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INTEGRATED AND SUSTAINABLE SYSTEM FOR MULTI-WASTE VALORIZATON

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Abstract

The general aim of this paper is to demonstrate the sustainable management of a broad spectrum of wastes (livestock, industrial, agri-food, agricultural and non-recyclable fraction proceeding from waste treatment plants) in an integrated plant. The technological development and practical application of the "Mixed Plant" concept is used to achieve this objective. In order to valorize in a joint form all the above-mentioned categories of waste, two different processes have been integrated. The first one is an anaerobic digestion system for the transformation of biodegradable organic waste into biogas. The second one is a low-temperature pyrolysis (chemical) treatment for the valorization of the non-recyclable plastic waste fraction and other non-organic waste streams. Biogas together with pyrolysis gas fraction (syngas) will be used as fuel in an adapted co-generation engine. So as to close the recycling cycle with a minimum environmental impact, and as an added value, the digestate obtained in the anaerobic reactor will be valorized as a slow-release fertilizer (struvite), the liquid fraction obtained during the pyrolysis gas distillation process will be valorized as second generation biofuels and the solid fraction generated in the pyrolysis process (ashes) will be transformed into carbon pellets (biofuel). The ultimate aim of this model is to reduce costs associated with waste treatment processes, thereby optimizing waste management, not only from the environmental perspective, but also from the financial point of view.

Key words: anaerobic digestion, energy optimization, mixed plant, pyrolysis, waste valorization

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1. Introduction

The pace of life in today's society has led to an increase in the consumption of resources, particularly those with a short lifecycle. This in turn has resulted in an exponential increase in the quantity of waste going to landfill (Sarkady et al., 2013). In the European Union countries, over 250 Mt of municipal solid waste (MSW) are produced each year, with an annual growth of 3%. In 1990, each individual in the world produced an average of 250 kg of MSW generating in total 1,300 Mt of MSW (Beede and Bloom, 1995). Ten years later, this amount almost doubled leveling at 2,300 Mt.

Different MSW treatment options have different type of impacts; however, environmental soundness of the technology should be accounted in the long time perspective. Pyrolysis-gasification is one of the emerging technologies which have lower environmental impact than the traditional incineration process (Al-Salem et al., 2009).

On the other hand, biomass is considered as a renewable energy source because its supplies are not limited (Saidur et al., 2011). While biomass biological processing is usually very selective and produces a small number of discrete products in high yield using biological catalysts, thermal conversion often gives multiple and often complex products, in very short reaction times with inorganic catalysts

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often used to improve the product quality or spectrum (Brigdwater, 2011).

Thermal treatment has been applied during thousands of years for charcoal production but it has become a technology of considerable interest on the last 30 years. The main reason is the process directly produces high yields of byproducts which can be used directly in a variety of applications. Fast pyrolysis process occurs at moderate temperatures of around 400-500 °C (Czernik and Bridgwater, 2004; Panepinto and Genon, 2014).

In parallel to this, intensified livestock farming has meant an increase in livestock waste, which represents a serious environmental problem if not managed appropriately. The concurrent growth in demand for livestock products suggest that by 2020, annual production will have to be increased by another 100 million tons of meat and 200 billion liters of milk. Such a large growth will require more than the simple adaptation of current livestock waste managing and treatment practices as they exist in Europe and North America (Martínez et al., 2009).

A wide range of technologies and systems are potentially available to treat manures (Burton and Turner, 2003) but few were adopted on a large scale because of: heavy operating costs and investment without an equivalent return; poor adaption for the livestock farm; their complexity and impracticality for the livestock operator; and further environmental problems, such as odors (Martínez et al., 2009).

The objective of an integrated system is to be both, environmentally and economically sustainable (White et al., 1999). Treatment facilities are complex systems of unit operations and streams, thereby how mass and energy flow throughout the process is primordial to obtain an overall perception and these insights will be used as a consistent basis for developing cost-effective pollution-prevention solutions (El-Halwagi, 1997). The main advantage of process integration is to consider a system as a whole (i.e. integrated or holistic approach) in order to improve its design and/or operation.

