Heat integration in regeneration process of molecular sieves including heat storages.

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Abstract:

Molecular sieves are materials often used in the petroleum industry, especially for the purification of gas streams and in the chemical industry for separating compounds and drying reaction starting materials. They are also used in the filtration of air supplies for breathing equipments.

When saturated, the molecular sieve should be regenerated so that it can be used again. The regeneration in typical cyclic systems consists of the adsorbate removal from the molecular sieve bed by heating and purging with a carrier gas. Sufficient heat must be applied to raise the temperature of the adsorbate, the adsorbent and the vessel to vaporize the liquid and offset the heat of wetting the molecular-sieve surface. After regeneration, the molecular sieve should be cooled, and this is achieved by using the same gas stream as for heating but without heat input.

The objective of this paper is to study the heat integration possibilities in the process of regeneration and cooling of a molecular sieve using heat storages. We will be trying to find the optimum design of the heat integration network including heat storages and heat exchangers. This is done using a MILP (Mixed Integer Linear Programming) formulation of the multi period heat integration problem. The inputs of the algorithm are the temporal variation of the operational gas' temperature and of its heat capacity over the whole cycle and the desired maximum number of heat storages to be used. The outputs of the algorithms are the heat storages temperatures, their capacities and the net heating and cooling energy demanded to satisfy all the energy needs of the process.

The study will consider many simulations; in each the number of heat storages used is different. The results of each case will be shown and discussed and compared with the reference case, where all energy demands (heating and cooling) are satisfied using hot and cold utilities.

Keywords:

Heat integration, multi-period, heat storage, MILP formulation, regeneration of molecular sieves.

1. Definition of molecular sieves

A molecular sieve is a material with very small holes of precise and uniform size. These holes are small enough to block large molecules, while allowing small molecules to pass. Many molecular sieves are used as desiccants.

Molecular sieves are used in different types of applications [1-3]. They are used for the purification of gas streams in petroleum industry. For example, in the liquid natural gas industry, the water content of the gas must be reduced to very low values to prevent it from freezing (which causes blockages) in the cold section of liquid natural gas plants.

Molecular sieves are also used in chemistry applications for compounds separation and drying reaction starting materials. They are also used in filtration of air supplies for breathing apparatus.

Molecular sieves used for dehydration need to be dried before being used again. This drying process is energy consuming. This paper treats the possibility of heat integration including heat storages [4-6] in the drying process of a molecular sieve.

2. Presentation of the case study

As said previously, this paper treats the drying process of a humid molecular sieve after being used in dehumidification processes in the petroleum industry. This drying process is done in two steps:

- First, hot dry air is blown to the molecular sieve. It leaves the molecular sieve after being charged with particles of water. The molecular sieve is then left hot and dry.
- Then dry air at lower temperature is blown to cool the molecular sieve.

This molecular sieve is now ready to be used again in dehumidification processes.

An example of a drying process of a molecular sieve is plotted in Figure 1. This figure represents the variation of the air temperatures Tin, blown to the molecular sieve and Tout, after having left the molecular sieve. The process, as shown in the figure, is divided in two phases. In the first phase, the dry air at 290 °C is blown to the molecular sieve and leaves charged with humidity at a lower temperature Tout. The second phase is the cooling phase where the air is blown at 45 °C and leaves at a higher temperature Tout.

The heating and cooling utilities needed in this application are the following:

- Heating the dry air from a temperature of 45 °C to 290 °C to be blown to the molecular sieve in the first phase.
- Cooling the rejected humid air resulting from the drying process in the first phase from Tout to 45 °C.
- Cooling the rejected air resulting from the cooling process from Tout to 45 °C in the second phase (the cooling phase).



Figure 1: Variation in time of the air temperature used for molecular sieve drying.

To be able to study the heat integration possibilities by using a multi period approach, the total time of the cycle is divided into equal time intervals; in each interval the inlet and outlet temperatures of air are given. To be more accurate, the output temperature in each period is calculated as the average of the output temperature (Equation 1) on the whole period between two instants t and t+dt.

$$Tout_av = \frac{Tout_{(t)} + Tout_{(t+dt)}}{2}$$
(1)

A first estimation of the maximum heat integration potential in this case is done by integrating all the fluxes over the whole cycle. The pinch analysis [8] is then applied in energy term and not in heat flux Figure 2.



