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# START-UP PERFORMANCE OF A MESOPHILIC ANAEROBIC DIGESTER WITHOUT EXTERNAL INOCULUMS

The objective of anaerobic digester start-up is to achieve steady-state operation and the required reduction performance in the shortest possible time. The present study aims to assess a novel strategy to start-up pilot scale mesophilic anaerobic digester with internal inoculums; the operational parameters such as feed concentration and operating temperature were also evaluated. Three pilot scale anaerobic digester systems were designed and manufactured. They were equipped with all necessary instruments for measurements, operation and control. Each system consists of a feed tank, a reactor (anaerobic digester), a gas storage tank, and a displaced water collecting tank. The digester start-up was achieved successfully and smoothly without any operational problems. The feed concentration had minor effects on the performance of the digester start-up while the operating temperature has approximately no effect.

## 1. INTRODUCTION

Start-up is a critical step in the operation of anaerobic digestion, and it requires much attention so that the reactor be kept in good conditions. Some researches focused on the start-up of anaerobic digestion revealed that the inoculum type and ratio are important factors for a successful start-up. It is well recognized that digested sludge taken from another working digester is a good inoculum for a stable start-up of anaerobic digestion [1]. In many cases, however, start-up relies on the internal inoculum inside the substrate by cultivating microorganisms throughout the anaerobic self-degradation. It has been reported that start-up without external inoculums caused significant accumulation of volatile fatty acids (VFAs) and took longer for the organic loading rate to be raised during the early days of operation [2].

Mesophilic anaerobic digestion is a biological process which requires time and careful management during start-up. Good planning, continuous monitoring and slow changes are

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the three key factors for a successful start-up [3]. Careful monitoring and control of pH are essential until the digester attains pH in the 6.8–7.2 range [4].

Various strategies are utilized to start-up mesophilic digesters. Kobayashi et al. [1] filled the digester with waste activated sludge (WAS) diluted to 15 g/dm$^3$ as total solid (TS) with tap water. At first, the reactor was operated in a batch mode. Then, fresh WAS was fed to the reactor. The hydraulic retention time (HRT) was gradually shortened from 100 days through 50 days to 30 days. While Hatzigergiou et al. [3] introduced two start-up strategies, the first strategy was used by the Upper Occoquan Sewage Authority (VA) which consists of the following: the digester was filled with primary sludge and seed. Unfortunately there are no data available on the exact amount of seed used. All that is available is the monitoring data during the start-up that indicate that the amount of seed used was not adequate for the feed rates to that digester. On the other hand, the second strategy was used by the Alexandria Sanitation Authority (VA) which consists of the following: the digester was filled with plant effluent that was then heated to mesophilic digestion temperature. Then the digester was seeded with digested biosolids. The volume of the seed solids was equal to 4.5% of the total volume of one digester. Once the digester was seeded, primary sludge was added at a very slow rate.

Therefore, there is a need for a strategy to enhance the start-up of anaerobic digestion from internal inoculum for optimum, stable operating conditions. The present study is an attempt to start-up the anaerobic digester without any seed, even though full feed with thickened sludge, just the digester should be filled with treated wastewater at the beginning. Also in the present study, the effect of feed concentration as well as operating temperature on the digester start-up performance will be evaluated.

## 2. EXPERIMENTAL

*Test rig.* The experiments were carried out using a continuous operation, pilot scale digester. Three pilot scale anaerobic digesters were designed and manufactured. They were equipped with all necessary instruments for measurements, operation and control. The experimental set up was constructed at El Kinayat WWTP Zagazig, Sharkia, Egypt. Each system consisted of a feed tank, a reactor (anaerobic digester), a gas storage tank, and a displaced water collecting tank. The volume of the produced biogas was measured by the water displacement method [5]. A schematic illustration of the experimental test rig is shown in Fig. 1.

