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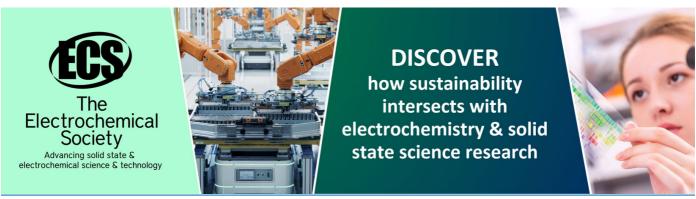
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# Kinetics study of methane production from anaerobic digestion of sludge and wastewater recycled pulp and paper

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Abstract. Methane yield is one of the important factors that indicate the anaerobic digestion (AD) process efficiency. The AD is an available treatment of recycled pulp and paper sludge (RPPS) and recycled pulp and paper wastewater (RPPW). This process protects the environment and produces methane, which is considered as renewable energy. In this study, the anaerobic treatment of the RPPW and RPPS were studied using 1L completely stirred tank reactor (CSTR) at laboratory scale with mesophilic conditions. However, a kinetic study was carried out using four models in order to compare and adjust the experimental results. The experimental results show that the RPPW and RPPS methane productions were between 691 and 945 mL/g based on volatile solid (VS), and the corresponding digestion time was about 26 days for the both experiment. The corresponding digestion time could be understood through the methane production kinetic models by digestion experiments within a short time. Different kinetics models were tested including Richards, Logistic, modified Gompertz and Cone models. The obtained results show the modified Gompertz model as the suitable model since it gives the lowest deviation from experimental results. Therefore, the proposed model could be used to accurately predict behavior of AD processes.

#### 1. Introduction

The AD is a recommended technology that can be applied to reduce the pollution, by the reduction of organic matter, and to produce methane as a renewable energy that we can exploit in different way [1, 2]. The recycled pulp and paper is a growing industry which plays a significant part in the growth of the economy in the world [2, 3]. However this industrial sector consumes a significant amount of resources, raw materials and energy. Furthermore, it produces a considerable amount of organic waste, which is not suitable for the production of new paper [4]. Correspondingly, the biodegradable organic fractions and hydrolysis rates of substrates are two important parameters for anaerobic biodegradation. In fact, these parameters determine, to a certain extent, both the design and economic details of biomethane plants. A huge quantity of the sludge produced by paper mills is considered as one of the most serious environmental problems [5]. In general term, we use the anaerobic batch tests as standard method for the determination of methane yield potential [6]. Structured kinetic models are used for the simulation of biomethane production from anaerobic treatment by biodegradation of RPPS and RPPW to produce renewable energy. The structured models include multiple stages of the material degradation in CSTR reactors which have made it possible to predict the actual response of the process.

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[7,8]. Compared to the Richards, Logistic, Gompertz and Cone model, the modified Gompertz model was commonly used as it was correlated with four biochemical reaction parameters, A,  $\mu_m$ ,  $\lambda$  and R. The Kinetic study of mesophilic AD show that the corresponding equations could be used for design CSTR for digesting organic wastes from pulp and paper industries, food processing industries, treatment plants wastewater [9,3].

The kinetic model of methane production by AD is a practical way to understand the ultimate technique of methane production in the standard condition [3]. In the literature, several kinetic studies of the AD process show an efficient application for monitoring and controlling the stability parameters, using different kinds of reactors and substrates. The kinetic model equations could be used to predict dynamic simulation of the AD on the principle of a complex matrix series for constant kinetic values [9].

The purpose of this paper is to apply kinetic models on the AD process of RPPS and RPPW. The digester used in this study is a CSTR on batch mode. A number of modified kinetic models as Richards, Logistic, Gompertz and Cone, have been used to evaluate their applicability on experimental results of methane production of RPPW and RPPS experiments. In addition, the purpose of this paper is to describe the kinetics parameters of methane production.

