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Biochemical methane potential (BMP) tests: Reducing test time by early parameter estimation

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ABSTRACT

Biochemical methane potential (BMP) test is a key analytical technique to assess the implementation and optimisation of anaerobic biotechnologies. However, this technique is characterised by long testing times (from 20 to >100 days), which is not suitable for waste utilities, consulting companies or plants operators whose decision-making processes cannot be held for such a long time. This study develops a statistically robust mathematical strategy using sensitivity functions for early prediction of BMP first-order model parameters, i.e. methane yield (B_0) and kinetic constant rate (k). The minimum testing time for early parameter estimation showed a potential correlation with the k value, where (i) slowly biodegradable substrates ($k \leq 0.1\ d^{-1}$) have a minimum testing times of $\geq$15 days, (ii) moderately biodegradable substrates ($0.1 < k < 0.2\ d^{-1}$) have a minimum testing times between 8 and 15 days, and (iii) rapidly biodegradable substrates ($k \geq 0.2\ d^{-1}$) have testing times lower than 7 days.

1. Introduction

Anaerobic digestion (AD) is a competitive treatment technology for the management of organic-rich wastes since it transforms organic matter into renewable energy in the form of methane-rich biogas and a stabilised organic mulch fertiliser (Appels et al., 2008; Batstone and Jensen, 2011). Biochemical methane potential (BMP) test is the most used methodology by academic and technical practitioners to determine the maximum methane production (B_0) of a certain substrate (Raposo et al., 2011). This batch assay determines B_0 by recording the methane produced when the substrate is mixed with an active anaerobic inoculum until only a small volume of methane is produced (Angelidaki et al., 2009; Holliger et al., 2016). BMP testing is today the most reliable method to determine B_0, which is a key parameter to assess the implementation feasibility of a full-scale AD plant as well as its optimisation (e.g. co-digestion, pre-treatment) (Angelidaki et al., 2009; Lesteur et al., 2010; Carrere et al., 2016; Koch et al., 2016; Sol and Lansing, 2013; Ward, 2016). Moreover, BMP test can also be used to estimate the kinetic constant of the rate limiting step (e.g. hydrolysis rate for highly particulate substrates) which is needed to achieve optimal design and operation of anaerobic digesters (Batstone et al., 2002, 2003; Batstone and Jensen, 2011; Lopez et al., 2015). However, BMP tests of highly particulate substrates are very time consuming with testing time ranging from 20 days to over 100 days (Raposo et al., 2012). The long testing time makes BMP testing not practical for waste and water utilities, consulting companies, and AD plants operators, which decision-making processes cannot be held for a month or longer time.

Two strategies have been evaluated to decrease the testing time needed to obtain reliable B_0 and kinetic constant values: (i) the development of new and faster methods, such as near-infrared spectroscopy and aerobic respirometry (Lesteur et al., 2010; Ward, 2016), and (ii) the statistical treatment of BMP data for parameters early prediction (Ponsá et al., 2011; Strömberg et al., 2015). The second strategy is a more conservative approach; however, it can be carried out using current BMP equipment and it is readily applicable. Strömberg et al. (2015) proposed the early prediction of B_0 by using an interactive programmed algorithm with 6 different models. The algorithm returns the most suitable model when the experimental data reaches the two established criteria: (i) the relative root mean squared error between the predicted values and the observed values is below 10% and (ii) the coefficient of determination (R^2) is higher than 0.9. Strömberg et al. (2015) algorithm could predict B_0 in 6 days or less, with the exception

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of agricultural waste which required 8 days. The Monod-type, the quadratic Monod-type and the first-order model with variable time tendency were the models that could better predict B_0. However, these empirical models contain, besides B_0, other parameters with no physical meaning. Similarly, Ponsá et al. (2011) found that the biogas generated at 14 days of testing was linearly correlated with the B_0 (at 100 days) of municipal solid wastes, facilitating the B_0 early prediction. However, these studies have only focused on B_0 early prediction, while little attention has been paid to early estimation of the degradation kinetics based on the statistical treatment of BMP data.

