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# Utilisation of Waste from Digesters for Biogas Production

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## 1. Introduction

### 1.1 Is the waste from digesters (digestate) an excellent organic fertilizer?

A prevailing opinion of bio-power engineers as well as in literature is that wastes from digesters in biogas production are an excellent fertiliser and that anaerobic digestion is to some extent an improvement process in relation to the fertilising value of organic materials used for biogas production. These opinions are apparently based on the fact that in anaerobic stabilisation of sludge the ratio of organic to mineral matters in dry matter is approximately 2:1 and after methanisation it drops to 1:1. Because there is a loss of a part of organic dry matter of sludge in the process of anaerobic digestion, the weight of its original dry matter will decrease by 40%, which will increase the concentration of originally present nutrients. In reality, anaerobic digestion will significantly release only ammonium nitrogen from the original material, which will enrich mainly the liquid phase due to its solubility; the process will not factually influence the content of other nutrients (Straka 2006).

The opinion that waste from anaerobic digestion is an excellent fertiliser is also due to the observation of fertilised lands. The growths are rich green and juicy. They have a fresh appearance – this is a typical sign of mineral nitrogen, including larger quantities of water retention by plants due to the nitrogen. However, the content of dry matter is changed negligibly, which shows evidence that the fertilisation is inefficient.

If organic matter is to be designated as organic fertiliser, it has to satisfy the basic condition: it has to be easily degradable microbially so that it will release necessary energy for soil microorganisms.

### 1.2 Mineralisation of organic matter in soil

This microbial transformation of organic matter in soil is mineralisation when organic carbon of organic substances is transformed to  $\text{CO}_2$  and from mineralised organic matter those mineral nutrients are released that were already contained in organic matter in mineral (ionic) form and those that were in it in organic form.  $\text{CO}_2$  is an important fertiliser in agriculture; it is the basic component for photosynthetic assimilation, for the formation of new organic matter produced by plants. As plants can take up only nutrients in mineral form ( $\text{K}^+$ ,  $\text{NH}_4^+$ ,  $\text{NO}_3^-$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{H}_2\text{PO}_4^-$ ,  $\text{HPO}_4^{2-}$ ,  $\text{SO}_4^{2-}$  etc.) and nutrients in organic form (e.g. protein nitrogen, phosphorus of various organophosphates), it is not accessible to plants, and besides its main function – energy production for the soil microedaphon – the mineralization of organic matter in soil is an important source of mineral nutrients for

plants. It is applicable solely on condition that organic matter in soil is easily mineralisable, i.e. degradable by soil microorganisms.

### **1.3 Gain from mineralising organic fertiliser for farmers: energy for soil microorganisms and release of mineral nutrients for plant nutrition**

What we appreciated more for organic fertilisers? Gain of energy and enhancement of the microbial activity of soil or savings that are obtained by the supply of mineral nutrients? Unfortunately, simplified economic opinions cause each superficial evaluator to prefer the gain of mineral nutrients released from organic matter. Such a gain is also easy to calculate. The calculation of the gain from an increased microbial activity of soil is difficult and highly inaccurate. Nevertheless, a good manager will unambiguously prefer such a gain. It is to note that the microbial activity of soil is one of the main pillars of soil productivity, it influences physical properties of soil, air and water content in soil, retention of nutrients in soil for plant nutrition and their losses through elution from soil to groundwater. A biological factor is one of the five main factors of the soil-forming process; without this process the soil would not be a soil, it would be only a parent rock or perhaps a soil-forming substrate or an earth at best.

Hence, it is to state that the release of mineral nutrients for their utilisation by plants during mineralisation of organic fertiliser in the soil produces an economically favourable effect but it is not the primary function of organic fertiliser, its only function is the support of microedaphon. The effect of mineral nutrients is replaceable by mineral fertilisers, the energetic effect for the microbial activity of soil is irreplaceable.

### **1.4 What influences the quality of digestate as a fertiliser?**

The digestate, the waste from digesters during biogas production, is composed of solid phase and liquid phase (fugate). We have demonstrated that the solid phase of the digestate is not an organic fertilizer because its organic matter is very stable and so it cannot be a relatively expeditious source of energy for the soil microedaphon (Kolář et al. 2008). Neither is it a mineral fertilizer because available nutrients of the original raw material and also nutrients released from it during anaerobic digestion passed to the liquid phase – fugate. The digestate, and naturally the fugate, have a low content of dry matter (fugate 0.8 – 3% by weight) and this is the reason why analytical data on the ones to tens of weight % of available nutrients given in dry matter foster an erroneous opinion in practice that these wastes are excellent fertilizers. In fact, fugates are mostly highly diluted solutions in which the content of the nutrients that are represented at the highest amount, mineral nitrogen, is only 0.04 – 0.4% by weight.

The surplus of water during fertilization with this waste increases the elution of this nutrient in pervious soils while in less pervious soils the balance between water and air in the soil is impaired, which will have negative consequences.

The quality of the digestate as an organic fertiliser (labile, not organic material that is hard to decompose) substantially influences not only the microbial decomposability of the input material but also the level of anaerobic digestion in the digester. In the past when the sludge digestion was carried out in municipal waste treatment plants in digesters at temperatures of 18°C-22°C (psychrophilic regime), the decomposability of the substrate after fermentation was still good, therefore the digested sludge was a good organic fertiliser. These days we work with less decomposable substrates in mesophilic ranges (around 40°C) or even in thermophilic conditions. The degree of decomposition of organic matter during fermentation is consequently high and the digestate as organic fertiliser is practically worthless.

### 1.5 A hopeful prospect – IFBB process

It would be ideal to realize biogas production from the liquid phase only – it would be possible to introduce high performance UASB (Upflow Anaerobic Sludge Blanket) digesters and to achieve the large saving of technological volumes but the concentration of substances in the liquid phase should have to be increased. The solid phase of substrates, which cannot be applied as an organic fertilizer after the fermentation process, would be used as biomass for the production of solid biofuels in the form of pellets or briquettes. But it would be necessary to reduce its chlorine content to avoid the generation of noxious dioxins and dibenzofurans during the burning of biofuel pellets or briquettes at low burning temperatures of household boilers and other low-capacity heating units. Wachendorf et al. (2007, 2009) were interested in this idea and tried to solve this problem in a complex way by the hot-water extraction of the raw material (at temperatures of 5°C, 60°C and 80°C) followed by the separation of the solid and liquid phase by means of mechanical dehydration when a screw press was used. This procedure is designated by the abbreviation IFBB (Integrated Generation of Solid Fuel and Biogas from Biomass). These researchers successfully reached the transfer ratio of crude fibre from original material (grass silage) to liquid phase only 0.18, which is desirable for biogas production, but for more easily available organic substances influencing biogas production, e.g. nitrogen-free extract, the ratio is 0.31. The transfer of potassium, magnesium and phosphorus to the liquid phase ranged from 0.52 to 0.85 of the amount in fresh matter, calcium transformation was lower, at the transfer ratio 0.44 – 0.48 (Wachendorf et al. 2009). Transformation to the liquid phase was highest in chlorine, 0.86 of the amount in original fresh matter, already at a low temperature (5°C). The transfer of mineral nitrogen to the liquid phase before the process of anaerobic digestion is very low because there is a minute amount of mineral N in plant biomass and the major part of organic matter nitrogen is bound to low-soluble proteins of the cell walls. Nitrogen from these structures toughened up by lignin and polysaccharides is released just in the process of anaerobic digestion. Because in the IFBB process also organic nitrogen compounds (crude protein – nitrogen of acid detergent fibre ADF) are transferred to the liquid phase approximately at a ratio 0.40, the liquid phase, subjected to anaerobic digestion, is enriched with mineral nitrogen.

Like Wachendorf et al. (2009), we proceeded in the same way applying the IFBB system for the parallel production of biogas and solid biofuels from crops grown on arable land. The IFBB technological procedure is based on a high degree of cell wall maceration as a result of the axial pressure and abrasion induced with a screw press.

## 2. Crucial problems

### 2.1 The first problem: organic matter of digestate is poorly degradable in soil, its labile fractions were already utilised in a digester

The point is that the digestate is not an organic fertiliser because its organic substance is poorly degradable. But its liquid fraction contains a small amount of mineral nutrients, mainly of nitrogen. The fugate (and also the digestate) can be considered as a very dilute mineral fertiliser, nitrogenous fertiliser. However, the agriculture sector is exposed worldwide to an enormous pressure on economic effectiveness while the costs of machinery, fuels and agricultural labour force are very high in relation to the price of agricultural products. Therefore the chemical industry helps farmers to save on transportation and application costs incurred by fertilisation when highly concentrated

mineral fertilisers are produced. Even though they are substantially more expensive, from the aspect of cost accounting their use will finally pay off. Before the manufacture of town gas from coal using the ammonia water ended, farmers took the waste containing 1% of ammonia nitrogen only exceptionally even though it was practically free of charge.

With the current output of a biogas plant 526 kW (Chotýčany, South Bohemia) and daily dose of a substrate to the digester 46 t and practically identical production of digestate the daily production of mineral nitrogen is approximately 40 kg, which amounts to a relatively high value per year, almost 15 t of mineral nitrogen, but the dilution is unacceptable.

## **2.2 The second problem: the digestate contains much water and therefore the solution with plant nutrients is very dilute.**

If this waste is applied as a fertiliser, the water surplus increases the elution of this nutrient into the bottom soil in pervious soils. In impervious soils and in less pervious soils the imbalance between water and air in the soil is deteriorated with all adverse consequences: aerobiosis restriction, reduction in the count of soil microorganisms, denitrification and escape of valuable nitrogen in the form of  $N_2$  or N-oxides into the atmosphere. Soil acidification takes place because organic substances are not mineralised under soil anaerobiosis and they putrefy at the simultaneous production of lower fatty acids. These soil processes result in a decrease in soil productivity. Currently, its probability is increasingly higher for these reasons:

1. As a consequence of global acidification the frequency of abundant precipitation is higher in Europe throughout the year.
2. As a result of rising prices of fuels, depreciation on farm machinery and human labour force farmers apply digestates or fugates in the closest proximity of a biogas plant. It causes the overirrigation of fertilised fields even though the supplied rate of nitrogen does not deviate from the required average.

The problem of an excessively high irrigation amount has generally been known since long: it occurred in Berlin and Wrocław irrigation fields after irrigation with municipal waste water in the 19<sup>th</sup> and 20<sup>th</sup> century, in the former socialist countries after the application of agricultural and industrial waste waters and of slurry from litterless operations of animal production. Even though nobody surely casts doubt on the fertilising value of pig slurry or starch-factory effluents, total devastation of irrigated fields and almost complete loss of their potential soil productivity were quite normal phenomena (Stehlík 1988).