#### 2. Material and methods

#### 2.1. Inoculum characterization

Starting the process of AD requires the use of an inoculum rich in bacteria that will degrade the organic matter. The inoculum improves the production of biogas; at the same time it reduces the retention time. The inoculum used in all experiments comes from a mesophilic anaerobic digester treating sludge from a wastewater treatment plant in Morocco. Table 1 shows its main physicochemical characteristics. Initially, the digester was fed with  $7g\ VS\ /\ L$  of inoculum, which is equivalent to  $342\ mL$ .

# 2.2. Substrate characterization

The sludge and the wastewater used in the experiment were recovered from a recycled pulp and paper manufactory in Morocco [10,1]. The sludge was kept after gravitational static decantation, at laboratory scale, in order to eliminate the supernatant and recover only the deposit of the effluent "sludge". Inoculum, sludge, and wastewater analysis were performed according to the "Standard Methods for the Examination of Water and Wastewater" (12). Indeed, recent values show that the COD of pulp and paper effluent corresponds to values in the range of (3770 - 9330mg  $L^{-1}$ ) and BOD<sub>5</sub> in the range of (816-2495 mg  $L^{-1}$ ) [11].

Parameters	Unit	Inoculum	Wastewater recycled	Sludge recycled pulp
			pulp and paper	and paper
рН		$7.2 \pm 0.11$	7.5	7.1
COD	mg/l	$7741.12 \pm 140$	7910.5	9230
$BOD_5$	mg/l		4430.5	6100
COD/BOD <sub>5</sub>			1.51	1.94
Alk	mgCaCO <sub>3</sub> /l	2750	2250	1875
TS	g/l	$29.05 \pm 0.63$	55.70	105.07
VS	g/l	$9.71 \pm 0.46$	35.25	55.22
MS	g/1	$19.339 \pm 0.43$	20.457	49.855
VS (% TS)	%	33.42	63.28	52.55

**Table 1**. Substrates and inoculums characteristics.

#### 2.3. The anaerobic digester

The reactor used in the laboratory for AD, consisted of 1L Pyrex CSTR (Continuous Stirred-tank reactor), equipped with magnetic stirring. It worked in batch mode. This reactor is characterized by four orifices, the first to insert the substrate, the second one for the ventilation of biogas, the third for inert gas injection (nitrogen) to maintain anaerobic conditions, and the last one to remove effluents. The schematic diagram of experimental set up is shown in Fig. 1. [12,3].

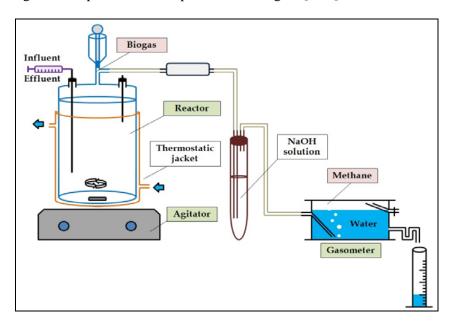


Figure 1. Schematic diagram of CSTR reactor system.

The temperature of reactor was maintained by means of a thermostatic jacket containing water at 37°C for mesophilic conditions. The volume of methane produced during the digestion process was measured by using 1L Boyle-Mariotte reservoir connected to the reactor. To remove the CO<sub>2</sub> produced during the process, tightly closed bubblers containing a NaOH solution (6 N) were connected between the two elements. It is necessary sometimes to close the pipe of the NaOH solution that links the reservoir to the reactor. The methane volume produced can be determined from the volume of water ejected from the reservoir (volume corrected in STP conditions: 0°C and 1 atm).

#### 2.4. Methane measurement

The produced methane is measured by the displacement of water using a cylindrical gas-meter which capacity is one liter linked to a graduated tube of the same volume. The volume of water in the tube is equivalent to the methane produced. The cumulative methane volume is standardized with taking account the effect of atmospheric pressure, the water vapor temperature, and the pressure of the room at the time of measurement, the results are expressed under normal conditions (0  $^{\circ}$  C at 760 mm Hg) [13].