BMP kinetic constant rate (k) early prediction requires the selection of a mathematical model before the experiment is finished. Many kinetic models have been used to describe the methane production of BMP tests (Vavilin et al., 2008; Donoso-Bravo et al., 2010). Among them, the first-order kinetic model is the most widely used due to its simplicity, and because it is able to reflect the cumulative effect of all the reactions occurring during the actual process (Batstone et al., 2002; Vavilin et al., 2008). Additionally, the first-order maintains the parsimony principle of using a reduced number of parameters. The simultaneous early prediction of B_0 and k cannot be made during the period of time where both parameters are correlated, i.e. when B_0 and k are mathematically related by a functional relationship (changes in the value of one variable can be balanced by changes in the value of another variable) (Li and Vu, 2013). Therefore, the two parameters of the first-order model (B_0 and k) may only be identifiable after a certain period of time that ensures enough no-proportionality between sensitivity functions. In this aspect, sensitivity analysis is a suitable tool for assessing parameter identifiability for a simple mathematical model, like the first-order model (Mclean and Mcauley, 2012; Li and Vu, 2013).

The present study aims to develop a mathematically robust methodology for the simultaneous early prediction of BMP test first-order model parameters, i.e. maximum methane production (B_0) and kinetic constant rate (k).

2. Materials and methods

2.1. BMP assays

BMP tests were carried out at mesophilic conditions following the procedure described by Angelidaki et al. (2009). BMP tests were performed in triplicate in 160 and 240 mL serum bottles sealed with rubber septa and aluminium caps. The serum bottles contained inoculum and the amount of substrate required to achieve an initial inoculum-to-substrate ratio of 2 (VS-basis). Blank assays containing only inoculum were used to correct for the background methane potential of the inoculum. Next, the headspace of each bottle was flushed with 99.9% N_2 for one minute (4 L/min). Finally, the bottles were placed in an incubator set at 37 °C. Serum bottles were manually mixed by swirling before each sampling event. At each sampling event, the accumulated volumetric methane production was calculated from the increase in gas pressure and methane concentration in the headspace. Methane yields are reported at standard conditions (i.e. 0 °C and 1 bar).

Six different waste sources, commonly treated by anaerobic digestion, have been selected for this study based on industrial interest and diversity criteria. Specifically, the BMP data set consisted of a mix of already published data and genuine results including: two different sewage sludges (Astals et al., 2013; Jensen et al., 2014), one primary sludge (Peces et al., 2016), two slaughterhouse wastes (paunch and blood) (Astals et al., 2014), pig manure from two different locations (this study), and a mixture of sewage sludge with glycerol (0.25% of glycerol in weight-basis) (this study).

2.2. Sensitivity analysis of the first-order kinetic

The BMP cumulative methane production can be frequently described by a first-order model (Eq. (1)) (Angelidaki et al., 2009):

$$B(t) = B_0(1 - e^{-kt}) \tag{1}$$

where B(t) is the methane production over time (ml CH_4/gVS); t is the independent variable, time (d); B_0 is the maximum methane production (ml CH_4/gVS); and k is the kinetic parameter (d^{-1}).

According to Beck and Arnold (1977), B_0 and k (model parameters) can only be estimated by using experimental data from operational time regions where the sensitivity functions are not proportional between them. The sensitivity functions are the partial derivative of the model equation with respect to each parameter. Taken into account Eq. (1), the sensitivity functions for B_0 (Lb_0) and k (Lk) are Eqs. (2) and (3), respectively.

$$Lb_0 = \frac{dB(t)}{dB_0} = 1 - e^{-kt} \tag{2}$$

$$Lk = \frac{dB(t)}{dk} = B_0 t e^{-kt} \tag{3}$$

For the operational region where the sensitivity coefficients are proportional between them, the relationship can be expressed as for Eq. (4):

$$Lb_0 = CLk \tag{4}$$

where C is the constant of proportionality. Therefore, Eq. (4) can be also expressed as:

$$(1 - e^{-kt}) = C'(te^{-kt}) \tag{5}$$

where C' is $C \cdot B_0$.

