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# Optimization of anaerobic digestion for mechanically pretreated waste paper

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## ABSTRACT

In the anaerobic digestion of lignocellulosic materials such as waste paper, the accessibility of microorganisms to the fermentable sugars is restricted by their complex structure. A mechanical pre-treatment with a Hollander beater was assessed in order to reduce the biomass particle size and to increase the feedstock' specific surface area available to the microorganisms, and therefore improve the biogas yield. The mechanical pretreatment has been applied to a batch of office paper previously shredded and inoculated with pre-incubated sludge in order to minimize the inoculum contribution and improve the gas measurements precision. Pretreatment of paper waste for 60 min improves the methane yield by 20.60%, from a value of 210.30 ml/gVS correspondent to untreated paper waste to 253.63 ml/gVS. 30 min pretreatment decreases the methane yields of waste paper by 5.24%. A response surface methodology was used in order to evaluate the effect of the beating time and feedstock/inoculum ratio on the methane yield. An optimum methane yield of 253.62 ml/gVS resulted at 55 min beating pretreatment and a F/I ratio of 0.3. The net energy of the process was always positive making the pretreatment of waste paper for anaerobic digestion economically feasible

## KEYWORDS

Biogas, waste paper, Hollander beater, methane, pretreatment, anaerobic digestion

## 1. INTRODUCTION

Paper and cardboard are a heterogeneous mixture of plant material such as cellulose, hemi-cellulose, lignin and filling material such as clay and calcium carbonate. Chemical additives (i.e. rosin, alum, starch) are added to modify quality of the material and its properties such as brightness, opacity, or glossiness. Cellulose is the major biodegradable fraction of waste paper but lignin is a recalcitrant compound for anaerobic digestion and reduces the bioavailability of the cellulose [1]. The lignin content in commercial paper ranges between 2% in office paper and 25% in newspaper. Some paper such as currency paper is composed by almost 100% cellulose. Residual contents of chemicals used during processing, such as talc or sodium silicate may still be found in the paper product and consequently also in waste paper [2–4]. In Europe the per capita consumption of paper and board was 137 kg in 2012, in United Kingdom the total consumption was 1,0095,000 tonnes [5]. The biggest source of recovered paper is industry and businesses with the 52% of the total, this covers the

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converting losses (cuttings and shavings) and returns of unsold newspapers and magazines. Around 10% comes from offices, and the remaining 38% from households [6].

Waste paper is mainly disposal to landfill, becoming the major contributor to municipal solid waste by both volume (reaching the 50%) and weight. In United Kingdom, the space for approved and licensed landfills will run out by 2018. This fact alongside with leaching and greenhouse gases emissions from the landfills requires other ways of waste paper treatment. A major way of paper waste recycling is in paper mills, but some other uses are being investigated such as construction materials [7,8], animal bedding [9], composting [10] or as a fuel [11,12]. Many studies have been carried out about the anaerobic digestion of pulp and paper sludge [13–16] and municipal solid waste (MSW) (partially composed by paper and cardboard) [17,18]. In anaerobic digestion, hydrolysis appears to be the rate-limiting step for the whole process [19]. During this stage the degradation of recalcitrant compounds like lignin, cellulose and the cell walls occur. Hydrolysis depends on multiple factors such as the particle sizes of the substrate, pH and enzymatic permeability of the substrate’s membranes. The availability of the substrates for the enzymatic attack will be achieved through the increment of the specific surface area and breakdown the crystalline structure. In recent years different technologies for biomass pretreatment have been developed in order to increase the availability of substrate for anaerobic digestion [20,21]. Breaking down lignin, disrupting the crystalline structure of cellulose and increasing its surface can be attained by pre-treatment methods, so that micro-organisms can more easily access the cellulose [22]. Although performing pre-treatment makes the process more complicated and expensive, it can improve the process efficiency and reduce the whole cost so that a positive energy balance can be obtained compared with non-pre-treated biomass [23,24]. Mechanical, ultrasounds, microwave, thermal, chemical and biological are the main pretreatment methods applied. Only two pretreatment techniques have been reported in the literature to improve the biodegradability of paper and cardboard: mechanical and biological. The mechanical pretreatment consisted in shred the paper and cardboard fraction of municipal solid waste before anaerobic digestion but it has no significant effect on biogas yields and on kinetics [25]. Better results were obtained when filter paper, waste office paper, newspaper and cardboard were pretreated with a thermophilic cellulose-degrading consortium (MC1). After 55 days of anaerobic digestion, the methane yield of pretreated filter paper, waste office paper, newspaper and cardboard were 277, 287, 192, and 231 ml CH4/gVS respectively, with corresponding increases of 33.2%, 34.1%, 156.0%, and 140.6% [26]. However biological pretreatments are slow processes, usually with residence times of 10–14 days, they require large amount of space and each feedstock requires a specific enzyme, forcing to study an enzyme-substrate specificity [24].