This study investigates, on the one hand, the advantages of two-phase anaerobic digestion for treating a mixture of livestock and agri-food wastes using two semi-continuous digesters operated at mesophilic temperature (37 ± 1 °C). Moreover, the operation of a low-temperature catalyzed pyrolysis system has been analyzed. Besides, the use of energy and resources optimization in the global facilities has been performed by an integration process.

2. Material and methods

The “Mixed Plant” concept proposes the treatment and recovery, at the same facility, of a wide range of waste types: farm/livestock waste, industrial waste, agricultural and forest biomass and the non-recyclable fraction from waste treatment centers (Fig. 1).

2.1. Waste analysis

Total and volatile solid concentration (TS, VS), ammonium nitrogen, phosphate, fat content and pH were determined following Standard Methods (APHA, 2005) recommendations. Ammonium nitrogen and phosphate were analyzed colorimetrically with an UV-Visible Spectrophotometer (Shimadzu, UV-1603, Japan). Mg was measured with an Inductively Coupled Plasma Optical Emission Spectrometer (Varian, 720-ES, US). C, N, H and S contents in waste samples were determined by UNE-CEN/TS 15104 EX with a LECO Truspec CHN(S) elemental analyzer. Oxygen content was not measured directly but was estimated assuming that no other elements were present in the wastes.

Wastes intended for thermal treatment were previously submitted to a thermogravimetric analysis in a DTG-60H Shimadzu instrument (Japan).

2.2. Anaerobic digestion module

Two-phase anaerobic digestion was the system chosen for the anaerobic co-digestion of manure and other waste streams from the agri-food industry. There is evidence that a two-phase digestion design, where acidogenesis takes place in one digester and methanogenesis in another, can achieve better operating performance than traditional digestion systems (Göblös et al., 2008; Hidalgo et al., 2014; Shen et al., 2013; Wang and Banks, 2003; Wust, 2003; Yang et al., 2013).

The anaerobic digestion block in the “Mixed Plant” (Fig. 2) is comprised of a continuous stirred tank acidification reactor (AR, 450 mm inner diameter and 700 mm height), a continuous stirred tank methanogenic reactor (MR, 800 mm inner diameter and 1,200 mm height), and a digested effluent tank. The acidogenic and methanogenic reactors were constructed with 1:5 volumetric ratios to maintain shorter hydraulic retention time (HRT) in the AR as comparable with longer HRT in the MR.

To provide a feed of uniform composition to the digestion system, the organic waste should be shredded and homogenized in a tank (0.5 m³), before entering the acidogenesis digester.

Biogas was measured using two wet gas flow meters. A Varian CP-4900 Micro-GC with a thermal conductivity detector was used to analyze biogas composition.

2.3. Low-temperature catalyzed pyrolysis module

The low-temperature catalyzed pyrolysis system is basically made up of a main block for thermochemical treatment in controlled conditions (Fig. 3). The internal diameter of the rotary-kiln pyrolyzer is 0.30 m and its length is 0.85 m.

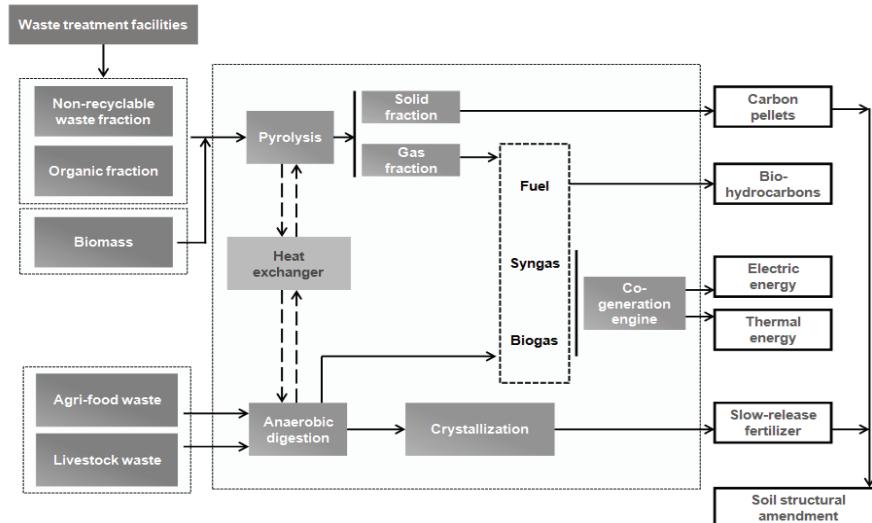


Fig. 1. Schematic diagram of the integrated “Mixed Plant”