In Eq. (5), a functional relationship between both sensitivity functions occurs from t = 0 until a certain period of time (threshold time), which depends on k. In this study, a coefficient of determination (R^2) of 0.80 between both sensitivity functions was chosen as a criterion to define when the proportionality is lost. The combination of Eq. (5) and the $R^2 < 0.8$ criterion, allows obtaining the relationship between the kinetic rate and the threshold (Figs. 1 and S1 at supplementary data). As can be seen in Fig. 1, the relationship between k and threshold time is well represented by a potential model (Eq. (6)). In this study, Eq. (6) will be used to determine the threshold time (i.e. the minimum testing time). The mathematical methodology used is further described in the supplementary information section S1.

$$\text{Threshold time (d)} = 1.892k^{-0.908} \tag{6}$$

2.3. Parameters estimation

The average data from triplicates was used to estimate B_0 and k of each BMP. Matlab® function "fitnlm" was used to carry out the non-linear regression of the first-order model (Eq. (1)). This function minimises the mean squared differences between the experimental data and the model predictions.

For each BMP test, the parameter estimation was done by three different approaches: (1) using all the experimental data (common approach); (2) using all the experimental data between t = 0 until the calculated threshold time; and (3) using three data points ("balanced threshold sampling"): the initial time, the threshold time and the average time between them. The latter strategy is chosen to reduce the contribution of the initial experimental data belonging to the proportional region while aligning with the sampling strategy proposed by D-optimal (Grijspeerdt and Vanrolleghem, 1999; Valencia et al., 2013). Parameters confidence

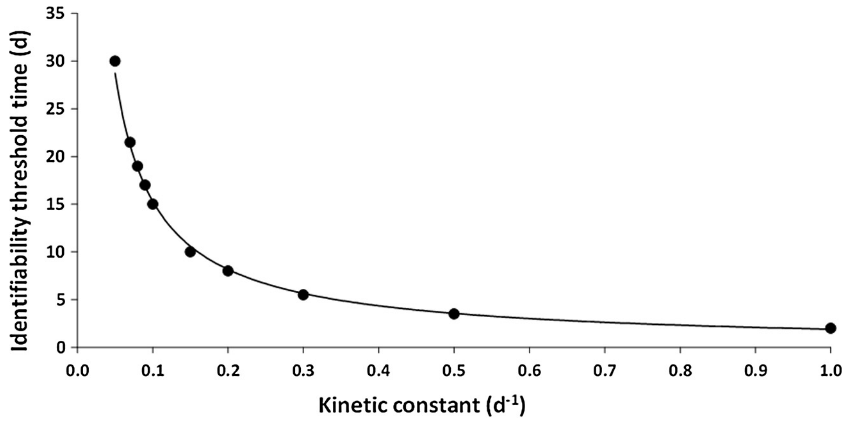

Fig. 1. Minimum testing time required to make both first-order model parameters identifiable. Dots (●) represent the calculated threshold times combining Eq. (5) and the $R^2 < 0.8$ criterion. Solid line (–) represents the potential model (Eq. (6)) that defines the threshold time as function of the kinetic constant (k).

intervals were estimated at the 95% confidence level using a two-tailed *t*-test. Adjusted coefficient of determination (R^2_{adjust}) was used to describe "goodness of fit" between the experimental observations and the model predicted outcomes (Montgomery, 2013).