This paper investigates the improvements provided by a Hollander beater pretreatment. This technique is based on the same ‘comminution’ concept proposed by all other mechanical treatments. The Hollander beater has never been used as mechanical pretreatment machine on paper wastes. Seeing that this proposed pretreatment has already proved its effectiveness when applied to seaweed biomass with an improvement in biogas yield up to 20% [27], in this study it has been applied to paper wastes in batch mode.

## 2. MATERIALS AND METHODS

### 2.1 Feedstock and inoculum

Waste paper was collected from recycle bins at the School of Computing and Engineering at the University of West of Scotland (UWS) in Paisley, Scotland. This paper was mostly one side printed and was cut by an office shredder Fellowes Powershred C-320 in 0.6 x 29.7 cm pieces. The sludge used as inoculum was provided by the Energen Biogas Plant (Cumbernauld, Scotland), and stored in a fridge at 4°C. The total solids (TS) and volatile solids (VS) of the waste paper were calculated by duplicate and were obtained by submitting random samples of pretreated algae at 105°C (for TS) and 550°C (for VS) until constant weight. The sludge's characterization is provided by the supplier. The methane production is provided in terms of volume per gram of VS (ml/gVS). The characterization of the paper and the sludge is detailed in Table 1.

Table 1. Waste paper and sludge characterization.

| Parameters | Sludge | Untreated paper | 30 min pret. paper | 60 min pret. paper |
|---|---|---|---|---|
| Total Solids (%) | 4.70 | 94.79 | 2.51 | 2.53 |
| Volatile Solids (%) | 62.98 | 98.6 | 97.1 | 97.3 |
| Ash content (%) | 37.02 | 1.4 | 2.9 | 2.7 |

### 2.2 Hollander beater pretreatment

The machine is composed of an oval vessel divided along its major axis by a partition that did not reach the walls, so an elliptic channel is formed. In one of the sides of the channel is placed a bladed drum that spins above a bedplate, churning pulp up over the back fall where it slides down creating momentum to round the curve and continue the loop [28–30]. Samples were taken at 30 and 60 minutes of beating pretreatment. The samples were taken from the bend before the bladed drum in the middle of both the width and height of the channel to take the most representative sample.

### 2.3 Bioreactors

Reactors were fed with a fixed amount of 200ml of sludge (inoculum), while different quantities of pulp (beated paper) were required to have different F/I ratios as (0.3, 0.5 and 0.7). The pH was adjusted to 7.00±0.15 with potassium dihydrogen phosphate (KDP) as a buffer solution. The reactors corresponding to the untreated samples were feed with shredded paper. In order to assess the inoculum contribution to the methane production, control batches were prepared in the same way except for the paper addition. Flasks were daily shaken during the process in order to favour the degasification of the substrate and the contact between the biomass and the inoculum. Each test was conducted twice, and the average results were reported in this paper.

### 2.4 Design of experiments

The experiment is planned according to a response surface methodology (RSM) for two factors, beating time (BT) and feedstock/inoculum ratio (F/I) with three levels; the response is the methane production per g of volatile solids (ml/gVS). The statistical study is performed using the software Design Expert v.9. A second order polynomial is used,

$$Y = b_0 \sum b_i x_i + \sum b_{ii} x_{ii}^2 + \sum b_{ij} x_i x_j \quad (1)$$

where the values of the model coefficients $b_0$, $b_i$, $b_{ii}$ and $b_{ij}$ are estimated using regression analysis. The adequacy of the models is tested through the analysis of variance (ANOVA). The statistical significance of the models and of each term is examined using the sequential F-test and lack-of-fit test. If the Prob. > F of the model and of each term in the model does not exceed the level of significance (in this case $\alpha = 0.05$) then the model may be considered adequate within the confidence interval of $(1 - \alpha)$.

### 2.5 Energy balance calculation

In order to calculate the energy balance related to the use of the pretreatment, a series of equations were employed following the procedure in [31].

### 2.6 Methane production rate

An exponential model (Equation 2) was used to describe the progress of cumulative methane production obtained from the batch experiments.

$$M(t) = F(1 - e^{-kt}) \tag{2}$$

where M(t) is the cumulative methane production (ml/gVS), F is the maximum methane production (ml/gVS), k is the methane production rate constant (d-1), and t is the time (d). Biodegradability results were compared after a significance statistical analysis by using analysis of variance (ANOVA) for a single factor. Statistical significance was established at $p < 0.05$ level.