## **2.3 Fundamental issues to solve**

A further part of this study should help solve these crucial problems:

1. What is the rational utilisation of digestate and/or fugate and separated solid fraction of digestate in the agriculture sector that are generated by current biogas plants if we know that their utilisation as fertilisers is rather problematic?
2. What are the prospects of utilisation of wastes from biogas production and what modifications in the technology of biogas production from agricultural wastes should be introduced?
3. What problems should be solved by researchers so that the promising utilisation of wastes from biogas production could be realised?
4. What is the optimum form of utilisation of wastes from biogas plants and why?



### 3. Information

#### 3.1 Current optimum utilisation of digestate from biogas plants in the agriculture sector

##### 3.1.1 Biodegradability (lability) and stability of organic matter

How many labile components of organic matter are lost during anaerobic digestion in a biogas plant can be demonstrated by determination of the degree of organic matter lability. For this purpose a number of methods can be used that are mostly based on resistance to oxidation or on resistance to hydrolysis. Oxidation methods are based on oxidation with chemical oxidants, e.g. with a solution of  $K_2Cr_2O_7$  in sulphuric acid at various concentrations – 6 M + 9 M + 12 M (Walkley 1947, Chan et al. 2001) or with a neutral solution of  $KMnO_4$  at various concentrations (Blair et al. 1995, Tirol-Padre, Ladha 2004). The degree of organic matter lability is evaluated from the amount of oxidizable carbon in per cent of its total amount in particular variously aggressive oxidation environments or the reaction kinetics of the observed oxidation reaction is examined while its characteristic is the rate constant of the oxidation process.

In 2003 was proposed and tested the method to evaluate the kinetics of mineralisation of the degradable part of soil organic matter by the vacuum measurement of biochemical oxygen demand (BOD) of soil suspensions using an Oxi Top Control system of the WTW Merck Company, designed for the hydrochemical analysis of organically contaminated waters (Kolář et al. 2003). BOD on the particular days of incubation is obtained by these measurements whereas total limit  $BOD_t$  can be determined from these data, and it is possible to calculate the rate constant  $K$  of biochemical oxidation of soil organic substances per 24 hours as the rate of stability of these substances. A dilution method is the conventional technique of measuring BOD and also rate constants. It was applied to determine the stability of soil organic substances but it was a time- and labour-consuming procedure. The Oxi Top Control method was used with vacuum measurement in vessels equipped with measuring heads with infrared interface indicator communicating with OC 100 or OC 110 controller while documentation is provided by the ACHAT OC programme communicating with the PC, and previously with the TD 100 thermal printer. Measuring heads will store in their memory up to 360 data sentences that can be represented graphically by the controller while it is also possible to measure through the glass or plastic door of the vessel thermostat directly on stirring platforms. The rate of biochemical oxidation of organic substances as the first-order reaction is proportionate to the residual concentration of yet unoxidised substances:

$$dy/dt = K (L - y) = KL_z \quad (1)$$

where:

$L$  = total BOD

$y$  = BOD at time  $t$

$L_z$  = residual BOD

$k, K$  = rate constants

By integrating from 0 to  $t$  of the above relation the following equation is obtained:

$$L_z = L \cdot e^{-Kt} = L \cdot 10^{-kt} \quad (2)$$

In general it applies for BOD at time  $t$ :

$$y = L (1 - 10^{-kt}) \quad (3)$$

where:

$y$  = BOD at time  $t$

$L$  = BOD<sub>total</sub>

$k$  = rate constant [24 hrs<sup>-1</sup>]

Used procedure is identical with the method of measurement recommended by the manufacturer in accordance with the Proposal for German Uniform Procedures DEV 46<sup>th</sup> Bulletin 2000 - H 55, also published in the instructions for BOD (on CD-ROM) of WTW Merck Company.

The decomposition of organic matter is the first-order reaction. In these reactions the reaction rate at any instant is proportionate to the concentration of a reactant (see the basic equation  $dy/dt$ ). Constant  $k$  is the specific reaction rate or rate constant and indicates the instantaneous reaction rate at the unit concentration of a reactant. The actual reaction rate is continually variable and equals the product of the rate constant and the instantaneous concentration. The relation of the reaction product expressed by BOD at time  $t$  ( $y$ ) to  $t$  is the same as the relation of the reactant ( $L - y$ ) at time  $t$  and therefore the equations

$$(L - y) = L \cdot e^{-kt} \quad (4)$$

and

$$y = L (1 - e^{-kt}) \quad (5)$$

are analogical.

If in the graph the residual concentration of carbon is plotted on the y-axis in a logarithmic scale  $\log (L - y)$  and the time in days from the beginning of experiment is plotted on the x-axis, we will obtain a straight line, the slope of which corresponds to the value  $-k/2.303$ .

The quantity of the labile fraction of organic matter can also be assessed by determination of soluble carbon compounds in hot water (Körschens et al. 1990, Schulz 1990) and their quality by determination of the rate constant of their biochemical oxidation (Kolář et al. 2003, 2005a, b).

Hydrolytic methods are based on resistance of the organic matter different aggressive ways of hydrolysis that is realised at different temperature, time of action and concentration of hydrolytic agent, which is usually sulphuric acid. Among many variants of these methods the hydrolytic method according to Rovira et Vallejo (2000, 2002, 2007) in Shirato et Yokozawa (2006) modification was found to be the best. This method yields three fractions: labile LP1, semi-labile LP2 and stable LP3. The per cent ratio of these three fractions, the sum of which is total carbon of the sample  $C_{tot}$ , provides a very reliable picture of the degree of organic matter lability.

Of course, there are a lot of methods based on the study of organic matter biodegradability in anaerobic conditions. First of all, it is the international standard ISO C<sub>D</sub> 11734: Water quality - evaluation of the "ultimate" anaerobic biodegradability of organic compounds in digested sludge - Method by measurement of the biogas production, and particularly a very important paper using the Oxi Top Control measuring system manufactured by the German company MERCK for this purpose (Süssmuth et al. 1999).

Tests of methanogenic activity (Straka et al. 2003) and tests examining the activity of a microbial system (Zábranská et al. 1985a, b, 1987) are methods that can describe the degree of organic matter lability in its ultimate effect. Our long-time work experiences in the

evaluation of a huge amount of various analyses for the study of organic matter lability have brought about this substantial knowledge:

1. The study of the ratio of organic matter labile fractions, i.e. of their quantity, is always incomplete. A more authentic picture of the situation can be obtained only if information on the quality of this labile fraction is added to quantitative data. Such a qualitative characteristic is acquired in the easiest way by the study of reaction kinetics of the oxidation process of this fraction. The process of biochemical oxidation and the calculation of its rate constant  $K_{\text{Bio}}$  are always more accurate than the calculation of its rate constant of oxidation by chemical oxidants  $K_{\text{CHEM}}$  (Kolář et al. 2009a).
2. It applies to current substrates for biogas production in biogas plants that with some scarce exceptions the degree of organic matter lability is very similar in both aerobic and anaerobic conditions. In other words: organic matter is or is not easily degradable regardless of the conditions concerned (Kolář et al. 2006).
3. A comparison of various methods for determination of organic matter lability and its degradability in the anaerobic environment of biogas plant digesters and also for determination of digestate degradability after its application to the soil showed that hydrolytic methods are the best techniques. They are relatively expeditious, cheap, sample homogenisation and weighing are easy, and the results correlate very closely with methods determining the biodegradability of organic matter directly. E.g. with the exception of difficult weighing of a very small sample and mainly its homogenisation the Oxi Top Control Merck system is absolutely perfect and highly productive – it allows to measure in a comfortable way simultaneously up to 360 experimental treatments and to assess the results continually using the measuring heads of bottles with infrared transmitters, receiving controller and special ACHAT OC programme for processing on the PC including the graph construction. But its price is high, in the CR about 4 million Kč for the complex equipment. Hydrolytic methods require only a small amount of these costs and are quite satisfactory for practical operations (Kolář et al. 2008). However, for scientific purposes we should prefer the methods that determine anaerobic degradability of organic matter, designated by  $D_C$ .

The substrate production of methane  $V_{\text{CH}_4\text{S}}$  [the volume of produced methane ( $V_{\text{CH}_4\text{c}}$ ) after the subtraction of endogenous production of methane ( $V_{\text{CH}_4\text{e}}$ ) by the inocula] was determined by an Oxi Top Control Merck measuring system.

The calculation is based on this equation of state:

$$n = p \times V/RT \quad (6)$$

where:

$n$  = number of gas moles

$V$  = volume [ml]

$P$  = pressure [hPa]

$T$  = temperature [°K]

$R$  = gas constant 8.134 J/mol °K

and the number of  $\text{CO}_2$  and  $\text{CH}_4$  moles in the gaseous phase of fermentation vessels is calculated:

$$n_{\text{CO}_2 \text{ g CH}_4} = (\Delta p \times Vg/RT) \times 10^{-4} \quad (7)$$

$$\Delta p = p_1 - p_0 \quad (8)$$



where:  $p_0$  = initial pressure

Fermentation at 35° C and continuous agitation of vessels in a thermostat lasts for 60 days, the pressure range of measuring heads is 500 – 1 350 kPa and the time interval of measuring pressure changes is 4.5 min. Anaerobic fermentation is terminated by the injection of 1 ml of 19% HCl with a syringe through the rubber closure of the vessel to the substrate. As a result of acidification CO<sub>2</sub> is displaced from the liquid phase of the fermentation vessel. The process is terminated after 4 hours. The number of CO<sub>2</sub> moles is calculated from the liquid phase:

$$n_{CO2\ l} = \{[p_2 (V_g - V_{HCl}) - p_1 \times V_g] / RT\} \times 10^{-4} \quad (9)$$

The injection of 1 ml of 30% KOH into the rubber container in the second tube of the fermentation vessel follows. The sorption of CO<sub>2</sub> from the gaseous phase of the vessel is terminated after 24 hours and the total number of CO<sub>2</sub> moles in gaseous and liquid phases is calculated from a drop in the pressure in the vessel:

$$n_{CO2\ l, CO2\ g} = \{[p_3 (V_g - V_{HCl} - V_{KOH}) - p_2 (V_g - V_{HCl})] / RT\} \times 10^{-4} \quad (10)$$

where:

$\Delta p$  = difference in pressures [hPa]

$V_g$  = the volume of the gas space of the fermentation vessel [ml]

$p_1$  = gas pressure before HCl application [hPa]

$p_2$  = gas pressure before KOH application [hPa]

$p_3$  = gas pressure after KOH application [hPa]

$R$  = gas constant = 8.134 J/mol °K

$T$  = absolute temperature = 273.15 + X °C

$V_{HCl}$  = the volume of added HCl [ml]

$V_{KOH}$  = the volume of added KOH [ml]

Based on the results, it is easy to calculate the number of CO<sub>2</sub> moles in the gaseous phase and by the subtraction from  $n_{CO2\ g\ CH_4}$  the number of moles of produced methane:

$$n_{CH_4} = (n_{CO2\ g\ CH_4} + n_{CO2\ l}) - n_{CO2\ l\ CO2\ g} \quad (11)$$

The total number of moles of the gases of transported carbon:

$$n_{CO2\ g\ CH_4} + n_{CO2\ l} = n_{total} \quad (12)$$

Baumann's solution A + B in deionised water of pH = 7.0 is used as a liquid medium (Süssmuth et al. 1999).