The temperature is determined by means of the thermometer, the pressure is considered as constant throughout the process, which is equal to the atmospheric pressure. From the law of perfect gases, expressed under normal and experimental conditions:

At normal condition:

$$P_N V_N = nRT_N$$

With:

$$K = \frac{P_{N}V_{N}}{T_{N}} \quad (1)$$

At ambient conditions:

$$P_R V_R = nRT_R$$

With:

$$K = \frac{P_R V_R}{T_R} \qquad (2)$$

The relation (1) and (2) we have:

$$\frac{P_R V_R}{T_R} = \frac{P_N V_N}{T_N} \quad (3)$$

The final formula obtained to be applied is:

$$V_{N} = V_{R} \frac{(P_{alm} - P_{\nu})}{P_{N}} \frac{T_{N}}{T_{n}}$$
(4)

With:

 $V_N$ : Normal volume of methane (0 ° C, 1 atm) (NmL),  $V_R$ : Volume of real methane (mL),  $P_N$ : Pressure at normal conditions (0 ° C, 1atm),  $P_R$ : Actual pressure at experimental conditions (mbar),  $P_{atm}$ : Atmospheric pressure in mmHg. P: Vapor pressure at room temperature mmHg,  $T_N$ : Temperature at normal conditions (0 ° C (273 K), 1atm),  $T_R$ : Actual temperature at experimental conditions (° C (K)) [14].

#### 2.5. Sigmoidal models

A number of sigmoidal models are found in the literature; such as the Gompertz, Richards, Standard and Logistic models [14,15]. Modeling microbial growth can evaluate several parameters; such as specific growth rate and latency. The bacterial growth curve showed the minimal phase (in our case zero) and the enhancement of the maximum rate of growth  $(\mu_m)$  in the case named as lag phase  $(\lambda)$  [16]. Thus, when the Bacterial growth reaches its limit, this shows that the growth curve reaches the maximum limit A [16].

The following curve shows three essential parameters for understanding the biological process of organic matter degradation.  $\mu_m$  presents the tangent of the linear curve during methane production;  $\lambda$  shows the intersection of the curve with the x-axis, and a shows the intersection of the curve with the y-axis (also presents the maximum cumulative methane).

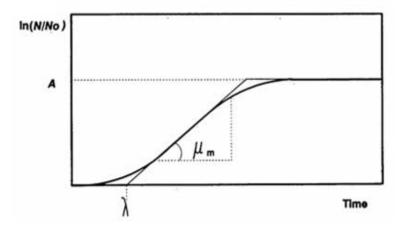


Figure 2. Typical curve of methane production

Model	Equation	Modified equation
Logistic	$y = \frac{a}{\left[1 + a \exp(b - cx)\right]}$	$y = \frac{A}{\left[1 + \exp\left(\frac{4\mu_m}{A}(\lambda + t) + 2\right)\right]}$
Gompertz	$y = a \exp\left[-\exp(b - cx)\right]$	$y = A \exp \left\{ -\exp \left[ \frac{\mu_m \cdot e}{A} (\lambda - t) + 1 \right] \right\}$
Richards	, ,	
	$y = a \left\{ 1 + \nu \exp \left[ k \left( \tau - x \right) \right] \right\}^{\left( -\frac{1}{\nu} \right)}$	$y = A \left\{ 1 + v \exp\left(1 + v\right) \cdot \exp\left[\frac{\mu_{m}}{A} \left(1 + v\right) \left(1 + \frac{1}{v}\right) \cdot (\lambda - t)\right] \right\}^{\left(-\frac{1}{v}\right)}$
Stannard	$y = a \left\{ 1 + \exp \left[ -\frac{(I + kx)}{2} \right] \right\}^{-p}$	$\int_{\mathbb{R}^{n}} \left( 1 + K \right) \exp \left( 1 + K \right) \exp \left( \frac{1}{p} \right)$

**Table 2.** Models use in bacterial growth and their modified forms according to Zwietering et al. [13].

Y: cumulative specific methane production (mLCH<sub>4</sub> g VS/L), A: maximum specific methane production potential (mLCH<sub>4</sub>/gVS),  $\mu$ m: max. Specific methane production rate (mLCH<sub>4</sub>/gVS.d), e: Exp (1),  $\lambda$ : lag-phase (days), t: incubation time (days), v: shape coefficient.