## 3. RESULTS AND DISCUSSION

### 3.1 Methane yield

The experiment parameters, beating time (BT) and feedstock/inoculum ratio (F/I) are checked in three levels. BT varies between 0-60 min and F/I between 0.3-0.7. The response is the methane production in ml per g of volatile solids (ml/gVS). The values of the methane volume obtained were converted into standard conditions (101.3kPa, 273.15K). As the biogas produced is saturated with water vapour, the water content was removed from the results as well. The inoculum contribution to biogas production was never higher than 10%. The experimental results of the assays are shown in Table 2, while the methane production from waste paper beating pretreatment is shown in Figure 1.

Table 2. Experimental results obtained at the end of the biodegradability tests

| Ratio F/I | BT (min) | Methane yield (ml/gVS) | k ($d^{-1}$) | pH |
|---|---|---|---|---|
| 0.3 | 0 | 210.30±8.15 | 0.12±0.01 | 7.13±0.07 |
| | 30 | 199.28±7.50 | 0.18±0.01 | 6.65±0.14 |
| | 60 | 253.63±12.30 | 0.14±0.01 | 7.04±0.08 |
| 0.5 | 0 | 132.84±7.69 | 0.20±0.01 | 7.05±0.06 |
| | 30 | 120.97±9.31 | 0.24±0.01 | 6.70±0.20 |
| | 60 | 215.19±9.96 | 0.10±0.01 | 6.98±0.10 |
| 0.7 | 0 | 107.72±4.20 | 0.24±0.01 | 6.89±0.27 |
| | 30 | 112.97±12.50 | 0.21±0.01 | 6.98±0.06 |
| | 60 | 175.65±11.55 | 0.09±0.01 | 7.03±0.04 |

The methane yield decreased with increased ratio F/I for all pretreatment times. For the untreated paper, the methane yield decreased by 36.84% from 210.30 ml/gVS correspondent to ratio 0.3 to 132.83 ml/gVS for ratio 0.5. For 60 min pretreated paper, the methane yield at ratio 0.7 was 175.65 ml/gVS, which was a 30.74% less than for a ratio of 0.3. Knowing the optimum F/I ratio allows a better exploitation of the feedstock. Feeding the reactor with high quantities of biomass that the inoculum is not able to process lead to a loss of feedstock, that is not digested. The 0.3 F/I ratio is also the more accurate level for study the present data and the effect of the pretreatment.