Figure 2: Pinch analysis applied to the integrated problem of drying of a molecular sieve.

This analysis shows that for a 10 K pinch between the fluxes, the minimum hot and cold utilities required after all the possible exchanges were made between all the fluxes is 2 792 MJ for cooling and 6 257 MJ for heating. The amount of heat exchanged between the different fluxes is almost 7 924 MJ.

It should be noted that the integration, which assumes that the fluxes can be used in a heat exchanger at any period, is not feasible in practice but constitutes only a theoretical target. Hence, heat storages have to be used to allow the heat exchange between non-simultaneous fluxes and thereby try approaching the theoretical target. The methodology presented in this paper allows proposing practical design of the heat storage system. More precisely, it permits to determine the temperature, capacity and variation of the energy stored within time.

3. Mathematical formulation

The proposed methodology to address the problem of heat integration in batch plants is formulated as a Mixed Integer Linear Programming (MILP) optimization problem. The model optimizes a linear objective function that is subject to linear constraints.

The heat integration problem in batch plants consists on finding the heat exchangers' network that is capable of energetically integrate the variable fluxes in time. Knowing that the fluxes are variable in time, thermal storage is the solution to obtain maximum heat recuperation over the whole cycle.

3.1. Description of heat integration network

An intermediate fluid circulates in the network and its role is to transport heat from the hot fluxes to the cold fluxes or from/to the thermal storages (Figure 3).

At this point the integration problem is divided into sub problems depending on the type of thermal storage used. In this paper we will treat one type of thermal storage: the case where the fluid circulating in the network is itself stored in tanks at constant temperature and variable volume [7].



Figure 3: Heat integration network including thermal storages of the fluid circulating in the network.

3.2. Inputs and outputs of the model

The inputs of the model are the following:

- The number of fluxes to heat "NFF" and those to cool "NFC";
- Time of calculation is discretized to a certain number of periods "NP", each period's duration is "tp";
- The discretized temperature range parameters (maximum temperature, minimum temperature and node temperatures);

- The pinch of the heat exchangers between the fluxes and the circulating fluid in the network "Pinc";
- The reference temperature, as well as the temperature of the hot source and cold source for exergy calculations;
- The maximum number of desired heat storages "nb_Stocks".

The outputs of the model are the following:

- The number and temperature of thermal storages used, in addition to their maximal capacity and the variation of the heat stored as a function of time;
- The heat exchanged between the different fluxes in each period of time;
- The net heating and cooling utilities needed in each period of time.

3.3. MILP formulation of the optimization problem

Each storage tank is filled or emptied depending on the heat or cooling demands of the fluxes. Hence the volume of the fluid stored in any thermal storage varies in time leading to a corresponding mass flow in each branch of the network.

Many parameters are pre calculated in the algorithm:

- The theoretical number of storages (NS) and their temperatures (Equation.2) (Figure 4):

$$TS[k] = T\left[floor\left(\frac{k+1}{2}\right)\right] + if(i \mod 2 = 0) then (-Pinc) else (+Pinc)$$
(2)



Figure 4: Storages theoretical temperatures.

Where TS[k] is the temperature of the heat storage tank k. T[i] is the temperature of the node i of the fluxes temperature range. Pinc is the pinch of the exchanger between the intermediate fluid circulating in the network and the operating fluxes of the process.

These temperatures are then arranged from the highest to the lowest temperature.

The constraints on variables are the following:

- Energy balance applied to each interval of temperature 'i' of each hot flux 'j' at a certain period of time 'p' : heat that exists in the flux will be either rejected to a cold utility or exchanged with the fluid circulating in the network between two thermal storages at lower temperatures.

$$CP_{c}[i, j, p] \times (T[i] - T[i + 1]) = UF[i, j, p] + (if T[i]) \geq (TS[k] + Pinc) and T[i + 1] \geq (TS[k + 1]) + Pinc)) \sum_{k=1}^{NS-1} DebF_d[k, i, j, p] \times (TS[k] - TS[k + 1])$$
(3)

Where UF is the cooling power and DebF_d is the partial heat capacity flow (m.Cp) that exchanges with the hot flux (the Cp of the intermediate fluid is assumed constant). This is represented by the Figure 5.



