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A bioremediation case of an ex-quarry area restored by paper sludge

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ABSTRACT

Most paper industry waste is in the form of sludge from paper production and recycle process paper. There has been an increasing use of paper sludge in environmental restoration, a practice that requires particular attention.

This issue presents a case which demonstrates how the biogas production related to this kind of recovery system can represent a problem for environmental protection and public health.

The case history relates to a former quarry area restored by means of paper sludge. After the filling, a substantial quantity of biogas was produced, with an external diffusion to sensible target as well.

Initial investigations showed that the area was characterized by a large amount of paper mill sludge made unstable by anaerobic conditions. To date there are no proven technologies for this kind of treatment. In this case, for safety and naturalization as agricultural area, new methods of bioremediation were used and, in particular, an innovative physical, mechanical and biological intervention, based on bio-stabilization of paper mill sludge. The treatment is site-specific, based on the in-site paper sludge biostabilisation. To complete the intervention and in order to demonstrate its validity an important monitoring activity was performed, testing all the phases affected by the biological transformation.

1. Introduction

A correct management of paper industry waste is becoming very important in order to reduce the environmental impact of paper production, in particular reducing the amount of waste for final disposal. Paper mill sludge, are classified as non-hazardous waste by the Eu and Italian framework; its use as environmental restoration is in that way very important (Monte et al., 2009). However, as noted in some cases, there is a real risk of an uncontrolled biogas production from the waste with consequent danger to human health and environment.

Paper mill sludge has an high organic content, because it consists mainly of cellulose and wood fiber materials and it's widely used for environmental restoration due to its stability under aerobic conditions. However, laboratory tests show that there is a mismatch between its stability under aerobic and anaerobic conditions. It can be seen that it's very stable in relation with an aerobic degradation, which is a very slow degradation, but at the same time, it presents an easy anaerobic degradation with a consequent production of methane (Lin et al., 2011).

In this issue an interesting case history is reported, related an ex-quarry area characterized by a large amount of paper mill sludge made unstable because of its storage in anaerobic conditions. In this case, for the safety and the naturalization as agricultural area Amek, an Italian enterprises, has provided new method of bioremediation and in particular an innovative and site specific physical, mechanical and biological intervention, based on bio-stabilization of paper mill sludge (AMEK, 2008). This technology shows important advantages both from an economic and environmental point of view and requires human intervention only in the preparation phase, without water or electricity utilisation. Some of these advantages are: noise and air emissions minimization, reduction of visual impact at the end of operations at the site, global warming mitigation, technology implementation and management costs reduction, accelerated recovery of the areas for agriculture.

1.1. Paper sludge

The paper industry waste are biomass and are suitable for recovery, both of matter and energy. The main waste generated from production of paper is in the form of sludge, not classified as hazardous waste. The sludge is usually produced by the process of water purification, both physical, chemical, and biological, while the production process of the paper itself does not produce substantial waste; normally processing waste such as scrap and rag can be directly reused in the production process. As for the residue of the process of recycling waste paper, the focus is on pulper waste (arising from the separation of impurities from the crude fiber) and de-inking sludge, produced only when it involved removing ink from recovered paper. These residues are necessary to extract the pulp fiber used to make new paper; however they are on average less than 10% of the waste avoided. About 400,000 tons of the latter type of sludge are produced every year in Italy (Assocarta, 2010).

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The characteristics of residues from production of paper makes them suitable to be reused.

Paper mill sludge is increasingly used in the production of other paper, in the brick, cement construction and conglomerates and operations of environmental restoration as, for example, coverage of landfills or quarries. In addition, this sludge, subjected to drying, can be used in paper mills for energy production, contributing to the energy requirements for production facilities to disposal plants and subtracting large quantities of waste. The amount of waste that ends up in landfills is continually decreasing. Even in Europe, most countries in the CEPI (Confederation of European Paper Industries) have observed a growing use of more efficient options for the recovery of paper industry waste and a continued decline in the proportion of waste sent to landfills. However, the legislation penalizes such waste and unfortunately still does not allow a full exploitation of the high potential of these resources. Not all states recognize paper industry waste as a source of renewable energy and in some cases it's easier to put that waste in landfill.

