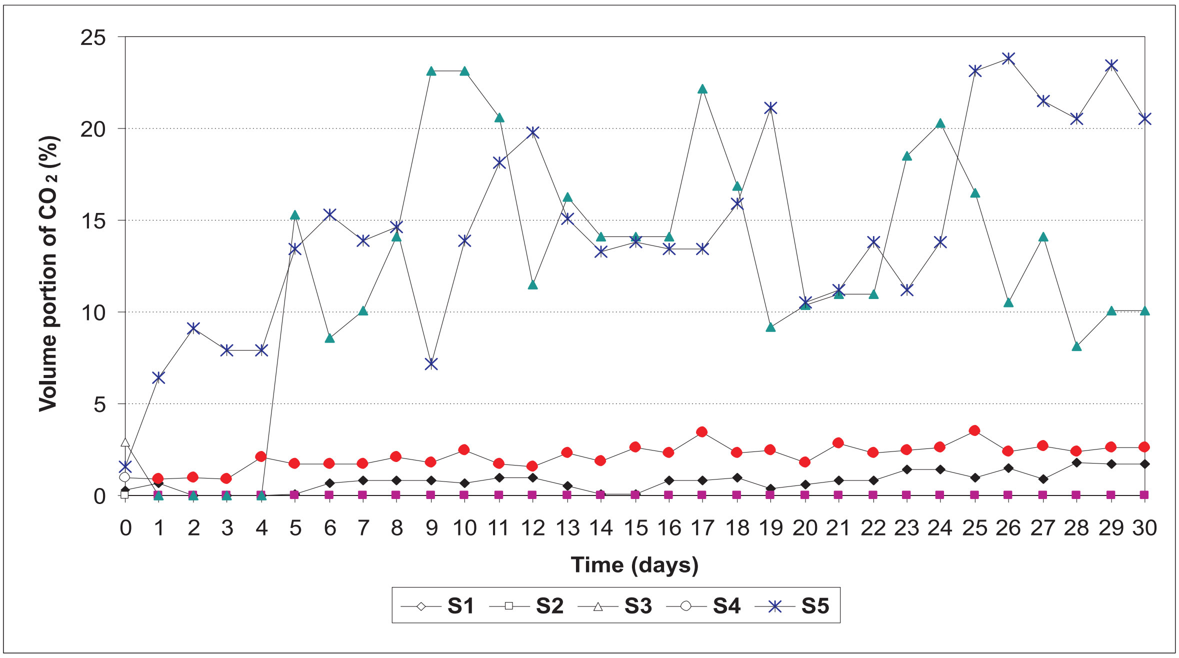

Fig. 3: Comparison of the typical dynamics of CO_2 emission from tested sludges (S1–S5).

smell either. The other materials were significantly more stable, manifesting practically no emissions.

A comparison with a similar investigation of biodegradation properties of chemi-mechanical and de-inking sludges (Palko *et al.* 2001) confirmed the fact that between 10 and 30% of the originally present organic matter is liable to biodegradation.

Subsequent geomechanical tests of sludge samples also showed some favourable properties, which may be still further improved by the addition of ash, obtained from sludge or wood residues.

For illustration, average values of the most important geomechanical parameters of a typical chemi-mechanical as well as de-inking sludge are collected in Table 5.

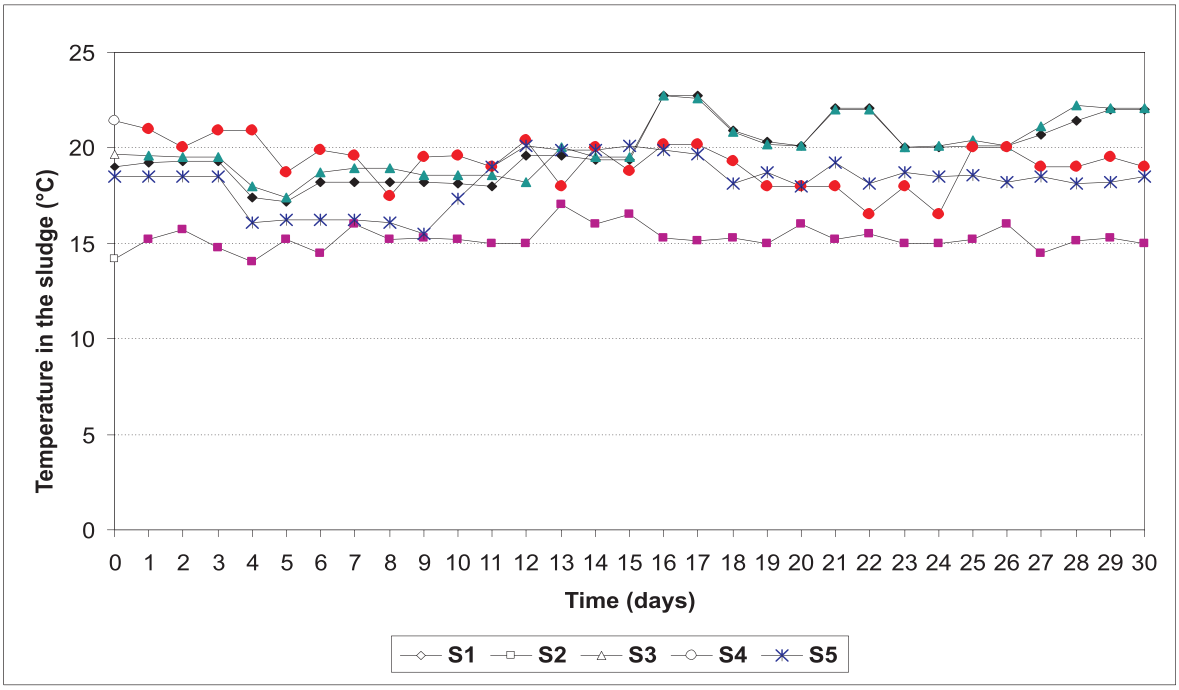

Fig. 4: Comparison of the typical dynamics of H_2S emission from tested sludges (S1–S5).