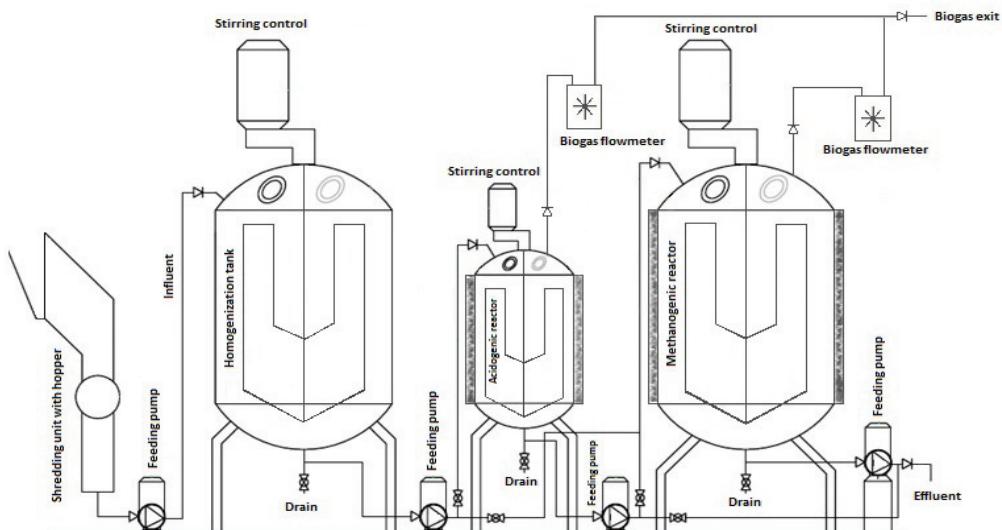


Fig. 2. Two-phase anaerobic digestion experimental set up

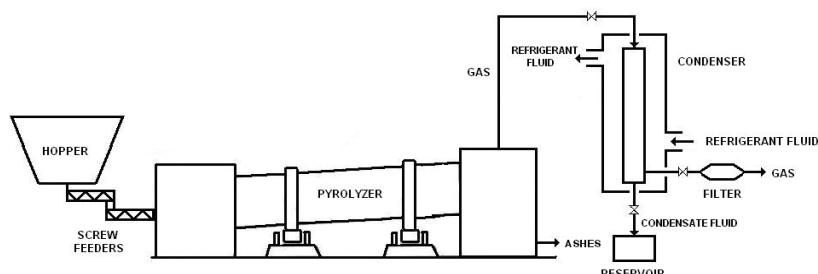


Fig. 3. Pyrolysis experimental set up

In this study, temperature varied in the range from 350 to 550°C and the kiln rotation rate was adjusted to 3 rpm.

2.4. Crystallization assays

The digest obtained in the methanogenic reactor will be fed to a crystallization system to be converted to struvite (magnesium ammonium

phosphate). The recovered product can be used as the alternative of agricultural fertilizer or as the phosphate rock for phosphate industry (de-Bashan and Bashan, 2004; Rahman et al., 2014).

In order to investigate the effects of pH value, magnesium dosage and reaction time on struvite crystallization, lab-scale batch experiments were carried out. Batch experiments were performed on a

magnetic stirrer with a stirring rate of 800-900 rpm at room temperature of 20-22 °C. NaH₂PO₄•2H₂O, MgCl₂•6H₂O and CaCl₂•2H₂O were used as P, Mg and Ca sources, respectively.

Throughout the reaction the pH value of the mixture was kept at the defined constant value (± 0.02) by NaOH supplement. Samples of the solution were removed at frequent intervals and filtrated with 0.45 µm membranes. Struvite crystals were dried naturally at room temperature.