From a regulatory standpoint, in Italy, for this type of product you refer Annex 1 sub-annex 1: "General technical standards for the recovery of materials from non-hazardous waste" of the Ministerial Decree of 5 February 1998 "Identification of non-hazardous waste subject to the simplified recovery procedures under Articles 31 and 33 of Legislative Decree 5 February 1997, No. 22. The decree shows the type of sludge and the EWC codes corresponding to" sludge from paper industry"; it describes the origin and characteristics of the waste, or waste from "purification process water" and "waste water of paper industry", with characteristic "mud shoveled."

It also lists the possible recovery activities including use for environmental restoration (the percentage of sludge used in combination with the ground must not exceed 30% (w/w) for sludge with at least 27% of dry matter). The sludge should have the following characteristics: total Hg $\leq$ 1.5 mg/kg DM, total Cd $\leq$ 1.5 mg/kg DM, Cr VI $\leq$ 0.5 mg/kg DM, total Ni $\leq$ 30 mg/kg DM, total Pb $\leq$ 40 mg/kg SS, total Cu $\leq$ 150 mg/kg DM, total Zn $\leq$ 500 mg/kg DM.

Article 5 of the decree defines the activities of "environmental restoration" as activities that "involve the restoration of damaged areas in order to return to productive or social uses through morphological remodeling" (code R10). This classification derives from recovery operation defined in waste framework directive (Council Directive 2008/98/EC). About 26% of paper mill waste is used for environmental restoration, type of recovery which is relevant in our case history.

Paper sludge are classified as: paper de-inking sludge, paper mill sludge cake, paper primary sludge, paper residue sludge and granulates, paper sludge, paper-mill sludge, primary sludge, pulp & paper secondary sludge, sludge, biological sludge, clarifier sludge, deinking sludge (ISPRA, 2010).

1.2. Bioremediation as environmental site restoration

In situ bioremediation consists in the use of indigenous microorganisms, heterotrophic, aerobic and anaerobic, in order to degrade contaminants with the ultimate goal of obtaining harmless chemicals. This technology is applied to soils and water, and is a cheaper and more efficient alternative to the standard method "pump and treat", used to clean the aquifers and soils contaminated with chlorinated solvents, hydrocarbon fuels, explosives, nitrates and metals toxic. The contaminant in this case is seen as a source of energy for microorganisms. The result of each degradation process depends on the microbial population (concentration of biomass, diversity of the population, enzyme activity), on the substrate (physical and chemical characteristics, molecular structure, concentration) and on a set of environmental factors (pH, temperature, content moisture, Eh, the ability of electron acceptors, carbon and energy sources). Some pollutants such as hydrocarbons are degraded by aerobic fuel, others such as carbon tetrachloride by anaerobic and others can be degraded under both conditions, such as trichloroethane (http://www.apat.gov.it/site/it-IT/Temi/Siti_contaminati, 2010).

Accelerated in situ bioremediation consists in adding substrate or nutrients, oxygen or other soil to an aquifer to stimulate the growth of a target of a consortium of bacteria. Generally these are indigenous, but crops of fungi, bacteria and other microbes can be introduced into the aquifer from other relevant sites in sufficient numbers to complete the process of biodegradation or bioaugmentation. The accelerated bioremediation is used where you want to increase the rate of biotransformation of contaminants. The type of correction required depends on the metabolic target pollutant present. This technique requires only the addition of oxygen, while anaerobic often requires the simultaneous addition of an electron donor (lactate or benzoate, for example) as an electron acceptor (nitrate or sulfate for example). Chlorinated solvents often require the adoption of a carbon substrate to stimulate reductive dechlorination. The objective of the accelerated in situ bioremediation is to increase the biomass for the entire aquifer volume occupied by the contaminant, for completion of the dissolved contaminants biodegradation and adsorbed.

Accelerated bioremediation technology degrades faster the contaminant, but also presents a greater investment in materials, equipment and manpower. We have many benefits as: the contaminants can be transformed completely into harmless substances, such as carbon dioxide, water; the time required for dealing with pollution of soil by using bioremediation can often be faster than when the "pump and treat" treatment is used and the cost is often lower than other remedial options; the treatment area concerned may be greater than that of other technologies, because the treatment can reach areas that might otherwise be inaccessible.

We have also some limits in this kind of bioremediation. The limits depend on the particular site where some contaminants may not become completely innocuous products. If the biotransformation stops at an intermediate compound, this may be more toxic or more mobile than the last, while some contaminants do not biodegrade (recalcitrant).