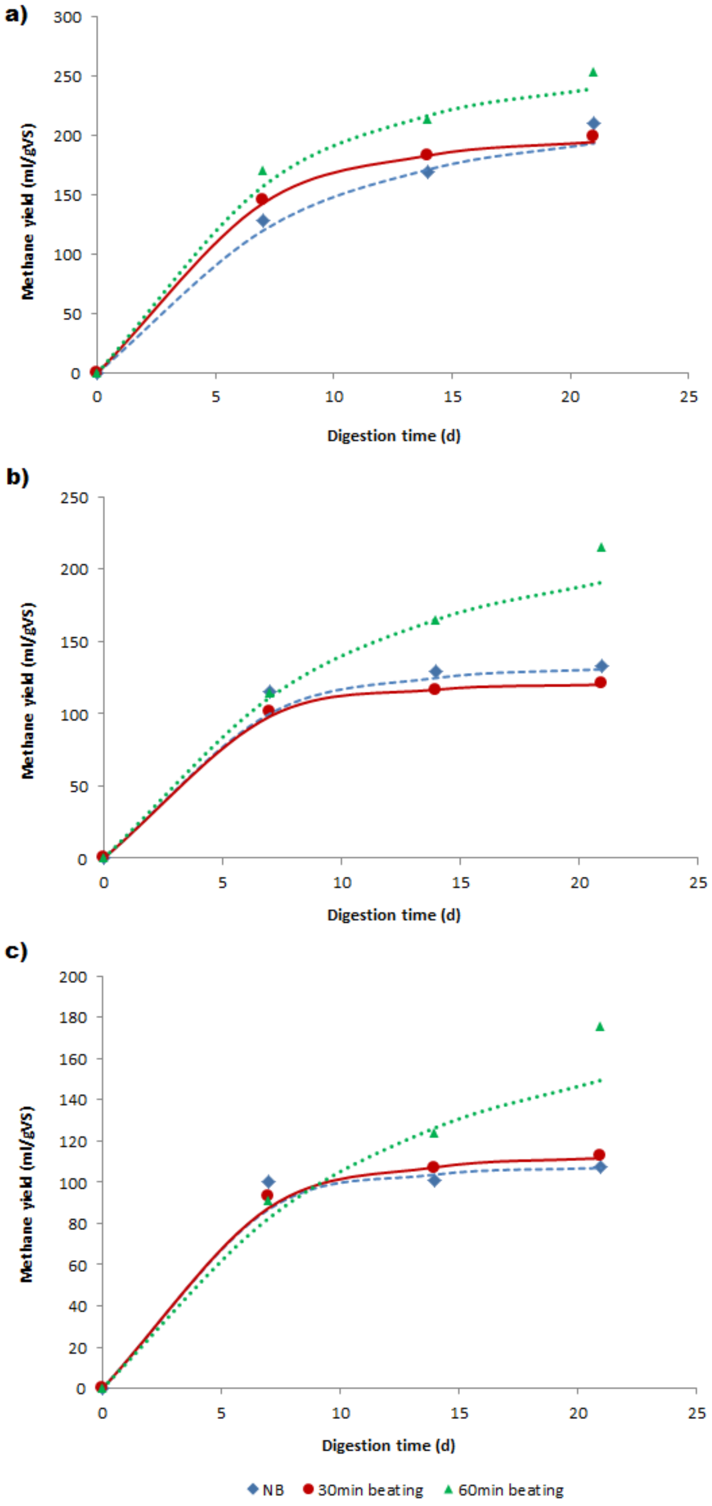

Figure 1. Methane yield: a) ratio F/I 0.3, b) ratio F/I 0.5 and c) ratio F/I 0.7.

At the early stages of the degradation (day 7) for a F/I ratio of 0.3, the methane yield from 30 min beated samples is 13.46% higher than for untreated material. 60 min beated paper produced 42.66% more methane than untreated biomass on the same day. These improvements continued in day 14 of digestion, when 30 min pretreatment improved the methane yield by 8.25% and 60 min pretreatment by 26.21%. The methane yields improvements on day 14 are roughly the half of improvements in day 7, and at the end of the digestion only 60 min pretreatment achieved a positive effect on the methane yield. This trend can be explained due to that the first step of lignocellulosic materials degradation is hydrolysis of the cellulose. It takes place at the surface of the cellulose fibres, therefore more beated samples achieved more specific surface area accelerating the hydrolysis.

At the end of the degradation, the methane yield for a ratio F/I of 0.3 decreased by 5.24% when the paper waste was beated for 30 min. When the pretreatment time was increased to 60 min, the methane yield increased by 20.60% from 210.30 ml/gVS correspondent to the untreated paper to 253.62 ml/gVS. The present result from non beated paper is consistent with the data from Eleazer et al [32], where office paper yield 220.39 ml$CH_4$/gVS.

A short beating time do not disrupt paper structure enough to lead to an improvement in methane production. Because the waste paper has passes through refining during its preparation, its structure is already disrupted to a large degree.

The small reduction in methane production may be due to the removal of ink and paper additives during the beating pre-treatment. These components could be metabolized and produce methane. The removal of ink from the paper is visible to the naked eye; a layer of microbubbles accumulates near the rotor with a distinctive black colour. The majority are inorganic and therefore do not produce biogas ($CaCO_3$, clay, talc...), others are organics and miscible with water therefore they will be present both in dry paper and in the beated pulp. Finally, there is other organic additive immiscible in water, which is the one removed during the pretreatment; rosin, composed of abietic acid ($C_{19}H_{29}COOH$) and present both in the paper and the ink. Rosin and its salts are collected on the surface of the microbubbles in the same way as soap. Once the organic layer has been form around the air bubbles, it is probable that further quantities of abietic acid would also collect in the same place. There is a tendency of ink to accumulate where the recirculating biomass is forced downwards to pass underneath the rotating roll, and where bubbles of air are created by the downward stroke of the bars. The bubbles flow upward against the flow of the biomass at this point, and the abietic acid is stripped from the biomass in a similar way to that used in industry in froth flotation equipment. Rosin is a biodegradable material [33,34], however only aerobic degradation data is available [35]. If it assumed that rosin is anaerobically degradable as well, its removal during beating will lead to a decrease in methane production. This problem with the ink and paper additives will not happen in the industrial process, as the entire pretreated batch will be fed to the reactor, both the pulp and the supernatant.

Beating pre-treatment seems start to be effective after 60 min being that methane production for 60 min treatment is higher than for both untreated and 30 min treated paper. Longer beating times disrupt the feedstock structure making it more available for microorganisms. If the effect of structure disruption is higher than the loss of additives and ink, the beating pre-treatment of waste paper will be feasible.

### 3.2 Energy balance

The energy balance was calculated in terms of total energy. The highest net energy (2.19 Wh/gVS) was achieved at 60 min beating pretreatment and a F/I ratio of 0.3. Although the energy consumed by the Hollander beater increases with the beating time, the net energy also increases with the beating time because the energy produced from the pretreated algae is higher than the energy consumed by the pretreatment. The net energy for 60 min pretreatment was higher than the corresponding from untreated feedstock while for 30 min pretreatment the net energy was always lower than the net energy from untreated material (Figure 2), meaning that 30 min pretreatment was not energy efficient. The lowest value for net energy was achieved at 30min pretreatment time and ratio F/I of 0.7, meaning these conditions are the less desired for the process' energy efficiency.