2.5. Process integration methodology

The identification of a suitable process depends on the cooling and heating requirements of the respective unit operations. Thereby, the integration of the processes and Pinch analysis have been applied. The sequence of the proposed methodology should be: i) identifying cooling and heating requirements of the whole process; ii) generation of options and scenarios how requirements can be satisfied; iii) classify the generated options.

Pinch methodology is based on thermodynamic principles and offers a systematic approach to optimum energy integration in a process (Foo et al., 2012). Due to the generation of a heat integration scheme associated with this technique, improvements in the process are obtained. Firstly, for a Pinch methodology analysis, all the process streams that need to be cooled and all those which need to be heated in the target process are identified. Therefore, the streams, their flow rates, thermal properties, phase changes, and the temperature ranges through which they must be heated or cooled, are identified. Then, all the process streams that are to be heated, their temperatures, and enthalpy change of the temperature range or phase changes are tabulated. By Eqs. (1, 2), the enthalpy change rate for each stream is obtained.

$$\Delta H_i = \left(\sum_{Hot-streams} CP_{Hot} - \sum_{Cold-streams} CP_{Cold} \right)_i \cdot \Delta T_i \quad (1)$$

$$CP_i = \dot{m}_i \cdot Cp_i \quad (2)$$

where: ΔH_i = enthalpy increase of the temperature range ($T_{i-1} - T_i$); ΔT_i = temperature range ($T_{i-1} - T_i$); CP = heating value of the streams in the range ΔH_i ; \dot{m}_i = mass flow rate; Cp_i = heat capacity.

Each temperature interval includes one or more of the streams to be heated.

Table 1. Waste characterization

	<i>Physico-chemical parameters</i>					<i>Elemental composition^a</i>						
	pH	TS	VS	VS/TS	Fat content	%C	%H	%N	%P	%S	%Ash	%O ^b
Waste mix	-	g·L ⁻¹	g·L ⁻¹	-	%	40.4	5.7	2.5	0.4	0.6	26.0	24.4

^a % dry-matter weight basis. ^b %O estimated.

Over each temperature interval, the enthalpy change rates are added. The resulting values allow plotting of the temperature versus enthalpy rate. By this plot, a composite curve of all the streams that require a heat source is obtained. A composite curve of the streams to be cooled is achieved with the same information and procedures. Thermodynamic methods are used to obtain enthalpy changes rather than absolute enthalpies. ΔT_{min} is the smallest temperature difference between the hot and the cold streams in the heat exchanger. The location of ΔT_{min} on the composite diagram is where the two curves most closely approach each other in temperature, when measured in a vertical direction (Dunn and El-Halwagi, 2003).

3. Results and discussion

3.1. Co-digestion of livestock and agri-food wastes

The reactor was fed with a selected waste mixture of livestock and agri-food residues. Table 1 gathers the results obtained from the characterization of this waste stream. In order to find the optimum operating conditions for organic matter removal and methane generation, an experiment consisting of four runs with successively decreasing HRT in acidogenic and methanogenic reactors and, thus, total HRT, was performed. The system was operated at the HRT showed in Table 2. The results obtained during the process run of different HRT are plotted in Fig. 4. The maximum total solids removal efficiency of 81.9% was achieved at 20 days of HRT (Fig. 4a).