*Design and dimension of the reactor.* Each reactor (working volume 240 dm$^3$) was constructed from steel in a cylindrical shape with 64 cm in diameter and 100 cm high. Each reactor had two ports on the top flange – one for sludge feeding and the other for biogas collection. Both ports had the diameter of 2.54 cm. The digester was provided with two centrifugal pumps – one for sludge feeding and the other for digested sludge

withdrawn (although the withdrawn of digested sludge normally achieved by raw sludge displacement, in the present study it was achieved by pumping system to prevent clogging). The digester contents were mixed using sludge circulation method.
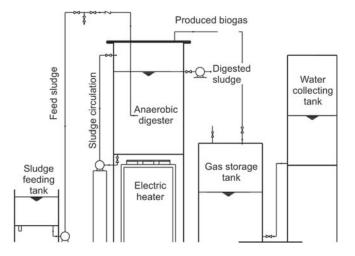


Fig. 1. Schematic illustration of the experimental test rig

*Anaerobic digester operation.* The digester start-up can be divided into preparation and operation phases. During the preparation phase (the first 4 days) the digesters were filled with primary clarifier effluent, allowed to reach operating temperature, and mixing was started [6]. During the operation phase, the digesters were fed with thickened sludge (mixture of primary and humus trickling filter sludge). The main characteristics of feed sludge are summarized in Table 1. The sludge was screened to prevent clogging during the transfer from the feed tank to the digesters [7]. Digesters were fed and digested sludge was withdrawn continually each 2 h, at a hydraulic retention time of 20 days by centrifugal pumps [6].

Table 1

Characteristics of the influent sewage sludge

| Parameter | COD [g $O_2$/dm$^3$] | TS [g/dm$^3$] | VS [g/dm$^3$] | Alkalinity [mg CaCO$_3$/dm$^3$] | pH | VFA [mg/dm$^3$] |
|---|---|---|---|---|---|---|
| Range | 30–70 – | 38–72 | 20–50 | 850–1870 | 5.7–7.5 | 800–2700 |
| Average | 50 | 55 | 35 | 1360 | – | 1750 |

Each reactor was heated using an external electric heater at the bottom of the reactor. The heater was equipped with a temperature control system. The produced biogas was

discharged to a storage system. The storage tank was filled with water, in which the displaced volume represents the volume of the biogas produced.

The three digesters were operated in parallel to evaluate the start-up strategy as well as to study the effect of other operational parameters. To study the effect of feed concentration one of the digesters (AD1) was fed with thickened sludge, while the feed of the other digester (AD2) was diluted sludge. The diluted sludge was prepared by diluting thickened sludge with primary treated wastewater in 1:1 ratio. Kalloum et al [5] proposed dilution of feed sludge to avoid the medium acidification during the start-up period. The operating temperature of the AD1 and AD2 reactors was 37.5±0.5 °C [2]. To study the effect of operating temperature, the third digester (AD3) was fed with diluted sludge as AD2 and it was operated at 32±0.5 °C

*Analysis and measurement*. To monitor the performance of the anaerobic reactors, influent and effluent sludge samples were analyzed by measuring the concentration of chemical oxygen demand (COD), total solids (TS), volatile solid (VS), and alkalinity, according to the standard methods for the examination of water and wastewater [8]. Volatile fatty acids (VFA) were determined by the titration method by Kappa [9]. For the alkalinity and VFA, the samples were centrifuged, and filtered through 0.45 µm filter membrane [10].

## 3. RESULTS AND DISCUSSION

The digester performance was evaluated by monitoring the following parameters: the volume of biogas produced, the COD removal ratio, the solid removal ratio. The digester stability was evaluated by monitoring the VFA, alkalinity, and pH.
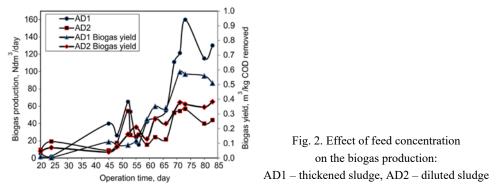


Fig. 2. Effect of feed concentration
on the biogas production:
AD1 – thickened sludge, AD2 – diluted sludge