3. Results

3.1. Traditional regression

The parameter estimation carried out using all the experimental points (traditional regression analysis) of the eight substrates under study gave kinetic constant values from 0.08 to 0.39 d^{-1} (Table 1). $R^2_{adjusted} > 0.98$ for all BMPs indicated that the first-order model fits well the experimental data (Fig. 2). The lowest k value (0.08 d^{-1}) was obtained for the lignocellulose-rich paunch, followed by pig manure (0.14 and 0.20 d^{-1}) and mixed (primary and secondary) sewage sludge (0.18 and 0.23 d^{-1}). For blood, a protein-rich substrate, k value was 0.28 d^{-1}, while primary sludge k value was estimated at 0.31 d^{-1}. The highest k value (0.39 d^{-1}) was estimated for the co-digestion mixture between sewage sludge and glycerol. The difference on k values between sewage sludge and the sewage sludge co-digestion mixture was attributed to the addition of an easily biodegradable substrate like glycerol (Jensen et al., 2014). The experimental and modelled data for each substrate is shown in the supplementary information section S2.

3.2. Threshold regression

The minimum testing time required to make both first-order model parameters (B_0 and k) identifiable was obtained by applying the k values from the traditional regression to Eq. (6). Thus, the minimum testing time ranged from 4.5 days for the sewage sludge and glycerol co-digestion mixture to 19 days for paunch. The minimum testing time for common substrates in anaerobic digestion such as sewage sludge and pig manure was around 10 days.

4. Discussion

4.1. Parameters identifiability

High $R^2_{adjusted}$ values (>0.98) indicates that the first-order model (Eq. (1)) is able to properly describe BMP experimental data, which is in agreement with most published data (Vavilin et al., 2008; Angelidaki et al., 2009; Donoso-Bravo et al., 2010). The loss of proportionality ($R^2 < 0.80$) between both first-order model sensitivity functions (Eqs. (2) and (3)) was used to determine the minimum testing time needed to make both first-order parameters (B_0 and k) identifiable (Fig. S2). The minimum testing time obtained under these conditions shows a strong potential relationship with k, with an R^2 close to 1 (Fig. 1 and Eq. (6)). Fig. 1 shows that the minimum testing time for substrates with k values higher than 0.2 d^{-1} is less than a week, while for substrates with k values of 0.1 d^{-1}, and below, the minimum testing time is two weeks or more. The asymptotic behaviour of Fig. 1 to the y-axis indicates that small decreases in the degradation kinetics will led to significant increase of the minimum testing time. This fact can clearly explain why Strömberg et al. (2015) and Ponsá et al. (2011) needed lower testing times to predict the maximum methane yield for highly biodegradable substrates than for slowly biodegradable substrates. Interestingly, the potential relation between the threshold time and k (Fig. 1) is similar to found by Koch and Drewes (2014), when examining the relationship between the time needed to reach the 1%

Table 1
Non-linear regression results: traditional sampling, threshold sampling, and balanced threshold sampling.

	Experimental	Traditional regression				Threshold regression				Balanced threshold regression		
Substrate	B_0 (ml CH_4/g VS)	B_0 (ml CH_4/g VS)	k (d^{-1})	R^2_{adj}	Time (d)	B_0 (ml CH_4/g VS)	k (d^{-1})	R^2_{adj}	Threshold time (d)	B_0 (ml CH_4/g VS)	k (d^{-1})	R^2_{adj}
Sewage sludge 1	361.8 ± 17	352.8 ± 18	0.23 ± 0.05	0.992	33	423 ± 236	0.17 ± 0.16	0.990	7.5	351 ± 1	0.24 ± 0.01	0.999
Sewage sludge 2	437 ± 55	428 ± 11	0.18 ± 0.04	0.984	56	563 ± 256	0.12 ± 0.08	0.973	9.5	415 ± 1	0.20 ± 0.01	0.999
Primary sludge	337 ± 22	330 ± 11	0.31 ± 0.04	0.991	24	430 ± 151	0.20 ± 0.22	0.992	5.5	362 ± 1	0.27 ± 0.01	0.999
Pig manure 1	228 ± 8	307 ± 16	0.14 ± 0.02	0.992	67	299 ± 36	0.15 ± 0.03	0.998	11.5	288 ± 1	0.16 ± 0.01	0.999
Pig manure 2	148 ± 14	141 ± 10	0.20 ± 0.04	0.988	37	148 ± 43	0.19 ± 0.09	0.990	8.0	134 ± 1	0.23 ± 0.01	0.999
Blood	422 ± 25	419 ± 6	0.28 ± 0.08	0.994	38	512 ± 127	0.20 ± 0.08	0.993	10.0	446 ± 1	0.25 ± 0.01	0.999
Paunch	232 ± 19	252 ± 17	0.08 ± 0.02	0.984	38	331 ± 93	0.05 ± 0.02	0.983	19.0	245 ± 1	0.09 ± 0.01	0.999
Sewage sludge and glycerol mixture	311 ± 21	288 ± 9	0.39 ± 0.06	0.983	60	294 ± 89	0.39 ± 0.24	0.988	4.5	280 ± 1	0.42 ± 0.01	0.999