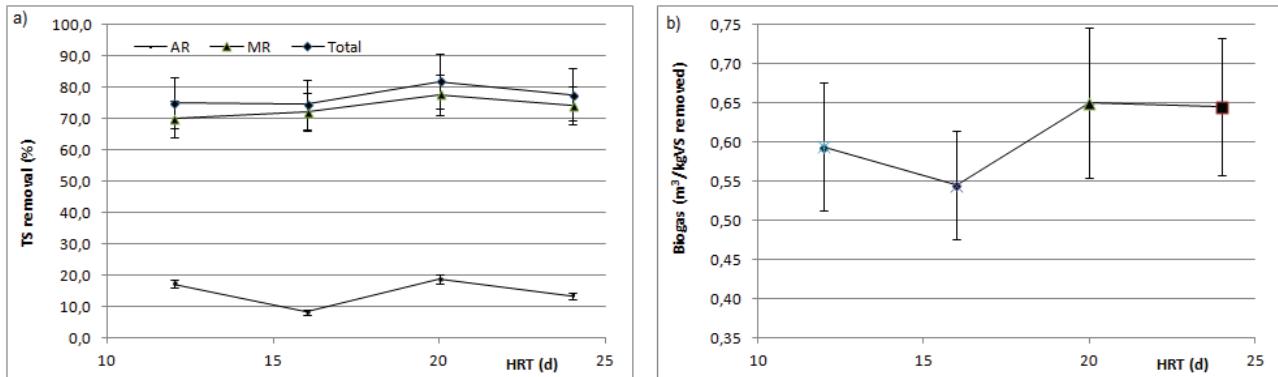
Biogas production for the whole system is shown in Fig. 4b. The average methane content in the biogas ranged between 63 and 65% during the experiments. The average maximum biogas production of 0.65 m³ kgVS⁻¹ removed (Standard Deviation, SD: 14.8%) was achieved at 20 days of HRT. The statistical calculation results indicated that the SD for methane production in the stable periods of the two-phase system was always lower than 15%. According to Shen et al. (2013), it means that the anaerobic digestion was stable under the whole experimentation.

3.2. Thermal treatment of waste mixtures

Pyrolysis characteristics of two real waste mixtures were investigated by thermogravimetric analysis.

Table 2. Experimental HRT

	<i>HRT acidogenic reactor (d)</i>	<i>HRT methanogenic reactor (d)</i>	<i>Total HRT (d)</i>
Run 1	4	20	24
Run 2	2	18	20
Run 3	1	15	16
Run 4	0.5	11.5	12

**Fig. 4.** AD system performance at different HRT: a) TS removal; b) biogas production

The first waste stream was a mix from an Ecopark (ECOMIX) with low efficiency triage, comprising in different proportions of a mixture of organics, inerts, glass, agricultural biomass and non-recyclable plastics. The second waste stream was non-recyclable plastic (NRP) with trace amounts of other wastes. Both showed a very similar compositional balance as shown in Table 3.

Table 4 shows the results of the thermogravimetric analysis. It was observed that while the weight loss for the two streams is quite similar at 100 °C (thus, the moisture is near the same), the weight loss for NRP is the double than for ECOMIX at 400 °C. Furthermore, the ECOMIX pyrolysis produces large amount of ashes, mainly due to the inert fraction.

The low temperature pyrolysis, within the range of 350-450 °C, is mainly devolatilization by the plastic cracking as a result of producing high molecular weight hydrocarbons (ranging from C5-C50). The high temperature pyrolysis within the range of 650-1000 °C is mainly cracking the primary devolatilization products and the secondary gas phase for low molecular weight hydrocarbons (ranging from C1-C9), as reported by Ray and Thorpre (2007). The thermal process in the "Mixed Plant" has the purpose of obtaining a mainly gaseous fraction (80-90%) to produce a gas that is equivalent to natural gas in terms of calorific value and composition. Mainly, gaseous fraction composition is high-methane content, ethane and butane.

According to these objectives, it is clear from the Table 4 that the NRP mix will give the higher operation yields since the proportion of volatile gases at moderate operational temperatures (400 °C) is the highest.

In the second part of the experimentation, the NRP mixture was fed to the pilot-scale catalyzed pyrolysis reactor. The product yields of solid, liquid and gas fraction from the process at 400 °C were, respectively, 10%, 7% and 83%. The study shows the following conclusion: it is possible to decrease the energy required for thermal degradation of the waste and to optimize the cracking of the polymer chains in a controlled manner, maximizing the gaseous fraction if the properties of the catalyst used are controlled.