Figure 2 illustrates the volume of biogas produced from the digesters AD1 and AD2. The biogas yield expressed as $m^3$/kg COD removed is also presented. The volume of biogas produced during the first 59 day of operation was fluctuating. This may be due to the changed in organic loading rate, while the biogas yield was generally increased

slightly with operation time. Also the amount of biogas was small during the first 59 days, then it was increased. The measured values were expressed as $Ndm^3$ (normal cubic decimeter). The normal conditions are atmospheric pressure and 25 °C.
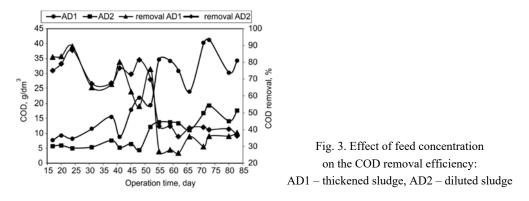
The amount of biogas produced from AD1 increased from 42 $Ndm^3$/day after 59 day to 160 $Ndm^3$/day after 73 day of operation. The amount of biogas produced from AD2 increased from 15.6 $Ndm^3$/day after 59 day to 57 $Ndm^3$/day after 73 day of operation. The biogas yield also increased for AD1 from 0.26 to 0.57 $m^3$/kg COD after 59 and 73 day of operation respectively, while these values were 0.13 and 0.36 $m^3$/kg COD for AD2. The biogas yields at 73 day of operation remain around these values up to 83 day of operation that indicate steady state condition. The volume of biogas produced per kilogram of COD removed provides a measure of the transformation of organic matter into biogas. It also reflects proper operation and the absence of gas leakage in the system. This value is consistent to the published results of [11], who reported a production of 0.55 $m^3$ biogas/kg COD removed.

The organic loading rate was about 1.75 kg of VS per day/$m^3$ of the digester for AD1 after 73 day of operation. The corresponding biogas production yield was 1.18 and 0.71 $m^3$/kg of VS removed for AD1 and AD2, respectively. This represent good yield rate for the digesters used in this experiments. Stroot et al. [2] evaluated the start-up of anaerobic digester operated at OLR 3.7 (kg VS per day/$m^3$), and indicated that the steady state was achieved at biogas production yield of 0.7 $m^3$ biogas/kg VS removed. Young et al. [7] examined steady state sewage sludge anaerobic digester operated at 20 days HRT, 35±2 OC and average organic loading rate of 1.43 kg of VS per day/$m^3$ of the digester, which results in 0.77 $m^3$ biogas yield/kg of VS removed. A gas production of 0.8–1.1 $m^3$/kg of VS removed is an indication of proper digestion [6] and the values obtained in the current study fall in that range.

The above mentioned biogas production indicated that the digesters produced a significant amount of biogas after the first 55 days of operation, while the steady state operation was reached after 73 day. Hatzigeorgiou et al. [3] reported that steady state of anaerobic digester during start-up was achieved after 116 day of operation. Kobayashi et al. [1] evaluated the start-up of anaerobic digester of waste activated sludge applying internal inoculums. The start-up was divided into four intervals; at first operation was batch without feed, then continuous operation at 100 day HRT, after that the HRT was reduced to 50 days and finally operated at 30 day. Their study concluded that each of the operating parameters stabilized after 100 day from start-up. On the other hand, Stroot et al. [2] compared the effect of different inoculums in digester start-up, and stated that the steady state was achieved for the digesters after 75 day of operation. This result is consistent with the results of the present research that was achieved without the addition of inoculums or any other chemicals.

Figure 3 illustrates the COD removal as well as the effluent COD from AD1 and AD2 digesters. The COD removal was high for the first day of operation. This is due to

mixing effect as the digesters were filled with primary treated wastewater, so the influent sludge was just diluted. During the operation, the sludge total solids concentration inside the digester increased, and the effect of dilution decreased, thus the removal efficiencies were decreased and reached minimum values after 55 day of operation. These values are 27 and 42% for AD1 and AD2, respectively.



Fig. 3. Effect of feed concentration
on the COD removal efficiency:
AD1 – thickened sludge, AD2 – diluted sludge