The standard addition of the inoculum corresponds roughly to an amount of 0.3% by volume (aqueous sludge from the anaerobic tank of the digester). Instead of Baumann's solution it is possible to use a ready-made nutrient salt of the MERCK Company for this system.

The operation of the Oxi Top Control measuring system was described in detail by Süssmuth et al. (1999).

Methane yield was calculated from the substrate production of methane  $V_{CH_4S}$  by division by the initial quantity of the added substrate:

$$Y_{CH_4g} = \frac{(V_{CH_4C} - V_{CH_4e})}{S} = \frac{V_{CH_4S}}{S} [l / g] \quad (13)$$

where:

$V_{CH4C}$  = methane yield of C-source

$V_{CH4e}$  = methane yield of the added inoculum

$S$  = substrate quantity at the beginning [g]

Lord's test and other methods suitable for few-element sets and based on the R range of parallel determinations were used for the mathematical and statistical evaluation of analytical results including the computation of the interval of reliability.

Anaerobic degradability is given by the equation:

$$D_c = \frac{C_g}{C_s} \cdot 100 \quad (14)$$

where:

$C_s$  = total C content in the sample

$C_g$  = C content in methane released during the measurement of anaerobic degradability

The value of  $C_g$  is computed from the substrate production of methane  $V_{CH4S}$ :

$$C_g = \frac{12 p V_{CH4S}}{RT} \quad (15)$$

(because 1 mol  $CH_4$  contains 12 g C)

where:

$K$  = temperature (°K)

$R$  = gas constant

$P$  = pressure

$V_{CH4S}$  = the volume of produced methane after the subtraction of endogenous production by the inoculum from total production

This method, which determines organic matter lability in anaerobic conditions, is so exact that it allows to investigate e.g. the digestive tract of ruminants as an enzymatic bioreactor and to acquire information on its activity, on feed utilisation or digestibility and on the influence of various external factors on the digestion of these animals (Kolář et al. 2010a) or to determine the share of particular animal species in the production of greenhouse gasses (Kolář et al. 2009b).

At the end of this subchapter dealing with the degree of organic matter lability and its changes after fermentation in a biogas plant these experimental data are presented:

A mixture of pig slurry and primary (raw) sludge from the sedimentation stage of a municipal waste water treatment plant at a 1 : 1 volume ratio was treated in an experimental unit of anaerobic digestion operating as a simple periodically filled BATCH-system with mechanical agitation, heating tubes with circulating heated medium at a mesophilic temperature (40°C) and low organic load of the digester (2.2 kg org. dry matter/m<sup>3</sup>) and 28-day fermentation.

Acid hydrolysis of sludge, slurry and their mixture was done before and after anaerobic fermentation. The hydrolysis of samples was performed with the dry matter of examined sludge and its mixture with pig slurry including the liquid fraction after screening the material through a 250-µm mesh sieve. The method of hydrolysis according to Rovira and Vallejo (2000, 2002) as modified by Shirato and Yokozawa (2006): 300 mg of homogenised sample is hydrolysed with 20 ml of 2.5 M  $H_2SO_4$  for 30 min at 105°C in a pyrex tube. The

hydrolysate is centrifuged and decanted, the residues are washed with 25 ml water and the wash water is added to the hydrolysate. This hydrolysate is used to determine Labile Pool I (LP I).

The washed residue is dried at 60°C and hydrolysed with 2 ml of 13 M H<sub>2</sub>SO<sub>4</sub> overnight at room temperature and continuous shaking. Such an amount of water is added that the concentration of the acid will be 1 M, and the sample is hydrolysed for 3 hours at 105°C at intermittent shaking. The hydrolysate is isolated by centrifugation and decantation, the residue is washed again with 25 ml of water and the wash water is added to the hydrolysate. This hydrolysate is used for the determination of Labile Pool II (LP II). The residue from this hydrolysis is dried at 60°C and Recalcitrant Pool (RP) is determined from this fraction.

$C_{tot}$  is determined in all three fractions.

Degradability of organic matter of the test materials was studied by modified methods of Leblanc et al. (2006) used to examine the decomposition of green mulch from *Inga samanensis* and *Inga edulis* leaves. These authors conducted their study in outdoor conditions (average annual temperature 25.1°C) and we had to modify their method in the cold climate of this country. At first, the liquid phase of sludge, slurry and mixture was separated by centrifugation; the solid phase was washed with hot water several times and separated from the solid phase again. By this procedure we tried to separate the solid phase from the liquid one, which contains water-soluble organic substances and mineral nutrients. Solid phases of tested organic materials were mixed with sandy-loamy Cambisol at a 3:1 weight ratio to provide for inoculation with soil microorganisms and volume ventilation of samples with air. After wetting to 50% of water retention capacity the mixtures at an amount of 50 g were put onto flat PE dishes 25 x 25 cm in size. The material was spread across the surface of the dish. Cultivation was run in a wet thermostat at 25°C, and in the period of 2 – 20 weeks dishes were sampled in 14-day intervals as subsamples from each of the four experimental treatments. The agrochemical analysis of the used topsoil proved that the content of available nutrients P, K, Ca and Mg according to MEHLICH III is in the category “high” and  $pK_{KCl} = 6.3$ . After drying at 60°C for 72 hours the content of lipids, crude protein, hemicelluloses, cellulose, lignin, total nitrogen and hot-water-insoluble dry matter was determined in the dish contents.

After twenty weeks of incubation organic substances were determined in the dish contents by fractionation into 4 degrees of lability according to Chan et al. (2001).

The content of hemicelluloses was calculated from a difference between the values of neutral detergent fibre (NDF) and acid detergent fibre (ADF), lignin was calculated from ADF by subtracting the result after lignin oxidation with KMnO<sub>4</sub>. Because ADF contains lignin, cellulose and mineral fraction, it was possible to determine the cellulose content by ashing the residue in a muffle furnace and by determination of mineral fraction. These methods were described by Van Soest (1963), modifications used by Columbian authors (Leblanc et al. 2006) were reported by López et al. (1992).

Ion exchange capacity [mmol.chem.eq./kg] was determined in dry matter of the examined materials according to Gillman (1979), buffering capacity was determined in samples induced into the H<sup>+</sup>-cycle with HCl diluted with water at 1 : 1 and washed with water until the reaction to Cl<sup>-</sup> disappears. In the medium of 0.2 M KCl the samples were titrated to pH = 7 with 0.1 M NaOH and buffering capacity was calculated from its consumption.

Tab. 1 shows the analyses of a mixture of pig slurry and primary sludge used in the experiment. Obviously, compared to the values reported in literature our experimental materials had a somewhat lower content of organic substances in dry matter, and perhaps

this is the reason why anaerobic fermentation reduced the content of organic substances by 39% only although the usual reduction by 45 – 65% for primary sludge was expected as reported in literature (Pitter 1981) and by 40 – 50% for pig slurry (Stehlík 1988). As a result of the organic dry matter reduction the content of nutrients in sludge after anaerobic fermentation is higher, nitrogen content is lower by about 20%. In this process organic nitrogen is converted to  $(\text{NH}_4)_2\text{CO}_3$ , which partly decomposes into  $\text{NH}_3 + \text{H}_2\text{O} + \text{CO}_2$  and partly passes into the sludge liquor. Roschke (2003) reported that up to 70% of total nitrogen might pass to the ammonium form at 54% degradation of organic substances of dry matter. Even though concentrations of the other nutrients in dry matter of the aerobically stabilised sludge increased as a result of the organic dry matter reduction, their content in the sludge liquor also increased (Tab. 2).

		Pig slurry	Primary sludge	Mixture of slurry and sludge before methanisation	Mixture of slurry and sludge after methanisation
Organic substances		65.1 ± 2.6	62.7 ± 2.4	64.1 ± 2.4	36.9 ± 1.5
Total nutrients	N	6.2 ± 0.2	2.6 ± 0.1	3.9 ± 0.2	3.1 ± 0.1
	P	1.6 ± 0.1	0.7 ± 0.0	1.1 ± 0.0	1.3 ± 0.1
	K	2.3 ± 0.1	0.2 ± 0.0	1.2 ± 0.0	1.2 ± 0.0
	Ca	2.8 ± 0.1	2.6 ± 0.1	2.5 ± 0.1	2.8 ± 0.1

Table 1. The analysis of experimental pig slurry and primary sludge, mixture of pig slurry and primary sludge before methanisation in a digester and after methanisation in % of dry matter (pig slurry and primary sludge were mixed for anaerobic digestion at a 1:1 volume ratio). (Sample size n = 6, interval of reliability of the mean for a significance level  $\alpha = 0.05$ )

	[%] A	[%] B	[mg/l] Before fermentation	[mg/l] After fermentation
Total N	8.40	55.20	246.2 ± 14.7	994.7 ± 59.6
Ammonia N	52.60	90.80	153.7 ± 8.4	907.2 ± 48.2
Total P	12.20	25.30	134.5 ± 8.7	176.3 ± 11.6
Total K	19.90	28.10	172.9 ± 10.4	184.1 ± 11.0

Table 2. The analysis of the liquid fraction (sludge liquor) of a mixture of pig slurry and primary sludge from a waste water treatment plant (1 : 1) before fermentation and after fermentation in mg/l. The values A and B express % in the liquid phase of the total amount of sludge before and after fermentation (Sample size n = 5, interval of reliability of the mean for a significance level  $\alpha = 0.05$ )

Taking into account that the amount of water-soluble nutrients in the sludge liquor and organic forms of N and P dispersed in the sludge liquor in the form of colloid sol (but it is a very low amount) is related not only to the composition of the substrate but also to technological conditions of anaerobic digestion, digester load and operating temperature, it is evident that the liquid fraction of anaerobically stabilised sludge contains a certain amount of mineral nutrients, approximately 1 kg N/m<sup>3</sup>, besides the others, although

differences in the concentration of P and K in the liquid fraction before and after fermentation are generally negligible. It is a very low amount, and there arises a question whether the influence of the liquid fraction on vegetation is given by the effect of nutrients or water itself, particularly in drier conditions. After anaerobic digestion the solid phase of sludge still contains a high amount of proteins and other sources of organic nitrogen that could be a potential pool of mineral nitrogen if the degradation of sludge after fermentation in soil is satisfactory.