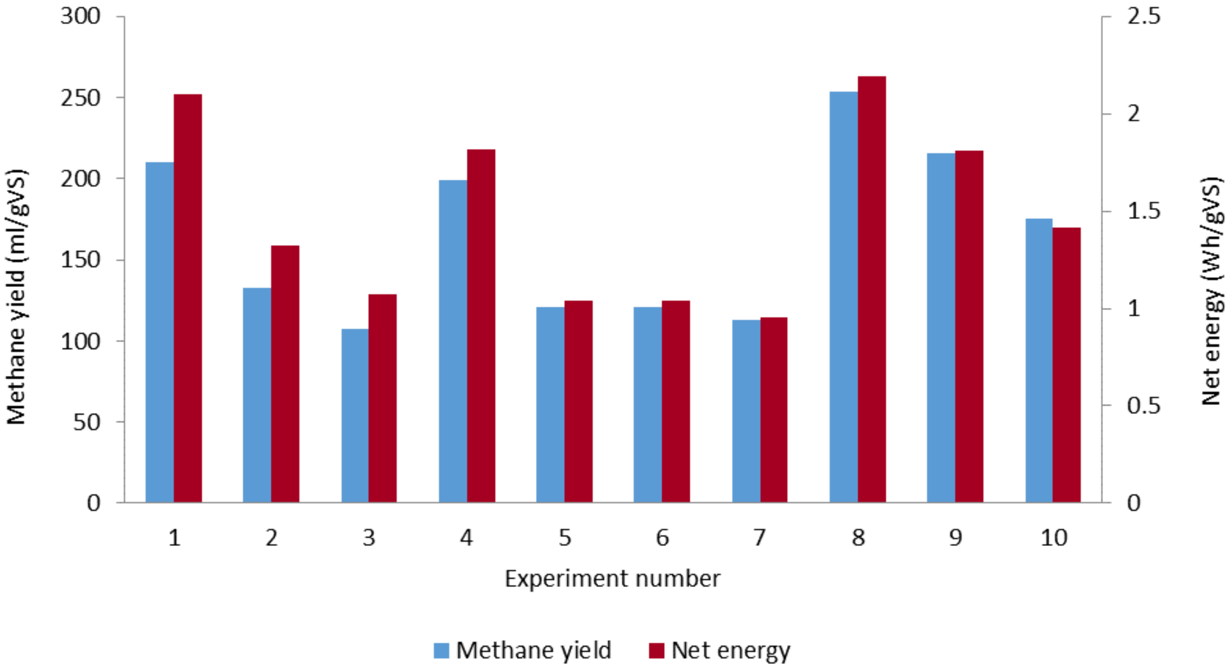

Figure 2. Bar-diagram of methane yields and net energy.

### 3.3 Experimental design

The experimental factors, F/I and BT were checked in three levels. Beating time varies between 0 and 60 minutes and ratio feedstock/inoculum varies between 0.3 and 0.7. The responses were the methane production given in ml per g of volatile solids (ml/gVS) and the net energy (Wh/gVS). Parameters and results are presented in Table 3.

*Methane yield.* For the optimization through the RSM of the methane yield, the model F-value of 36.43 implies the model is significant. The model terms of $R^2$ = 0.9785, adjusted-$R^2$ = 0.9517, predicted-$R^2$ = 0.8127, all these values are very close to 1 and so indicate the adopted model is adequate. The adequate precision, which measures the signal to noise ratio is 17.44. A ratio greater than 4 indicates an adequate signal. Values of Prob>F less than 0.0500 indicate model terms are significant. In this case A, B, AB, $A^2$ and $B^2$ are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The final mathematical model associated to the response in terms of actual factors in Equation 3.

$$\begin{aligned}Methane\ yield &= 401.86 - 2.12BT - 812.85F/I \\ &+ 1.02BT * F/I + 0.04BT^2 + 559.70(F/I)^2\end{aligned} \tag{3}$$

Table 3. Experimental factors and responses for the methane model estimation

| Experiment number | Experimental factors | | Response | |
|---|---|---|---|---|
| | BT (min) | Ratio F/I | Methane yield (ml/gVS) | Net energy (Wh/gVS) |
| 1 | 0 | 0.3 | 210.30 | 2.10 |
| 2 | 0 | 0.5 | 132.84 | 1.32 |
| 3 | 0 | 0.7 | 107.72 | 1.07 |
| 4 | 30 | 0.3 | 199.28 | 1.82 |
| 5 | 30 | 0.5 | 120.97 | 1.04 |
| 6 | 30 | 0.5 | 120.97 | 1.04 |
| 7 | 30 | 0.7 | 112.97 | 0.96 |
| 8 | 60 | 0.3 | 253.63 | 2.19 |
| 9 | 60 | 0.5 | 215.19 | 1.81 |
| 10 | 60 | 0.7 | 175.65 | 1.81 |