When applied inappropriately, the injection wells can be blocked due to the abundant microbial growth resulted from the addition of nutrients, electron donors or electron acceptors. Accelerated bioremediation is difficult to implement in aquifers with low permeability, because the transport of nutrient additives is limited. Heavy metals and toxic concentrations of organic compounds can inhibit the activity of indigenous microorganisms. Bioremediation usually requires a population of microorganisms that can't acclimate to develop recent contamination or recalcitrant compounds. Low temperatures slow down the process of remediation. The layers of warm soil can be used to cover the soil surface, thus increasing the temperature and rate of degradation. The length of time required for processing can vary from six months to five years and depends on many factors specific to the site in question.

1.3. The case history

The site was formerly a quarry area situated in the town of Imola (North Italy) and was used for mining of clay material in the eighties. About 6 years ago, the area has been affected by an environmental restoration, which provided for the use of 60,000 ton paper industry sludge according to waste recovery activities as R10 "land treatment and treatment resulting in benefit to agriculture and ecology". The types of sludge used were: paper mill sludge; paper residual sludge and granulates; clarifier sludge; de-inking sludge, in the following defined as PS (paper sludge).

One year later the end of restoration efforts, there were two explosions in a country house situated about 80 m from the former quarry and a subsequent inspection determined the explosion source to be biogas. Audits conducted under the supervision of ARPA ER have confirmed that its origin was due to production and migration of biogas from the restored area. It was also found that:

1. The sludge transferred belonged to the types allowed;
2. In several places the sludge had not been mixed with the soil by the percentages set by the reference regulation (30%, w/w);
3. The depth of wells was inconsistent, but much greater than the declared depth and came up to about −9 m from ground level.

High concentrations of methane gas were found within the recovery area and about 100 m away, close to sensitive targets. The storage of sludge led to an accelerated process of anaerobic degradation of organic matter (mainly cellulose), resulting in the production of large quantities of biogas.

2. Materials and methods

In order to describe paper sludge conditions and behavior and to define a proper treatment methodology, a preliminary characterization of buried PS mixed with soil was performed. It consisted in chemical–physical analysis, observations and respirometric analysis.

2.1. Sampling

Sampling procedure of mixture soil/buried PS was defined in order to check mixing distribution and to assess pilot intervention performance. They were taken in large amount for respirometric tests by means of a bucket and by Geoprobe system (slotted PVC well screen pipe ID 1 in. and 1.20 m long) for chemical characterization to minimize disturbance (Marroni et al., 2010).

Also samples of tailored compost and a mixture PS with compost (1:2 v/v) were prepared for respirometric characterization. Compost was produced from urban organic wastes from separated collection, agro-industrial wastes and lignocellulosic matter and additivated with enzymes according to Amek patent (2008) with Total Organic Carbon 25% DM and C:N about 18.

Sampling was scheduled at $t = 0$, and 14 months after pilot treatment for respirometric analyses.

The samples were collected from two different trenches and inside (ins.) and outside (out) of them. The fresh sample was subdivided in two homogeneous for the chemical–physical characterization and for the biostability lab scale tests. The samples were processed within 1–4 days after reception.

2.2. Chemical–physical characterization

Dry matter (DM) content, electrical conductivity, nitrogen as Total Kijeldhal (TKN) and ammonium ($NH4^+$-N) were determined on wet samples, after homogenization. Dry matter was used for pH and nitrate (DX120, DIONEX, Sunnyvale, CA, USA) measurements; it was used also for Volatile Solids (VS) determination, as loss on ignition at 550 °C for 2 h.

Total Organic Carbon (C) was measured by analyzer RC412 (LECO, Milano, Italy) on 40 °C oven dried samples, after grounding below 0.2 mm and sieving (500 μm). Also fibrous parts, that could not be ground, were weighted.

Chemical analytical determinations were performed according to Italian Standard Methods for fertilizers and compost (Trinchera et al., 2006; ANPA, 2001).