Figure 5: Energy balance applied to an interval of temperature i of a hot flux j at a certain period of time.

- Energy balance applied to each interval of temperature 'i' of each cold flux 'j' at a certain period of time 'p': the heat needed is either supplied by a hot utility or by the fluid circulating in the network between two thermal storages at higher temperatures.

$$CP_{f}[i, j, p] \times (T[i] - T[i + 1]) = UC[i, j, p] + (if TS[k])$$

$$\geq (T[i] + Pinc) and TS[k + 1]$$

$$\geq (T[i + 1])$$

$$+ Pinc)) \sum_{k=1}^{NS-1} DebC_d[k, i, j, p] \times (TS[k] - TS[k + 1])$$
(4)

Where UC is the heating power and DebC_d is the partial heat capacity flow that exchanges with the cold flux.

- Energy balance applied to each thermal storage 'k' at a certain period of time 'p': the storage heat capacity *Stock*[*k*, *p*] is calculated in kJ/K. The heat power stored in a thermal storage 'k' at the period of time 'p' is equal to the difference of circulating fluid heat capacity flows entering and exiting this storage.

$$Stock[k, p + 1] = Stock[k, p] + tp[p] \times (-xc[k - 1, p] - xf[k - 1, p] + \frac{UCS[k, p]}{TS[k] - TS[k+1]} - \frac{UFS[k, p]}{TS[k-1] - TS[k]} + \frac{UFS[k-1, p]}{TS[k-1] - TS[k]})$$
(5)

Where UCS[k,p] and UFS[k,p] are respectively the heating and cooling powers of the utilities between two storage temperatures TS[k] and TS[k + 1].

xc[k-1,p] is the heat capacity flow entering the storage 'k' after having exchanged with the cold flux or exiting the storage to exchange heat with the cold flux. xf[k-1,p] is the heat capacity flow entering the storage 'k' after having exchanged heat with the hot flux or exiting the storage to exchange heat with the hot flux.

- Node law applied to all the heat capacity flows entering or exiting each knot 'k' in the circuit at a certain period of time 'p' (Figure 6):

$$DebF[k+1,p] - DebF[k,p] + xf[k,p] = 0$$
(6)



Figure 6: Node law applied to each node in the network (law of mass conservation).

Where xf and xc have positive values if the flow is entering the knot and negative values if it is leaving it.

- Energy balance applied to interval 'k' between two thermal storages TS[k] and TS[k+1]:

$$DebF[k,p] = \sum_{\substack{i=1\\NT-1}}^{NT-1} \sum_{\substack{j=1\\NFF}}^{NFC} DebF_d[k,i,j,p]$$
(8)

$$DebC[k,p] = \sum_{i=1}^{NT} \sum_{j=1}^{NT} DebC_d[k,i,j,p]$$
(9)

Where DebF_d and DebC_d are the partial heat capacity flows. *DebF* and *DebC* are the total heat capacity flows in the network.

- A constraint that imposes the equality between the initial state of a thermal storage 'k' and its final state:

$$Stock[k,1] = Stock[k,NP+1]$$
(10)

- A constraint that forces every storage 'k' to be emptied at least for one period of time: p=NP+1

$$\sum_{p=1} bS_n[k,p] \le NP$$
(12)

Where $bS_n[k, p]$ is a binary that is equal to 1 if the storage k has a positive value at a period 'p' and 0 if this storage is empty (i.e. value = 0). Knowing that there are NP periods of time, each storage k should have NP+1 values, one of them is imposed to be 0.

- A constraint that forces the number of storage to be under a certain parameter desired by the user:

$$\sum_{k=1}^{k=NS} bS[k] \le nb_Stocks$$
(13)

Where bS is a binary that is equal to 1 if the storage does exist and equal to 0 if it doesn't. nb_Stocks is the maximum number of storages desired by the user.