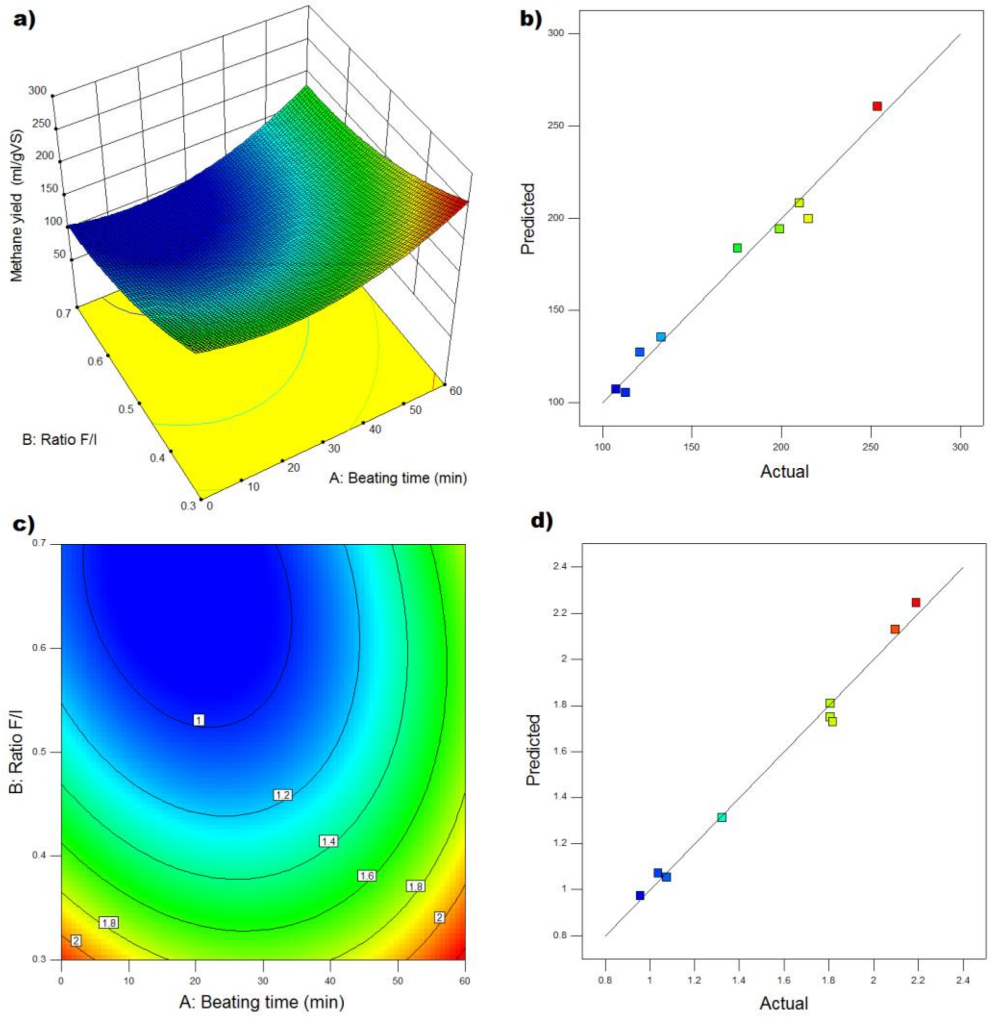

Figure 3. Response surface plot in 3D for methane yield (a), scatter diagram for methane yield (b), response surface plot in 2D for net energy (c) and scatter diagram for net energy (d).

The response surface obtained from the model illustrated in Figure 3a shows that higher methane yields are obtained at high beating times and low F/I ratios. The predicted vs. actuals plot (Figure 3b) shows that these values were distribute near to a straight line and a satisfactory correlation between them is observed. This demonstrates that the model can be effectively applied for mechanical pretreatment with a Hollander beater for paper waste. The perturbation plot in Figure 4a shows how the methane yield is affected by the input variables beating time and F/I ratio, both variables have an exponential effect on the methane production. Increasing B (F/I ratio) the methane yield will decrease exponentially. The effect of the beating time in the early stages of digestion (until day 14) is almost linear with a minimum around at day 9. After day 14 the methane yield increases exponentially with the beating time. The effect of pretreatment has a similar behaviour at low and high F/I ratios (Figure 4b). For a F/I ratio of 0.7, the methane yield achieved a minimum around 27 min of pretreatment, for ratio F/I of 0.3 the minimum is achieved at around 23 min.