2.3. Respirometric methods

Solid phase characterization was performed by means of respirometric methods. Aerobic stability was checked measuring Dynamic Respirometric Index - DRI (mgO_2/kg VS*h) according to UNI/TS 11184 (2006) and Binner (2003) by adopting the Costech Instrument 3022 Respirometer (Costech International SpA, Cernusco sul Naviglio, Milano, Italy). It was determined on samples of 20-30 kg under humidity and pH standardized conditions as described in Adani et al. (2004). Also the cumulative oxygen consumption at/for 96 h DRI_{96cum} (mgO_2/kg VS) were determined (Grilli et al., 2008).

To assess the source term for biogas and its evolution during time, the Biochemical Methane Potential (BMP) over 30 and 100 days proposed by Environment Agency England and Wales (2005) was adopted; the application methods are reported in Grilli et al. (2008). The BMP experiments (Nm^3 CH_4/ton VS) were carried out in 500 mL capacity reactor using as inoculum granular sludge taken from an anaerobic digester treating paper mill waste and wastewaters. Methane production was periodically measured using the water displacement method together with alkaline solution trap. All samples were tested in triplicate in accordance with the methods reported by Environment Agency England and Wales (2005).

Fig. 1. View of a piezometer used for in situ gas monitoring.

2.4. Gas phase measurements

In combination with bioremediation pilot intervention, monitoring wells were created by inserting (6 m deep and 1 in. diameter) piezometers in the interested area (Fig. 1); they were perforated (4–5 m depth) to collect the gases. Gas phase composition was measured by means of portable instrument (GA 2000 PLUS, Geotechnical, Warwickshire, UK), an infrared gas analyzer which can monitor methane, carbon dioxide and oxygen as % v/v.

3. Results

In the following the results of the case history are reported in terms of investigation methodology and new restoration technology application.

3.1. Soil/sludge characterization

An example of chemical–physical characterization is reported in Table 1; first column describes the mixture soil/PS before bioremediation treatment. Carbon, SV and moisture figures show the characteristics of a sludge with very poor mixture with soil. The peculiarities are related to the presence of fibrous parts, cellulosic parts not yet degraded after 2 years disposal and a C:N ratio about 100 in combination with high C content (Dall'Ara et al., 2008a). Data are consistent with paper sludge composition, as available on specialized data set (www.ecn.nl/phyllis, 2011).

Table 1
Chemical characterization of buried paper sludge (PS) on wet basis inside biopile in pilot intervention at $t = 0$ and after 10 months; initial sample was characterized by about 50% of fibrous parts, not sieved.

Parameters	$T = 0^a$	$T = 300$ days
DM (%)	53.6	64.0
VS (% DM)	27.3[a]	19.7
pH	6.9	7.6
Conductivity (dS/m)	0.52	0.77
TOC (C) (%)	12.8[a]	8.8
TKN (%)	0.11	0.54
Nitrogen ($NH4^+$-N) (%)	0.02	0.09
C/N	116	17.6

[a]Measures performed in sieved parts.

In terms of initial reactivity, the results of respirometric characterization of initial samples are reported in Table 2, as DRI, oxygen consume and BMP_{100} for PS/soil mixture, tailored compost and biopile (a 1:2 mixture of PS and compost). Data are referred to VS, in order to compare different matrices avoiding the effects of different moisture and ashes contents.

Figures show a PS stable under aerobic conditions, a compost not yet stabilized and a reactivity even higher of the biopile. Threshold/Reference values for organic matter stability have been defined by Adani et al. (2004) in 1000 mgO_2/(kg VS*h) and 45.000 mgO_2/(kg VS), so to define it "a very stable material". DRI determination on other samples of tumbed sludge were in the range 120–420 mgO_2/(kg VS*h) (Grilli et al., 2008). Also BMP for both PS and compost were high; the mixture of sludge and compost caused an increment of BMP in comparison with the single component. Therefore a fraction of organic matter had been transformed in prompt degradable fraction. This availability could be explained by the microbiological composition of the tailored compost: it had a good amount of total heterotrophic bacteria (about 10^9 CFU/g total solids), with 20% of (aerobic) cellulolosolitic bacteria but also by its contribution for nutrients, in particular Nitrogen (Dall'Ara et al., 2008b).

In terms of evolution over time, after 14th treatment month, BMP_{100} showed a significant decrease to 40–70 (Nm^3 CH_4/ton VS), inside trenches. In both monitored opencuts the methane production potential was reduced by more than 60%. Instead, after the same treatment time the methane production of the outside opencuts samples was in the range 104–133 (Nm^3 CH_4/ton VS) leading to a nearly 25% reduction of the methanogenic activity (Grilli et al., 2008).