3.4. Function to optimize

The objective of the heat integration is to reduce net heating and cooling demands. Hence, the function to minimize is the sum of the hot and cold exergy consumption in the process:

$$Minimize: \sum (Ex_c + Ex_f + ExS_c + ExS_f)$$
(14)

Where:

$$Ex_{c} = \sum_{i=1}^{NT-1} \sum_{j=1}^{NFF} \sum_{p=1}^{NP} UC[i, j, p] * tp[p] * (1 - \frac{T_{ref}}{T_{SC}})$$
(15)

$$ExS_{c} = \sum_{i=1}^{NT-1} \sum_{j=1}^{NS} \sum_{p=1}^{NP} UCS[i, j, p] * tp[p] * (1 - \frac{T_{ref}}{T_{SC}})$$
(16)

 Ex_c and ExS_c are the exergies required for heating.

The exergies consumed to satisfy the cooling needs, using a cold utility at a certain temperature T_{SF} which is lower than the reference temperature T_{ref} , are calculated in the Equations (17) and (18).

$$Ex_{f} = \sum_{\substack{i=1\\NT-1}}^{NT-1} \sum_{\substack{j=1\\NS}}^{NP} \sum_{\substack{p=1\\NP}}^{NP} UF[i,j,p] * tp[p] * \left(1 - \frac{T_{SF}}{T_{ref}}\right)$$
(17)

$$ExS_{f} = \sum_{i=1}^{NT} \sum_{j=1}^{NS} \sum_{p=1}^{MT} UFS[i, j, p] * tp[p] * (1 - \frac{T_{SF}}{T_{ref}})$$
(18)

It should be noted that in this case, only the exergy consumed is calculated. In future works, the exergy destroyed will be taken into account too and the function to minimize is the sum of the consumed and destroyed exergies in the whole process.

4. Optimization scenarios

To study the heat integration possibilities four scenarios are proposed, each with a different maximum number of heat storages. The algorithm detailed in section 3 will be used and results are compared.

It should be noted that the following parameters were given to the algorithm:

- The number of periods is 31 of 1 000 seconds each.
- The number of fluxes to heat is 18 and of those to cool is 31.
- The heat capacity of all the flows is equal to 3.216 kW/K.
- The pinch of the heat exchangers between the fluid circulating in the network and the operating fluxes is considered to be 5 K.
- For exergy calculations: the reference temperature is equal to the cold source temperature which is equal to 15 °C. The hot source temperature is considered to be 900 °C.
- The maximum number of heat storages is a parameter that will be changed from a simulation to another.

5. Results

Many simulations using the algorithm described in section 3 are done. In each simulation, the maximum number of heat storages is varied. Then the exergy consumed and destroyed is calculated for each solution and plotted in the Figure 7. It should be noted that if no heat integration is done, the total exergy needed for this application is almost 10 820 MJ. This value is not represented in the Figure 7.



Figure 7: Exergy required to accomplish the integrated drying process as a function of the number of thermal storages used.

The continuous line in the graph represents the minimum theoretical required exergy of the integrated problem in section 2. I.e. this is the target value of exergy to reach. The results show that just by proceeding to a heat exchange between the hot fluxes and cold fluxes in the first 18 periods of time, there's a saving potential of 3 984 MJ of exergy (this case is represented on the graph by the point at zero storages). In other terms, if only heat exchangers are installed between the simultaneous fluxes, 36.8 % of the total spent exergy is saved.

The usage of two heat storages reduces the spent exergy by 54.4 %. This is represented on the graph by the point at 2 heat storages.

As it is noticeable also on the graph, the values of exergy for the cases where the number of storages is above three are very close. This means that increasing the number of thermal storage tanks does not have a major effect on reducing the exergy demands of the process.

To understand the type of solutions given by the algorithm, the case with two maximum heat storages will be detailed and discussed.

Supposing that under pressure water is the intermediate fluid circulating in the network, Figure 8 shows the variation of the volume of water stored in both of the storages in one cycle. The storages are situated at 240.2 $^{\circ}$ C and 56 $^{\circ}$ C.