Material	Proportion		
	LP I	LP II	RP
Primary sewage sludge	68 ± 5	23 ± 2	9 ± 1
Pig slurry	59 ± 5	15 ± 2	26 ± 2
Mixture of primary sludge and pig slurry at a 1:1 volume ratio	63 ± 5	20 ± 2	17 ± 1
Mixture of primary sludge and pig slurry at a 1:1 volume ratio after methanisation	18 ± 2	16 ± 1	66 ± 5

Table 3. Proportions of the three pools of carbon in experimental materials, as determined by the acid hydrolysis method of Rovira and Vallejo (2002), (Sample size n = 4, interval of reliability of the mean for a significance level  $\alpha = 0.05$ ), (Materials including the liquid fraction were used)

The results of hydrolysis in Tab. 3 prove that pig slurry has 59% of its total carbon in LP I, which indicates great lability, corresponding to the hydrolysability of cereals and grasses according to Shirato and Yokozawa (2006). Primary sewage sludge is still better from this aspect, having almost 70% C in LP I. The degree of lability of the sludge and slurry mixture is relatively high and corresponds to the component ratio. After methanisation carbon content in LP I of the sludge and slurry mixture decreases to less than a third of the original amount and carbon of non-hydrolysable matters increases even almost four times in the RP fraction. The sum of LP I and LP II, i.e. the labile, degradable fraction of carbon compounds of the sludge and pig slurry mixture, was reduced by anaerobic digestion from 83% to 34%, that means approximately by 50%. These are enormous differences and they prove that mainly very labile organic substances are heavily destroyed by the anaerobic process even though a reduction in the content of organic substances during anaerobic fermentation is lower (by 39% in our experiment).

Tab. 4 shows the analysis of raw materials (sludge and pig slurry) and their mixture before and after anaerobic fermentation while Tab. 5 shows the analysis of their liquid fraction. The same results (Tab. 4) are provided by the incubation of the solid phase of sludge, pig slurry and their mixture before and after anaerobic fermentation when incubated with soil at 25°C and by the contents of lipids, crude protein, hemicelluloses, cellulose, lignin, total nitrogen and hot-water-insoluble dry matter; the same explicit conclusion can be drawn from the results of the fractionation of organic matter lability of the experimental treatments after 20-week incubation with soil according to Chan et al. (2001) shown in Tab. 5. A comparison of the results in Tab. 3 and 5 indicates that as a result of the activity of microorganisms of the



added soil in incubation hardly hydrolysable organic matter was also degraded – differences between the most stable fractions F 3 and F 4 in Tab. 5 are larger by about 73% after anaerobic fermentation while in the course of acid chemical hydrolysis the content of non-hydrolysable fraction was worsened by anaerobic fermentation because it increased by about 290%. But it is a matter of fact that the soil microorganisms are not able to stimulate the anaerobically fermented sludge to degradation as proved by more than ¾ of total carbon in fraction 4.

	I Before incubation (25° C)				II After incubation (25°C, 20 weeks)			
	A	B	C	D	A	B	C	D
Lipids (petroleum ether extractable compounds) [%]	8.60 ± 0.69	14.27 ± 1.14	10.82 ± 0.86	2.01 ± 0.15	7.97 ± 0.65	13.50 ± 1.09	10.39 ± 0,85	2.08 ± 0,17
Proteins (Berstein) [%]	13.43 ± 1.30	17.95 ± 1.62	15.31 ± 1.60	8.50 ± 0.93	11.81 ± 1.20	16.10 ± 1.53	13.89 ± 1.42	8.50 ± 0.98
Hemicelluloses [%]	1.82 ± 0.19	5.03 ± 0.73	3.32 ± 0.61	0.70 ± 0.60	1.43 ± 0.11	4.23 ± 0.51	2.89 ± 0.30	0.69 ± 0.10
Cellulose [%]	7.45 ± 0.92	11.18 ± 1.33	9.61 ± 1.05	6.03 ± 0.95	5.42 ± 0.82	9.27 ± 0.98	7.96 ± 0.94	6.05 ± 0.83
Lignins [%]	4.84 ± 0.62	5.16 ± 0.84	4.99 ± 0.75	5.18 ± 0.92	4.83 ± 0.91	5.18 ± 1.07	4.98 ± 0.84	5.20 ± 0.91
Total N [%]	1.59 ± 0.06	2.70 ± 0.11	2.29 ± 0.10	1.07 ± 0.04	1.51 ± 0.06	2.50 ± 0.11	2.14 ± 0.09	1.08 ± 0.05
Hot-water insoluble dry matter [%]	98.25 ± 2.94	98.26 ± 2.95	98.25 ± 2.95	98.23 ± 2.92	89.05 ± 2.67	85.17 ± 2.60	87.26 ± 2.58	98.20 ± 2.93
Ion exchange capacity [mmol chem. eq./kg]	48 ± 3	55 ± 3	53 ± 3	145 ± 9	50 ± 3	61 ± 4	55 ± 4	168 ±10
Buffering capacity [mmol chem. eq./kg]	62 ± 4	69 ± 4	65 ± 4	157 ± 9	65 ± 4	72 ± 4	70 ± 4	179 ± 11

Table 4. The content of selected organic substances (%) and ion exchange and buffering capacity of the solid phase of primary sludge (A), pig slurry (B), sludge and pig slurry mixture at a 1:1 ratio before fermentation (C) and after fermentation (D) before and after 20 weeks of incubation with sandy-loamy Cambisol topsoil at a 3:1 ratio at 25°C in dry matter (Sample size n = 4 /hot-water-soluble dry matter n = 7/, interval of reliability of the mean for a significance level  $\alpha = 0.05$ )

	Unfermented primary sludge	Unfermented pig slurry	Mixture A	Mixture B	Soil only
Fraction 1 (12 N H <sub>2</sub> SO <sub>4</sub> )	59.84 ± 7.18 (32.00)	55.38 ± 6.52 (28.40)	54.09 ± 6.50 (30.05)	2.65 ± 0.30 (2.60)	1.30 ± 0.17 (7.22)
Fraction 2 (18 N - 12 N H <sub>2</sub> SO <sub>4</sub> )	42.45 ± 5.13 (22.70)	35.76 ± 4.26 (18.34)	34.22 ± 4.10 (19.01)	9.28 ± 1.10 (9.07)	0.80 ± 0.09 (4.44)
Fraction 3 (24 N - 18 N H <sub>2</sub> SO <sub>4</sub> )	27.34 ± 3.28 (14.62)	20.18 ± 2.53 (10.35)	20.30 ± 2.42 (11.28)	11.13 ± 1.33 (10.91)	3.70 ± 0.44 (20.56)
Fraction 4 (TOC = 24 N H <sub>2</sub> SO <sub>4</sub> )	57.37 ± 6.85 (30.68)	83.67 ± 10.01 (42.91)	71.39 ± 8.55 (39.66)	78.97 ± 9.40 (77.42)	1.22 ± 1.42 (67.78)

Table 5. The fractionation of organic carbon (g/kg) of primary sludge, pig slurry, and sludge and slurry mixture at a 1:1 ratio before fermentation (A) and after fermentation (B) in a mixture with sandy-loamy Cambisol (3 : 1) in dry matter after 20 weeks of incubation at 25°C by the modified Walkley-Black method according to Chan et al. (2001) with a change in H<sub>2</sub>SO<sub>4</sub> concentration. (The values given in brackets are % of the C fraction in total dry matter carbon) (Sample size n = 5, interval of reliability of the mean for a significance level α = 0.05)

The table results document that 20-week incubation decreased more or less the per cent content of examined organic substances except lignin (total N 5 – 8%, cellulose 17 – 25%, hemicellulose 13 – 22%, proteins 9 – 12%, lipids 4 – 7%, and the content of hot-water-insoluble dry matter by 10 – 15%) factually in all experimental treatments except the treatment of the anaerobically fermented mixture of primary sludge and pig slurry where a reduction in these matters is low or nil. Hence, primary sludge, pig slurry and their mixture can be considered as organic fertilisers but only before anaerobic fermentation. We recorded a substantially lower degree of degradation of selected organic substances in sludge, pig slurry and their mixture during incubation with 25% of sandy-loamy soil (5 – 25%) than did Leblanc et al. (2006) with phytomass of *Inga samanensis* and *Inga edulis* leaves, who reported about 50% degradation of total mass, hemicelluloses and nitrogen in mass. We are convinced that it is caused by a very different content of hemicelluloses in our materials compared to the materials used by the above-mentioned authors. No easily degradable hemicelluloses are present in sewage sludge or in pig slurry any longer, and obviously, only more stable forms pass through the digestive tracts of animals and humans. It is also interesting that after anaerobic fermentation and after 20-week aerobic cultivation at 25°C only the compounds (lipids + proteins + hemicelluloses in mixture II D account roughly for 11%) that could be considered as labile remained in the mixture of slurry and sludge. These are apparently their more stable forms as confirmed by the results in Tab. 5 which illustrate that to approximately 11% of organic carbon compounds it is necessary to add the % proportions of the first and second fraction on the basis of oxidisability according to Chan et al. (2001). Literary sources report that the sum of lipids, proteins and hemicelluloses in the

anaerobically stabilised sludge from municipal waste water treatment plants amounts to 13% – 39.6% of dry matter, so it is quite a general phenomenon.

The ion exchange capacity of sludge, pig slurry and their mixture before fermentation, before incubation and after incubation is very low and does not reach the values that are typical of sandy soil. It is increased by anaerobic fermentation along with incubation markedly but practically little significantly to the level typical of medium-textured soils. The same relations were observed for buffering capacity, which is not surprising. The results document that degradability of the organic part of anaerobically stabilised sludge worsened substantially and that it cannot be improved very markedly by the use of soil microorganisms and soil.