3.3. Struvite crystallization

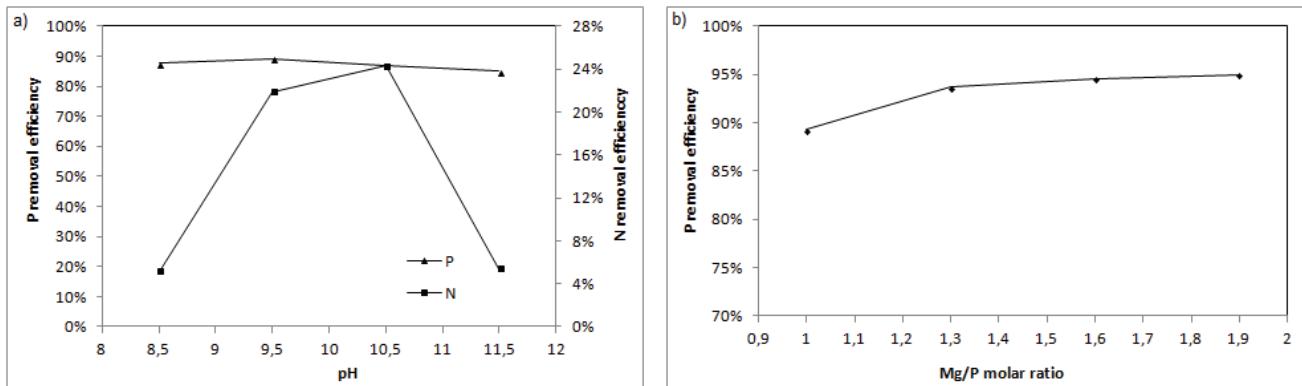
A series of experiments were performed with the digestate proceeding from the two-phase anaerobic reactor under the conditions of pH range of 8.5-11.5 and N:P:Mg molar ratio of 8:1:1. The removal efficiencies of N and P at the reaction time of 25 min are shown in Fig. 5a. The P removal efficiency increases at pH range of 8.5-9.5 up to 90% and slightly decreases at pH range of 9.5-10.5. The highest removal efficiency of only 24.3% is reached at pH 10.5. To study the effect of Mg²⁺ on the struvite crystallization, Mg/P molar ratios of 1.0 and 1.9 were adopted at pH 10.5, N:P molar ratio of 8:1 and reaction time of 25 min.

Table 3. Elemental composition (<http://www.infinitve.com/>)

<i>Waste</i>	<i>% N</i>	<i>% C</i>	<i>% H</i>	<i>% S</i>	<i>% O</i>	<i>%Ashes</i>
ECOMIX	1.51	35.79	5.20	0.16	27.11	30.23
NRP	1.12	41.57	6.59	0.15	32.79	17.78

Table 4. Thermogravimetric analysis (weight losses) (<http://www.infinitve.com/>)

Waste	100 °C	150 °C	200 °C	300 °C	400 °C	600 °C	800 °C	1.000°C
ECOMIX	5.79	6.64	9.41	25.35	35.92	43.19	45.34	48.87
NRP	4.43	5.24	8.47	-	73.99	83.31	85.07	90.10

**Fig. 5.** Removal efficiencies of phosphate and/or ammonium nitrogen: a) effect of pH value; b) effect of Mg/P molar ratio**Table 5.** Streams thermodynamic properties

Stage	Stream	T out (°C)	T in (°C)	m (kg/h)	Cp (kcal/kg·°C)	CP (kcal/h·°C)	ΔH (kcal/h)
Anaerobic digestion	Hot	37	20	742	1.199	890	15,122
Pyrolysis	Hot	400	20	1,000	0.336	336	127,680
Crystallization	Cold	20	37	685	1.199	821	-13,959
Gas cooling	Cold	37	400	900	0.295	265	-96,240

Table 6. Modified temperatures (T*)

Stage	Original T (°C)		Modified T, T* (°C)	Sequence
	out	in		
Anaerobic digestion	37		27	T4
		20	10	T6
Pyrolysis	400		390	T2
		20	10	duplicate
Crystallization	20		20	T5
		37	37	T3
Gas cooling	37		37	duplicate
		400	400	T1

Table 7. Temperature intervals

T* (°C)	Intervals Δ T* _i T* _{i-1} -T* _i (°C)	Streams			
		Anaerobic digestion (D)	Pyrolysis (P)	Crystallization (C)	Gas cooling (G)
T* ₁ = 400	-	-	-	-	-
T* ₂ = 390	Δ T ₁₋₂ = 10				Cold
T* ₃ = 37	Δ T ₂₋₃ = 353		Hot		Cold
T* ₄ = 27	Δ T ₃₋₄ = 10		Hot	Cold	
T* ₅ = 20	Δ T ₄₋₅ = 7	Hot	Hot	Cold	
T* ₆ = 10	Δ T ₅₋₆ = 10	Hot	Hot		