On the other hand, the effect of biological action increased and after the first 55 days the removal ratio for AD1 was increased to 36% at 73 day of operation. The removal ratios for AD2 approximately did not change until the 73 day. This indicated that AD2 reached steady state faster than AD1. The COD removal efficiencies at 73 day of operation remain around these values up to 83 day of operation that indicating steady state condition. The COD removal efficiencies achieved at steady state is approximately consistent with that of Cacho [11] who conducted experimental work on mesophilic digester, operated at 20 day HRT, and the concentration of COD in the influent was 56 $g/dm^3$. That digester achieved 46% COD removal.
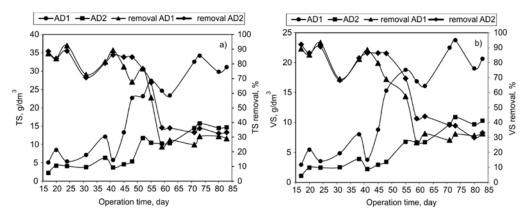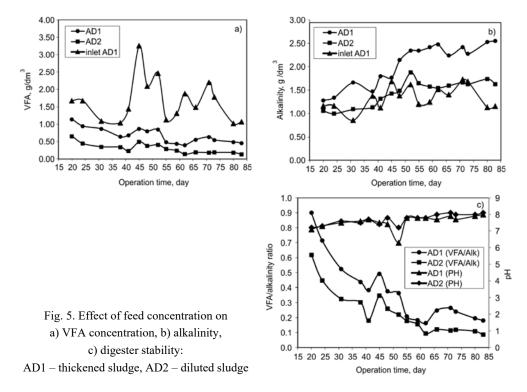


Fig. 4. Effect of feed concentration on TS (a) and VS removal (b):
AD1 – thickened sludge, AD2 – diluted sludge

Figures 4a, b illustrate the total solids and volatile solids removal efficiencies as well as the effluent solid concentration from AD1 and AD2. The TS removal was high from the first day of operation due to dilution effect. These values are decreased and reached minimum values after 59 day of operation – 23.5 and 36.5 % for AD1 and AD2, respectively. After the 59 days the removal ratio for AD1 was increased to reach 30.5% after 73 day of operation. The removal ratios for AD2 approximately did not change until the 73 day this indicated that AD2 reached steady state faster than AD1. The VS removal approximately has the same trend as TS (Fig. 4b). The removal efficiencies of volatile solids for AD1 and AD2 after 73 day of operation were 32% and 37%, respectively. The volatile solid removal efficiencies at 73 day of operation remain approximately around these values up to 83 day of operation indicating steady state conditions. The presented VS removal values are around that of Yue [12] who used mesophilic digester. The VS concentration in the influent was 29.2 $g/dm^3$, while the volatile solid removal was 32.5%.

Fig. 5. Effect of feed concentration on
a) VFA concentration, b) alkalinity,
c) digester stability:
AD1 – thickened sludge, AD2 – diluted sludge

The digester stability was evaluated by monitoring the VFA, alkalinity, and pH. Figure 5a shows the influent and effluent concentration of VFA, which is considered as intermediate product before biogas ($CH_4$) is formed by methanogenses bacteria. The concentration of influent VFA ranged from 1050 to 3260 $mg/dm^3$. As shown in Fig. 5a,

the effluent concentrations for the two digesters decreased upon increasing operation time, and that values did not exceed 1000 mg/dm$^3$ during all the period of start-up. During the first days of operation, the dilution prevents the acid accumulation until the biological action activated. The concentration of VFA on AD1 reached 550 mg/dm$^3$ after 73 day of operation, that value for AD2 was 190 mg/dm$^3$, this may be due to the low concentration of feed sludge on AD2. These values continued to decrease during the last period of operation that indicates steady state operation.

Figure 5b shows the alkalinity. It is considered as the buffer to the digester pH. The concentration of influent alkalinity ranged from 0.85 to 1.7 g CaCO$_3$/dm$^3$. The effluent concentrations for the two digesters increased upon increasing time of operation, and these values were not smaller than 1 g CaCO$_3$/dm$^3$ during all the period of start-up. During the first days of operation, the dilution prevents acid accumulation until the biological action activated, thus the alkalinity was maintained high enough. The concentration of CaCO$_3$ on AD1 reached 2.27 g/dm$^3$ after 73 day of operation, for AD2 – 1.62 g/dm$^3$, this may be due to the lower concentration of sludge on AD2 than on AD1. In sewage sludge digesters, sufficient alkalinity is generally found in the range of (2–5 g CaCO$_3$/dm$^3$) to prevent pH value from falling below the limit for methanogenesis inhibition [13]. Alkalinity enables the digester to better handle a sudden increase in organic load without process deterioration [6, 14].

Figure 5c shows pH for the two digesters which was within the range of 7–8. Kalloum et al. [5] stated that after 25 day of start-up, pH was stable around 7. This value is within the acceptable pH range of 6.1–8.3, which is suitable for sludge digestion. Below pH = 6.1, volatile acids become toxic to methane-forming microorganisms. On the other hand for pH above 8.3, the dissolved ammonia becomes toxic to methane-forming microorganisms [13, 6]. The increase in pH may be attributed to the increase in the alkalinity as well as the VFA consumption.