We have to draw a surprising conclusion that sludge as a waste from the processes of anaerobic digestion is a mineral rather than organic fertiliser and that from the aspect of its use as organic fertiliser it is a material of much lower quality than the original materials. We cannot speak about any improvement of the organic material by anaerobic digestion at all. Their liquid phase, rather than the solid one, can be considered as a fertiliser. If it is taken as a fertiliser in general terms, we do not protest because besides the slightly higher content of mineral nutrients available to plants (mostly nitrogen) it has the higher ion exchange capacity and higher buffering capacity than the material before anaerobic fermentation, but this increase is practically little significant.

### **3.1.2 Digestate composting**

#### **3.1.2.1 What is compost?**

Similarly like in the evaluation of digestate when the daily practice has simplified the problem very much because the main functions of mineral and organic fertilisers are not distinguished from each other, the simplification of the problem of composting and application of composts has also led to an absurd situation. In many countries the compost is understood to be a more or less decomposed organic material, mostly from biodegradable waste, which contains a certain small amount of mineral nutrients and water. The main requirement, mostly defined by a standard, is prescribed nutrient content, minimum amount of dry matter, absence of hazardous elements and the fact that the particles of original organic material are so decomposed that the origin of such material cannot be identified. Such 'pseudo' composts are often offered to farmers at a very low cost because the costs of their production are usually paid by producers of biodegradable waste who want to dispose of difficult waste.

The producers of such composts often wonder why farmers do not intend to buy these composts in spite of the relatively low cost. It is so because the yield effect of fertilisation with these composts is minimal, due to a low content of nutrients it is necessary to apply tens of tons per 1 ha (10 000 m<sup>2</sup>), which increases transportation and handling costs. In comparison with so called "green manure", i.e. ploughing down green fresh matter of clover, lucerne, stubble catch crops and crops designed for green manure, e.g. mustard, some rape varieties, etc., the fertilisation with these false composts does not have any advantage. The highly efficient decomposing activity of soil microorganisms, supported by equalising the C : N ratio to the value 15 – 25 : 1, works in the soil similarly like the composting process in a compost pile where the disposal of biodegradable material is preferred at the cost of a benefit to farmers.

What should the real compost be like? It is evident from the definition: the compost is a decomposed, partly humified organomineral material in which a part of its organic

component is stabilised by the mineral colloid fraction. It is characterised by high ion-exchange capacity, high buffering capacity and is resistant to fast mineralisation. The reader of this text has surely noticed that the nutrients have not been mentioned here at all. Of course, they are present in the compost, their amount may be higher or a lower, but it is not important. It is crucial that the compost will maintain nutrients in the soil by its ion-exchange reactions and that it will protect them against elution from topsoil and subsoil layers to bottom soil or even to groundwater, no matter whether these plant nutrients originate from the compost itself or from mineral fertilisers or from a natural source – the soil-forming substrate in the soil-forming process. In the production of such “genuine” compost it is necessary to ensure that organic matter of the original composted mixture will be transformed not only by decomposing mineralisation, exothermic oxidation processes but also partly by an endothermic humification process that is not a decomposing one, but on the contrary, it is a synthetic process producing high-molecular, polycondensed and polymeric compounds, humic acids, fulvic acids and humins, i.e. the components of soil humus. It is to note that we should not confound the terms “humus” and “primary soil organic matter”; these are completely different mixtures of compounds, of quite different properties! Humus is characterised by high ion-exchange capacity and very slow mineralisation (the half-time of mineralisation of humic acids in soil conditions is 3 000 – 6 000 years!) while primary organic matter, though completely decomposed but not humified, has just opposite properties. Sometimes it may have a high sorption capacity but not an ion-exchange capacity.

The high ion-exchange capacity of humified organic matter is a cause of other two very important phenomena: huge surface forces of humus colloids in soil lead to a reaction with similarly active mineral colloids, which are all mineral soil particles of silicate nature that are smaller than 0.001 mm in size. These particles are called “physical clay” in pedology. The smaller the particles, the larger their specific surface, which implies their higher surface activity. Clay-humus aggregates are formed, which are adsorption complexes, elementary units of well-aerated, mechanically stable and elastic soil microaggregates that may further aggregate to macroaggregates and to form the structured well-aerated soil that has a sufficient amount of capillary, semi-capillary and non-capillary pores and so it handles precipitation water very well: in drought capillary pores draw water upward from the bottom soil while in a rainy period non-capillary pores conduct water in an opposite direction. The basic requirement for soil productivity is met in this way. It is often much more important than the concentration of nutrients in the soil solution (and hence in the soil).

The other important phenomenon related to ion-exchange properties of compost or soil is buffering capacity, the capacity of resisting to a change in pH. Soils generally undergo acidification, not only through acid rains as orthodox ecologists often frighten us but also mainly by electrolytic dissociation of physiologically acid fertilisers and intensive uptake of nutrients from the soil solution by plants. By the uptake of nutrient cations plants balance electroneutrality by the  $H^+$  ion, which is produced by water dissociation, so that the total electric charge does not change. If it were not so, each plant would be electrically charged like an electrical capacitor. The humus or clay or clay-humus ion exchanger in compost or in soil, similarly like any other ion exchanger, behaves in the same way as the plant during nutrient uptake: when any ion is in excess in the environment, e.g.  $H^+$  in an acidifying soil, the plant binds this  $H^+$  and exchanges it for another cation that was bound by it before. The

H<sup>+</sup> ion is blocked in this way and the pH of soil does not change. High buffering capacity is a very favourable soil property and is typical of soils with a high content of mineral or organic colloid fraction, i.e. of heavy-textured soils and of organic soils with a high degree of humification of soil organic matter.

As described above, it is quite obvious what soils should be fertilised with real genuine composts preferentially: these are mainly light sandy and sandy-loam soils in which mineralisation processes are so fast due to high aeration that the organic matter of potentially applied organic fertilisers factually “burns”. Mineral nutrients are released from an organic fertiliser but very soon there is a lack of necessary organic matter in such a soil. Energy for the soil microedaphon is not sufficient, ion-exchange capacity is low because decomposed organic matter fails to undergo humification. Such a soil does not hold water while rainfall quickly leaches nutrients from the soil. Only the application of genuine composts can markedly improve the productivity of these soils. Their clay-stabilised organic matter resists the attack of oxygen excess and remains decomposable, so it is able to maintain the required microbial activity of soil.

### 3.1.2.2 How is “genuine” compost produced?

Modern production of industrial composts is based on an idea that the compost is a substrate for plants with nutrient content. This is the reason why attention is mainly paid to the mechanical treatment of organic material – grinding, crushing and homogenisation. A homogenised blend, enriched with nutrients, applied water and/or compost additives, is subjected to fast fermentation. It is turned at the same time and homogenised again. The turning ensures a new supply of oxygen and if the compost has a sufficient amount of easily degradable organic matter, the temperature during composting increases up to 50 – 60°C, which allows a desirable breakdown of particles of the original organic material. The product acquires a dark colour, it is loose, often has a pleasant earthy smell while the odour of the original organic material is not perceptible any more. Farm sludge is often added to the compost formula as a nitrogen source or the improper C to N ratio is adjusted by the addition of mineral nitrogenous fertilisers. Slurry and liquid manure are used as an N and water source and sometimes limestone is added to prevent acidification. The aeration of the fermented pile of materials is provided by the addition of inert coarse-grained materials, mainly of wood chips, crushed straw, rubble, undecomposable organic waste and other materials available from local sources, whereas the use of horizontal and vertical ventilation systems is less frequent. It is often the type of “aeration” additive which explicitly shows that the compost producer prefers waste processing to the interest of future users of their products, farmers and productivity of their soils. The ion-exchange capacity of these composts is about 40 – 80 mmol chem. eq. 1000 g<sup>-1</sup> and it is very low. It characterises a light, little fertile sandy soil.

How is the real “genuine” compost produced? The following principles should be observed:

1. Organic material of the compost formula should have a high degree of lability. If the compost producer does not have a sufficient amount of such very easily degradable organic material, its lability should be enhanced by saccharidic waste.
2. The C : N ratio should be adjusted to the value 10 – 15 : 1, not to total C and total N, but to the value of C<sub>hws</sub> and N<sub>hws</sub> (hot water extractable carbon and nitrogen). Obviously, it is not worth adding to the compost a nitrogen source e.g. in waste polyamide because this nitrogen is not accessible. It is a flagrant example but we have detected many times that the C : N ratios are completely different from those the compost producers suppose them to be.



3. The compost formula should have a high proportion of buffering agent. It should always be ground limestone or dolomite, it should never be burnt or slaked lime. Do not economize on this additive very much. It will be utilised excellently after the application of this compost to soils.
4. Stabilisation of organic matter should be ensured by a sufficient amount of the clay mineral fraction. It must not be applied in lumps, but in the form of clay slurry, clay water suspension, used also for the watering of the blend of compost materials. Concrete mixers are ideal equipment for the preparation of clay slurry.
5. The compost blend should be inoculated by healthy fertile topsoil. Soil microorganisms are adapted in a different way than the microorganisms of the intestinal tract of animals. Therefore slurry and liquid manure are sources of water and nitrogen but they are not a suitable inoculant even though they are often recommended in literature for this purpose.
6. The basic requirement is to reach a high temperature (55 – 60°C) during composting and to maintain the second phase of temperature (40 – 50°C) for a sufficiently long time. This process will be successful only at a sufficiently high amount of highly labile organic matter in the compost formula, at a correct C : N ratio, at a correct water to air ratio in the pile (the moisture during fermentation should be maintained in the range of 50 – 60% of water-retention capacity) and at a reduction in heat losses. Heat losses of the compost into the atmosphere through the pile surface are relatively small. The highest quantity of heat is lost by conducting the heat through the concrete or the frozen ground of the compost pile, and mainly by an aerating system if it is installed.
7. Humification processes, formation of humus acids and humins or their precursors at least, occur rather in later stages of fermentation and so we should accept that the good compost cannot be produced by short-term fermentation. Old gardeners fermented composts for 10 – 12 years, but their composts reached the ion-exchange capacity of 300 – 400 mmol chem. eq. 1000 g<sup>-1</sup>.

### 3.1.2.3 How is the digestate used in compost production?

If besides decomposing exothermic processes synthetic endothermic processes are also to take place in compost when high-molecular humus substances (fulvic acids, humic acids and humins) are formed, these conditions must be fulfilled: very favourable conditions for the microflora development must exist in compost, and minimum losses and the highest production of heat must be ensured. For this purpose it is necessary to use a high admixture of buffering additive (limestone) in the compost formula, sufficient amount of very labile organic matter, thermal insulation of the base of fermented material because the heat transfer coefficient does not have the highest value for transfer from the composted pile into the atmosphere but mainly into solid especially moist materials, i.e. into concrete, moist or frozen earth, clay, bricks, etc. At a sufficient amount of labile fractions of organic matter the maximum heat production can be achieved only by a sufficient supply of air oxygen. Beware of this! The ventilation through vertical and horizontal pipes provides sufficient air for aerobic processes in the fermented material but at the same time the ventilation is so efficient that a considerable portion of reaction heat is removed, the material is cooled down and the onset of synthetic reactions with the formation of humus substances does not occur at all.