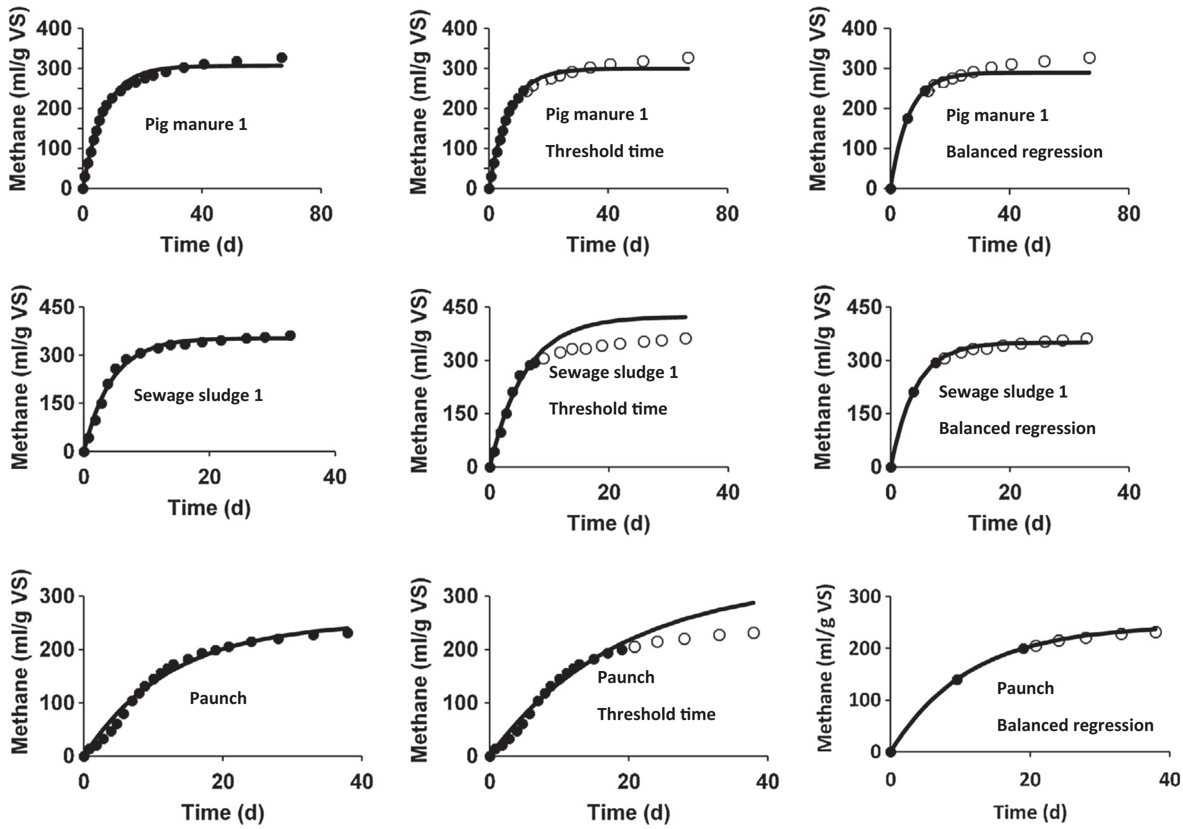

Fig. 2. Regression curves for pig manure 1, sewage sludge 1, and paunch using the three different sampling strategies. Black dots (•) represent the experimental data used depending on the regression analysis approach.

BMP termination criterion (German Guideline VDI 4630) with the hydrolysis constant.

The minimum testing times suggested by Strömberg et al. (2015) are lower than the ones obtained in this study. For instance, the minimum testing time needed for sewage sludge by Strömberg et al. (2015) is 4 days while in this study 8 and 10 days are recommended for sewage sludge 1 and 2, respectively. Although the difference can be attributed to the different models used, the criterion used in this study to decide when both sensitive functions have lost their proportionality is conservative ($R^2 < 0.80$). For instance, if the R^2 criterion is increased to 0.90 the minimum testing time sewage sludge 1 and 2 is reduced to 5.5 and 7 days, respectively. However, a more conservative approach is preferred by the authors to avoid inaccurate predictions while still managing relatively low testing times. The influence of the R^2 value criterion on the minimum testing time is shown in the supplementary information section S3.

The $R^2 < 0.80$ criterion guarantees that the proportionally between both sensitivity functions has been lost (see Fig. S2 in supplementary information), since Grijspeerdt and Vanrolleghem (1999) recommended applying visual criteria or regression analysis (without providing any standard criteria like a R^2 threshold value) to ensure the no proportionality between sensitivity functions.

4.2. Parameters predictions robustness

As can be seen in Table 1, there is no statistically significant difference between the experimental value of B_0, and the one predicted from the traditional regression. However, major differences are observed in five out of eight of the substrates when comparing the experimental value of B_0 and the predicted using all experimental data obtained at times lower than the threshold time calculated. Such