*<u>Net energy.</u>* The net energy model F-value of 28.83 implies the model is significant. There is only a 0.31% chance that an F-value this large could occur due to noise. The model terms of R2 = 0.9730, adjusted-R2 = 0.9392, predicted-R2 = 0.7645 are very close to 1 and so indicate the adopted model is adequate, also the adjusted-R2 and predicted-R2 are in reasonable agreement with a difference between them lower than 0.2. The adequate precision, which measures the signal to noise ratio is 28.915. A ratio greater than 4 indicates an adequate signal and the model can be used to navigate the design space. Values of Prob>F less than 0.0500 indicate model terms are significant, in this case A, B, AB, A2 and B2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. The final mathematical model associated to the response in terms of actual factors (Equation 4) determined by the software is shown below.

$$\begin{aligned} Net\,energy &= 4.00 - 0.03BT - 8.09F/I \\ &+ 0.01BT * F/I + 4.45^{-4}BT^{2} + 5.56(F/I)^{2} \end{aligned} \tag{4}$$

The Figure 3c shows the response surface obtained from the net energy model, the net energy increases with increasing beating times and decreasing F/I ratios, in a similar behaviour that methane yield. The predicted vs. actuals values shows in Figure 3d are distributed near to a straight line which prove a satisfactory correlation between them and demonstrates that the net energy of the process can be effectively modelled. The perturbation plot in Figure 4c shows how the net energy is affected by the input variables beating time and F/I ratio, both variables have an exponential effect on the methane production. Increasing B (F/I ratio) the methane yield will decrease exponentially. The net energy decreases with increasing beating times until about day 13. After this minimum, net energy increases exponentially with the beating time. The effect of the ratio F/I is more evident for a non-pretreated paper (Figure 4d), where increasing F/I led to a drop in the net energy of the process. For a 60 min pretreated paper, the net energy decreases with increasing ratios F/I until a value of F/I of 0.6, after that the net energy remain almost constant.