The P removal efficiencies at the end of reaction are shown in Fig. 5b. It shows that the P removal efficiency increases with the increase of Mg/P molar ratio. The P removal efficiencies reach 93.7% at the Mg/P molar ratio of 1.3, but they do not increase significantly any more with further increase of Mg/P molar ratio.

3.4. Savings by process integration

The first step in the application of the Pinch analysis was the identification of the process streams as hot and cold streams. The possible heat exchange will be limited by the approach temperature between

them. In this case, the streams of the process and their thermal properties are shown in Table 5.

The next step in the Pinch analysis was to select the minimum approach temperature ΔT_{\min} . The minimum approach temperature can be used as a parameter to determine the optimal size of the heat values for evaluating the total area required for heat exchange and energy recovery that is generated for each case. The ΔT_{\min} selected in this case was 10 °C.

Then, an energy balance between the process streams was performed and the corrected temperatures (T^*) defined. These T^* values are obtained by reducing the initial and target temperatures of the hot streams by ΔT_{\min} while the temperatures of the cold streams remain unchanged (Table 6). The corrected temperatures define an ordered list of n_{r+1} increasing temperatures (Table 7).

A temperature interval is defined by two successive temperatures: from T^*_{i-1} to T^*_i .

Considering the heat cascaded from the system at a temperature higher than T_i , the energy balance may be written for each temperature interval. The heat cascade model computes the minimum energy required to balance the needs of the cold streams when recovering the maximum energy from the hot streams by counter-current heat exchange and cascading the heat excess to the lower temperatures. Then, enthalpy of each interval was calculated by Eqs. (1–2). Results of these calculations are shown in Table 8. According to Table 8, the process has an excess energy of 32,603 kcal h⁻¹. From these results, a heat cascade diagram was constructed. Initially, it was assumed that there was no external heat source ($Q_1=0$).

Table 8. Enthalpy for each temperature interval

T^* (°C)	Intervals ΔT^*_i T^*_{i-1} - T^*_i (°C)	$\sum CP_{Hot} - \sum CP_{Cold}$	ΔH_i (kcal/h)	
$T^*_1 = 400$	-	-	-	
$T^*_2 = 390$	$\Delta T_{1-2} = 10$	- CP_G	-2,651	Energy deficit
$T^*_3 = 37$	$\Delta T_{2-3} = 353$	$CP_p - CP_G$	25,019	Energy excess
$T^*_4 = 27$	$\Delta T_{3-4} = 10$	$CP_p - CP_C$	-4,851	Energy deficit
$T^*_5 = 20$	$\Delta T_{4-5} = 7$	$CP_D + CP_p - CP_C$	2,831	Energy excess
$T^*_6 = 10$	$\Delta T_{5-6} = 10$	$CP_D + CP_p$	12,255	Energy excess
		TOTAL ΔH_i (kcal/h)	32,603	

Heat flowing from a temperature level (T_i) to the next level (T_{i+1}) was evaluated by Eq. (3) balance.

$$Q_{i+1} = Q_i + \Delta H_i \quad (3)$$

The results of this energy balance are shown in Table 9. Examining the heat flows between the intervals a negative value is noticed. This would imply that the heat cascade violates the Second Law of Thermodynamics since heat is being transferred from a low to a high temperature region. In order to ensure a feasible heat cascade, an external source of heat is required, and all the heat flows must be increased by 2,651 kcal h⁻¹. The resulting heat cascade diagram satisfies now the natural flow of heat ($\Delta H_i \geq 0, \forall i$). The values of the heat flows entering and leaving the heat cascade represent the quantities of hot and cold utilities required for the value of ΔT_{\min} assumed. Pinch point is given by T^*_2 , since in the Pinch point there is no energy transfer.

The result can be observed in Table 9. Finally, it is necessary to reset the correction of temperature and calculate the accumulated ΔH for hot and cold streams, separately (Table 10).