difference appears when the BMP curve deviates from the exponential ideal behaviour, which has been related to phenomena like tailing (e.g. pig manure 1) and sigmoidal shape (e.g. paunch). Contrariwise, in most cases, the difference between the experimental value of B_0 and the obtained from a balanced threshold regression B_0 is insignificant (Table 1). The percentage difference between the predicted B_0 by the balanced threshold regression for the conflicting substrates is 7% for primary sludge and 12% for pig manure 1. This ~10% difference is considered acceptable from a practical point of view.

Regarding the *k* values, in most cases, there is no statistical difference between the *k* values obtained from the three different regression approaches (calculated confidence intervals overlap). However, the confidence interval provided by the threshold regression is much larger than the confidence region obtained from the traditional and the balanced threshold regression. These results highlight that the balanced threshold regression (3-points regression) gives better results (less noise in parameter estimation) than the threshold regression, which uses all data set between t = 0 and t = threshold time. The balanced threshold regression allows minimising the influence of the experimental data from the proportional region in the regression analysis while improving the quality of parameters estimation (i.e. higher accuracy and lower confidence intervals). The present results clearly show that the balance threshold regression is a feasible tool for *k* and B_0 early prediction. Finally, it is worth to mention that methane yields and kinetic constant rates reported in the literature for the substrates under study are highly variable (Table 2) (Raposo et al., 2012). The values obtained in this study are within the literature ranges. Based on the *k* literature values, a minimum BMP testing time for different substrates is suggested (Table 2).

Table 2
Literature kinetic constant rate values of some common anaerobic digestion substrates and the subsequent threshold time.