3.2. Intervention methodology

The intervention performed is configured as a physical, mechanical and biological.

This technique requires only the use of:

- Compost "designed"

- Enzyme mixture of vegetable origin, both produced using the methodology stated in a patent application.

A biological treatment was made based on natural attenuation, biostimulation with the proper elements of bioremediation. Indeed, in biostimulation, nutrients in the form of organic fertilizers (compost) are introduced into the soil to increase the indigenous microbial population. In this case, they are also used as substances to activate bioremediation, to assist and accelerate the degradation of contaminants in a state of inertia. The degradation is carried out by micro-organisms already present in the soil, particularly by microaerophilic microorganisms. A particular type of compost was used: it is a "designed" compost, manufactured for the treatment of specific contaminants at specific sites, in this case enriched by microorganisms that break down cellulose. In this way, it also has the properties of any quality compost with a high degree of maturity (which has an active biomass with high metabolic activity of such degradation), with specific features for the material degradation. The "Compost Bioremediation is a procedure which is validated by the EPA (U.S. Environmental Protection Agency) in different contexts, such as sites contaminated with hydrocarbons, solvents, pesticides, heavy metals, oil products and explosives.

The adoption of a "designed" compost determines a positive effect on the general conditions of soil formation, contributing, in particular, in a relatively short time, to restore the same quality of pre-existing humus. It is a stable "material" with a high content of organic matter, qualificated as fertilizer and substrate.

The physical, biological and technical proposals were developed according to three types of intervention:

- Trenches "Biofilter", which act as a chimney for the escape of gas convection and at the same time allow the biochemical transformation of methane into carbon dioxide, catalyzed by the materials used and promoted. In the excavation, a "designed" and selected enzyme mixture was introduced to the compost campaign plan. Given the lower specific gravity compared to the cover materials, a lower lithostatic pressure is obtained by replacing the original material and topsoil with compost and enzyme mixture, avoiding the creation of pockets of gas and promoting the degassing of existing ones.

- Trenches "biopiles" excavation, in which are introduced layers of "designed" compost, the enzyme mixture and the mud removed during the excavation itself The biopiles are then covered with about 1 m of the original layer. The hat in this case is completely or partially replaced with compost enriched with enzyme mixtures in order to decrease the lithostatic pressure, acting on the material and thereby degassing the grave, but also, given the greater porosity of the compost compared to the cover layer establishing aerobic conditions, favorable for aerobic bio-stabilization process. The objective of this type of trench is the activation of methanogenic fermentation, namely to reduce the methane production of sludge, accelerating their aerobic degradation.

- Biopiles outside, the extracted mud is mixed with "designed" compost, in an amount properly estimated on the base of the chemical characteristics of the material; it's also additived with enzyme mixture and subjected to composting. At the end of the bio-stabilization and maturation processes, the obtained material is used in situ for the area restoration and its recovery for agriculture.

3.3. Pilot in situ intervention

On the basis of initial soil/PS mixture characterization, as a preliminary step, a pilot in situ intervention was performed in June

Table 2
Dynamic respirometric index (DRI) results and BMP at 100 days (BMP_{100}) for extracted paper sludge, compost used for bioremediation and of the mixture (biopile).

Components	DRI_{mean}	DRI_{mean}	O_2 Consumption at 96 h	BMP_{100}
	mgO_2/(kg VS*h)	mgO_2/(kg DM*h)	mgO_2/(kg VS)	Nm^3 CH_4/ton VS
Buried paper sludge (PS)	345	103	22,256	180
Compost	3215	1501	166,188	330
Biopile (PS and Compost 1:2)	8798	1608	248,468	350