When sufficiently frequently turning the fermented material, the safest method of compost aeration and ventilation is the addition of coarse-grained material while inert material such as wood chips, chaff and similar materials can be used. It is however problematic because

inert material in the fermented blend naturally decreases the concentration of the labile fraction of organic matter, which slows down the reaction rate of aerobic biochemical reactions and also the depth of fermentation is reduced in this way. It mainly has an impact on the synthetic part of reactions and on the formation of humus substances while the influence on decomposing reactions is smaller.

It would be ideal if during compost fermentation in a microbially highly active environment the inert aeration material were able not only to allow the access of air oxygen into the fermented material but also to decompose itself at least partly and to provide additional energy to biochemical processes in the pile in this way.

These requirements are excellently met by the solid fraction of digestate from biogas plants. It aerates the compost and although it lost labile fractions of organic matter in biogas plant digesters, it is capable of further decomposition in a microbially active environment. It releases not only energy but also other mineral nutrients. So this waste is perfectly utilised in this way. The average microbial activity of even very fertile, microbially active soils is not efficient enough for the decomposition of this stable organic material when the solid phase of digestate is used as an organic fertiliser. The decomposition rate is slow, especially in subsequent years, and therefore the resultant effect of the solid fraction of digestate as an organic fertiliser is hardly noticeable. The combination of anaerobic decomposition in the biogas plant digester and aerobic decomposition in compost could seem paradoxical, and some agrochemists do think so. The preceding exposition has shown that it is not nonsense.

Now let us answer the question: what dose of the solid fraction of digestate should be used in the compost formula? It depends on many factors: on the amount of the labile fraction of organic component and mainly on the degree of its lability (which can be determined in a reliable way by the above-mentioned method according to Rovira and Vallejo 2002, 2007, Shirato and Yokozawa 2006), on the aeration and porosity of materials used in the compost formula, on the number of turnings, on prevailing outdoor temperature, water content, degree of homogenisation and on other technological parameters.

In general: the higher the amount of the labile component of organic matter and the higher its lability (e.g. the content of saccharides and other easily degradable substances), the higher the portion of the solid fraction of digestate that can be used.

Now short evidence from authors own research is presented:

The basic compost blend was composed of 65% fresh clover-grass matter from mechanically mown lawns, 10% ground dolomite, 2% clay in the form of clay suspension, 20% solid phase of digestate (obtained by centrifugation with fugate separation) or 20% crushed wood chips and 3% PK fertilisers. The C : N ratio in the form of  $C_{hws} : N_{hws}$  (hot-water-soluble forms) was 15 : 1, nitrogen was applied in  $NH_4NO_3$  in sprinkling water that was used at the beginning of fermentation at an amount of 70% of the beforehand determined water-retention capacity of the bulk compost blend. Inoculation was done by a suspension of healthy topsoil in sprinkling water. Fermentation was run in a composter in the months of April – November, and the perfectly homogenised material was turned six times in total. Water loss was checked once a fortnight and water was replenished according to the increasing water-retention capacity to 60%. The formation, amount and quality of formed humus substances were determined not only by their isolation and measurement but also by their specific manifestation, which is the ion-exchange capacity of the material. The original particles of composted materials were not noticeable in either compost (with the solid part of digestate and with wood chips), in both cases the dark coloured loose material with pleasant earthy smell was produced. Tab. 6 shows the analyses of composted materials and

composts. The digestate was from a biogas plant where a mix of cattle slurry, maize silage and grass haylage is processed as a substrate. The material in which the aeration additive was polystyrene beads was used as compost for comparison.

	Solid phase of digestate	Wood chips	Compost		
			PS	Wood chips	Solid phase of digestate
C <sub>FA</sub> [mg.kg <sup>-1</sup> ]	0	0	38	84	178
C <sub>HA</sub> [mg.kg <sup>-1</sup> ]	0	0	15	20	62
C <sub>HA</sub> : C <sub>FA</sub>	-	-	0,39	0,24	0,35
Ion-exchange capacity T [mmol chem. eq.kg <sup>-1</sup> ]	51	12	72	64	224

Table 6. The content of fulvic acid carbon (C<sub>FA</sub>), humic acid carbon (C<sub>HA</sub>), their ratio and ion-exchange capacity T of the solid phase of digestate and wood chips at the beginning of fermentation and of composts with polystyrene (PS), wood chips and solid phase of digestate

The results document that the ion-exchange capacity, and hence the capacity of retaining nutrients in soil and protecting them from elution after the application of such compost, increased very significantly only in the digestate-containing compost. The ion-exchange capacity of this compost corresponds to the ion-exchange capacity of heavier-textured humus soil, of very good quality from the aspect of soil sorption. The compost with wood chips produced in the same way does not practically differ from the compost with polystyrene but it does not have any humic acids and the ion-exchange capacity of these composts is on the level of light sandy soil with minimum sorption and ion-exchange properties. However, the total content of humus acids in the compost with the solid phase of digestate is very small and does not correspond to the reached value of the ion-exchange capacity of this compost. Obviously, precursors of humus acids that were formed during the fermentation of this compost already participate in the ion exchange. Humus acids would probably be formed from them in a subsequent longer time period of their microbial transformation. If only humus acids were present in composting products, at the detected low concentration of C<sub>FA</sub> + C<sub>HA</sub> the T value of the compost with the solid phase of digestate would be higher only by 1 – 1.2 mmol.kg<sup>-1</sup> than in the compost with polystyrene or wood chips. Because it is more than a triple, other substances obviously participate in the ion exchange.

3.1.3 Use of digestate for improvement of heavy-textured soils

Optimum values of reduced bulk density O<sub>r</sub> for soils are around 1.2 g.cm<sup>-3</sup>, but more important is the minimum value of bulk density for the restriction of root growth which is about 1.7 – 1.8 g.cm<sup>-3</sup> for light soils and only 1.40 – 1.45 g.cm<sup>-3</sup> for heavy-textured clay soils. Bulk density O<sub>r</sub> is a crucial parameter for the assessment of the soil compaction rate as an important negative factor of soil productivity. Bulk density of topsoil in the range of 0.95 – 1.15 g.cm<sup>-3</sup> shows loose topsoil while the value > 1.25 g.cm<sup>-3</sup> indicates heavily compacted topsoil.

Another important value of soil is soil aeration  $V_z$ . It is expressed in volume % as the difference between porosity  $P_o$  and momentous soil moisture  $W_{obj}$ .

$$V_z = P_o - W_{obj}. \quad (16)$$

Optimum aeration e.g. for grasslands is 10% by volume, for soils for barley growing it is already as much as 24% by volume. Soil porosity  $P_o$  is the sum of all pores in volume per cent, in topsoils it is around 55%, in subsoil it decreases to 45 – 35%. Sandy soils have on average  $P = 42\%$  by vol., out of this 30% are large pores and 5% are fine pores while heavy-textured clay soils have the average porosity of 48% by vol., out of this only 8% are large pores and 30% are fine pores. Fine pores are capillary and large pores are non-capillary ones. Cereals should be grown in soils with 60 – 70% of capillary pores out of total porosity and 30 – 40% of non-capillary pores. Forage crops and vegetables require the soils with 75 – 85% of capillary pores and only 15 – 25% of non-capillary pores out of total porosity.

Ploughing resistance  $P$  is also significant. It is a specific resistance that must be overcome during cutting into and turning over the soil layer. It is expressed by the drawbar pull measured dynamometrically on the coupling hook of a tractor. It is related to the texture and moisture of soil, to its content of organic substances and ploughing depth. Ploughing resistance for light soils is 2 – 4 t.m<sup>-2</sup>, for heavy-textured soils it is 6 – 8 t.m<sup>-2</sup>. The units kp.dm<sup>-2</sup> are also used. For sandy soils the ploughing resistance of 25 – 28 kp.dm<sup>-2</sup> is usual, for clay soils it is 70 kp.dm<sup>-2</sup>.

Hence heavy-textured soils are more responsive to the higher reduced bulk density of soil when roots develop poorly, they need more non-capillary pores to allow for the better infiltration of precipitation water, they also need higher aeration because they are mostly too moist and many aerobic processes including the microbial activity take place with difficulty. Of course, the high ploughing resistance is not desirable either for the economics of soil cultivation or for the production process of any crop. Therefore it is necessary to improve heavy-textured soils and the question is how. Organic fertilisers are not sufficient; peat was used previously but now it is banned to use it for the reason of the peat bog conservation, and synthetic soil amendments (Kriliun, Flotal etc.) are currently too costly for the agriculture sector. An excellent material for the improvement of heavy-textured soils is the solid phase of digestate if ploughed down at higher doses than those used for the application of farmyard manure or compost, i.e. 100 – 150 t.ha<sup>-1</sup>. Even though we cannot expect any great release of mineral nutrients from organic matter of the solid phase of digestate due to high stability of this material, the improvement and aeration of heavy-textured soil with better conditions for the microbial activity of soil and undisturbed root growth often bring about a higher yield effect than is the yield effect of nutrients from high-quality organic fertilisers as shown by the results of this field trial:

When we still believed that the solid phase of digestate was an organic fertiliser, we laid out an exact field trial on a heavier-textured, loamy-clay soil with medium to good reserve of available nutrients. The trial had two treatments: the one treatment was fertilisation with the solid phase of digestate only (after fugate centrifugation) and the other treatment was the application of only mineral fertilisers in the form of pure salts at such a dose that the level of these easily available nutrients to plants was the same as the amount of unavailable or little available nutrients in the treatment fertilised with digestate. We wanted to find out from the yield of the grown crop what amount of mineral nutrients would be released from the digestate in comparison with completely available nutrients in the first year and in subsequent years of the crop rotation: early potatoes – winter barley – red clover – oats. We



intended to compare the digestate with other organic fertilisers, e.g. farmyard manure which in the first year mineralises about a half of its nutrients bound in organic matter. But the result we obtained was surprising: in the first year the yield of early potatoes was higher by 12% in the digestate treatment although nobody could doubt that this treatment had a lower amount of nutrients than the variant fertilised with pure salts. The only explanation is that the higher yield effect in the digestate treatment was not caused by the higher input of nutrients but by the improvement in physical properties of heavy-textured soil that surely occurred as seen in Tab. 7. The favourable effect of the heavy-textured soil improvement on yield was positively reflected in subsequent years also in other crops of the crop rotation that were fertilised in both treatments in the same way, i.e. mineral fertilisers were applied. We drew a conclusion that in practice the yield effect is often ascribed to digestate nutrients although it is caused by better soil aeration and better root growth due to soil loosening after the application of digestate.