Figure 5c shows also the VFA/alkalinity ratio. For AD1, it was high, equal to 0.9 after 20 day of operation, that value was 0.6 for AD2. These values decreased to around 0.2 for the two digesters after 55 day, and finally became 0.24 for AD1 and 0.12 for AD2 after 73 day of operation. The VFA/alkalinity ratios continued to decrease during the last period of operation indicating a steady state operation. Kalloum et al. [5] in their found that the value of VFA/alkalinity ratio was lower than 0.5 during the digestion process.

Young et al. [7] monitored the VFA/alkalinity ratio for a steady state digester, and concluded that the buffering capacity is sufficient when this ratio is maintained below 0.4. This indicated that after 55 days of operation the two digesters were stable and start--up was achieved successfully, while the digesters required another 18 days to reach steady state.

The biogas produced from the digesters AD2 and AD3 was collected in order to examine the temperature effect. The biogas yield for AD3 digester was 0.22, 0.35, 0.38 m$^3$/kg COD removed, after 66, 71, 73 day of operation, respectively. The biogas

production for AD2 digester was 0.234, 0.38, and 0.37 m³/kg COD removed, after 66, 71, 73 day of operation, respectively. The biogas production at 73 day was approximately the same up to 83 day of operation. This indicated that as the temperature increased from 32 to 37.5 °C, the biogas production increased by about 3%. Ryan [15] examined the effect of temperature on the mesophilic digester performance, and concluded that increasing the temperature from 30 to 35 °C produced similar amounts of biogas.
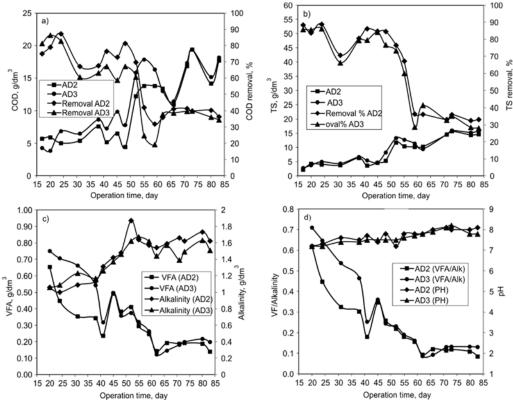


Fig. 6. Effect of temperature (AD2 – 37.5 °C, AD3 – 32 °C) on:
a) COD removal, b) TS removal, c) VFA and alkalinity, d) pH

Figure 6a shows the COD removal as well as the effluent COD from AD2 and AD3. Up till 62 day of operation, the COD removal for AD2 digester was higher than that for AD3 digester by approximately 6%. After 62 days, the two digesters approximately removed the same percent of COD. The TS removal and the effluent TS from AD2 and AD3 are shown in Fig. 6b. Up to 70 day of operation, the TS removal for AD2 digester was higher than that for AD3 digester by approximately 3%, while after 70 days the two digesters approximately removed the same percent of TS.

As shown in Fig. 6c, the VFA concentration for AD2 digester was lower than that for AD3 digester during the first 40 day of operation, while during the rest of the operation period the two digesters approximately contained the same amounts of VFA. The two digesters have approximately the same contents of alkalinity during as well.

Figure 6d shows pH for the two digesters. It was within the range of 7–8. The values of the VFA/alkalinity ratio for AD2 digester were lower than those for AD3 digester during the first 40 days of operation, reaching the same level at longer period of time.

## 4. CONCLUSIONS

The study aims to a better understanding of the start-up of anaerobic digester and assesses a novel strategy to start-up the digester without any external additions or high skills requirements. The only requirement of the proposed strategy is to start by filling the digester with treated wastewater, then normal operation by feeding the thickened sludge to the digesters. Also the operational parameters such as feed concentration and operating temperature were evaluated. The experimental set up consisted of three similar pilot scale mesophilic anaerobic digesters; each digester had a cylindrical shape with a capacity of 0.24 $m^3$, the digesters were operated at a municipal wastewater treatment plant from which the feed sludge was taken.