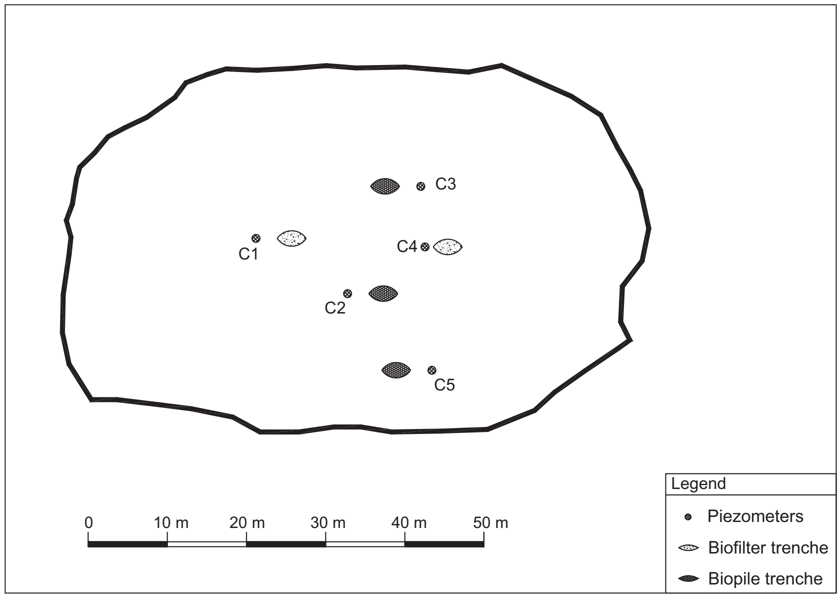

Fig. 2. Scheme of pilot intervention.

2006. It involved a 130 m^2 out of the almost 5 hectares of the site and consisted in 2 biofilter trenches (pits) and 3 biopiles of about 9 m^2 each. At the same time, 5 piezometers for gas monitoring were located (Fig. 2). After 16–18 months after pilot intervention, the whole site was biotreated according to the technology described.

The results of biogas monitoring over time in the dedicated wells are reported in Fig. 3, where mean value for methane, carbon dioxide and oxygen are reported over 50 months. The trends show an immediate decrease in methane and carbon dioxide concentration in the few months after pilot treatment, followed by a subsequent reset to initial situation. Only after whole area treatment (18th month) there was stable decrease in CH_4 and CO_2 content; after an additional 12 months, methane figures are lower than 5% (v/v), which is Lower Explosion Level - LEL. Oxygen reached at the same time figures around 20%, atmospheric value.

Gas trend is in agreement with oxido-reduction conditions measured in suspended water tables; in fact pH was stable around 6.6, Eh (mV) values were about −100 mV in June 2006, indicating conditions favorable to methanogenesis. Eh reached the range (100–200) mV after 48 months (Marroni et al., 2010).

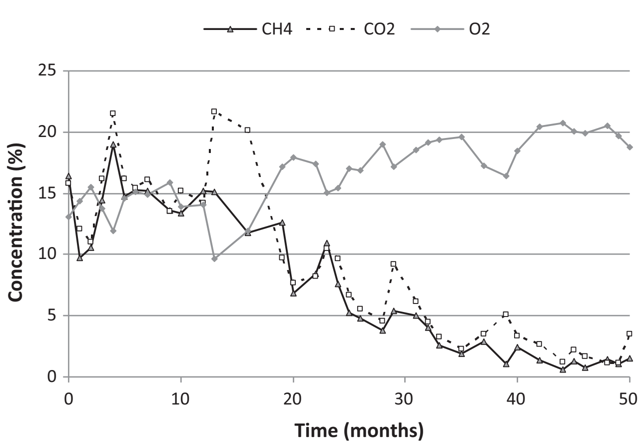

Fig. 3. Biogas and oxygen trend over time expressed as mean value in the 5 piezometers located in pilot intervention area. Treatment of the whole area was completed in the 16–18.

4. Conclusions

The innovative methodology developed for the permanent safety of this area showed the reduction of methane production and concentration in the site. The technology was developed specifically for matrices with high organic matter content focused on the different behavior of paper sludge in aerobic or anaerobic conditions.

This methodology can be applied to other restoration sites, with characteristics similar to the studied ones, both to put in safety conditions and eliminate hazardous conditions when biogas has been produced beyond control and in sites to prevent biogas production and migration. With proper adaptations, the knowledge and the information received from this experience can be the basis for a technology that can be applied also to old landfill sites, where lignocellulosic matter constitutes an important fraction of disposed waste and which contributes to biogas production also for long term. In fact some old landfills were managed as quarries, and they have to be managed also for more than 30 years aftercare; biogas concentration is not enough to be exploited for energy production, there are difficulties in its burning and therefore new different solution have to be defined.

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