These two equations are used from the kinetics study of author Dong Li, Kinetics of methane production and hydrolysis in AD of corn stover, to give a relationship between methane production and organic matter degradation in terms of volatile solids [17].

$$y = A \exp\left\{-\exp\left[\frac{\mu_{m} \cdot e}{A}(\lambda - t) + 1\right]\right\}$$

$$VS_{T,t} = VS_{NS} + \left(VS_{T,0} - VS_{NS}\right) * \exp\left(-K_{H,S} \times t\right)$$
(6)

Where  $VS_{T,t}$  is the total VS content (g/L),  $VS_{NB}$  is the non-biodegradable VS content (g/L, or %  $VS_{T}$ ),  $VS_{T,0}$  is the initial total VS.

Adjustment of the pairs of experimental data (t,  $VS_T$ ,t) by non-linear regression allowed the calculation of the fraction of  $VS_{NB}$  and  $K_{H.B.}$ 

### 3. Results and discussion

Cone

In order to characterize the kinetics of the two experiments, and thus to facilitate the comparison between the different kinetic models applied to the production of methane; the previous models were used to adjust the experimental data. Figure 3 shows the variation of the accumulated methane volume as a function of time for the two types of the treated substrate. The results show that methane production increases with increasing time. This phenomenon is due to the organic matter degradation, which emerges in form of biogas in 26 days. This is equal to the substrate degradation time for both waste types. The methane yields in this study were higher than that of previous studies [18]. The methane yield increased in 10 days from 0.003 to 0.09 L  $CH_4/g$  COD.

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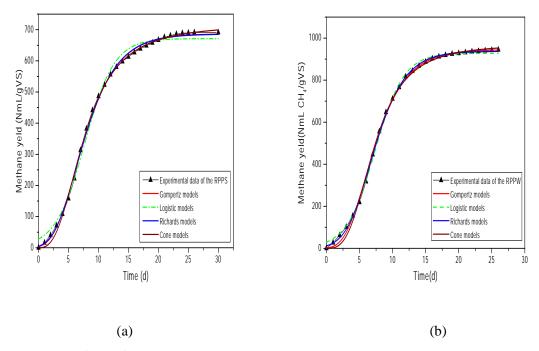
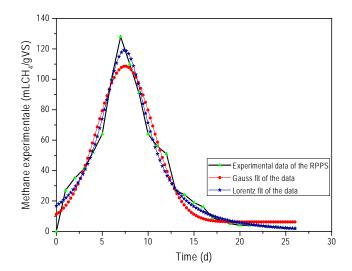
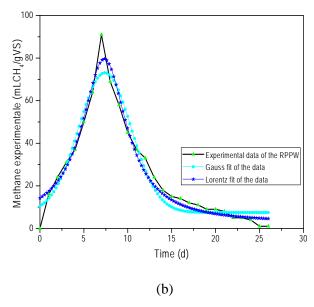


Figure 3. Cumulative methane production of: (a) RPPS, (b) RPPW.

The volume of methane produced is an important parameter used for the control and monitoring of the AD process. Indeed, a substantial production of methane reflected the proper functioning of the digester. Figure 3 represents the total volume of methane produced for both: AD of RPPS and RPPW. The efficient method to determine the methane production for different organic matter concentrations is the kinetic study using a several models [17].

The figure shows a rapid evolution of methane production with a high rate of bacteria growth during the first 10 days, which means that the bacteria are well adapted to the substrate. This growth rate decreases during the last days for both experiments.