The novel strategy to start-up the digester was successful. The digester start-up was achieved after 55 day of operation, while the steady state condition was reached after 73 day of operation. During the start-up period, the removal of COD and solid was achieved from first day of operation by dilution effect (due to mixing with treated wastewater). After accumulation of biomass, the removal was due to biological reaction. Also the dilution effect due to starting with treated wastewater was the key factor of digester stability, the VFA was lower than 1 $g/dm^3$ along the start-up period and there was no acid accumulation. Also the VFA to alkalinity ratio decreased, and after 55 day of operation it was around 0.2, which indicated stable operation of the digester. The feed concentration has minor effects on the performance of digester start-up. The digester with diluted feed performed 5% removal ratio better than that with concentrated feed. Also the diluted feed digester was more stable than that with concentrated feed. On the other hand, the biogas production from the digester with concentrated feed was higher than that with diluted feed. Finally the results indicated that the operating temperature had minor effects on the digester performance during the first 55 days of operation, and almost no effect after that period.

## REFERENCES

[1] KOBAYASHI T., YASUDA D., LI Y., KUBOTA K., HARADA H., YU D.H., *Characterization of start-up performance and archaeal community shifts during anaerobic self-degradation of waste-activated sludge*, Biores. Technol., 2009, 100 4981.

[2] STROOT P.G., MCMAHON K.D., MACKIE R.I., RASKIN L., *Anaerobic codigestion of municipal solid waste and biosolids under various mixing conditions*. I. *Digester performance*, Water Res., 2001, 35 (7), 1804.

[3] HATZIGEORGIOU M., OWSENEK B., ALKEMA T., SIEGER R., PALLANSCH K., *Start-up of Anaerobic Mesophilic Digesters*, Proc. Water Environment Foundation, 2006, https://doi.org/10.2175/193864706783710569.

[4] SURYAWANSHI P.C., CHAUDHARI A.B., BHARDWAJ S., YEOLE T.Y., *Review Paper Operating Procedures for Efficient Anaerobic Digester Operation*, Res. J. Animal., Vet. Fish. Sci., 2013, 1 (2), 12.

[5] KALLOUM S., KHELAFI M., DJAAFRI M., TAHRI A., KAÏDI K.A., TOUZI A., *Study of start-up of a continuous digester on a laboratory scale treating the sludge issued from wastewater treatment plant in Adrar city (south west of Algeria)*, Rev. Energ. Renouv., 2012, 15 (2), 229.

[6] *Operation of Municipal Wastewater Treatment Plants*, Water Environment Federation, McGraw-Hill, New York 2008.

[7] YOUNG C.S., SANG J. K., JUNG H. W., *Mesophilic and thermophilic temperature co-phase anaerobic digestion compared with single-stage mesophilic- and thermophilic digestion of sewage sludge*, Water Res., 2004, 38, 1653.

[8] *Standard Methods for the Examination of Water and Wastewater*, 20th Ed, American Public Health Association (APHA), Washington 1999.

[9] BUCHAUER K., *A comparison of two simple titration procedures to determine volatile fatty acids in influents to waste-water and sludge treatment processes*, Water SA, 1998, 24 (1).

[10] YAN C., BO F., YAN W., QIAN J., HE L., *Reactor performance and bacterial pathogen removal in response to sludge retention time in a mesophilic anaerobic digester treating sewage sludge*, Biores. Technol., 2012, 106, 20.

[11] CACHO R., *Anaerobic Digestion of Excess Municipal Sludge Optimization for Increased Solid Destruction*, PhD Dissertation, Division of Research and Advanced Studies of the University of Cincinnati, 2005.

[12] YUE H., *Laboratory studies on the temperature-phased anaerobic digestion of mixtures of primary and waste activated sludge*, PhD Dissertation, Iowa State University, Ames, Iowa, 1997.

[13] FERRER I., VAZQUEZ F., FONT X., *Long term operation of a thermophilic anaerobic reactor: process stability and efficiency at decreasing sludge retention time*, Biores. Technol., 2010, 101, 2972.

[14] *Wastewater Engineering, Treatment, and Reuse*, 4th Edition, Metcalf & Eddy, Inc., McGraw-Hill, New Delhi 2003.

[15] GRANT R., *Enhanced biogas production through the optimization of the anaerobic digestion of sewage sludge*, Master Thesis, Tuscaloosa, Alabama, 2011.