It should be noted that the process is cyclic, in other words it is repeatedly done over the whole production time.



Figure 8: Volume variation within time in m^3 of the thermal storages used in the heat integration of the drying process.

In the first 18 periods of time (first phase), there are simultaneous cooling and heating demands. In this phase, the behaviour of the thermal storages changes between the beginning and the end of this phase. We can see on the graph that the high temperature storage tank (240.2 $^{\circ}$ C) is emptied between the first and tenth period of time. The low temperature storage tank (56 $^{\circ}$ C) is simultaneously filled at the same rate. Afterwards, the volumes of these storage tanks remain almost constant.

A reversed behaviour is observed in the second phase (from period 18 to 31). In this phase, the high temperature storage tank is filled from period 18 to period 26 while the low temperature storage tank is filled at the same rate. Afterwards, the volumes of both storages remain constant.

Due to this change in storages' behaviour in both of the phases, each phase is divided into 2 subphases. The results of the simulations also allow analyzing the behavior of the network in each phase, more specifically in each sub-phase. This allows us to determine the architecture of the heat integration network all over the cycle. Figure 9 shows the topology of the heat exchangers network.



Figure 9: The heat integration network scheme in the drying process of a molecular sieve.

As previously said, the operating scenarios of this heat integration network vary from a sub-phase to another. Figure 10 represents the heat integration network operation in the sub-phases of the first phase.



Figure 10: Heat integration scenario in the first phase of the drying process.

In this phase, both cooling and heating demands exist and it's divided as already mentioned into two sub-phases represented on Figure 10.a and Figure 10.b respectively. In the first sub-phase (Figure 10.a), a water mass flow rate m3 leaves the high temperature storage tank (240.2 °C) and exchange its heat with the cold fluxes of the process. The rest of the heat (if it exists) is exchanged with a cold utility, and the water mass flow rate m3 is filled in the low temperature storage tank (56 °C). This flow rate has a function to transport the heat already stored from the previous cycle at high temperature to the cold fluxes, responding by that to a large portion of the heating demands of the process. At the same time, a mass flow rate m2 is heated from the lowest temperature in the network T1 to a certain temperature T3 by exchanging heat with the hot fluxes of the process. This mass flow rate exchanges then a part of its heat with the cold fluxes and then it's cooled using the cold utility to the temperature T1 to continue its circulation in the network. This sub-network's main function is to ensure the cooling demands of the hot fluxes of the process. Simultaneously, a mass flow rate m1 is heated from a certain temperature T2 to T3 by heat exchange with the hot fluxes, and then it's heated using a hot utility to the highest temperature in the network T4. Then its heat is exchanged with the cold fluxes which cools it to the temperature T2 to continue its circulation in the network. This sub-network's main function is to ensure the heating demands of the cold fluxes of the process.

In the second sub-phase (Figure 10.b), the volumes of thermal storages are constant which means the mass flow rate m3 exchanged between them doesn't exist anymore. Two mass flow rates continue to circulate in the two sub-networks described above.

In the second phase (Figure 11), only cooling demands exist. In the first sub-phase, a water mass flow rate m1 leaves the low temperature storage tank to be heated to a certain temperature T3 by exchanging heat with the hot fluxes of the process. Heating up to the temperature of the storage (240.2 °C) is done using a hot utility. The mass flow rate m1 is then stored in the high temperature storage tank. This sub-network has the function to store the heat available in this phase to be used in the next cycle. At the same time a certain water mass flow rate circulates between the lowest temperature of the network T1 and a temperature T3 by exchanging heat with the fluxes to cool. This heat is then exchanged with a cold utility and the flow continues to circulate in the sub-network. This sub-network's function is to exchange with the cold utility the available heat that is not stored in this phase. In the second sub-phase (Figure 11.b), the storage tanks' volumes are constant, no heat is stored and then only the mass flow rate m2 continues to circulate in the sub-network.



Figure 11: Heat integration scenario in the second phase of the drying process.