The resulting T/H plot is a single curve representing all the hot streams, known as the hot composite curve. A similar procedure gives a cold composite curve of all the cold streams. The composite curves can be observed in Fig. 6. The overlap between the composite curves represents the maximum amount of heat recovery possible within the process.

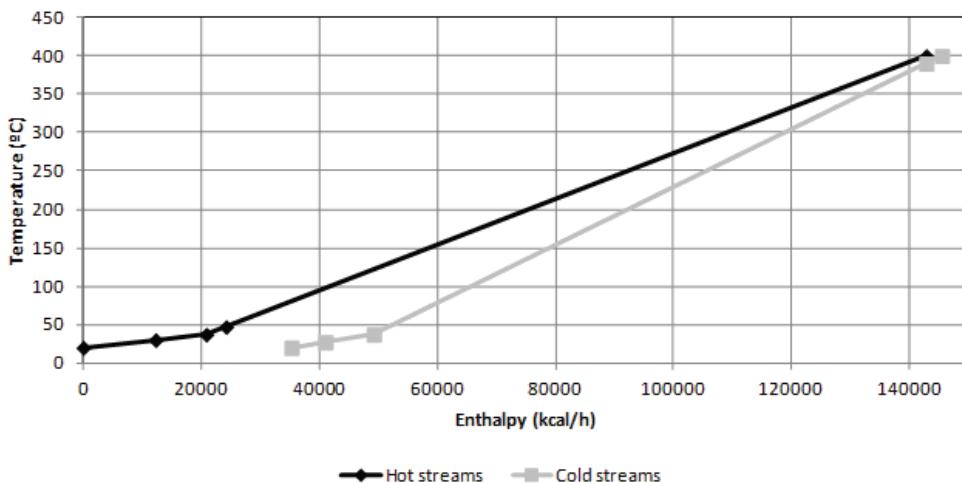
The “overshoot” at the bottom of the hot composite represents the minimum amount of external cooling required and the “overshoot” at the top of the cold composite represents the minimum amount of external heating (Hallale, 2001). A summary of the results Pinch analysis is shown in Table 11. Using the Pinch analysis method, the energy integration of main streams valorization process was achieved.

Table 9. Heat cascade

T^* (°C)	ΔH_i (kcal h ⁻¹)	Corrected ΔH_i (kcal h ⁻¹)	
$T^*_1 = 400$	0	2,651	Energy provided as heating (Q_H)
$T^*_2 = 390$	-2,651	0	PINCH POINT
$T^*_3 = 37$	22,368	25,019	
$T^*_4 = 27$	17,517	20,168	
$T^*_5 = 20$	20,348	22,999	
$T^*_6 = 10$	32,603	35,254	Energy removed as cooling (Q_C)

Table 10. Accumulated enthalpies in each interval

T_{Cold} (°C)	ΔH_i (kcal/h)	T_{Hot} (°C)	ΔH_i (kcal/h)
$T_6 = 10$	-	$T_6 = 20$	0
$T_5 = 20$	35,254	$T_5 = 30$	12,255
$T_4 = 27$	41,002	$T_4 = 37$	20,834
$T_3 = 37$	49,213	$T_3 = 47$	24,194
$T_2 = 390$	142,802	$T_2 = 400$	142,802
$T_1 = 400$	145,453	$T_1 = 410$	-

**Fig. 6.** Hot and cold process curves**Table 11.** Pinch analysis results

Minimum energy to provide as heating (kcal/h)	2,651
Minimum energy to remove as cooling (kcal/h)	35,254
PINCH POINT (°C)	
Assumed	390
Hot	400
Cool	390

The heat exchanged between the process streams was calculated and the minimum energy to provide as heating and to remove as cooling was determined.

4. Conclusions

The “Mixed Plant” concept, including a biological treatment together with a thermal treatment, allows the management and valorization at the same facility, of a wide range of wastes.

The energy integration in the main streams of the whole process leads to an optimization of the utility global consumptions. The results obtained from this study provide fundamental information for scaling up a high-performance integrated “Mixed Plant” in the future.

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