**Figure 4**. Experimental methane:(a) methane production rate during the AD of sludge recycled paper mill, (b) methane production rate during the AD of wastewater recycled paper mill.

The results of the simulation of methane-produced volume are illustrated in Figure 4. The results show that the simulations are in agreement with the experimental results. However, the little variations observed are related to the change in organic matter degradation and the substrate nature.

Figure 4 shows the variation of the simulated and experimental results of the produced methane volume over time. This depends on the nature, composition, and biodegradability of industrial waste. In this study, two equations were applied for the experimental data to test which was compatible with our case study. The graph shows the experimental data and the simulation of Gauss and Lorentz equation. We deduce from the correlation vector and the variables of each equation that Lorentz is well adjusted to our study by a correlation vector of R<sup>2</sup> equal to 97.1%, which is higher than the one of Gauss which is equal to 94.71%. In this case, for experimental data of recycled wastewater paper mill; Lorentz equation is compatible by a correlation vector equal to 95.54% which is higher than the one of Gauss which is equal to 93.01%.

# 3.1. Kinetic study of methane production

Table 3 shows the values of the kinetic models used, as well as their standard deviations obtained in the two experiments. The standard deviations are respectively less than 5% and 10% for both types of waste studied. This suggests that the proposed models are well adjusted to the experimental data. The authors summarize the adjustment errors of 10% or month is a good adjustment for methane production obtained from the kinetic study by deferent model [19, 20].

The table shows the kinetic model variables applied to the experimental result. Each model gives an idea of the quantity of methane produced as a function of eliminated gVS; the volume of methane produced each day, and the hydraulic retention time  $\lambda$  (day).

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Parameter	Models	Recycled paper mill wastewater	Recycled paper mill sludge
$V_0 = (mLCH_4 / gVS)$	Experimental	691	945
$A(mLCH_4/gVS)$	Richards	685.96±3.32	937.28±3.17
	Logistic	671.4±5.8	927.9±4.5
	Gompertz	687.13±3.23	945.37±4.4
	Cone	716.37±4.99	970.8±8.3
$\mu_{m}(mLCH_{4}   gVS.d)$	Richards	38.24±2.5	67.65±9.5
	Logistic	68.35±2.9	$107.9 \pm 2.8$
	Gompertz	67.4±1.3	$106.71 \pm 2.23$
	Cone	-	-
$\lambda(days)$	Richards	2.47±0.16	2.84±0.08
, , ,	Logistic	2.81±0.23	3.06±0.12
	Gompertz	$2.5 \pm 0.1$	$2.75 \pm 0.1$
	Cone	$2.72 \pm 0.07$	$3.116 \pm 0.11$
V	Richards	0.052	0.376
	Logistic	-	-
	Gompertz	-	-
	Cone	0.13	0.138
$R^2$	Richards	0.9986	0.9993
	Logistic	0.9941	0.9981
	Gompertz	0.9987	0.99865
	Cone	0.9987	0.99755
%errorV <sub>0</sub> andA	Richards	-0,73	-0,82
	Logistic	-2,84	-1,81
	Gompertz	-0,56	0,04
	Cone	3,67	2,73

**Table 3**. Kinetic parameters of average cumulative methane production curves.

From the result obtained by the kinetic study of recycled pulp and paper sludge with nonlinear regression, we observe that the theoretical methane volume varies between 927.9 to 970.8 mL CH<sub>4</sub>/gVS, the correlation vector is between 99.755% to 99.934%, and the time required for the degradation of organic matter is from 2.75 to 3.116 days. It can be concluded from these results that the model of Gompertz fits the experimental data because the difference between the experimental and theoretical value given by this model is very low compared to other models. In the literature indicate that the most frequently used kinetic model for methane production is the Gompertz model [21,22,23].