Substrate	k (d^{-1})	Threshold time (d)	Reference
Sewage sludge	0.17–0.60	9.5–3.5	Vavilin et al. (2008) Batstone et al. (2002) Donoso-Bravo et al. (2010)
Primary sludge	0.23–0.40	7.5–4.5	Siegrist et al. (2002) Donoso-Bravo et al. (2010)
Pig manure	0.07–0.17	21.5–9.5	Vavilin et al., 2008 Pham, 2013
Paunch	0.10–0.23	15.5–7.5	Jensen et al. (2014a, 2016)
Waste activated sludge	0.16–0.30	10–6	Wang et al. (2013) Ruiz-Hernando et al. (2014)
Crops residues	0.009–0.094	136.5–16.5	Vavilin et al. (2008)
Algae	0.032–0.11	43.5–14.5	Gavala et al. (2003) Passos et al. (2014)
Slaughterhouse waste	0.28–0.35	6.5–5	Jensen et al. (2015) Vavilin et al. (2008)

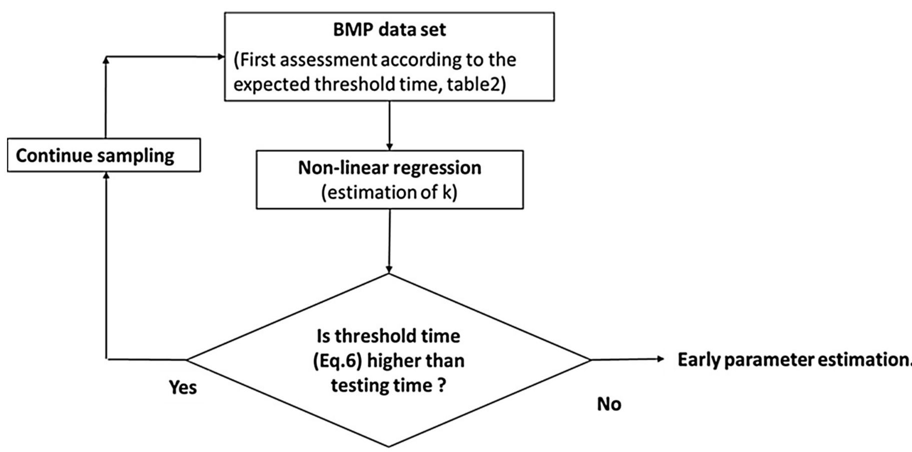

Fig. 3. Diagram flow for BMP first-order model parameters early prediction.

4.3. Proposal for BMP parameters early prediction

An iterative strategy to minimise the experimental effort in the determination of BMP first-order model parameters (B_0 and k) is proposed (Fig. 3). Depending on the substrate type, the minimum testing time can be estimated to set the first iteration (Table 2). In general, substrates can be merged in three major group depending on the kinetic constant rate value: (i) slowly biodegradable substrates ($k < 0.1\ d^{-1}$) with minimum testing times of, at least, 15 days, (ii) moderately biodegradable substrates ($0.1 < k < 0.2\ d^{-1}$) with minimum testing times between 15 and 8 days, and (ii) rapidly biodegradable substrates ($k \geq 0.2\ d^{-1}$) with minimum testing time lower than 7 days. Once the BMP test is run for the minimum estimated time, a non-linear regression is applied to experimental data to estimate both model parameters. With the value of k obtained, the minimum time required for the BMP test is calculated by Eq. (6). If the time is lower than the sampling time used, early parameter estimation is achieved. On the contrary, the BMP test should continue because the early prediction cannot be yet achieved.

5. Conclusions

A mathematically robust strategy using sensitivity functions for early prediction of BMP first-order model parameters has been developed. The minimum testing time for parameters early prediction showed a potential correlation with the substrate kinetic constant rate, with minimum testing time ranging from 5 to 15 days. This study also concluded that a balanced regression (3-experimental points) gives better results than the regression that uses all experimental data until t = minimum testing time. The balanced regression allows minimising the influence of the experimental data from the proportional region in the regression analysis while improving the quality of parameters estimation.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.wasman.2017.10.009.

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