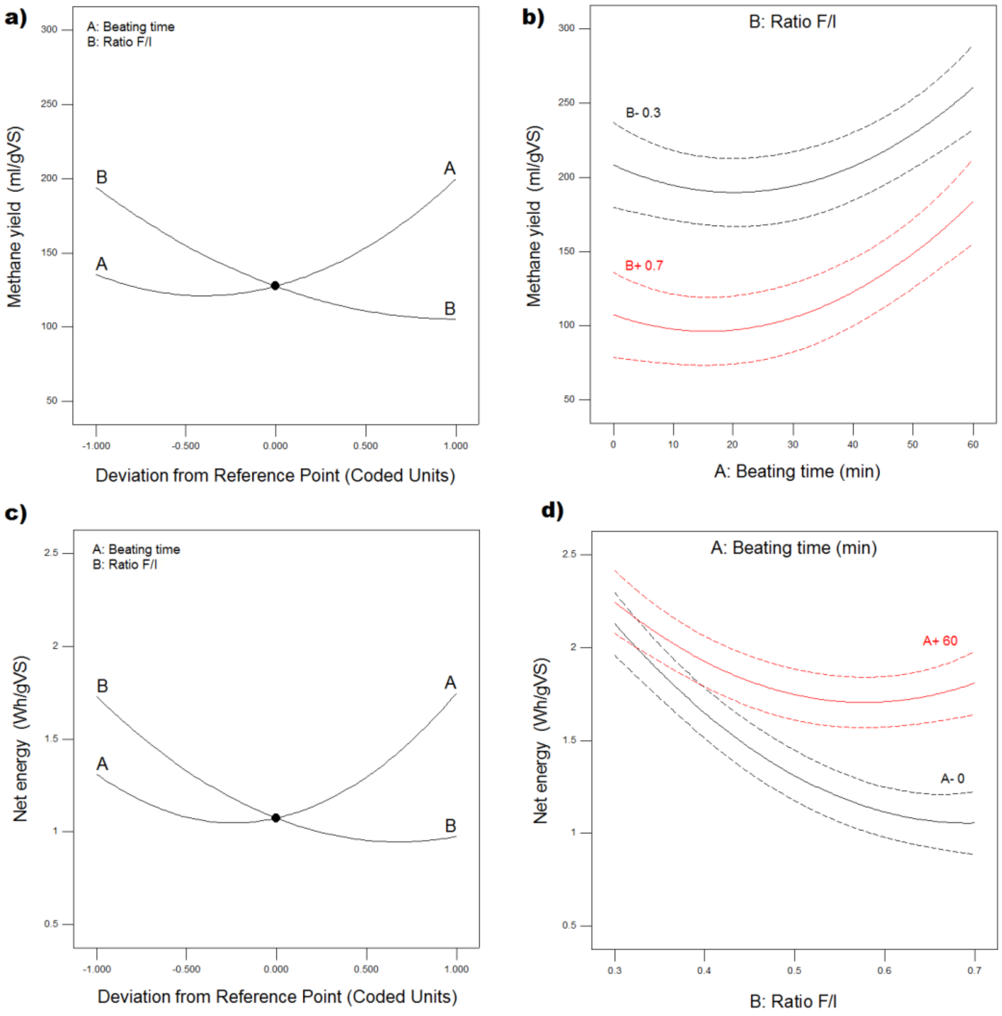

Figure 4. Perturbation plot for methane yield (a), interaction plot for methane yield (b), perturbation plot for net energy (c) and interaction plot for net energy (d).

*Methane yield optimization*. Based on the response surface model showed in Equation 3, which describes the effects of process parameters on the methane production, an optimization study was conducted using Design-expertV9 software. The optimization criteria combine the productivity with the cost of the process, the methane yield was maximized with level 5 and beating time was minimized with level 1 while F/I ratio was permitted to vary in the same range as in Table 3.

The optimal methane yield of 245.37 ml/gVS from the numerical optimisation was found at BT= 55 min and F/I ratio= 0.3, allowing 16.67% extra methane when compared to the maximum methane production for untreated paper. The graphical optimization allows a selection of the optimum process parameters by means of visual inspection. The yellow areas on the overlay plot (Figure 5) that represent the values that meet the proposed criteria is delimited by the curves corresponding to the optimization criteria set by the authors.

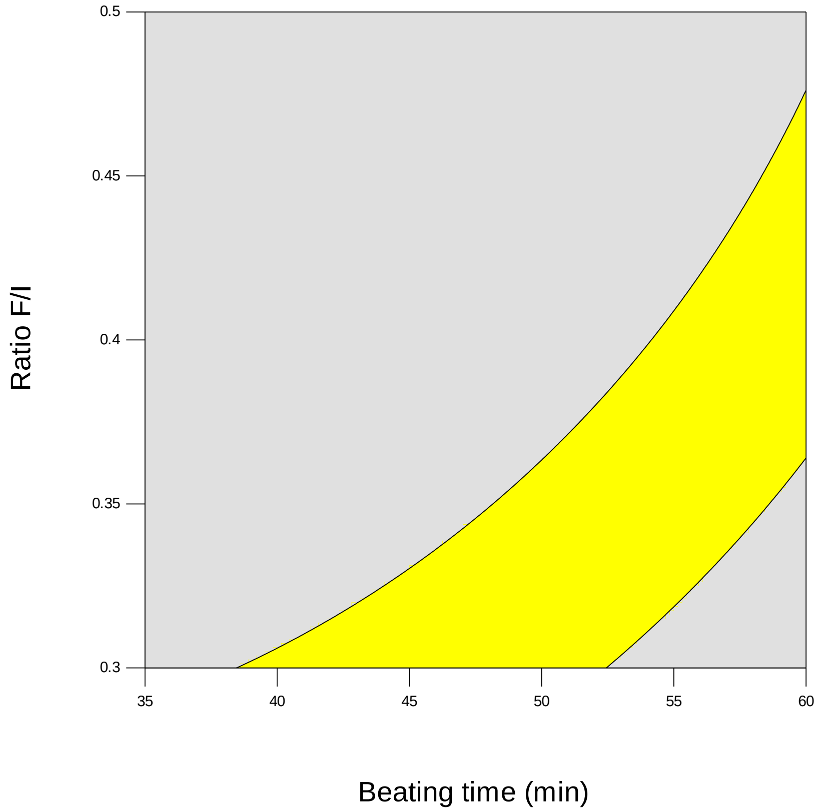

Figure 5. Graphical optimization for maximizing methane yield

## 4. CONCLUSIONS

The experimental work shows the methane yields obtained from the digestion of office paper inoculated with sludge from a biogas production plant. Pretreated office paper with a Hollander beater for 60 min improved the methane yield by 20.60%. The methane yield decreased by 5.24% for a 30 min pretreatment due to the loss of ink and paper additives during the pre-treatment. The highest methane yields were achieved at F/I ratio 0.3 for all pretreatment times and the net energy of the process was positive for all process conditions with a maximum of 2.19 Wh/gVS at 60 min beating pretreatment and a F/I ratio of 0.3. An optimization study was performed to reduce the operating costs and time associated to the pretreatment and maximize the productivity. The aim is maximizing the methane production while minimizing the pretreatment time. An optimized methane yield of 245.37 ml/gVS was achieved for 55 min of beating pretreatment and a F/I ratio of 0.3. The study proved the Hollander beater pretreatment increases the anaerobic biodegradation of waste paper and the process is economically feasible as positive net energy values were achieved.

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