6. Conclusion

In this paper, an algorithm for heat integration of batch plants is presented. The mathematical formulation as well as the inputs and outputs of this algorithm are detailed.

This heat integration algorithm was applied to a study case in which the possibility of heat integration in the drying process of molecular sieve used in petroleum applications.

The results compare the different amounts of exergy needed to accomplish the heat integration while responding to the heating and cooling demands of the process. The result is a significant reduction in the amount of exergy used to accomplish the drying process while energetically integrating the operating fluxes. The results show also a further reduction in the exergy expenses when using two thermal storages to store heat between the different periods of time of the process. Furthermore, it was made clear that the increase in the number of thermal storages does not induce a larger decrease in the exergy spent to accomplish the process.

Then, the case where two heat storages is detailed, the variation of every storage volume is shown and the heat integration scheme was explained.

After exergetically analyzing the study case, future works will rely on economic studies of the solutions. This will help to make the decision of whether it is profitable to install thermal storages and heat exchangers. It will help also to determine the sizing of these storages, heat exchangers and utilities used, allowing to calculate the payback time of the investment on such a heat network installation.

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Nomenclature

Notions		
(a) mod (b)	The remainder after the division of (a) by (b)	-
floor (a)	The largest integer that is directly inferior or equal to the real a	-
MILP	Mixed Integer Linear Programming	-
Terms		
bS	Binary determining if the storage exists or not	-
bS_n	Binary determining if the storage is empty or not at a certain period of time.	-
Ср	Specific heat capacity	kJ/(kg.K)
ĊP	The heat capacity flow	kW/K
DebC	The total heat capacity flow in the network exchanging heat with all the temperature intervals of all the cold fluxes.	kW/K
DebC_d	The partial heat capacity flow in the network exchanging heat with an interval of temperature of a determined cold flux	kW/K
DebF	The total heat capacity flow in the network exchanging heat with all the temperature intervals of all the hot fluxes.	kW/K
DebF_d	The partial heat capacity flow in the network exchanging heat with an interval of temperature of a determined hot flux.	kW/K
Ex	Exergy of the utility to supply directly the fluxes	kJ
ExS	Exergy of the utility to supply the intermediate fluid circulating in the network	kJ
m	Mass flow rate	Kg/s
nb_Stocks	The maximum number of heat storages required by the user	-
NFC	Number of hot fluxes to cool in the process	-
NFF	Number of cold fluxes to heat in the process	-
NS	Number of theoretical heat storages	°C
NT	The number of temperature nodes in the range of temperature of the operating fluxes	-
Pinc	Pinch of the heat exchanger between the intermediate fluid in the network and the operating fluxes	K
Т	The temperature of a node in the operating fluxes range of temperature	°C
Tin	The air inlet temperature in the drying process	°C
Tout	The air outlet temperature in the drying process	°C
Tout_av	The average outlet temperature in a certain period of time	°C
tp	The duration of each period of the process	S
T_{ref}	The reference temperature for exergy calculations	Κ
ΤŚ	Heat storage temperature	°C
T_{SC}	The temperature of the hot source used in exergy calculations	К
T_{SF}	The temperature of the cold source used in exergy calculations	Κ
UC	The heating power of the hot utility directly exchanged with the cold flux	kW

UCS	The heating power of the hot utility exchanged with the intermediate fluid circulating in the network	kW
UF	The cooling power of the cold utility directly exchanged with the hot flux	kW
UFS	The cooling power of the cold utility exchanged with the intermediate fluid circulating in the network	kW
хс	The heat capacity flow entering the storage after having exchanged with the cold flux or exiting the storage to exchange heat with the cold flux	kW/K
xf	The heat capacity flow entering the storage 'k' after having exchanged heat with the hot flux or exiting the storage to exchange heat with the hot flux.	kW/K
Subscriptions		
с	Related to the hot flux	-
dt	The time between two instants of the process	S
f	Related to the cold flux	-
t	The instant t of the process	S
Index (between		
brackets)		
i	The node in the temperature range of the operating fluxes	-
j	The number of the operating flux	-
k	The number of the heat storage	-
р	The number of the period of time	-