	Clay-loamy soils	
	initial	improved by digestate
Reduced bulk density $O_r$ [g.cm <sup>-3</sup> ]	1.43	1.38
Soil aeration $V_z$ [% by vol.]	18.5	22.4
Total porosity $P_o$ [% by vol.]	43.9	43.8
Proportion of large pores in total porosity [%]	22.7	28.1
Ploughing resistance $P$ [kp.dm <sup>-2</sup> ]	63	50

Table 7. Bulk density  $O_r$ , aeration  $V_z$ , total porosity  $P_o$ , proportion of large pores in total porosity and ploughing resistance  $P$  in a heavy-textured clay-loamy soil and after its improvement with the dose of 150 t-ha<sup>-1</sup> of the digestate solid phase

**3.2 Perspective utilisation of digestate with a modification of conventional technology of biogas production**

Perspective utilisation of digestate is connected with envisaged modifications of the technology of biogas production in agricultural biogas plants. These plants have digesters for the solid phase only or the most frequent are liquid (suspension) digesters. These are digesters without partition wall where the biomass of microorganisms is carried by the processed substrate. In reactor systems for the technological processing of waste from chemical and food technologies and from the technology of municipal and industrial waste water treatment those digesters are preferred where the biomass of functional microorganisms is fixed onto a solid carrier or onto partition walls of apparatuses. It is often granulated and is maintained in the digester as a suspended sludge cloud. These reactors may be affected by short-circuiting and therefore they are sensitive to the particle size of the processed substrate but they withstand a much higher organic load than the digesters without partition wall. Of course, the reactor is smaller, cheaper and more efficient. Hence a perspective modification of the biogas production technology in agricultural biogas plants is gradual transition to the procedures of anaerobic digestion that are currently used in industrial plants for the treatment of organic waste water. The promising utilisation of digestate from such digesters is mainly the manufacture of solid fuels in the form of pellets



that are prepared from the solid phase of agricultural waste before the proper aerobic digestion of the material for a biogas plant. The first proposal of this type is the IFBB procedure, the principle of which was explained in Chapter 1.4. The liquid phase from the preparation of processed material, which is destined for anaerobic fermentation in digesters with partition wall, could be used as a liquid or suspension fertiliser but researchers would have to solve the cheap method of nutrient concentration in this waste. The current price of Diesel fuel, machinery and human labour and low purchase prices of agricultural products do not allow the application of highly diluted fertilisers and in fact handling of water.

The problem is that a small biogas plant is only scarcely profitable. Hence economic reasons favour large-capacity plants with the volume of digesters 5 000 – 10 000 m<sup>3</sup>. In such large plants the reactors with partition wall would be unjustifiably expensive and therefore in these large-capacity facilities for biogas production it is necessary to use reactors without partition wall. The utilisation of their digestate should be based on this scheme: separation of digestate – concentration of fugate and its utilisation as a liquid mineral nitrogenous fertiliser. The solid phase of digestate should be used as an inert aeration component in compost production and as a material for the improvement and aeration of heavy-textured soils.

In any case, researchers must resolve a cheap method of nutrient concentration in fugate.

A number of different reactors are available for small to medium-sized biogas plants with the treatment of material according to IFBB that were developed on a research basis mainly in the sixties to the nineties of the twentieth century. At first, these were reactors with suspension biomass, e.g. mixing contact anaerobic reactor (ACR – AG), its innovation was a membrane anaerobic reactor system (MARS) and sequencing batch reactors (SBR). Then reactors with immobilised biomass were developed that are divided into reactors with biomass on the surface of inert material and reactors with aggregated (granulated) biomass. The former group is divided into upflow reactors and downflow reactors. Reactors with a mobile filling are the third variant.

The latter group is divided into reactors with the internal separator of biogas and biomass, reactors with the external separator of biomass and reactors with partitions.

Further development brought about biofilm reactors where the biomass of microorganisms is fixed onto a solid carrier. These reactors are considered as facilities with the highest operating stability, very resistant to the fluctuation of operating conditions. But they do not usually allow for such a high load as reactors with suspension biomass. The oldest reactor of this series was an upflow anaerobic filter (UAF) reactor from 1967, then a downflow stationary fixed film reactor (DSFF) and downflow reactor with filling in bulk followed. Great progress was made by designing an anaerobic rotating biological contactor (ARBC) and fluidized bed reactor (FBR) in the eighties of the last century. A similar type of reactor, expanded bed reactor (EBR), also designated by AAFEB (anaerobic attached film expanded bed), is suitable to be operated at low temperatures. The detention time is only several hours and the portion of residual organic impurities is practically the same as in modern aerobic systems for the treatment of organically contaminated waters.

Further advance was the development of reactors with aggregated biomass. The most important representative of this group of digesters is an upflow anaerobic sludge blanket (UASB) reactor. It is a reactor with sludge bed and internal separator of microorganism biomass. The biggest reactor of this type (5 000 m<sup>3</sup>) processes waste water from the manufacture of starch in the Netherlands, it withstands the load of 12.7 kg chemical oxygen demand (COD) per 1 m<sup>3</sup>/day, 74% of organic matter is degraded and the detention time is

33 hours only. Besides the UASB reactor these reactors belong to this group: hybrid upflow bed filter (UBF) reactor, anaerobic baffled reactor (ABR), expanded granular sludge bed (EGSB) reactor, internal circulation (IC) reactor and upflow staged sludge bed reactor (USBB), often also called biogas tower reactor (BTR), and other design models of the UASB reactor.

At the end of this chapter it is to note that modern anaerobic reactors have almost amazing outputs – unfortunately, the more perfect the reactor, the more expensive, and also their advantage over huge digesters without partition wall we have got accustomed to in biogas plants is gradually disappearing. The selection of modern anaerobic reactors is also more difficult than the selection of conventional technology of reactors without partition wall, because they are mostly rather specific to the substrate to be processed. They also have higher demands on processing, attendance and checks.

The perspective possibility of using modern anaerobic reactors for biogas production in smaller plants and the simultaneous solution to the use of the digestate solid phase as a raw material for the production of solid pelleted biofuels initiated our study of the IFBB procedure (Chap. 1.4.) for the substrate commonly used in biogas plants in the CR. The results of our experimental work are presented below:

The IFBB technological procedure is based on a high degree of cell wall maceration as a result of the axial pressure and abrasion induced with a screw press. Reulein et al. (2007) used this procedure for dehydration of various field crops; it is also known from the technologies of processing rapeseed, sugar beet and leguminous crops for the production of protein concentrates (Telek and Graham 1983, Rass 2001) and in biorefineries for the extraction of lactic acid and amino acids (Mandl et al. 2006).

The basic substrate contained 37.5% by weight of cattle slurry and 62.5% by weight of solid substrates, i.e. a mixture of chopped maize silage and grass haylage of particle size max. 40 mm mixed at a 4.75 : 1 ratio, i.e. 51.6% of silage and 10% of haylage. In total, the substrate accounted for 19.3% of dry matter. This substrate at 15°C is designated by A. A portion of this substrate was mixed with water at a weight ratio of 1 : 5, put into a thermostat with a propeller stirrer at 15°C and intensively stirred for 15 minutes. Analogically, the other portion was also mixed with water at a substrate to water ratio of 1: 5 and put into a thermostat at a temperature of 60°C with 15-minute intensive stirring again. The sample of the substrate with water 15°C was designated by B, the sample with water 60°C was designated by C. The liquid phase from substrate A was separated by centrifugation while the liquid phases from substrate B and C were separated in a laboratory screw press for the pressing of fruits and vegetables. The separated liquid phases of substrates A, B and C were diluted with water to obtain a unit volume and the analytical results were recalculated to a transfer ratio in the liquid phase in relation to the content of particular nutrients in dry matter of the original substrate mixture.

The experiments conducted in an experimental unit of anaerobic digestion and in an equipment for IFBB made it possible to determine the content of mineral nutrients in substrate A after 42-day anaerobic digestion in mesophilic conditions (40°C), in the liquid phase of substrate A after anaerobic digestion, in the liquid phase of substrate B and C after recalculation to the dry matter content and concentration corresponding to substrate A, also after the process of anaerobic digestion under the same conditions (42 days, 40°C).

The above recalculations enable to clearly show the advantages of the IFBB process in nutrient transfer from solid to liquid phase when substrate A and 5 times diluted substrates B and C are compared, but they may unfortunately evoke a distorted idea about the real

concentration of nutrients in liquid phases. It is to recall that IFBB increases the mass flow and transfer to the liquid phase but with regard to the 5-fold dilution the nutrient concentration in liquid waste for fertilization continues to decrease. This is the reason why the table below shows the original, not recalculated concentrations in the fugate of fermented substrate A and in the fermented liquid phases of the same substrate in IFBB conditions designated by B and C, which document considerable dilution of these potential mineral fertilizers.

The solid phases of substrates A, B and C after anaerobic digestion were subjected to determination of organic matter hydrolysability in sulphur acid solutions according to Rovira and Vallejo (2000, 2002) as modified by Shirata and Yokozawa (2006); we already used this method to evaluate the degradability of a substrate composed of pig slurry and sludge from a municipal waste water treatment plant (Kolář et al. 2008).