From the results obtained by the kinetic study of recycled pulp and paper wastewater with nonlinear regression, we observe that the theoretical methane volume varies between 695.96 to 716.37 ml  $CH_4/gVS$ , the correlation vector is between 99.405 % to 99.876 %, and the time required for the degradation of organic matter is from 2.47 to 2.81 days. From the results, we concluded that the model of Gompertz fits the experimental data. The difference between  $V_0$  and A (the theoretical volume) is 0.56, which is very low compared to Richards, Logistic, and Cone models. The volume of methane produced per day for each experiment of sludge and wastewater from recycled pulp and paper, respectively, are 67.65 to 106.71 mL  $CH_4/gVS$  and 38.24 to 68.35 mL  $CH_4/gVS$ .

#### 3.2. Gompertz kinetics of hydrolysis and methane production

Fig. 5 displays both the experimental and simulated  $VS_{T,t}$  and  $G_t$  according to Eq. (6) and (7). The hydrolysis kinetic model of Eq. (6) did not fit the experimental data for both the recycled pulp and paper sludge and wastewater (RPPS, RPPW). Unfortunately, the experimental value of  $VS_{T,t}$  continued to fall, whereas the fitted curve leveled off. The values of  $VS_{NB}$  obtained using Eq. (6) were much greater than the experimental final  $VS_T$ . We concluded that Eq. (7) was not a good description of the entire hydrolysis process.

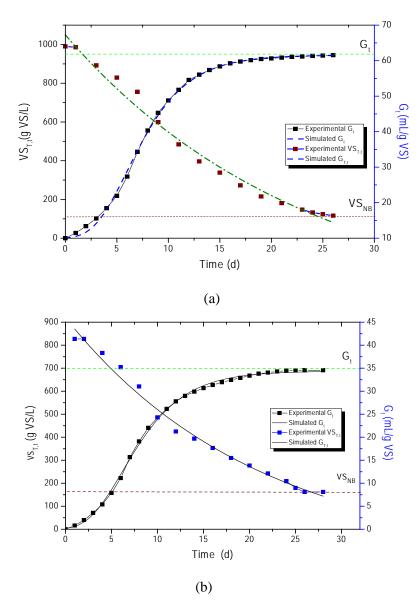


Figure 5. Gompertz kinetics of hydrolysis and methane production of: (a) RPPS, (b) RPPW

This phase, which is considered as the kinetic limiting step of the organic matter anaerobic degradation, is not a biological step strictly speaking, but a substrate reaction preparation step.

The hydrolysis rate under the best conditions depends mainly on the amount of material that remains to be hydrolyzed because the enzymes are in excess relative to the substrate. Under these conditions, its kinetics is comparable to that of a chemical-type reaction.

The hydrolysis constants determined experimentally for different types of organic matter, these constants result from analyzes of the substrate consumption or the production of VFA or even of biogas, under conditions allowing for hydrolysis kinetics determination. This is the case, for example, for methane production measurements which were carried out during the tests under controlled conditions in which all the limiting factors presented previously have been controlled and where hydrolysis proves to be effectively the same. Kinetically limiting step, that is to say in particular where

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acidogenesis or methanogens are not [24]. Kinetics determined in this way has already been applied to the production of methane obtained on different fractions of RPPS and RPPW from PBM tests.

The kinetic study built on the basis of various experimental results and describing the anaerobic degradation in two successive stages, has generally made it possible to account for these different phenomena. This representation was therefore sufficient to allow a relatively fine description of anaerobic degradation under laboratory conditions, taking into account the necessary balance between the main microbial populations.

#### 4. Conclusion

A mesophilic AD of wastewater and sludge recycled paper mill was evaluated using a 1-liter lab-scale CSTR anaerobic digesters. In this study, the Gompertz model shows a perfect fitting in kinetic modeling with a difference between  $V_0$  and A (0.04 and -0.56) for Sludge paper mill and wastewater respectively. Modified Gompertz model provided best fit for experimental data. From the results of wastewater paper mill experimental data, we conclude that Lorentz is better adjusted to our study by a correlation vector of  $R^2$  which is higher than Gauss. The ultimate methane production and the corresponding digestion time could only be understood thanks to the kinetic model of methane production.

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