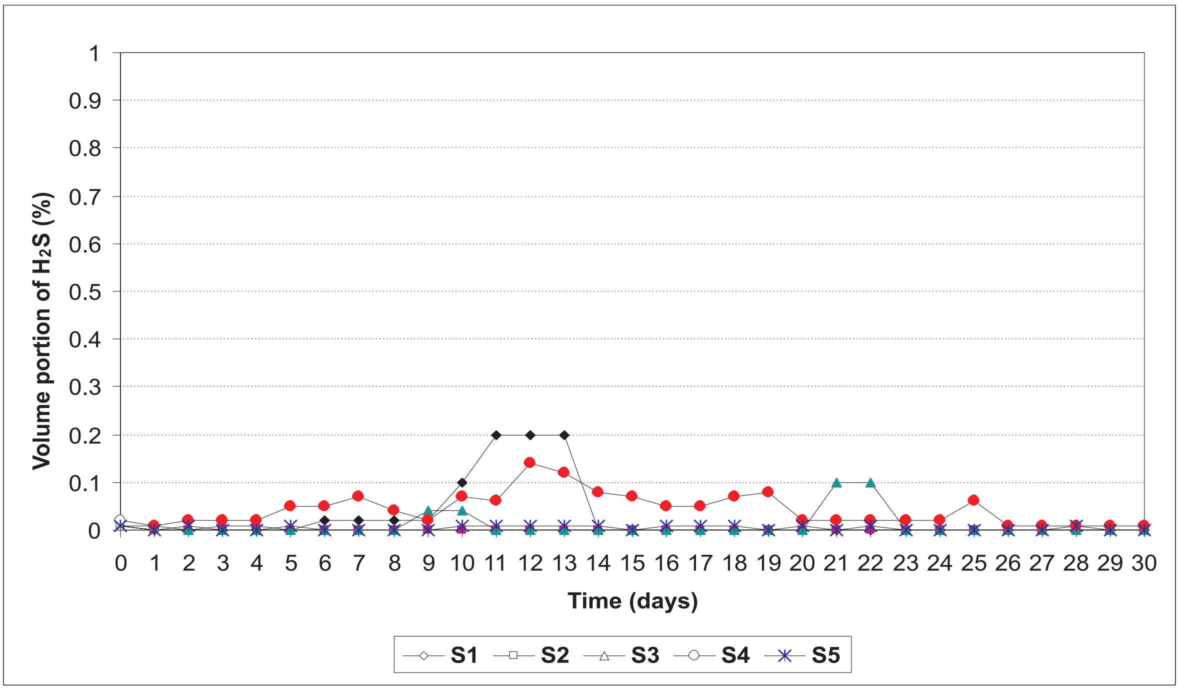

Fig. 5: Comparison of temperature profiles during biodegradation tests for sludges (S1–S5).

Table 5: Geomechanical properties of chemi-mechanical (S1) and de-inking (S4) sludges.

Sample	Uniaxial compressive strength (kPa)	Elastic modulus (MPa)	Poisson's ratio	Hydraulic conductivity coefficient ($m\ s^{-1}$)
S1	14	0.08–0.1	0.47–0.50	7.8×10^{-11}
S4	81	0.8–1.1	0.35–0.41	6.7×10^{-11}

Conclusion

It can be concluded that sludges subjected to landfill conditions during laboratory experiments are either not liable to biodegradation or else partial decomposition may take place deeper within the hydrodynamic layer. Subsequent chemical analyses (Tables 3 and 4) and measurements of released gas concentrations above the surface of these solids confirm the fact that in most cases they do not undergo any significant chemical and biochemical changes. Thus, there is practically no negative environmental impact such as emission of greenhouse gases and bad odours despite the relatively large content of organic fraction in sludges. Only those materials that contain large quantities of starch, which causes them to be more easily oxidized or readily attacked by micro-organisms, can be problematic. The latter are therefore usually incinerated. Incineration ash can be conveniently added to sludges that contain larger portions of organic particles, thus improving their geomechanical, chemical and stability characteristics. However, the addition of ash should not exceed 30% in order for the material to preserve its hydrodynamic properties. If the pH of leachates from sludge/ash mixtures increases above 8, the materials become even more resistant to microbiological degradation while at the same time their mechanical strength increases.

It has been actually proved by measurements of relevant geotechnical characteristics that paper mill sludges can be conveniently applied as alternative impermeable layers in landfill construction and closing where they can efficiently replace the much more expensive bentonite and compacted clay, which have similar geomechanical properties but a much higher price.

Such reuse of paper mill sludge may serve as a model example of efficient waste handling from both ecological and economical standpoints.

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