	Cattle slurry	Maize silage	Grass haylage	Substrate	Transfer ratio to liquid phase		
					A	B	C
Dry matter	6.4	28.9	18.7	19.3	0.06 ± 0.01	0.18 ± 0.04	0.20 ± 0.03
N-compounds (N x 6.25)	25.6	11.5	7.4	16.3	0.05 ± 0.01	0.20 ± 0.04	0.26 ± 0.05
Digestible nitrogen compounds	-	6.2	3.8	7.3	-	-	-
Nitrogen-free extract	-	52.8	48.6	49.9	0.30 ± 0.03	0.45 ± 0.05	0.48 ± 0.05
Crude fibre	-	25.7	29.8	18.0	0.01 ± 0.00	0.10 ± 0.00	0.10 ± 0.00
Fat	-	4.8	1.5	2.8	-	-	-
Organic substances	76.4	94.8	87.3	87.0	-	-	-
Mineral N (N - NH <sub>4</sub> <sup>+</sup> , NO <sub>3</sub> <sup>-</sup> )	2.4	< 0.1	> 0.1	1.0	0.74 ± 0.05	0.89 ± 0.06	0.95 ± 0.06
P	1.3	0.2	0.3	0.6	0.40 ± 0.05	0.52 ± 0.07	0.65 ± 0.08
K	5.3	1.4	1.7	2.9	0.57 ± 0.04	0.60 ± 0.04	0.79 ± 0.05
Ca	1.3	0.4	0.6	0.8	0.31 ± 0.06	0.38 ± 0.08	0.46 ± 0.08
Mg	0.5	0.2	0.3	0.3	0.38 ± 0.07	0.43 ± 0.08	0.55 ± 0.07
Na	0.1	< 0.1	<< 0.1	< 0.1	0.70 ± 0.08	0.77 ± 0.04	0.80 ± 0.08
Cl	0.3	0.2	0.2	0.2	0.77 ± 0.06	0.85 ± 0.05	0.85 ± 0.06

Table 8. Dry matter content in the fresh mass of used materials and their chemical composition in % dry matter. The transfer ratio of mass flow to the liquid phase from the fresh mass of substrate not diluted with water at 15°C (A), diluted with water at a 1:5 ratio at 15°C (B) and diluted with water at a 1:5 ratio at 60°C (C). Liquid phase A was separated by centrifugation, liquid phases B and C with a screw press.  
(Sample size n = 5, reliability interval of the mean for a significance level α = 0.05)

Table 8 documents that the IFBB procedure proposed by German authors for grass haylage is applicable to the typical substrate of Czech biogas plants, to a mixture of cattle slurry, maize silage and grass haylage. In agreement with German experience the observed transfer ratios are markedly higher at 60°C compared to 15°C of hydrothermal conditions but the value of transfer ratios to the liquid phase is generally lower in our experiments. We ascribe this fact to the properties of the material and also to the achieved axial force of the used press that was apparently lower even though the same perforation size of the conical part of the press (1.5 mm) and slope of the body (1 : 7.5) were used.

The results in Table 8 illustrate that the separation of the liquid and solid phase of the substrate that has not been subjected to anaerobic digestion yet by means of centrifugation only is rather imperfect from the aspect of the mass flow of components. The IFBB system (water dilution, intensive stirring at a temperature of 60°C and subsequent separation of the liquid and solid phase with a screw press) increases the transfer of organic and mineral substances into the liquid phase by about 15 – 20%, and it is also true of the saccharidic nitrogen-free extract and organic nitrogen compounds. This fact documents that the liquid phase has a higher amount of active, well-degradable organic material for anaerobic digestion, and so it is possible to expect not only the higher production of biogas but also more mineral nitrogen in the liquid after anaerobic digestion.

The high mass flow of alkaline metals and chlorine into the liquid phase, and on the contrary, the low transfer of calcium confirm the opinion of German researchers (Wachendorf et al. 2007, 2009) that the IFBB procedure largely increases the quality of biomass solid phase as a material for the production of solid fuels: the production of polychlorinated dioxins and dibenzofurans is reduced, waste gases will be less corrosive and the temperature of ash fusion will be higher.

Nitrogen compounds of the substrate dry matter account for 16.3%, i.e. these nitrogen organic compounds contain 2.6% of nitrogen in dry matter (Table 8). The content of mineral nitrogen in the substrate before anaerobic digestion was 1%. If the digestate contains 2.26% of mineral nitrogen in the same dry matter after anaerobic digestion, it is to state that during anaerobic digestion about a half of organic nitrogen mineralized and enriched the original 1% content of the substrate before the fermentation process. But the dry matter content decreased in the course of fermentation, and therefore the concentration of all nutrients in digestate apparently increased contrary to the original substrate. In our experiment the concentration of substrate dry matter decreased from 19.3% to 13.3% by weight during anaerobic digestion. The content of mineral nitrogen amounting to 3.28% at this dry matter content corresponds to 2.26% of min. N at the original dry matter content of 19.3% by weight (Table 9). The contents of the nutrients P, K, Ca and Mg in digestate dry matter after anaerobic digestion (Table 9) are apparently substantially higher than before fermentation. However, anaerobic digestion did not actually bring about any increase in the content of these nutrients, and the increased concentrations completely correspond to a reduction in dry matter content,  $19.3 : 13.3 = 1.45$ .

It is not a new fact, but Table 9 shows how this mineral nitrogen is transferred to the liquid phase of substrates B and C compared to substrate A. Obviously, the liquid phase of substrate B has a higher amount of mineral nitrogen than that of substrate A, and so the effects of the screw press, which already before anaerobic digestion enriched the liquid substrate B with colloidal solutions (sols) of nitrogen organic compounds from the crushed cell walls of the plant material that provided further mineral nitrogen during fermentation,

were significantly positive at the same temperature. It was still more evident in the liquid phase of substrate C while a conclusion can be drawn that a higher temperature contributes to a higher extraction of insoluble or partly soluble nitrogen organic compounds from which further mineral nitrogen is released after subsequent fermentation.

	Substrate A	Liquid phase of substrate A	Liquid phase of substrate after recalculation to dry matter content and concentration of substrate A	
			B	C
N	3.28	2.43	2.92	3.11
P	0.87	0.35	0.45	0.56
K	4.20	2.39	2.52	3.32
Ca	1.16	0.25	0.30	0.36
Mg	0.43	0.11	0.13	0.16

Table 9. Contents of mineral nutrients after anaerobic digestion (42 days, 40°C) in digestate (substrate A), in its liquid phase and in fermented liquids from IFBB in % dry matter by weight

Table 10 documents the original (before their recalculation) concentrations of mineral nutrients in the liquid phase of substrates A, B and C. These results indicate that liquid phase A can be considered as a highly diluted mineral fertilizer. Even though the IFBB process increases the concentration of nutrients (nitrogen) in the liquid phase before and after fermentation (liquid phase B and C), the dilution is very high. The recommended dilution with water, used by Wachendorf et al. (2009) and also in our experiments, produces liquid wastes diluted to such an extent after anaerobic digestion that they are practically hardly utilizable as a solution of mineral nutrients. Fugates are still rather problematic as mineral fertilizers, especially for applications in humid years and to soils with low microbial activity and consequently slow immobilization of mineral nitrogen, and naturally they are hardly applicable to pervious soils.

	Liquid phase		
	A	B	C
N	0.32 ± 0.03	0.09 ± 0.01	0.10 ± 0.01
P	0.05 ± 0.00	0.01 ± 0.00	0.02 ± 0.10
K	0.31 ± 0.04	0.08 ± 0.01	0.11 ± 0.15
Ca	0.03 ± 0.00	0.01 ± 0.00	0.01 ± 0.00
Mg	0.01 ± 0.00	0.00 ± 0.00	0.00 ± 0.00

Note: Statistical evaluation of this recalculation table is based on original data in Table 10.

Table 10. Contents of mineral nutrients after anaerobic digestion (42 days, 40°C) in the liquid phase of digestate A and in liquid phases B, C with the application of IFBB in % by weight of solutions that should be used for mineral fertilization  
(Sample size n = 4, reliability interval of the mean for a significance level  $\alpha = 0.05$ )

Table 11 shows the results of hydrolytic experiments with solid phases of substrates A, B and C. They confirm the previously observed fact in the work with the substrate consisting of a mixture of pig slurry and primary sludge from a municipal waste water treatment plant that



the solid phases of wastes from anaerobic digestion cannot be efficient as mineral fertilizers because of their very low degradability (Kolář et al. 2008). The IFBB process, which enriches the liquid phases with organic, easily degradable substances and improves biogas yields during the anaerobic degradation of only liquid phases, further depletes of these substances the solid phases of substrates and impairs their quality as organic fertilizers, even though it is not the case of an increase in the resistant component but only in worse hydrolysable LP II.

Solid phase of substrate	Proportion		
	LP I	LP II	RP
A <sub>1</sub>	43 ± 8	41 ± 7	16 ± 2
A <sub>2</sub>	22 ± 4	20 ± 3	58 ± 8
B	39 ± 6	44 ± 6	17 ± 3
C	31 ± 6	47 ± 8	16 ± 2

Note: Description of fractions according to the method of Rovira, Vallejo 2002:  
LP I = (labile pool I) = the reserve of very labile, easily hydrolysable organic substances expressed as % of the total amount of organic matter in a sample  
LP II = (labile pool II) = the reserve of intermediately labile, less easily hydrolysable organic substances in %  
RP = (recalcitrant pool) = the reserve of hydrolysis resistant, very hardly degradable organic substances in %

Table 11. Proportions of the three pools of carbon in the solid phase of substrate A before anaerobic digestion (A<sub>1</sub>), after anaerobic digestion (A<sub>2</sub>) and in the solid phase of substrate A<sub>1</sub> after IFBB procedure before anaerobic digestion at 15°C (B) and at 60°C (C) as determined by the acid hydrolysis method of Rovira and Vallejo (2002).  
(Sample size n = 4, reliability interval of the mean for a significance level α = 0.05).

Hence it is to state:  
We tested the Integrated Generation of Solid Fuel and Biogas from Biomass (IFBB) procedure proposed for ensiled grass matter from the aspect of suitability of its use for a typical substrate of new Czech biogas plants, a mixture of cattle slurry, maize silage and grass haylage. The agrochemical value of the liquid phase from a biodigester was also evaluated. We concluded that this procedure is suitable for the tested substrate and improves the agrochemical value of a fugate from biogas production. By chlorine transfer to the liquid phase it makes it possible to use the solid phase as a material for the production of solid biofuels with a reduced threat of the generation of polychlorinated dioxins and dibenzofurans during combustion. However, the concentration of mineral nutrients in the liquid phase during IFBB procedure is extremely low after anaerobic digestion as a result of dilution with water, and so its volume value is negligible.  
Here research must go on.

4. References

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## **Biofuel's Engineering Process Technology**

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This book aspires to be a comprehensive summary of current biofuels issues and thereby contribute to the understanding of this important topic. Readers will find themes including biofuels development efforts, their implications for the food industry, current and future biofuels crops, the successful Brazilian ethanol program, insights of the first, second, third and fourth biofuel generations, advanced biofuel production techniques, related waste treatment, emissions and environmental impacts, water consumption, produced allergens and toxins. Additionally, the biofuel policy discussion is expected to be continuing in the foreseeable future and the reading of the biofuels features dealt with in this book, are recommended for anyone interested in understanding this diverse and developing theme.

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